

# PROCEEDINGS OF THE INTERNATIONAL CONFERENCE ON SUGAR PALM AND ALLIED FIBRE POLYMER COMPOSITES 2021 (SAPC2021)

11 DECEMBER 2021

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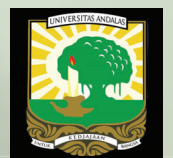
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*SAPC2021*

*International Conference on Sugar Palm and Allied Fibre Polymer Composites 2021*

**PROCEEDINGS OF THE  
INTERNATIONAL CONFERENCE  
ON SUGAR PALM AND ALLIED  
FIBRE POLYMER COMPOSITES  
2021 (SAPC2021)**

**11<sup>th</sup> DECEMBER 2021**

**Persatuan Pembangunan Dan Industri Enau Malaysia  
(PPIEM)**

*SAPC2021*

*International Conference on Sugar Palm and Allied Fibre Polymer Composites 2021*

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**PREFACE**

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Natural fibres are increasingly being utilized as reinforcement in composite materials due to the rising demand for renewable, cost-effective, and environmentally friendly materials in a spectrum of uses. Flax, kenaf, hemp, jute, coir, sisal, and abaca are the most widely used natural fibres as reinforcements. However, sugar palm (*Arenga pinnata*) fibre, as one of the natural fibres, is increasing in popularity as a reinforcement in composites such as biofibres, biopolymers, and biocomposites, even though it has been acknowledged in rural areas for decades for its versatile traditional usage. Thus, an international conference on Sugar Palm and Allied Fibre Polymer Composites (SAPC2021) was held which aims to provide a platform for knowledge sharing in research and technology of sugar palm and allied fibres polymer composites. It is a relevant platform for academics, researchers, policymakers and private

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### *International Conference on Sugar Palm and Allied Fibre Polymer Composites 2021*

companies to collaborate and to discuss issues related to research and technology in sugar palm and allied fibres where more than 10 countries participated including the United States, Brazil, Hungary, Czech Republic, Nigeria, Pakistan, India, Indonesia, Thailand and Malaysia.

Therefore, in this proceedings book, the works about advanced manufacturing, applications, and recent developments of sugar palm and allied fibre reinforced composites are presented. The book is comprised of five main fields. Field I deals with research related to sugar palm reinforced composites in various applications including automotive, aeronautics, food packaging and biomedical. Field II provides the material selections, characterisations and advanced development of natural fibre composites, followed by the treatments and modifications of natural fibre and its composites. Field III illustrates a number of recent applications of the bio-products including biosensors, military and green packaging. Plus, Part IV analyses the advanced manufacturing of biocomposites that has been utilized across the globe. The concepts above are applied in Part V to the development of various biocomposite technology related to bio nanocomposites. The research contained in this book is critical for the more efficient use of composites, as detailed up-to-date information is a pre-requirement. The information provides cutting-edge developments to the attention of young investigators to encourage further advances in the field of sugar palm fibres and other allied fibres in composites research.

In the preparation of the book, we have been helped by many colleagues. I wish to thank all the SAPC2021 committee and volunteers, who donate their time and strength in succeeding in this conference and proceedings book from scratch. Last, but not least, I wish to thank the students, undergraduate, graduate, and postdocs from various continents, who have joined our conference and shared their knowledge regarding their research activity with stimulating curiosity. Many comments, exercises, and complements of this book are the direct result of questions and comments that came from them.



# Foreword

Chairman

**Prof. Ir. Dr. Mohd Sapuan Salit**

**Persatuan Pembangunan dan Industri  
Enau Malaysia (PPIEM)**

In The Name of Allah, Most Gracious, Most Merciful.

Assalamualaikum Warahmatullahi Taala Wabarakatuh and Blessings.

All praise be to Allah, The Lord of both the worlds, and Salawat and Salam to Rasullullah ﷺ, the final messenger. I am most grateful to Allah, the most gracious and most merciful, for His blessings in giving us this precious opportunity to gather at this memorable event. International conference on Sugar Palm and Allied Fibre Polymer Composites (SAPC2021) is held for the first time to gather all industrial practitioners, academics, researchers, policy makers, students, administrators and funders, and to exchange ideas and information regarding sugar palm and allied fibre composites. In addition, this international conference also provides a platform to strengthen our relationship in sharing of our research findings with the research community.

On behalf of Society of Sugar Palm Development and Industry Malaysia or Persatuan Pembangunan dan Industri Enau Malaysia (PPIEM), I would like to express my appreciation to all the co-organizers who have worked together in making the long-awaited international conference a success. Furthermore, on behalf of PPIEM, I would also like to thank all the participants from all over the world for making this international conference a success. We feel very honoured because PPIEM has jointly organized this SAPC2021 with 12 organizations from Malaysia and abroad and 150 participants from 28 organizations from 10 countries, including USA, Brazil, Hungary, Czech Republic, Gambia, Nigeria, Pakistan, India, Indonesia, Thailand and Malaysia, have taken part to expand the sugar palm industry in Malaysia and around the world.

Last but not least, once again, I would like to congratulate all the students, participants, paper presenters and committee members for making this conference a success. Hopefully, this conference will be able to drive research and development in the sugar palm and allied fibre composite industry to a more advanced level.

Thank you.

A handwritten signature in black ink, consisting of a stylized 'S' followed by a horizontal line and a small flourish at the end.

Prof. Ir. Dr. Mohd Sapuan Salit

Chairman of Persatuan Pembangunan dan Industri Enau Malaysia (PPIEM)



# Foreword

**Chairman**

**Dr. Ahmad Ilyas Rushdan**

**International Conference on Sugar Palm  
and Allied Fibre Polymer Composite  
(SAPC2021)**

In The Name of Allah, Most Gracious, Most Merciful.

Assalamualaikum Warahmatullahi Taala Wabarakatuh and Blessings.

Alhamdulillah, all praises and gratitude always uttered to Allah, who blesses us, the only one Lord, the Most Merciful and the Most Gracious, and also Salawat and Salam to Rasullullah ﷺ, the final messenger. To be able to participate in this notable occasion is due to His blessings towards us. First and foremost, as a chairman, I take great pride in welcoming all the participants of the International Conference on Sugar Palm and Allied Fibre Polymer Composites (SAPC2021). I am very happy with the successful organization of the international conference, oral presentations and publication of the proceedings book.

The theme of the international conference SAPC2021 is Innovation for Sustainable Advanced Natural Fibre, which also is a step towards achieving our vision in becoming a world-class academic and research institution in order to produce human capital with first class mentality. I am very certain that this occasion will be able to provide a platform for all researchers, students, industry players and policy makers to exchange information, experiences and expertise in the field of sugar palm reinforced composites, natural fibre composites, biocomposite technology, bio-products and advanced manufacturing of biocomposites, hence, strengthening our relationships in knowledge sharing while at the same time provide the necessary thrust in joint research collaborations within the research society.

The concern about utilization of synthetic fibre and its effects on environmental health has brought researchers from various backgrounds working on solving the issue. Thus, green technology and renewable energy based on natural fibres have paved new and innovative ways for a sustainable future. In conclusion, it is my aspiration that this conference will be a foundation for the growth of new ideas related to sugar palm and allied fibre products towards a better tomorrow.

I would like to take this opportunity to thank the organizing committee for their commitment and superb drive in organizing this seminar. I would like also to acknowledge the cooperation of our respective co-organizers; from Malaysia, including Universiti Putra Malaysia, Selangor, and Universiti Teknologi Malaysia, Johor; and from abroad including Universidade Estadual de Londrina from Brazil, Technical University of Liberec from Republic Czech, University of the Gambia from Gambia, Kalasalingam Academy of Research and Education from India, National Textile University from Pakistan and King Mongkut's University of Technology

## *SAPC2021*

### *International Conference on Sugar Palm and Allied Fibre Polymer Composites 2021*

North Bangkok, Thailand. Plus, various organizations from Indonesia, Andalas University, University of Jember, and University of Mataram.

Here, I would also like to thank all the keynote and invited speakers for their insightful talks. Despite the pandemic, SAPC2021 was able to attract more than 88 presenters and 150 registrations in total for both presenters/participants from 10 countries (Malaysia, Pakistan, Indonesia, India, Thailand, Hungary, Brazil, Nigeria, United States and Czech Republic) by maximising the use of online technology and social media.

Thank you.

A handwritten signature in black ink, consisting of a large, stylized initial 'A' followed by a horizontal line and a small flourish.

Dr. Ahmad Ilyas Rushdan

Chairman of International Conference on Sugar Palm and Allied Fibre Polymer Composites (SAPC2021)

**KEYNOTE SPEAKER 1**



**Mohd Sapuan Salit**

Professor of Composite Materials, Faculty of Engineering, Universiti Putra Malaysia

**PROFILE**

**S.M. Sapuan** is a researcher and an A Grade Professor of composite materials at Department of Mechanical and Manufacturing, Universiti Putra Malaysia (UPM) and a Head of Laboratory of Biocomposite Technology, INTROP, UPM. He has a BEng. in Mechanical Engineering from University of Newcastle, Australia, an MSc in Engineering Design from Loughborough University, UK and PhD in Material Engineering from De Montfort University, UK. He is a Professional Engineer, a Society of Automotive Engineers Fellow, an Academy of Science Malaysia Fellow, a Plastic and Rubber Institute Malaysia Fellow, a Malaysian Scientific Association Fellow, an International Biographical Association Fellow and an Institute of Material Malaysia Fellow. He is an Honorary Member and past Vice President of Asian Polymer Association based in IIT Delhi and Founding Chairman and Honorary Member of Society of Sugar Palm Development and Industry, Malaysia. He is the co-editor-in-chief of Functional Composites and Structures, and member of editorial boards of more than two dozen journals. To date he has authored or co-authored more than 1800 publications including over 950 journal papers, 50 books, 175 chapters in book and other publications. He has successfully supervised 89 PhD and 70 MSc students and 15 postdoctoral researchers mainly in the areas of mechanical engineering and composites. He received nine Outstanding Researcher Awards from UPM. He was awarded ISESCO Science Award (Gold Medal) in Technology, Plastic and Rubber Institute Malaysia (PRIM) Fellowship Award and Forest Research Institute Malaysia (FRIM) First Prize Publication Award. He also received the Khwarizimi International Award, SEARCA Regional Professorial Chair award and Kuala Lumpur Royal Rotary Gold Medal Research Award. He also won two National Book Awards. He received the Endeavour Research Promotion Award by Teerthanker Mahaveer University/IEEE India, Citation of Excellence Award from Emerald, UK, the Malaysia's Research Star Award by Elsevier/Ministry of Education Malaysia and Publons Peer Review Award, from Publons, USA. Later, he received a Professor of Eminence Award from Aligarh Muslim University, India, Top Research Scientists' Malaysia Award from Academy of Science Malaysia, Gold in Invention and Innovation Awards, Malaysia Technology Expo and PERINTIS Publication Award from PERINTIS, Malaysia. He was listed among the World Top 2% Scientists by Stanford University, USA.

**ABSTRACT**

**RECENT DEVELOPMENT IN SUGAR PALM COMPOSITES**

Sugar palm (*Arenga pinnata* [wurmb. Meer]) is a plant mainly found in some Tropical regions such as Indonesia, Malaysia, India and Thailand. This tree is known as multipurpose tree because all parts of the tree can be utilized such as fibres for making roof, ropes, broom, and reinforcement for composites, starch for making flour and biopolymers, trunks for furniture and utensils, fruits for delicacy, etc. In the past, the fibres were used as ship cordage, roof for building, ropes in construction and for boat building. However, some unique properties of fibres are the major factors in using them as reinforcements in composites after bonding them with suitable polymer matrices. It was reported that the longer the fibres are soaked in the water, the better was their mechanical performance. Sugar palm fibre composites are reported to be used making different products such as boats, tables and chairs, solar panels, automotive components and food packaging. In the past, sugar palm fibres were used as reinforcements in synthetic polymers such as epoxy, polypropylene and used in structural and semi-structural applications. In the recent years, nanocellulose fibres made from sugar palm were developed and bonded with sugar palm starch biopolymers to produce bionanocellulose reinforced starch biopolymer composites mainly used for food packaging. The work is extended by incorporating essential oils to enhance antibacterial properties of the sugar palm composites.

KEYNOTE SPEAKER 2



**Tahir Dahlang**

Department of Physics, Hasanuddin University, Makassar 90245, Indonesia.

**PROFILE**

**Dahlang Tahir**, Ph. D, full Professor in Physics Department, Hasanuddin University, Makassar, Indonesia. Ph. D in 2010 at Physics Department, Chungbuk National University (CBNU), South Korea. Visiting scientist 2011 in Southern Denmark University, Denmark. The research interest covers lightweight materials, carbon based multifunctional composites and semiconductor and published 126 papers and 2 patent.

**ABSTRACT**

**STRUCTURAL AND MECHANICAL PROPERTIES OF BIOPLASTICS HYBRID BASED FIBBER BETWEEN PALM SUGAR FIBER (PSF)/PINEAPPLE LEAF FIBER (PLF) MODEL**

Plastic changes the world, and it is an amazing material. Plastic has many benefits which it is used every day and everywhere in this world. But with varied uses and benefits, plastic disposal has the threatening the environment. Biodegradable plastic may fulfil this purpose and may easily to reduce in environment. The problem of bioplastics is very low mechanical properties. In this study, we are developing new bioplastics with high mechanical performance using bioplastics cassava starch/chitosan/ ZnO with additional hybrid fibre using palm sugar fibre (PSF)/pineapple leaf fibre (PLF) as a function of ZnO concentration. The structural properties of composites were quantitative analysis from the X-ray diffraction (XRD) spectra. The values of the tensile strength, young's modulus, and elongation at break show that the highest for 10% ZnO – hybrid fibre bioplastic but for increasing up to 16% concentration of ZnO shows decrease sharply probably due to the rearrangement of interfacial bonding between ZnO and matrix. The bioplastics cassava starch/chitosan/ ZnO with additional hybrid fibre using PSF/PLF can be completely decomposed in ordinary soil and in seawater for less than 28 days. The bioplastics shows very good used for packaging of sliced bread with no fungal growth for 30 days. The results show that ZnO has a big role for antibacterial properties and hybrid fibre PSF/PLF for physical and mechanical properties of composite bioplastics starch/chitosan/ZnO/ hybrid fibre PSF/PLF.

**KEYNOTE SPEAKER 3**



**Béla Pukánszky**

Professor, Budapest University of Technology and Economics, Department of Physical Chemistry and Material Science, Laboratory of Plastics and Rubber Technology

**PROFILE**

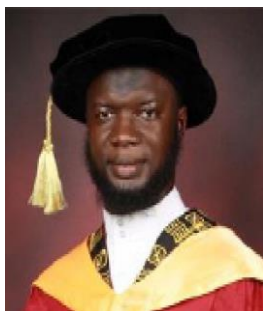
**Prof. Béla Pukánszky** graduated from the technical University of Budapest (Hungary) in chemical engineering, where he also received his PhD in 1976. From 1973 to 1978 he worked as a researcher at the Industrial Research Institute for Organic Chemistry (Hungary) and from 1978 he joined the Central Research Institute for Chemistry of the Hungarian Academy of Sciences (HAS) as an assistant scientific co-worker, latter scientific co-worker and finally as the head of a Department. In 1993, he was invited to the Technical University of Budapest where he became the head of the Department of Plastics and Rubber Technology. Latter the Department was merged with the Department of Physical Chemistry to create the Department of Physical Chemistry and Materials Science and he was the head of the merged unit for more than 10 years. He is still employed also at the research institute of HAS. He has produced more than 330 scientific publications, book chapters and several patents. His papers have been cited more than 8400 times and he has an *h* index of 55. Prof. Pukánszky is the member of numerous committees, is on the editorial panels of several scientific journals, and he is the full member of the Hungarian Academy of Sciences. He organized several international conferences with great success in the past; the last one was the fourth International Conference on Bio-based Polymers and Composites in 2018. He has many industrial contacts, and has been consulting international and Hungarian companies like Borealis, General Electric, Clariant, TVK, etc.

**ABSTRACT**

**ENGINEERING AND BIOCOMPOSITES FROM SUGAR PALM AND ALLIED FIBRES**

Polymers are often used in structural applications. Polymers are frequently modified in various ways including blending and the addition of particulate fillers or fibres. Because of environmental concerns, but also financial considerations, polymers are reinforced with natural fibres in increasing quantities. Sugar palm and allied fibres are potential reinforcements for commodity and biopolymers, but numerous factors must be considered before their applications, most of which are discussed in the presentation. Natural fibres are usually quite large and their aspect ratio varies in a wide range results in easy debonding of the matrix and the fibre leading to inferior strength. Moreover, the attrition of large particles takes place during the processing of the composite and the size as well as size distribution of fibres differ considerably from the initial values. The extent of reinforcement is determined by several factors including fiber length, aspect ratio and interfacial adhesion. This latter can be modified by coupling, by the use of functionalized polymers, maleated PE or PP in polyolefins and maleated PLA in poly (lactic acid). Coupling may increase composite strength, but even the application of the coupling agents mentioned above have some limitation. Accordingly, the estimation of interfacial adhesion is essential in the determination of the reinforcing effect of natural fibres and the efficiency of coupling. Several processes, including debonding, fibre fracture and fibre pull out may take place in natural fibre reinforced polymers and their analysis is essential for the optimization of composite properties. A crucial characteristic of structural materials including composites is impact resistance. However, the impact strength of natural fibre reinforced composites is usually quite small and cannot be improved by the usual way, the addition of elastomers. Hybridization results in considerable increase in impact resistance and stiffness.

**KEYNOTE SPEAKER 4**



**M.L. Sanyang**

Directorate of Research and Consultancy, University of The Gambia, Kanifing Campus, The Gambia

**PROFILE**

**Muhammed Lamin Sanyang** is currently appointed as Director of Research at the University of The Gambia (UTG). He has 12 years accumulative Professional working experience in the field of academic research and consultancy. Dr. Sanyang has published 16 Journal articles, 3 edited books, 10 International Conference papers and 10 book chapters on biopolymer and bio-composite related works. He also successfully completed 24 consultancy projects within the last 5 years. His research interest areas are Bio based materials, Environmental Engineering and Management. Dr. Sanyang pursued his Bachelor's Degree (BSc) in Petroleum Engineering at the National Taipei University of Technology (Taiwan). He later proceeded to complete his Master's (MSc) and Doctorate (PhD) degree in Environmental and Green Engineering, respectively at Universiti Putra Malaysia (UPM). He served as a post-doctoral fellow under the Laboratory of Biocomposite Technology at UPM for one year (Jun. 2016 -Jul. 2017). He also served as reviewer for many international journals such as Polymer Composite, BioResources, Polymers (MPDI), Polymer Bulletin, International Journal of Polymer Science, Current Analytical Chemistry etc. Dr. Sanyang has received numerous academic awards including the "Best PhD student award" for his outstanding researches and publications.

**ABSTRACT**

**ADVANCED GREEN MATERIALS: SUGAR PALM BIOPOLYMER BASED BIOCOSMOSITES**

Sugar palm starch like most other biopolymers is hydrophilic in nature due to either their hydroxyl or polar groups. The major challenges for the development of starches as packaging films are the shortcomings related to brittleness, processability, high moisture sensitivity, quick retro gradation, poor mechanical and barrier properties. In order to transform native sugar palm starch into high performance thermoplastic starch for packaging application, the aforementioned drawbacks should be addressed. Some of the approaches employed to tackle these roadblocks are; (1) the addition of different types and concentrations of plasticizer(s) into the starch matrix, (2) the combination of sugar palm starch with other polymers that possess better functional properties, and (3) the incorporation of cellulose fibres. The implementation of such modifications on the sugar palm starch contributes to the improvement of its functional properties as well as help in optimizing its full potential as an effective food packaging material.

**KEYNOTE SPEAKER 5**



**Nor Salwa Binti Hamdan**

Department of Skills Development, The Ministry of Human Resources Malaysia

**PROFILE**

**H.N. Salwa** was born on October 11, 1977, in Pahang, Malaysia, and is currently a Senior Assistant Director at the Human Resources Ministry Malaysia's Department of Skills. She received her bachelor's degree in Electrical/Electronic Engineering from Universiti Tenaga Nasional (UNITEN) in Kajang, Selangor, Malaysia, in 2000. She has worked as a Vocational Training Officer in public vocational institutions since 2002, initially at Institut Kemahiran Tinggi Belia Negara (IKTBN) Sepang. Later, she was assigned to the IKTBN/IKBN's Head Quarters at the Ministry of Youth and Sports Malaysia. In 2011, she was awarded a full scholarship by the Malaysia Public Service Department (JPA) to pursue her Masters of Science in Product Design Innovation at Aston University in Birmingham, United Kingdom, where she graduated in 2013. In September 2017, she was granted a full scholarship from JPA to pursue a PhD at the Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia, Malaysia. During her studies, she published three Q2, two Q3, and a scopus-indexed paper, as well as three chapters in three different books. She has now reporting back at work and has been assigned to the Ministry of Human Resources' Department of Skills Development

**ABSTRACT**

**LIFE CYCLE ASSESSMENT (LCA) OF SUGAR PALM: PLANTATION TO PRODUCT**

The depletion of non-renewable sources of raw material, i.e., petroleum-based polymer, initiates demand for a more environmentally friendly product, from conventional material to biopolymers. The bio-based packaging material is one of the alternatives to replace the synthetic plastic market. Sugar palm starch and sugar palm fibre are amongst alternative raw materials utilized in bio-based material to reduce petroleum-based food packaging material usage. The utilization of sugar palm-based biopolymer for the formation of packaging products is advantageous because they are biodegradable, environmentally friendly polymers and originated from renewable resources. Moreover, there is an increasing global environmental awareness and growing demand for cleaner and sustainable products and technologies due to environmental issues. For that reason, there is a necessity to understand and manage the assessment of a production process which includes raw material extraction to disposal phase. Life cycle assessment (LCA) is a technique to determine the overall potential of environmental impacts of a product established in the 1970s. This paper reports a life cycle assessment (LCA) analysis of a proposed natural fibre-reinforced biopolymer composite takeout food container. LCA can provide a holistic overview of the environmental impact whereby it covers the whole life cycle of a product from raw material extraction to the end-of-life stage. This paper focuses on the damage assessment of the entire product system, including disposal scenarios of the thermoformed sugar palm fibre (SPF)-reinforced sago starch composite takeout food container.

**KEYNOTE SPEAKER 6**



**Isma'ila Mukhtar**

Kano University of Science and Technology, Nigeria

**PROFILE**

**Engr. Dr. Isma'ila Mukhtar** was born in Yakasai quarters, Kano State of Nigeria on 6 th November 1977. Currently, he is a lecturer in the department of Mechanical Engineering, Kano University of Science and Technology, Wudil, Nigeria. He is a registered member of council for the regulation of engineering in Nigeria (R56552). He obtained his national diploma (ND) and higher national diploma (HND) in Mechanical Engineering from Kano State Polytechnic and Kaduna Polytechnic in the year 2000 and 2003 respectively. In 2008, he obtained his postgraduate diploma in Mechanical Engineering from Bayero University Kano. He later joined Universiti Putra Malaysia for his master's degree in Manufacturing Systems Engineering and graduated in May 2011. He joined the Faculty of Engineering, Universiti Putra Malaysia in February 2016 for his Doctor of Philosophy (PhD) in Materials Engineering and graduated in September 2018. His research focused on natural and synthetic fibre reinforced polymer hybrid composite for use as a structural component in an automobile. He published numerous journal articles and book chapters and presented in both national and international conferences.

**ABSTRACT**

**POTENTIAL OF SUGAR PALM FIBRE AND ALLIED FIBRE COMPOSITES FOR ADVANCED GREEN AUTOMOTIVE PARTS**

Light-weight and high-performance materials are crucial in an automotive engineering. This is due to the numerous benefits such as low fuel consumption, cost savings and weight reduction. Therefore, replacement of steel is necessary to achieve weight reduction especially for hybrid and electric vehicle as well as improvement in energy absorption. This could be achieved with light-weight material such as natural fibre composites and its hybrid composites. The trend of using natural fibre composite for automotive production is fast growing. Numerous findings on the use of sugar palm fibre and allied fibre composites for advanced green automotive parts were reported in literature. Though, the dominant use of natural fibre composites by far is in the interior parts, with few exterior composite automotive parts. But with newer production techniques and hybridization, structural and semi structural composites automotive parts are finding its way into automotive design. This short presentation will address the following questions. Where are the possible areas of the use of natural fibre composite parts in automobile? Can natural fibre composites serve as material for automobile structural components? What are the economic benefits of using natural fibre composites? This and many more will be highlighted and in addition, recent use of sugar palm fibre and allied fibre composites in automotive industry will be discussed and future trends will be outlined.

**KEYNOTE SPEAKER 7**



**Indrayana**

Palmasia Indo Perkasa & Sumber Mulya, Indonesia.

**PROFILE**

**Mr. Indrayana** is the owner of Palmasia Indo Perkasa, Indonesia (Export Company) and Sumber Mulya Perkasa, Indonesia (Local Company). He had finished his degree in management course at Fakultas Ekonomi, Universitas Kuningan, Jawa Barat, Indonesia. Currently, he is the founder and treasurer of Asosiasi Aren Indonesia (AAI), acts as founder and active member of Komunitas Aren Indonesia (KAI) and Masyarakat Aren Indonesia (MKI). He has 20 years of experience in the palm business. Palm exports were once made to Germany, China, and the Middle East. In addition, palm oil for brushes, roofing, filters, and brooms was exported to the Middle East (for roofing).

**ABSTRACT**

**THE DEVELOPMENT AND EXPANSION OF SUGAR PALM PRODUCTS IN INDONESIA**

Sugar palm fiber, probably the only black natural fiber in this world, has been cultivated and utilized widely in Indonesia. It has many advantages, maybe this is why they were the most widely used natural fibers by our ancestors at the ancient times, but because in the past the needs of human dressings were not as much as in modern times, so its use is still very simple in accordance with the needs of the modern era. Therefore, in this modern era, natural fibers, which have many advantages from their properties and characteristics, can be further developed for their use so that the fiber is more economically useful or aesthetically sustainable. However, because there is still a lack of literature, research and development of this black fiber, it is felt that many of its uses are not in accordance with the advantages of this fiber. After observing that these black fibers have different diameter sizes, namely fine, medium, coarse, and large like a stick, from the separation of these fibers they have different characters and properties, so that even more precise uses can be different. Thus, the processing technique must be refined again in order to maximize its benefits.

**INTERNATIONAL CONFERENCE ON SUGAR PALM AND ALLIED FIBRE POLYMER COMPOSITES 2021  
(SAPC2021)**

Time	Event						
0800-0835	Session Login & Preparation						
0835-0845	Welcoming Speech						
0845-0855	1. Dua recitation by Mr. Shah Faisal Khan Sherwani						
0855-0905	Opening Address and Officiation						
0905-0915	2. Welcoming speech by Chairman of SAPC2021						
0915-0920	<b>Dr. Ahmad Ilyas Bin Rushdan, Universiti Teknologi Malaysia, (UTM), Malaysia</b>						
0920-0930	3. Opening speech by President of PPIEM						
	<b>Prof. Ir. Dr. Mohd Sapuan Salit, Universiti Putra Malaysia (UPM), Malaysia</b>						
	4. Video montage presentation						
	5. Photo session						
0930-1000	<b>Chair:</b> Dr. Nuzaimah bte Mustafa	<b>Keynote Speaker 1 (SAPC2021: PR82): Prof. Dr. Tahir Dahlang, Universitas Hasanuddin, Indonesia</b>					
1000-1030		Title: Structural and Mechanical Properties of Bioplastics Hybrid Based Fiber between Palm Sugar Fiber (PSF)/Pineapple Leaf Fiber (PLF) Model					
		<b>Keynote Speaker 2 (SAPC2021: P08): Prof. Ir. Dr. Mohd Sapuan Salit, Universiti Putra Malaysia (UPM), Malaysia</b>					
		Title: Recent Development in Sugar Palm Composites					
1030	Session Login & Preparation						
Tracks	Sugar Palm Fibre Reinforced Composites & Advanced Manufacturing of Biocomposites	Natural Fibre Composites	Biocomposite Technology	Advanced Manufacturing of Biocomposites	Bio-Products	Sugar Palm & Bio-Products	
	<b>Room 1</b>	<b>Room 2</b>	<b>Room 3</b>	<b>Room 4</b>	<b>Room 5</b>	<b>Room 6</b>	
Chair	Dr. Mohd Nor Faiz Norrahim, UPNM, Malaysia	Dr. Noor Afeefah Nordin, UNITEN, Malaysia	Dr. Mohd Nurazzi bin Norizan, UPNM, Malaysia	Dr. Fatimah Atiyaah bt Sabaruddin, UPM, Malaysia	Ts. Azrena Abdul Karim, UiTM, Malaysia	Dr. Nazatul Asikin Muda, UiTM, Malaysia	
	<b>Invited Speaker</b>						

# SAPC2021

## International Conference on Sugar Palm and Allied Fibre Polymer Composites 2021

1030-1050		SAPC 2021: PR23 Dr. Mohd Nor Faiz Norrahim, UPM, Malaysia	SAPC 2021: PR78 Dr. Sanjay Mavinkere Rangappa, King Mongkut's University of Technology North Bangkok, Thailand	SAPC 2021: PR77 Prof. Dr. Khalina Abdan, UPM, Malaysia	SAPC2021: P45 Assoc. Prof. Dr. Edi Syams bin Zainudin, UPM Malaysia	SAPC2021: PR12 Assoc. Prof. Ts. Dr. Shukur Bin Hj. Abu Hassan, UTM, Malaysia	SAPC2021:PR70 Dr. Ir. Mochamad Asrofi, University of Jember, Indonesia
1050-1105		SAPC2021: PR22 Prof. Ts. Dr. Asma Awal, UiTM, Malaysia	SAPC2021: PR46	SAPC2021:PR73 Assoc. Prof. Dr. Yasir Nawab, National Textile University, Faisalabad, Pakistan	SAPC2021: PR03	SAPC2021: PR05	SAPC2021: PR02 Dr. Ahmad Ilyas Rushdan, UTM, Malaysia
1105-1120		SAPC2021: PR64	SAPC2021: PR08	SAPC2021: PR50	SAPC2021: PR06	SAPC2021: PR33	SAPC2021: PR48
1120-1135		SAPC2021: PR39	SAPC2021: PR31	SAPC2021: PR59	SAPC2021: PR10	SAPC2021: PR16	SAPC2021: PR71
1135-1150		SAPC2021: PR43	SAPC2021: PR14	SAPC2021: PR60	SAPC2021: PR11	SAPC2021: PR04	SAPC2021: PR25
1150-1205		SAPC2021: PR41	SAPC2021: PR15	SAPC2021: PR45	SAPC2021: PR28	SAPC2021: PR29	SAPC2021: PR13
1205-1220		SAPC2021: PR69	SAPC2021: PR44	SAPC2021: PR47	SAPC2021: PR38	SAPC2021: PR74	SAPC2021: PR09
1220-1250	<b>Chair:</b> Ts. Dr. Mohd Adrinata bin Shaharuzaman	<b>Keynote Speaker 3 (SAPC2021: PR83): Prof. Dr. Béla Pukaňszky</b> , Budapest University of Technology and Economics, Hungary					
		Title: Engineering and Biocomposites from Sugar Palm and Allied Fibers					
1250-1350	Break						
1350-1420	<b>Chair:</b> Ts. Dr. Mohd Adrinata bin Shaharuzaman	<b>Keynote Speaker 4 (SAPC2021: PR75): Dr. Muhammad Lamin Sanyang</b> , University of the Gambia, Gambia					
		Title: Advance Green Materials: Sugar Palm Biopolymer Based Biocomposites					
1420	Session Login & Preparation						
Tracks		Sugar Palm Fibre Reinforced Composites & Advanced Manufacturing of Biocomposites	Natural Fibre Composites	Biocomposite Technology	Advanced Manufacturing of Biocomposites	Bio-Products	Sugar Palm & Bio-Products
		<b>Room 1</b>	<b>Room 2</b>	<b>Room 3</b>	<b>Room 4</b>	<b>Room 5</b>	<b>Room 6</b>
Chair		Dr. Aisyah Humaira Alias, UPM, Malaysia	Dr. Nadlene Razali, UTEM, Malaysia	Ts. Dr. Ridzuan Mansor, UTEM, Malaysia	Dr. Mohd Shaiful Zaidi Bin Mat Desa, UMP, Malaysia	Dr. Siti Hasnah Kamaruddin, UiTM Malaysia	Assoc. Prof Dr Siti Norasmah Surip, UiTM, Malaysia
		<b>Invited Speaker</b>					

# SAPC2021

## International Conference on Sugar Palm and Allied Fibre Polymer Composites 2021

1420-1440		SAPC2021:PR09 Prof. Dr. Nasmi Herlina Sari, University of Mataram, Indonesia	SAPC2021:PR79 Prof. Dr.-Ing Hairul Abral, Universitas Andalas, Indonesia	SAPC2021: PR30 Assoc. Prof. Dr. T. Senthil Muthu Kumar, Kalasalingam Academy of Research and Education, India	SAPC2021:PR80 Prof. Dr. Michal Petru, Michal Petru, Liberec, Czech Republic	SAPC2021: PR53 Dr. Jéssica Fernanda Pereira, State University of Londrina, Brazil	SAPC2021:P12 Dr. Ainun Zuriyati Mohamed, UPM, Malaysia
1440-1455		SAPC2021: PR62	SAPC2021: PR27	SAPC2021: PR40	SAPC2021: PR24	SAPC2021: PR34	SAPC2021: PR72 Assoc. Prof. Dr. Khubab Shaker, National Textile University, Pakistan
1455-1510		SAPC2021: PR63	SAPC2021: PR57	SAPC2021: PR01	SAPC2021: PR26	SAPC2021: PR42	SAPC2021: PR07
1510-1525		SAPC2021: PR65	SAPC2021: PR32	SAPC2021: PR49	SAPC2021: PR17	SAPC2021: PR58	SAPC2021: PR19
1525-1540		SAPC2021: PR68	SAPC2021: PR36	SAPC2021: PR55	SAPC2021: PR37	SAPC2021: PR54	SAPC2021: PR52
1540-1555		SAPC2021: P34	SAPC2021: PR56	SAPC2021: PR51	SAPC2021: P19	SAPC2021: PR61	SAPC2021: PR18
1555-1610		SAPC2021: PR66	SAPC2021: PR67	SAPC2021: P10			
1610-1640	Chair: Assoc. Prof. Dr. Zulkiflle b. Leman	<b>Keynote Speaker 5 (SAPC2021: PR76): Engr. Dr Isma'lla Mukhtar</b> , Kano University of Science and Technology, Nigeria					
		Title: Potential of Sugar Palm Fibre and Allied Fibres for Advanced Green Composites Automotive Parts					
1640-1710		<b>Keynote Speaker 6 (SAPC2021: PR81): Dr. Nor Salwa Hamdan</b> , Jabatan Pembangunan Kemahiran, Kementerian Sumber Manusia (MOHR), Malaysia					
		Title: Life Cycle Assessment (LCA) of Sugar Palm : Plantation to Product					
1710-1740	Emcee: Dr. Mastura binti Taha/ Dr. Noryani binti Muhammad	<b>Keynote Speaker 7 (SAPC2021: PR84): Mr. Indrayana</b> , Palmasia Indo Perkasa & Sumber Mulya Perkasa, Indonesia					
		Title: The Development and Expansion of Sugar Palm Products In Indonesia					
1740-1800		Closing ceremony					
		1. Closing speech by Co-chairman of SAPC2021, <b>Assoc. Prof. Dr. Edi Syams bin Zainuddin (UPM), Malaysia</b>					
		2. Awarding ceremony					
1800	End						



**ADVISORY SCIENTIFIC COMMITTEE (International)**  
**INTERNATIONAL CONFERENCE ON SUGAR PALM AND ALLIED FIBRE**  
**POLYMER COMPOSITES 2021 (SAPC2021)**

**PERSATUAN PEMBANGUNAN DAN INDUSTRI ENAM MALAYSIA**

D/A: Jabatan Kejuruteraan Mekanikal dan Pembuatan,  
Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia.

Tel: 03-89466318

No. Pendaftaran: PPM-003-10-20062017

International:

1. PROF. DR. -ING. HABIL SUCHART SIENGCHIN  
King Mongkut's University of Technology North Bangkok, THAILAND.
2. PROF. DR. MICHAL PETRU  
Technical University of Liberec, THE CZECH REPUBLIC.
3. PROF. DR. SUZANA MALI  
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# **FULL PAPERS**

**KEYNOTE SPEAKER 1**  
**RECENT DEVELOPMENT IN SUGAR PALM COMPOSITES**

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**ABSTRACT**

Sugar palm (*Arenga pinnata* [wurmb. Meer]) is a plant mainly found in some Tropical regions such as Indonesia, Malaysia, India and Thailand. This tree is known as multipurpose tree because all parts of the tree can be utilized such as fibres for making roof, ropes, broom, and reinforcement for composites, starch for making flour and biopolymers, trunks for furniture and utensils, fruits for delicacy, etc. In the past, the fibres were used as ship cordage, roof for building, ropes in construction and for boat building. However, some unique properties of fibres are the major factors in using them as reinforcements in composites after bonding them with suitable polymer matrices. It was reported that the longer the fibres are soaked in the water, the better was their mechanical performance. Sugar palm fibre composites are reported to be used making different products such as boats, tables and chairs, solar panels, automotive components and food packaging. In the past, sugar palm fibres were used as reinforcements in synthetic polymers such as epoxy, polypropylene and used in structural and semi-structural applications. In the recent years, nanocellulose fibres made from sugar palm were developed and bonded with sugar palm starch biopolymers to produce bionanocellulose reinforced starch biopolymer composites mainly used for food packaging. The work is extended by incorporating essential oils to enhance antibacterial properties of the sugar palm composites.

*Keywords:* composite, natural fibre, sugar palm

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**KEYNOTE SPEAKER 2**  
**STRUCTURAL AND MECHANICAL PROPERTIES OF BIOPLASTICS HYBRID**  
**BASED FIBBER BETWEEN PALM SUGAR FIBER (PSF)/PINEAPPLE LEAF**  
**FIBER (PLF) MODEL**

Tahir Dahlang\*

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**ABSTRACT**

Plastic changes the world, and it is an amazing material. Plastic has many benefits which it is used every day and everywhere in this world. But with varied uses and benefits, plastic disposal has the threatening the environment. Biodegradable plastic may fulfil this purpose and may easily to reduce in environment. The problem of bioplastics is very low mechanical properties. In this study, we are developing new bioplastics with high mechanical performance using bioplastics cassava starch/chitosan/ZnO with additional hybrid fibre using palm sugar fibre (PSF)/pineapple leaf fibre (PLF) as a function of ZnO concentration. The structural properties of composites were quantitative analysis from the X-ray diffraction (XRD) spectra. The values of the tensile strength, young's modulus, and elongation at break show that the highest for 10% ZnO – hybrid fibre bioplastic but for increasing up to 16% concentration of ZnO shows decrease sharply probably due to the rearrangement of interfacial bonding between ZnO and matrix. The bioplastics cassava starch/chitosan/ ZnO with additional hybrid fibre using PSF/PLF can be completely decomposed in ordinary soil and in seawater for less than 28 days. The bioplastics shows very good used for packaging of sliced bread with no fungal growth for 30 days. The results show that ZnO has a big role for antibacterial properties and hybrid fibre PSF/PLF for physical and mechanical properties of composite bioplastics starch/chitosan/ZnO/ hybrid fibre PSF/PLF.

*Keywords:* Hybrid fibre, palm sugar fibre, pineapple leaf fibre, chitosan.

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**KEYNOTE SPEAKER 3**  
**ENGINEERING AND BIOCOMPOSITES FROM SUGAR PALM AND ALLIED FIBRES**

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**ABSTRACT**

Polymers are often used in structural applications. Polymers are frequently modified in various ways including blending and the addition of particulate fillers or fibres. Because of environmental concerns, but also financial considerations, polymers are reinforced with natural fibres in increasing quantities. Sugar palm and allied fibres are potential reinforcements for commodity and biopolymers, but numerous factors must be considered before their applications, most of which are discussed in the presentation. Natural fibres are usually quite large and their aspect ratio varies in a wide range results in easy debonding of the matrix and the fibre leading to inferior strength. Moreover, the attrition of large particles takes place during the processing of the composite and the size as well as size distribution of fibres differ considerably from the initial values. The extent of reinforcement is determined by several factors including fiber length, aspect ratio and interfacial adhesion. This latter can be modified by coupling, by the use of functionalized polymers, maleated PE or PP in polyolefins and maleated PLA in poly (lactic acid). Coupling may increase composite strength, but even the application of the coupling agents mentioned above have some limitation. Accordingly, the estimation of interfacial adhesion is essential in the determination of the reinforcing effect of natural fibres and the efficiency of coupling. Several processes, including debonding, fibre fracture and fibre pull out may take place in natural fibre reinforced polymers and their analysis is essential for the optimization of composite properties. A crucial characteristic of structural materials including composites is impact resistance. However, the impact strength of natural fibre reinforced composites is usually quite small and cannot be improved by the usual way, the addition of elastomers. Hybridization results in considerable increase in impact resistance and stiffness.

*Keywords:* Biocomposites, sugar palm, allied fibre.

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**KEYNOTE SPEAKER 4**  
**ADVANCED GREEN MATERIALS: SUGAR PALM BIOPOLYMER BASED**  
**BIOCOMPOSITES**

M. L. Sanyang\*

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**ABSTRACT**

Sugar palm starch like most other biopolymers is hydrophilic in nature due to either their hydroxyl or polar groups. The major challenges for the development of starches as packaging films are the shortcomings related to brittleness, processability, high moisture sensitivity, quick retro gradation, poor mechanical and barrier properties. In order to transform native sugar palm starch into high performance thermoplastic starch for packaging application, the aforementioned drawbacks should be addressed. Some of the approaches employed to tackle these roadblocks are; (1) the addition of different types and concentrations of plasticizer(s) into the starch matrix, (2) the combination of sugar palm starch with other polymers that possess better functional properties, and (3) the incorporation of cellulose fibres. The implementation of such modifications on the sugar palm starch contributes to the improvement of its functional properties as well as help in optimizing its full potential as an effective food packaging material.

*Keywords:* Composite, natural fibre, biocomposites, sugar palm biopolymer.

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**KEYNOTE SPEAKER 5**  
**LIFE CYCLE ASSESSMENT (LCA) OF SUGAR PALM: PLANTATION TO PRODUCT**

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**ABSTRACT**

The depletion of non-renewable sources of raw material, i.e., petroleum-based polymer, initiates demand for a more environmentally friendly product, from conventional material to biopolymers. The bio-based packaging material is one of the alternatives to replace the synthetic plastic market. Sugar palm starch and sugar palm fibre are amongst alternative raw materials utilized in bio-based material to reduce petroleum-based food packaging material usage. The utilization of sugar palm-based biopolymer for the formation of packaging products is advantageous because they are biodegradable, environmentally friendly polymers and originated from renewable resources. Moreover, there is an increasing global environmental awareness and growing demand for cleaner and sustainable products and technologies due to environmental issues. For that reason, there is a necessity to understand and manage the assessment of a production process which includes raw material extraction to disposal phase. Life cycle assessment (LCA) is a technique to determine the overall potential of environmental impacts of a product established in the 1970s. This paper reports a life cycle assessment (LCA) analysis of a proposed natural fibre-reinforced biopolymer composite takeout food container. LCA can provide a holistic overview of the environmental impact whereby it covers the whole life cycle of a product from raw material extraction to the end-of-life stage. This paper focuses on the damage assessment of the entire product system, including disposal scenarios of the thermoformed sugar palm fibre (SPF)-reinforced sago starch composite takeout food container.

*Keywords:* LCA, sugar palm plantation, SPF.

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**KEYNOTE SPEAKER 6**  
**POTENTIAL OF SUGAR PALM FIBRE AND ALLIED FIBRE COMPOSITES FOR**  
**ADVANCED GREEN AUTOMOTIVE PARTS**

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*Kano University of Science and Technology, Nigeria.*

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**ABSTRACT**

Light-weight and high-performance materials are crucial in an automotive engineering. This is due to the numerous benefits such as low fuel consumption, cost savings and weight reduction. Therefore, replacement of steel is necessary to achieve weight reduction especially for hybrid and electric vehicle as well as improvement in energy absorption. This could be achieved with light-weight material such as natural fibre composites and its hybrid composites. The trend of using natural fibre composite for automotive production is fast growing. Numerous findings on the use of sugar palm fibre and allied fibre composites for advanced green automotive parts were reported in literature. Though, the dominant use of natural fibre composites by far is in the interior parts, with few exterior composite automotive parts. But with newer production techniques and hybridization, structural and semi structural composites automotive parts are finding its way into automotive design. This short presentation will address the following questions. Where are the possible areas of the use of natural fibre composite parts in automobile? Can natural fibre composites serve as material for automobile structural components? What are the economic benefits of using natural fibre composites? This and many more will be highlighted and in addition, recent use of sugar palm fibre and allied fibre composites in automotive industry will be discussed and future trends will be outlined.

*Keywords:* Sugar palm fibre, allied fibre, composites, green, automotive.

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**KEYNOTE SPEAKER 7**  
**THE DEVELOPMENT AND EXPANSION OF SUGAR PALM PRODUCTS IN**  
**INDONESIA**

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*Palmasia Indo Perkasa & Sumber Mulya, Indonesia.*

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**ABSTRACT**

Sugar palm fiber, probably the only black natural fiber in this world, has been cultivated and utilized widely in Indonesia. It has many advantages, maybe this is why they were the most widely used natural fibers by our ancestors at the ancient times, but because in the past the needs of human dressings were not as much as in modern times, so its use is still very simple in accordance with the needs of the modern era. Therefore, in this modern era, natural fibers, which have many advantages from their properties and characteristics, can be further developed for their use so that the fiber is more economically useful or aesthetically sustainable. However, because there is still a lack of literature, research and development of this black fiber, it is felt that many of its uses are not in accordance with the advantages of this fiber. After observing that these black fibers have different diameter sizes, namely fine, medium, coarse, and large like a stick, from the separation of these fibers they have different characters and properties, so that even more precise uses can be different. Thus, the processing technique must be refined again in order to maximize its benefits.

*Keywords:* Development, sugar pam, products, commercialization.

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## INVITED SPEAKER 1

## NANOCELLULOSE THE NEXT SUPER VERSATILE MATERIAL FOR THE MILITARY

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## ABSTRACT

Military systems have become more complex, and the development of future advanced materials for defence applications have received much attention. Nanocellulose has been identified as a 'super versatile material' that will become a replacement material for many applications including in the military. Nanocellulose can be synthesized from discarded fibers that are left over from forestry and agricultural waste that can be transformed into high-value products. Thanks to its interesting properties, comprising low thermal expansion coefficient, high aspect ratio, highly crystalline, good mechanical and optical properties, nanocellulose has emerged as a new material class for high end military products such as bulletproof vests, fire-retardant materials, as a component of propellants, filtration material, and in textile, electronic and energy products. Therefore, in this review, current and future applications of nanocellulose in the military are critically discussed. This review is intended to impart to readers some of the excitement that currently surrounds nanocellulose research, it's fascinating chemical and physical properties and applications in the military.

*Keywords:* Nanocellulose, super versatile material, military, applications.

## INTRODUCTION

Global military expenditure sees largest annual increase in a decade. According to new data from the Stockholm International Peace Research Institute (SIPRI), the total global military expenditure rose to \$ 1917 billion in year 2019 which increased of 3.6 % from year 2018.[1] This is include a large spend on research and development (R&D) in the military. The R&D creates the foundation for new and improved technologies that underpin a wide range of applications. These applications include advanced materials for several purposes such developments on pharmaceuticals and medical treatments, combat corrosion and microbial contamination, aircraft, weapons, preserve food supplies for the army and work on portable energy sources and electronic to military equipment. The R&D in materials and processes will be required to improve existing materials and achieve breakthroughs in new materials and combinations. There are 13 criteria of properties needed for the military are as follows:[2]

- *Lightweight materials that provide equivalent functionality.*
- *Materials that enhance protection and survivability.*
- *Stealth materials.*
- *Electronic and photonic materials for high-speed communications.*
- *Sensor and actuator materials.*
- *High-energy-density materials.*
- *Materials that improve propulsion technology.*
- *Multifunctionality.*
- *Self-healing and self-diagnosing.*
- *Low total system cost.*
- *Low maintenance.*
- *High reliability.*
- *Environmental acceptability.*

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The newly developed technology of the material in military needs those properties, alone or in combination. Interestingly, nanocellulose could be the best candidate to meet those criteria. Nanocellulose materials have therefore emerged as promising materials and received tremendous demand to be used for broad applications such as for materials of construction,

packaging, automotive, energy, transportation, sensors and biomedical fields.[3]–[13] Nanocellulose is usually managed to improve the final properties of the material to meet those specific applications.

### APPLICATION OF NANOCELLULOSE IN MILITARY

#### Packaging

Nanocellulose has been widely studied as components of materials for a variety of packaging. Nanocellulose can be used in packaging to meet those military criteria of materials that enhance protection and survivability, environmental acceptability and multifunctionality as discussed in the introduction section. Mechanical properties of the packaging material of the military are one of the important aspects. Nanocellulose is known to increase the mechanical of the packaging material. Several types of polyolefins in packaging such as polyethylene, polypropylene, and polyethylene terephthalate were greatly increased on their mechanical performance with the additional small amount of nanocellulose usually 1 to 5 %. Table 1 shows the mechanical properties of composites which improved by the addition of nanocellulose.

TABLE 1 Mechanical properties of nanocellulose reinforced composites

Polymer matrix	Nanocellulose content	Improvement in mechanical properties (%)	Reference
Polyethylene	3 wt.%	Tensile strength – 57.7 %, Young's modulus – 92.7 %, Flexural strength – 198.2 %, Flexural modulus – 25.0 %	[6], [14]
Polypropylene	3 wt.%	Tensile strength – 34.2 %, Young's modulus – 175.9 %, Flexural strength – 27.8 %, Flexural modulus – 88.9 %	[4]
Polyamine/ Epoxy resin	0.7 wt.%	Tensile strength – 29.9 %, Young's modulus – 66.7 % Flexural strength – 30.6 %, Flexural modulus – 21.4 %	[15]
Polyvinyl alcohol/starch	10 % (v/v)	Tensile strength – 85.2 % while elongation at break decreased.	[16]
Polycaprolactone	3 – 12 wt.%	Slight improvement in tensile modulus and strain at break while retaining tensile strength ( <i>No quantitative data reported</i> )	[17]
Poly(styrene-co-butyl acrylate) copolymer	10 wt.%	Tensile modulus – 6142 %, tensile stress – 104.2 % and elongation at break decreased.	[18]

#### Energy devices

Recently, nanocellulose is one of the promising candidates for components used in electrochemical energy storage (EES) [19]. Due to the interesting characteristics of nanocellulose as discussed before and specifically good electrochemical properties, nanocellulose may use in emerging energy technology [20]. The good mechanical properties of nanocellulose can be integrated into composites of polymer electrolytes as a mechanical matrix. Besides that, the unique entangled structure of nanocellulose can be used as functional scaffolds to construct the electrode host, which benefits the transport of ion and achieves excellent cycle performance of electrodes [21]. Since nanocellulose is non-conductive materials, it must be functionalized with the conductive polymers, metallic particle or with the conductive carbon materials such as graphene, carbon black and carbon nanotube. The modified nanocellulose based EES may use in military application since it has good flexibility, lightweight, and durability [22]. Moreover, the rechargeable devices technology also received much attention in the military. The example of EES device from nanocellulose is rechargeable lithium-ion batteries, supercapacitor and solar cell. In this section, the application of nanocellulose for the military energy application are focused on the developments of supercapacitors, lithium-sulphur batteries and solar energy.

#### Fire retardant material

The development of fire retardant of nanocellulose composites is taking over in advanced engineering applications. Flammability characteristics of nanocellulose-reinforced thermoset nanocomposites had been explored and showed promising results. Nanocellulose composites show high levels of fire safety such as little smoke and toxic gases production of if exposed to fire. In this way, they protect the novel military products, infrastructure, the environment, and mainly aid in preventing loss of lives to fire.[23] To achieve this, additives or fillers called flame retardants such as halogen, boric acid, phosphorus, minerals, and nanometric compounds are incorporated into nanocellulose composites to prevent or minimize fire from causing damage. Solution impregnation and surface treatment are methods of incorporating fire-retardant agents towards the nanocellulose. In some cases, composites of nanocellulose were added to the polyurethane to make as fire retardant materials.[24]

#### Propellant

Nanocellulose can be used in the development of military propellant in order to have higher energy per unit mass than current explosives. Functionalization is a key factor of success for nanocellulose as a good propellant. There are several examples of R&D done on the development of nanocellulose as a propellant. Zhang et al. (2016)[25] fabricated double base-solid propellant using nanocellulose as filler. Due to its hydrophilic property, high aspect ratio and good suspension stability, their performance as double-base propellant are significantly improved as compared with conventional double base propellant. The tensile strength and elongation break increased by nearly 34% and 45%, respectively. While the burning rate of propellant increased by 27.5% and pressure coefficient decreased by 20%.

#### Filtration materials

Chemical and biological contaminants such as heavy metals, organic and inorganic compounds, and microbes are not only seriously affecting the environment but also threaten the army. In this regard, treatment of chemical and biological contaminated in air and water is very important in the military. A combination of biosorption and nanotechnology offers a new and greener way to remediate these contaminants. Nanocellulose has been identified as an excellent filtration material towards chemical and biological contaminants [26], [27].

#### Textiles

Textiles are major components of military equipment, and it includes a wide variety of functions. The choice and design of textile used for the fabrication of military equipment are challenging. There are several important components of environments need to be focused which are thermal protection, good durability, water repellent, withstand static propensity in numerous applications, able to endure tears and breaker, protect from ballistics and resistance from chemical and biological threats. Nanocellulose has been applied in modern textiles to give several benefits as mentioned above. Due to its superior mechanical properties than Kevlar, nanocellulose is useful for thermal insulation, sanitary napkins, tampons, diapers or as wound dressings [28]. Several innovations that have been discovered in this area. Mishra et al. (2014) [28] had discussed in their review the numerous applications of nanocellulose in the textile industry for the production of lightweight body armour and ballistic glass.

#### Military medical

Generally, nanocellulose has been widely used in medical fields such as tissue engineering, bone scaffold, wound healing, and drug delivery as shown in Fig. 1. However, in this section, only those applications that related to military were tackled.

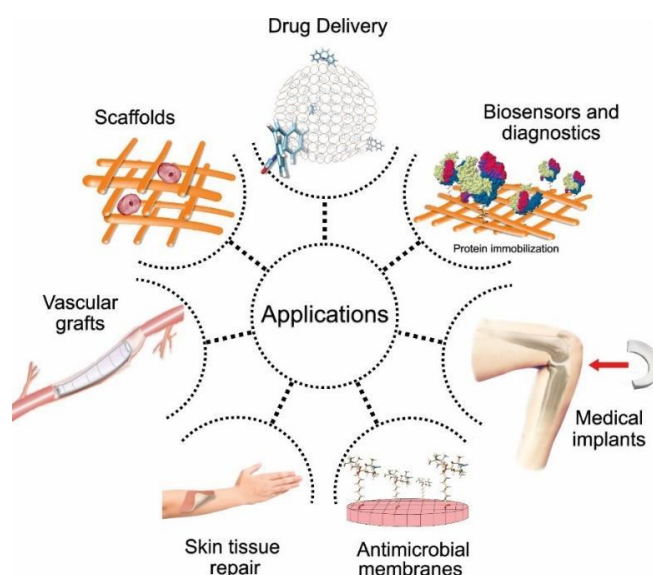


Fig. 1 Medical applications of nanocellulose. Reproduced with permission from ref. [29].

## CONCLUSIONS

In conclusion, the usefulness of nanocellulose for various application in the military is a promising and exciting area of current and future R&D. Several recent developments in the application of nanocellulose related to the military were reviewed. Although the effectiveness of nanocellulose as a new green biobased material in the military has been demonstrated through different works, several improvements are still needed. The R&D have to be conducted to the real military sample. For example, the effectiveness of nanocellulose as an antimicrobial material has to be tested on the several known biological warfare agents. Moreover, the developed nanocellulose based filtration materials need to be tested on the real sample of chemical warfare agents such as sarin and tabun. Another issue is related to the cost-effectiveness and availability of nanocellulose at the industrial level. Indeed, the energy consumption related to the production of nanocellulose is still an issue hampering the scale-up production of nanocellulose. However, progress has been accomplished in many reports which were focused on scale-up and reduce the production of nanocellulose.

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INVITED SPEAKER 2

PLANT TISSUE CULTURE: A MODERN BIOTECHNOLOGICAL  
TOOL TO IMPROVE PROPAGATION OF SUGAR PALM (*Arenga pinnata* WURMB  
MERR)

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ABSTRACT

This article discussed the vast potential and advantages of plant tissue culture technology as a modern, practical biotechnological approach to propagate sugar palm alternatively. Sugar palm has highly dormant seeds and promotes a significantly lower percentage of germination using the conventional propagation method. Thus, it is required to develop an alternative plan to resolve the issues and obtain effective propagation of sugar palm. The article also proposed suitable techniques of plant tissue culture technology to improve seeds germination and their potential to promote sugar palm breeding for many other technical objectives. The procedures of initiating plant tissue culture of sugar palm from previous research works are also discussed.

*Keywords:* plant tissue culture; modern biotechnology; sugar palm; alternative propagation, effective breeding.

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INTRODUCTION

Sugar palm (*Arenga pinnata* Wurmbe Merr) is an evergreen monoecious palm species most commonly found in tropical Asia regions, including Malaysia. Sugar palm trees are domesticated primarily for sweetened pulp, sugary sap, and industrial black gomuti (fiber) used mainly in the bio-composites and other engineering industries. The palm species is generally propagated and domesticated from seeds and sometimes suckers. They are known to have several propagation issues, particularly when natural sources are involved. Among the reported problems that hindered the effective propagation of untreated sugar palm seeds conventionally includes a relatively low germination rate and unpredictably long germination period, which may be taking from one month to more than a year after dispersal for sowing [1]. The dormancy problem restricted the commercialization and production of sugar palm on an extensive scale [2]. Propagation of sugar palm sexually also reported promoting low survival rate of seedling prior uprooting and transplantation following slow growth habit during the juvenile stage (first 1-3 years) [3].

In terms of germplasm management, the behavior of sugar palm seeds in storage was reported recalcitrant with less than 25% rate of survival when left in open storage for three months [4]. Recalcitrancy is a condition describing a seed that loses its viability if stored for any length of time, even under conditions that are generally conducive to seed longevity [5]. Considering the problem in sugar palm's generative propagation and its seeds management, an alternative solution to improve the conditions is necessary. One of the best methods to overcome this problem is by developing sugar palm seedlings through the plant tissue culture method. Plant tissue culture technology is a modern and most popular tool of agricultural biotechnology applied to solve diverse propagation issues of various plant species; among them, seed dormancy and recalcitrancy which are the most commonly reported problem for palm trees. The underlying principles involved in plant tissue culture is very simple, in which it dictates a necessity to isolate a plant part (organ, tissue, and cells) from the intact plant and provide the explants with an appropriate environment in the form of a suitable culture medium and a proper culture condition to which it can potentially grow under aseptic condition [6].

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There are many different types of plant tissue culture techniques, but the three most commonly in practice are callogenesis (callus culture), organogenesis (organ culture), and somatic embryogenesis (somatic embryo culture). Callogenesis is the culture of undifferentiated cell mass from heterogeneous structural tissues of plants exposed to specific plant growth regulators under *in vitro* conditions. Among the significant applications of callus culture in agriculture and horticulture includes the development of transgenic plants with improved agronomic traits (by the mechanism of somaclonal variation) as well as serving as a platform for large-scale production of sustainable secondary plant metabolites in pharmaceuticals, cosmetic food, and related industries [7].

Meanwhile, organogenesis is used as a general term for those types of culture in which an organized form of growth can be continuously maintained under the influence of specific plant growth regulators *in vitro*. Two types of organogenesis are (1) direct organogenesis which is initiated from definite plant organs or tissues (i.e., meristems, shoot tip, nodes, roots, and zygotic embryos), and (2) indirect organogenesis, which is initiated from callus cells. Direct organogenesis has great practical significance for plant propagation as it promotes the production of a higher rate of organized clusters of true-to-type clonal shoots with lower mutation possibility giving appropriate treatment. Regeneration of callus cells (indirect organogenesis), on the other hand, promoted random and unorganized plant regeneration, and there is a chance for somaclonal variation to happen. The organogenesis process is also not standardized for several recalcitrant plant species [8]. The culture of somatic embryogenic cells obtained from the embryogenic calluses is known as somatic embryogenesis. The potential application of somatic embryogenesis in plant improvement includes cloning zygotic embryos for repetitive somatic embryogenesis, synthesis of artificial seeds, source of regenerable protoplast system, a channel for genetic transformation studies, and conservation of plant genetic resources [9].

Presently, tissue culture research on sugar palm, specifically in Malaysia, is still very limited. Some reasons might be due to public ignorance and shunned popularity among researchers and government bodies towards the plant species compared to other commercially valuable commodities of the country such as oil palm, durian, and rubber. Thus, this article aimed to express the potential of plant tissue culture technology as an alternative method to effectively propagate sugar palm trees for commercial production.

### INITIATING PLANT TISSUE CULTURE FOR SUGAR PALM

The initiation of the plant tissue culture techniques on sugar palm usually starts with selecting suitable plant materials. The most preferred plant materials to be used as explants are zygotic embryos extracted from surface-sterilized fresh fruits of sugar palm. Surface sterilization is conducted by washing the plant materials under running tap water for 10-15 minutes, following soaking in 70-96% ethyl ethanol and 10-50% concentrations of sodium hypochlorite added with a few drops of surfactant depending on researchers' preferences. Other reported explants used to initiate plant tissue culture works of sugar palm includes seeds and intact plant parts obtained from aseptic seedlings [2], [10], [11]. Explants were then inoculated on a synthetic nutrient medium comprised of inorganic nutrient salts, carbon sources, vitamins, specific phytohormones, and gelling agents. Many types of plant tissue culture media are available in the market, but the most extensively used or 'so-called' standard tissue culture media is MS [12].

The success of plant tissue culture techniques depends primarily on the type and source of explants, medium pH, and an aseptic and controlled environment conducive to culture growth and development such as gas exchange, relative humidity, light requirement, and temperature [13]. In the effort to break the long dormancy period and low germination rate of sugar palm, Muda & Awal (2018) reported 30% and 100% germination rate of aseptic sugar palm seedling within eight weeks by culturing immature zygotic embryos as explant on basal MS medium and MS medium supplemented with specific plant growth regulators respectively instead of seeds [14]. While untreated seeds reported failed to germinate after four months ([15] or germinated at a significantly lower rate (17.15%) within an average of 313 days after sowing [3], *in vitro* method had been significantly proven to be more effective in reducing the germination period of sugar palm at a relatively higher rate.

Plant growth regulators are one of the essential factors in determining the success of plant tissue culture. Different type of plant growth regulators exhibits different physiological effect in different plants. These effects are determined by the type of the growth regulators, their concentration, the presence and absence of other growth regulators, and genetic makeup and the physiological status of the target tissue [16]. For instance, in a plant tissue culture study of sugar palm conducted by Muda et al. (2019), it was reported that the use of low concentrations of kinetin (1.0 mg/L Kin) in combination with Indolacetic acid (1.0 mg/L IAA) was found effective to promote optimum regeneration of clonal shoots and roots (direct organogenesis) from aseptic stem base explant [17]. Meanwhile, the use of 1.0 mg/L 6-Benzylaminopurine (BAP) together with 1.0 mg/L Naphthalene acetic acid (NAA) and 1.0 mg/L silver nitrate (AgNO<sub>3</sub>) gave rise to optimum clonal shoots via zygotic embryo explant.

In other studies, which were conducted to promote optimum callogenesis of sugar palm, it was observed that the inoculation of explants on MS medium supplemented with low concentrations of 2,4-Dichlorophenoxyacetic (2,4-D) in combination with either BAP or Kin had effectively triggered the built-up of embryogenic callus in masses [14], [15]. The exposure of the embryogenic calluses to the similar composition of culture media added with casein hydrolysate eventually gave rise to the production of different development stages of somatic embryos (somatic embryogenesis). Matured somatic embryos were sub-cultured onto fresh MS medium for regeneration into a complete clonal plant, and encapsulated for artificial seeds synthesis for germplasm conservation purposes [16].

### CONCLUSIONS

As an emerging technology, the plant tissue culture has a significant impact on agriculture and industry by solving various propagation issues of plants such as sugar palm and providing plants needed to meet the ever-increasing world demand. The considerable contribution of the mentioned technology as an indispensable tool in modern agriculture for the advancement of agricultural science in recent times is promising. It will surely give an outlook for a more fantastic future.

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## INVITED SPEAKER 3

## PROGRESS IN PLANT FIBER REINFORCED POLYMER COMPOSITES

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**ABSTRACT**

Environmental awareness across the world has motivated researchers to focus their attention on the use of cellulosic fiber as reinforcement in polymer matrices. Lignocellulosic fibers are an abundantly available resource in all countries, which is cheap and easily renewable. Also, due to their properties, cellulosic plant fibers exhibit a great potential for use in polymer reinforcement. As a result, considerable research and development efforts have been directed towards the use of cellulosic fibers as a reinforcing material in composites. The use of cellulosic fiber reinforced composites has continuously increased during recent years, which benefits the cultivation of fiber plants and the economy of the country. This research area continues to be of interest to both industry and academia, the use of cellulosic fibers in composite applications being investigated throughout the world. Cellulosic fiber reinforced composites are reasonably strong, lightweight, harmless to human health and the environment, and biodegradable, hence they have the potential to be used in various applications. Recent progress in cellulosic fiber composites research has illustrated their great potential as structural components in automobiles, aerospace structures, construction, and building, and so forth. This study is an effort to create awareness about the research works that have been published in the field of natural fiber composites. This short review briefly illustrates the main paths and results of major research published in the field of natural fiber reinforced polymer composites.

*Keywords:* Plant fiber, polymer composites.

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**INTRODUCTION**

Natural materials play an important role in the advancement [1], [2]. As a result, breakthroughs in biomaterials have influenced progress of mankind. Plant fibers are the oldest known fibers that people have cultivated and made, evoking their civilization and journey [3-5]. Plant fibers are widely accessible as agricultural crops in tropical locations. These plant fibers are extremely light, renewable, and cost-effective. Also, abundant availability and accessibility of plant fibers are the primary drivers of a growing interest in sustainable technologies. The investigation on plant fibers has accelerated in recent years, which could lead to a diversification of fiber sources as well as a reduction in material costs [6]–[8]. Because of the recent drop in fossil fuel output and increased environmental consciousness, the exploitation of natural fibers from plants has sparked a lot of interest and has become a critical topic. Plant fibers benefit both the industrial and rural economies in terms of economic and social benefits. Plant fibers have significant future growth potential due to strong market demand for environmentally sustainable materials, and government legislation requiring eco-friendly items. Plant fibers are made up of cellulose, hemicellulose, lignin, and pectin, which are bonded together by wax and other impurities, as well as moisture. Plant fibers can be extracted from stems, leaves, fruits, seeds, straws, and other parts of the plant [9]–[12]. Cellulose is defined as a non-branched macro-molecule containing chains of variable length of 1–4 linked  $\beta$ -D-anhydroglucopyranose units. It provides strength, stiffness, and structural stability to the fiber. Along with cellulose, one of the imperative constituents of natural cellulosic fibers is hemicellulose, which is the second most abundant family of naturally occurring polymers [12], [13]. Among the constituents of the cell wall, lignin is a highly branched polymer. It has been found to serve as matrix material in plants, along with hemicellulose, embedding cellulose fibers. Fiber extraction is a crucial step in fiber research that involves using proper techniques to remove or separate fibers from their source. The most commonly used chemical treatments are presented in Figure 1. For fiber extraction, mechanical, chemical, and retting processes are utilized, with the method chosen based on the type of plant material. Being hydrophilic, for preparing composites, natural fibers must first be treated to make them more compatible with hydrophobic thermosets and thermoplastics [14-18]. Chemical treatment of natural fibers allows reducing their hydrophilic characteristics. Surface treatments can lead to improved adhesion between

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the surface of the fiber and the polymer matrix. A variety of chemical and surface treatments have been reported by many researchers [19]–[22].

## BIOCOMPOSITES

The plant fiber reinforced polymer composites are called as “Green composites” or “Biocomposites” that may be simply discarded after usage without damaging the environment or creatures, indicating their eco-friendliness. Furthermore, as people become more aware of environmental issues, development toward green composites has become a major trend in industry and society. The plant fiber reinforced polymer composites have been widely used in a variety of applications for decades, owing to their promising potentials in comparison to synthetic materials [22], [23]. To address the environmental concerns raised by the use of synthetic fibers, academics and researchers are using plant fibers as a reinforcing material in polymer composites to replace synthetic fibers that are harmful to the environment. It is believed that the share of natural fiber composites in the market of engineering materials will reach 6500 million dollars in 2021. This type of composites is claimed to offer environmental advantages, such as reduced dependence on nonrenewable energy/material sources, lower pollutant emissions, enhanced energy recovery, and end-of-life biodegradability of components. Many components in various sectors are now made from natural fiber reinforced composite materials, which are mainly based on polymers with reinforcing natural fibers, primarily, jute, flax, hemp, kenaf, wood and so forth. In the aerospace sector, components such as tails, wings, propellers and helicopter fan blades are manufactured from natural fiber composites [24], [25]. The potential applications of natural fibers in other sectors such as automotive (door frames, door shutters, window frames, mirror casings), marine (boat hulls, fishing rods), building and construction (roofing sheets, bricks, furniture panels, storage tanks, pipelines), sports & leisure goods (ice skating boards, bicycle frames, baseball bats, tennis racket, fork, helmet, postboxes), electronics appliances (laptop and mobile cases, chip boards, projector and voltage stabilizer covers), as well as in pipes carrying coal dust, in construction of weapons and industrial fans, in the manufacture of textiles, paper and packaging [25].

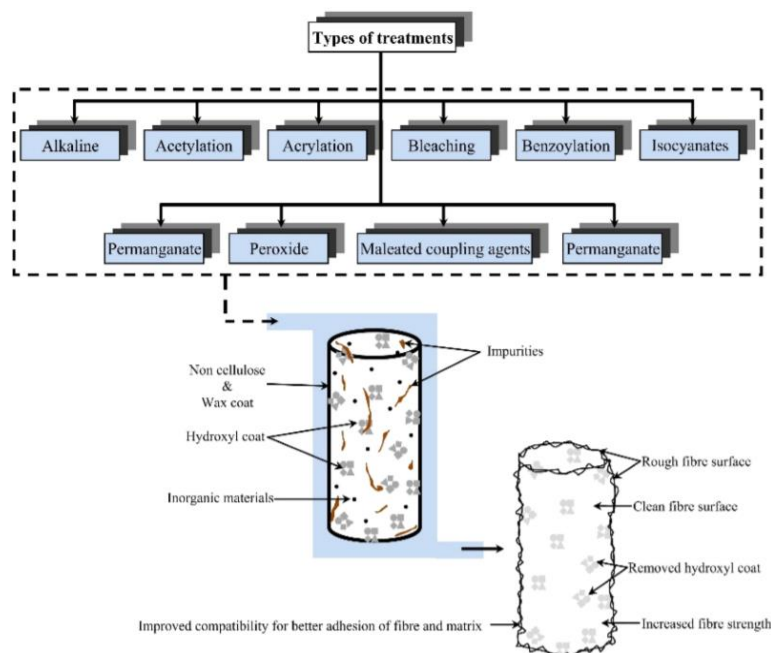


Figure 1: Different types of treatments on natural fibers

## CONCLUSIONS

Plant fiber based polymer composites are utilized for a variety of applications in everyday life due to their light weight, high mechanical strength, and acoustic and thermal insulation properties. Plant fiber based polymer composites are increasingly being used in a range of applications, including construction and building, locomotives, furniture, and the packaging industry. Plant fiber composites still have several limitations, such as moisture sensitivity and low tolerance to harsh operational circumstances, which restrict their service life. As a result, there is a significant effort to expand the use of plant fibers in other industries such as maritime, wind energy, aircraft, sports and recreation, and so on. Green materials are the future trend, and plant fiber is a big part of it. More research should be focused on getting better properties.

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## INVITED SPEAKER 4

## AGRICULTURE WASTE TO AUTOMOTIVE STRUCTURE: HYBRID CARBON AND SUGAR PALM YARN REINFORCED EPOXY COMPOSITES

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## ABSTRACT

This study aims to investigate the effect of fiber hybridization of sugar palm yarn with carbon reinforced epoxy composites. In this work, sugar palm yarn content at varying fiber loads of 5, 10, 15, and 20 wt.% using the hand lay-up process. The hybrid composites were fabricated from two types of fabric: sugar palm yarn of 250 tex and carbon fiber as the reinforcements, and epoxy resin as the matrix. The ratios of 85: 15 and 80: 20 were selected for the ratio between the matrix and reinforcement in the hybrid composite. The ratios of 50: 50 and 60: 40 were selected for the ratio between sugar palm yarn and carbon fiber. The mechanical properties of the composites were characterized according to the flexural test (ASTM D790) and torsion test (ASTM D5279). It was found that the increasing flexural and torsion properties of the non-hybrid composite at fiber loading of 15 wt.% were 7.40% and 75.61%, respectively, compared to other fiber loading composites. For hybrid composites, the experimental results reveal that the highest flexural and torsion properties were achieved at the ratio of 85/15 reinforcement and 60/40 for the fiber ratio of hybrid sugar palm yarn/carbon fiber-reinforced composites. The results from this study suggest that the hybrid composite has better performance regarding both flexural and torsion properties. The different ratio between matrix and reinforcement has a significant effect on the performance of sugar palm composites. It can be concluded that this type of composite can be utilized for beam, construction applications, and automotive components that demand high flexural strength and high torsional forces.

*Keywords:*, sugar palm yarn, carbon fiber, hybrid composites, flexural properties, torsion properties

## INTRODUCTION

Sugar palm fibre is available and randomly wrapped along with palm leaf ribs. If directly used, this may affect interaction with the chain of the matrix of composites. The tangling nature of sugar palm fibre itself becoming harder to be ignored. Leads the possibility of the fibres to become randomly oriented is high especially after the infusion of resin into the mould and during the lay-up process. Sugar palm if process in short and random, is not suitable for structural applications. The properties of the composite materials are mainly dependent on their respective fiber properties. Other than that, factors affecting the properties include microstructural parameters such as fiber diameter, fiber length, fiber distribution, fiber orientation, volume fraction of the fibers, and packing arrangement of the fibers [1]. In structural applications, fiber-reinforced composites have gained a lot of market potential for their varied uses. However, this market growth is limited due to the lack of toughness of fiber-reinforced composites. The mechanical properties of natural fiber-reinforced composites are significantly improved by the incorporation of synthetic fibers [2].

## MATERIALS AND METHODS

Sugar palm fibres gathered in Jempol (Negeri Sembilan, Malaysia) were used in this study. The carbon fibre was supplied by Sky Tech Malaysia Sdn. Bhd., and the epoxy resin (RTM grade, 40% styrene content, density of 1.025g/cm<sup>3</sup>) was purchased from CCP Composites Resins Malaysia Sdn. Bhd.

The sugar palm yarn with a linear density of 250 tex was produced by the spinning process, manually. The composite was prepared according to the mix ratio from the instruction labels. The mix ratio of 3A: 1B was used, where A is for epoxy and B is for hardener. The mixed resin was poured over the fibers and cured at room temperature for 24 h

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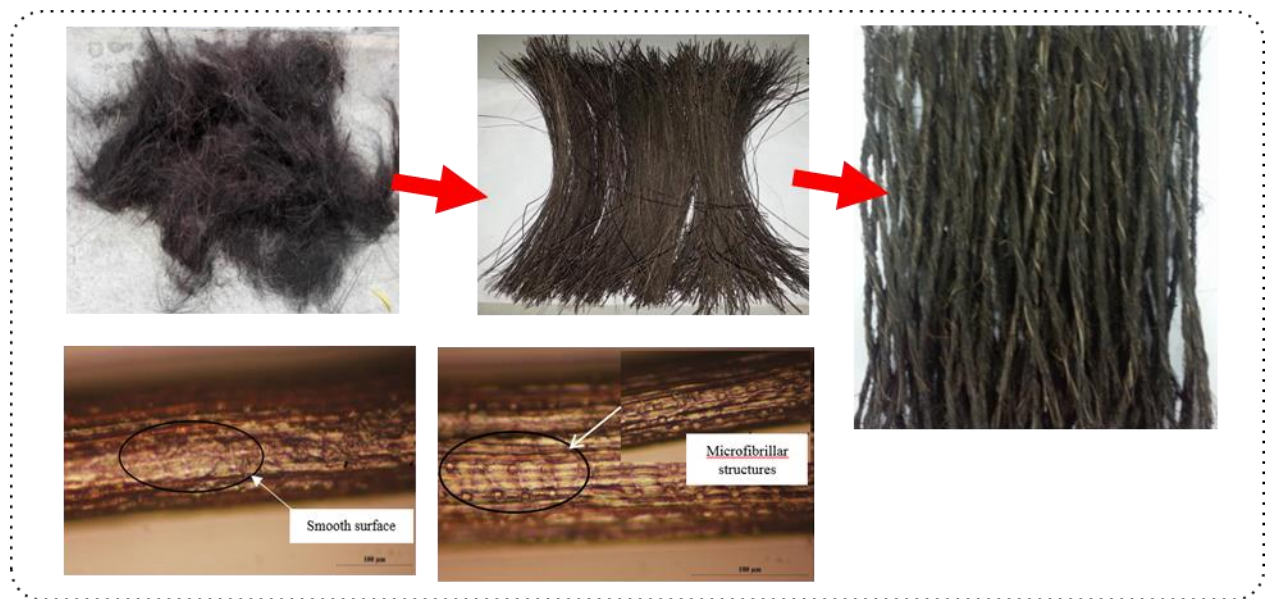


Fig. 1: Photograph of: a) raw sugar palm fiber, b) combed sugar palm fiber, c) sugar palm yarn fiber d) Image of a single fibre [3]

## RESULT AND DISCUSSION

The higher flexural strength and flexural modulus of non-hybrid composites were achieved at 15 wt % of sugar palm yarn fiber loading with 87 MPa and 3.3 GPa, respectively. For the hybrid composite, 15 wt % of reinforcement with the ratio of 60 : 40 revealed the highest flexural strength and flexural modulus, which were 118 MPa and 3.8 GPa, respectively. Hybrid composite shows a better result compare with non-hybrid composite.

## CONCLUSIONS

The hybrid composite of 250tex sugar palm yarn/carbon fibre could able to reach certain strength required of automotive component. The fabrication of hybrid composite is suggested to use other techniques in order to improve the hybridization and penetration of epoxy resin.

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## INVITED SPEAKER 5

## A REVIEW ON THE MORPHOLOGICAL CHARACTERIZATION OF VARIOUS NATURAL/SYNTHETIC FIBER HYBRID COMPOSITES

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## ABSTRACT

This article investigates the morphological behaviors of natural fiber-based hybrid composites. Natural fiber composites have raised greater attention due to three significant reasons such as low cost, biodegradability, and abundance. The morphology helps to investigate the characteristics of composite materials also explains how they fail. The morphological analysis ultimately determines the quality of composite structures. The quality of composites could be determined by inspecting (i) fiber-matrix adhesion, (ii) void content, (iii) fiber loading, (iv) lamina thickness, (v) resin-rich region over the fiber surface, (vi) fiber alignment, (vii) fiber pull-out, etc. This way is qualifying the composite materials in many industries: aerospace, automotive, construction and building materials, and marine. In this review paper, mechanical, physical, thermal, and structural properties of natural fiber-reinforced composites are studied with morphological analysis.

*Keywords:* Hybrid composites, morphological analysis, scanning electron microscopy, interface.

## INTRODUCTION

Reinforcement of two or more elements of different or same nature of fibers with the single or mixture of matrixes is called hybrid composites. Both the strength and properties of the composites depend on the fibers aspect ratio, individual characteristics of the fiber, fibers orientation, size of the fiber, binding properties of both fiber and matrix, and sequence of stacking of fiber [1]. Some of the techniques used for the morphological studies are Scanning Electron Microscope (SEM), Transferring Electron Microscope (TEM) and Atomic Force Microscopy (AFM). Transferring Electron Microscopes were used to study the microstructure changes on the thin films about the film orientation and the direction of the load apply [2]. Atomic Force Microscopy (AFM) were used to measure the different parameters such as mean roughness ( $S_a$ ) and root mean square ( $S_q$ ), which are called as localized AFM/ electrical measurements [3]. Scanning Electron Microscope (SEM) were used to measure the morphology of the polymer composites materials which were subjected to the mechanical and thermal loading conditions. The attraction towards the research on the natural/synthetic fiber hybrid composites were increased today [4]. The presence of the natural fiber reduces the harmfulness of the polymer composites towards the environment [5]. From the micrographs obtained, the different failure characteristics such as Fiber pullout, Fiber breakage, Matrix breakdown, Surface characteristics of the fiber, Micro buckling, Fiber rupture etc were investigated [6]. This article addresses the morphological characteristics of the hybrid composites in terms of the fiber matrix interfacial adhesion, compatibility between various natural fibers in the matrix, typical failures under various mechanical loads and fiber dispersion characteristics within the matrix.

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**Morphological characterization of natural-natural fiber hybrid composites**

Ramlee et.al [7] investigated the influence of adding sugarcane bagasse (SCB) into the oil palm empty fruit bunch (OPEFB)/phenolic composites at different weight ratios. The introduction of SCB fibers into the OPEFB/phenolic composites tends to reduce the fiber pull-out in the hybrid composites were observed. The fiber fracture was mainly observed for OPEFB fibers and the addition of high strength and stiff SCB helped to retain the tensile strength. Chaudhary et.al[8] developed the epoxy-based

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hybrid composite with jute/hemp/flax fibers and subjected them to tensile, flexural and impact load. Typical failure such as fiber pull-out, fiber breakage, matrix cracking and fiber matrix de-bonding was observed from the tensile fractured specimens. Premnath[9] fabricated sisal/jute-based epoxy hybrid composite in which the fibers were treated with 10% aqueous NaOH solution. It can be observed that treated fibers are well embedded into the matrix with only the epoxy matrix visible on the surface while the composite with untreated fibers has fibrous region. On the other hand, composite with treated fiber had more resin adhering to the surface of the fiber compared to the composite with untreated fibers where resin adhesion is lower.

**Morphological analysis of natural-synthetic fiber reinforced hybrid composites**

Negawo et al[10]. perfectly elaborated the effect of interplay composites on mechanical properties of ensete fiber (E)/E- glass woven fiber (G)/unsaturated polyester matrix composites. Results reported that, as expected, the mechanical properties of pure ensete fiber reinforced polyester matrix composites were lower than the pristine glass and their hybrid composites. In the case of hybrid composites, the asymmetrical ones (GGEE) performed less efficiently than the symmetrical ones (GEEG) because of the layering pattern. Researchers studied the effect of hybridization and NaOH treatment on the mechanical property of kenaf/glass polyester hybrid composites and obtained results that reported 15/15 v/v kenaf/glass fiber hybrid composites (NaOH treated) exhibited higher results[11]. Fig 1.2 shows the SEM photograph of the impact fracture surface of 15/15 v/v kenaf/glass fiber hybrid composites. It was observed that (i) good fiber-matrix interfacial bonding, (ii) fewer fiber-pullouts, and (iii) minimum matrix cracking.

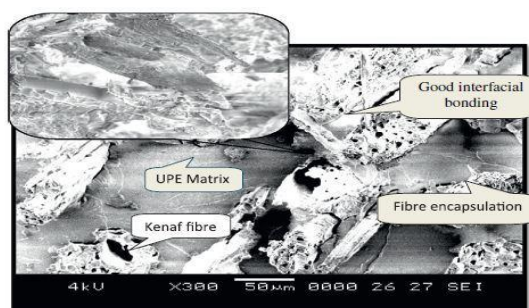


Fig 1.1. SEM micrograph of impact tested 15/15 v/v kenaf/glass fiber hybrid composites (NaOH treated)

**Conclusions**

This review paper surveyed the literature based on mechanical deformations of polymer matrix composites influenced by morphological analysis. It was further dealt with the mechanisms of failure of composite materials due to fiber pullouts, matrix cracking, resin-rich regions, interface quality, the interaction between fiber and matrix, crack formation, compatibility between the fiber and matrix. Hence morphological studies enhance the failure study of polymer composites subjected to the mechanical testings.

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## INVITED SPEAKER 6

## POTENTIAL BENEFITS OF DURIAN WASTE BIOMASS AS A RAW MATERIAL FOR BIOCOMPOSITE INDUSTRY

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## ABSTRACT

Biomass is a renewable and sustainable resource made up of organic compounds derived from animal or plant components. Agricultural products and residues are an example of constitute plant biomass, include tropical fruit wastes such as durian. Durian fruit has a significant quantity (more than 50%) of rind or peel and seed, both of which are discarded as waste. Long-term dumping of these wastes into the environment generates greenhouse gases during decomposition and also provides a breeding ground for bacteria and pests, which are contribute to the spread of plague. To mitigate a waste's environmental effect, researchers have focused on the recovery of health-promoting substances and conversion to other usable biomass in recent decades. This review aims to provide a comprehensive view of the actual trends in the usage of durian fruit wastes in order to encourage future research and discovery in unexplored areas. This review also discusses the potential for utilizing durian waste biomass as a raw material for composite materials, which would alleviate the fruit's waste management issue. The potential of durian fibre is highlighted, as well as the constraints associated with its application in composite manufacture.

*Keywords:* durian, fibre, biomass, composite properties

## INTRODUCTION

Durian (*Durio zibethinus*) is a popular seasonal tropical fruit consumed in South East Asia due to its unique taste and aroma. Durian is belonging to trees species of Malvales, order in Bombacae family, and genus of Durio and usually called as 'King of Fruits'. Durian fruit is high nutritional value and is high in dietary fibres, minerals, trace metals, sugar, vitamin C, potassium, as well as carbs, proteins, and lipids. In Malaysia, the peak season for durian is between June and August. Thailand and Malaysia are the world's leading durian growers and exporters, with yearly outputs of 600,000 and 300,000 metric tons, respectively. According to data published by Agricultural Department, durian is planted on a total of 70,286ha of land and the local output of durian in 2019 was 377,251 metric tons. The edible section of the durian fruit product accounts for just 22–30% of the total mass of the fruit, whilst the non-edible portions, such as the rind and seed, contribute up to 70% depending on the variety. From that total, around 255,353 metric tons of that being durian waste generated as a byproduct that generated an environmental problem. The durian wastes created by local or industry markets are generally burnt or likely disposed of in landfills, generating a number of issues in the society. As a result, effective management of these durian waste is required in order to minimize waste production. One method of decreasing the trash created by durian waste is to use it to make a useful product.

## PROPERTIES OF DURIAN WASTE

Durian fruit is made up of 20-35% flesh, 5-15% seeds, and 55-66 % husk (or rind/skin) [1] as shown in Figure 1. The largest part in the durian is the husk, with the exterior husk of the durian is green to brownish, with thick, sharp hexagonal spines, while the interior husk is white and loamy. Each durian husk includes four or five locules, each of which contains three edible and delectable pockets of creamy yellow flesh. The durian husk contains 11.27% moisture, 4.84% ash, 39.3% carbon, and 53.74% oxygen. The intricate structure of the mesocarp and exocarp in the durian shell is made up of bundles of fibres. The mix of cellulose (60.7%), hemicelluloses (22.1%), and lignin (22.1%) resulted in a complex fibre structure (17.2%) [2].

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Figure 1. Parts in durian fruit (Source: San Ha et al., 2020).

## DURIAN WASTE APPLICATION

Durian waste can become a valuable source for many applications. Cheek et al (2016) analysed and illustrated the uses of the durian peel/husk and seed in several applications [3] as shown in Figure 2. It can be observed that durian waste can be utilized in many applications such as polysaccharide gel, bioadsorbent, biocomposite, particleboard, polymer composite, dressing film, plates and binder.

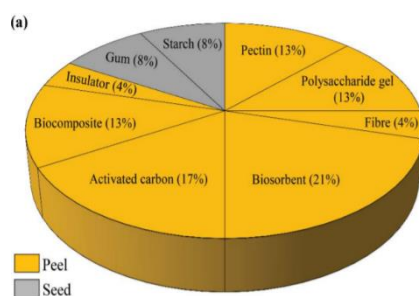


Figure 2. Application of durian waste [3].

Many studies have proven that the synthesis of activated carbon from durian husk exhibit toxic adsorbent properties by removing of organic chemicals and metal ions in aqueous solution such as toluene, lead ions, and chromium [4], [5]. On the other hand, Manickam et al. (2015) developed microstructure activated carbon (MAC) from durian husk that having an effective low-cost replacement for non-renewable coal based granular activated carbon (GACs) [6]. Lee et al. (2018) used a natural biopolymer from durian seed, durian seed gum (DSG), as a nutrient source and encapsulating agent [7]. In this study, DSG was used in synergy with reconstituted skim milk to be a complete nutrient source, for probiotic bacteria. They found that DSG was characterized to be a suitable nutrient source as it contains polysaccharides, organic acids, amino acids and fatty acids.

In composite industry, several research studies have been done on the utilizing durian waste. Charoenvai et al. (2013) manufactured particleboard from durian peel, with two objectives; to replace formaldehyde-based resin for particleboard manufacture and to disposing agricultural waste into economic value building materials [8]. Koay et al. (2018) produced plastic composite from recycled polystyrene (rPS) and durian husk fiber (DHF) using melt compound and compression moulding processes [9]. Manshor et al. (2014) produced a biocomposite from durian peel reinforced with polylactic acid (PLA) using extrusion and injection moulding method [10]. Aimi et al. (2014) was fabricated durian skin fiber reinforced polypropylene using extrusion and injection moulding processes [11]. Anuar et al. (2016) observed the loading on tensile and morphological properties of durian skin fibre (DSF) reinforced with PLA by adding epoxidized palm oil (EPO) as a plasticizer [12].

Chutrakulwong et al. (2020) synthesized silver nanoparticles (AgNPs) using the extract from mesocarp and endocarp of durian rind under photo-irradiation [13]. Due to some preferred properties of durian waste namely contains a high amount of water insoluble and water-soluble polysaccharides, which are long chains of carbohydrate molecules, these chemical components promote conversion of AgNPs to neutral silver atoms and also simultaneously contribute to stabilize the growth of AgNPs. A cellulose hydrogel sheet made out of durian husk with yeast phenolics for production of bandages was developed by Chen et al. (2021) [14]. The process extracts cellulose powder from the fruit's husks after they are sliced and freeze-dried, then mixes it with glycerol. This mixture becomes a soft hydrogel, which is then cut into bandage strips.

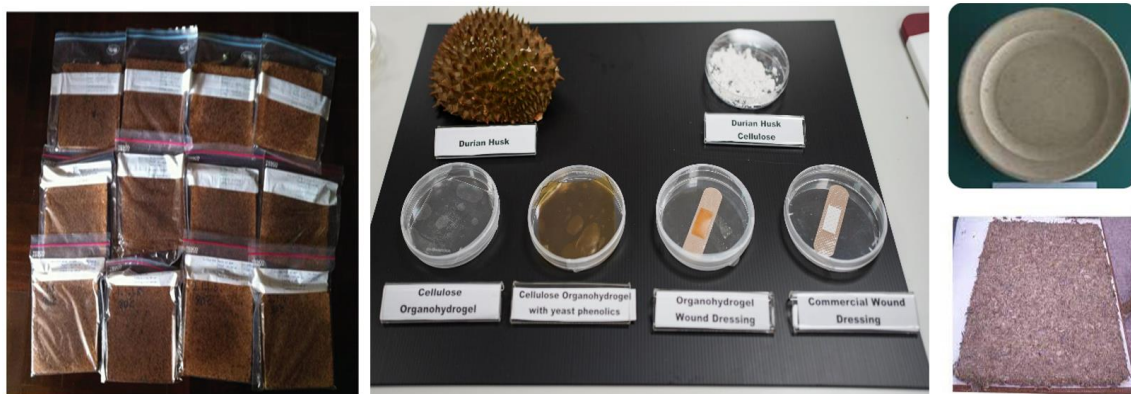


Figure 3. Some of application using durian waste.

## CONCLUSION

Durian waste can be a potential source for many applications. Durian waste are being mainly utilized as biosorbent, activated carbon, pharmaceutical products and biocomposite. Durian waste biomass provide unique properties and have potential as excellent material that can be used for many industries. Since numerous evidences showed remarkable properties in composite from durian waste, it is more worthwhile to be explored durian waste for matrix reinforcement applications such as in polymer composite. Hence, in order to fully utilize the potential use, treatment and hybridization must be considered to improve its performance by enhancing and altering the fiber quality.

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INVITED SPEAKER 7

**EFFECTS OF FIBERS STACKING SEQUENCE AND ITS ORIENTATION ON QSI BEHAVIORS OF CFRP/RAMIE/POLYURETHANE FABRIC INTERPLY HYBRID LAMINATE**

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**ABSTRACT**

Natural fibers have received major interests in polymers industry due to their cost-effectiveness, excellent strength, and rigidity as well as low specific weight compared to conventional glass fibers. To balance the advantages and shortcomings of natural fibers, hybridization of natural fibers and synthetic fibers with combination of polyurethane (PU) has been investigated. The purpose of this research is to evaluate the energy absorption of hybridization between carbon fibers reinforced polymer (CFRP)/Ramie/PU via QSI test. The effects of stacking sequence of interply hybrid laminate of CFRP/Ramie/PU have been examined. There are four groups of different stacking sequence have been used for QSI test, which are CFR1, CFR2, CFR3, and CFR4. CFR2 displays the highest energy absorption and specific energy absorption capabilities of 10.02 J and 291.90 J/Kg. In conclusion, it can be said that high energy absorption capability can be significantly influenced by the optimum fibers stacking sequence and orientation.

*Keywords:* Ramie Fiber, Polyurethane Fabric, Interply Hybrid, Energy Absorption, Quasi-Static Indentation (QSI) Behavior

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**INTRODUCTION**

Fiber-reinforced polymer (FRP) composites offer exceptional properties such as high durability, rigidity, damping property, flexural strength, and corrosion [1]. The material has high potential to substitute conventional metals in industry due to their behavior that suits the variety of applications. Carbon fibers-reinforced polymers (CFRP) for example, with outstanding properties have been widely used in automotive, civil, aerospace, sport goods etc. It also has been classified as advanced composite materials. Besides, the study of composites has shown significant improvement especially in hybrid composites as mentioned by Padmanabhan (2019) [2], that the weight of roof panel can be minimized and provides a good strength compared to regular panel by using natural fiber hybrid composite sheet material composed of 70% of unsaturated polyester resin and 30% of sugarcane bagasse and stainless-steel wire mesh. Moreover, Mahir et al. (2019) [3] have claimed that natural fibers exhibited many advantages over synthetic fibers including low specific weight, that resulting in greater strength and rigidity compared to glass fibers.

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Nowadays, most of the synthetic fibers' utilization have been replaced by natural fibers as reinforcement due to environmental concerns. Moreover, natural fibers possess outstanding properties including low-cost and abundant availability, renewable, biodegradable, low density, high toughness etc. as compared to synthetic fibers. Natural fibers such as Ramie fibers have been widely used in composite materials due to their higher tensile strength and elastic modulus than glass fiber [4]. However, one of the limitations of Ramie is the presence of hydroxyl groups that creating incompatibility problems with many synthetic polymers. This problem can be solved by conducting chemical treatment. Therefore, to balance the benefits and weakness of both synthetic and natural fibers, the hybridization composites between those two

types of fibers have been extensively studied. Moreover, it is believed with variety of combination and modification could lead to development of enhanced composite materials that are more sustainable in the future.

Besides, polyurethane (PU) has shown improvement in tensile properties of coating fabric in textile industry [5]. The ability of the PU fabric will be a great application in hybrid composites study. Due to that, considerably focused has been paid in the development of PU as additional material in the CFRP/Ramie hybrid composite to add extra strength and flexibility to the composite structures. Up to recently, there is no other studies were extensively discussed on the QSI behaviour of CFRP/Ramie/PU laminate composites. The energy absorption of the composite material needs to be determined since the composite often expose to external damage that cause the material failure. The data obtained in QSI experiments can be used to verify numerical attempts to classify damage mechanisms and their interaction during impact events [6]. Therefore, the new combination and sequence of the hybrid composites between CFRP, Ramie fiber and PU fabric should be conducted to address the energy absorption capability of the composite.

## MATERIALS AND METHODS

### Materials

The hybrid composites were prepared by using Cycom 934 prepreg carbon fiber supplied by Cytec Engineered Materials, polyurethane (PU) fabric supplied by Winson Shoes Industries Sdn. Bhd., Johor Bahru, and unidirectional ramie fiber supplied by Konsorsium Ramie Indonesia (KORI). All materials used in this study were cut into 300 x 300 mm each. There is no additional resin was used since the prepreg already contain epoxy. The materials were fabricated into interply laminate composites by using hot-press method using hot-press machine.

### Fabrication of Samples

The sample was hand lay-up one by one according to the desired sequence and orientation (Table 1). Based on the table, there are four samples were prepared with different sequence and combination with variation in Ramie and PU layers. If there is more than one layer of Ramie used, the orientation was varied into 0 and 90° since Ramie is unidirectional fibers. CFR3 and CFR4 is control sample where Ramie position in middle for CFR3 and PU fabric in the middle for CFR4. The samples were pressed in hot press machine for 60 minutes with temperature of 170°C.

### Quasi-static Indentation (QSI) Test

The experimental procedure to determine quasi static indentation response in laminated hybrid composite was referred to ASTM D6264/6264-17 [9]. The test samples with size of 100 mm x 100 mm were labelled accordingly to their variation, which are CFR1, CFR2, CFR3, and CFR4. The samples were tested using QSI test with displacement rate of 1.25 mm/min as suggested in ASTM D6264/6264-17 [9]. The Instron 600Dx- 600 kN Universal Testing Machine were used for the testing. An additional support fixture (Fig. 1(a)) was added to ensure that every laminated composite was tested in the same way. A hemispherical indenter (Fig. 1(b)) has been used for this test since this indenter geometry has produced a larger amount of internal damage than has been observed for similar indenter geometries with sharp tips. Then, the sample was clamped onto the support with a square metal plate that fit snugly into the larger square section of the support. Before the test began, the tip of the indenter was positioned at the center of the sample. The data was recorded at rates of 0.02 seconds for every sample.

TABLE 1: The stacking sequence and orientation of Ramie fiber

Sample group name	Number of ramies layers	Number of fiber carbons layers	Number of fiber Polyurethane Fabric	Fiber stacking sequence (R=ramie, Orientation of ramie C= carbon, F=Polyurethane Fabric) fibers.	of ramie
CFR1	1	2	2	C/F/R/F/C	0
CFR2	2	2	1	C/R/F/R/C	0/90
CFR3	1	2	0	C/R/C	0
CFR4	0	2	1	C/F/C	-

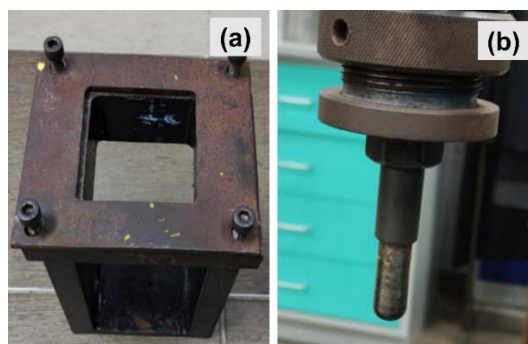


Fig. 1: (a) Support fixture and (b) Hemispherical indenter

## RESULT AND DISCUSSION

From Table 3 and Fig. 2, it can be obviously seen that the absorbed energy and specific energy absorption of CFR2 are the

highest as compared to other samples with values of 10.02 J and 291.90 J/Kg, respectively. However, there is insignificant different can be seen between CFR2 and CFR1, which is the absorbed energy of CFR1 is 18% lower than CFR2. In contrast to CFR1 and CFR2 (with thicker structure), CFR3 and CFR4 are the control samples and due to that, the comparison only between the control samples. It can be seen that, the control sample with addition of PU fabric shows better absorbed energy of 3.50 J as compared to CFR3 of 1.67 J. Essentially, the mechanical properties such as tensile strength have the biggest influence in determining the absorption energy of the composites. However, in this study, the thickness of the composites has shown great impact on the energy absorption. For example, CFR2 with thicker structure than CFR1 possessed higher energy absorbed [11]. Moreover, the variation in orientation of the unidirectional Ramie fibers shows great influenced on the energy absorption capacity. As can be seen, CFR2 is the only sample with two plies of Ramies that had advantages in orientation of 0/90°. This orientation will make the punctured of surface difficult as reinforcing fibers are designed to be loaded longitudinally rather than horizontally, the orientation of the fibers imparts highly directional qualities to the composite [12]. Based on literature, when compared to various layup sequences, it is noted that laminates with a (0/90°) layup sequence are the most effective in terms of impact resistance [11].

TABLE 3: Maximum Energy, Absorbed Energy and Specific Energy of Samples

Specimen	Absorbed Energy of Sample, $E_a$ (J)	Maximum Energy Sample, $E_{max}$ (J)	Average Mass of Sample (g)	Specific Energy Absorption of Sample, (J/Kg)
CFR1	8.21	8.29	32.67	251.19
CFR2	10.02	10.04	34.33	291.90
CFR3	1.67	1.68	20.67	80.93
CFR4	3.50	3.50	17.67	197.87

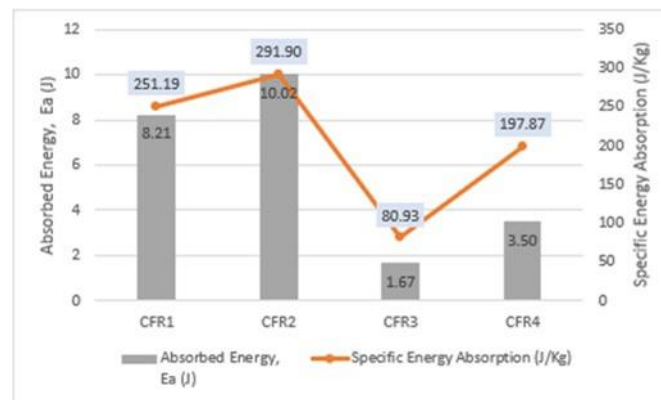


Fig. 2: Bar chart of absorbed energy and graph of specific absorbed energy of the CFRP/Ramie/PU interply hybrid laminates

## CONCLUSION

CFR2 exhibits the highest energy absorption and specific energy absorption among other samples with 10.02 J and 291.90 J/Kg, respectively. The control sample shows that CFR4 obtained higher energy absorption and specific energy absorption as compared to CFR3 with percentage different of 70.55% and 83.88%, respectively. Therefore, the samples with three different fibers configuration show that the tensile strength of the raw material can influence the energy absorption. Moreover, the results also revealed that the higher the thickness of the samples, the better the energy absorption capability. However, in control sample, it can be seen that thickness of the sample exhibits opposite behavior, which is the thicker the sample have lower energy absorption capability. In conclusion, energy absorption in this case does not solely influence by mechanical properties of the materials used. The influence of orientation from unidirectional Ramie fibers and thickness of the samples have shown significant impact toward the energy absorption capability.

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INVITED SPEAKER 8

**TENSILE PROPERTIES AND MORPHOLOGY OBSERVATION OF  
BIOCOMPOSITE PLASTIC FROM POLYLACTIC ACID (PLA) AND PINEAPPLE  
LEAF FIBER**

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**ABSTRACT**

The problem of plastic waste is a special topic for every country due to the higher amount of production in the environment. This has the potential to pollute and damage the environment. Synthetic plastic will decompose in the soil in more than 20 years and even up to 100 years, thereby reducing soil fertility. Therefore, this study aims to create environmentally friendly plastic made from natural materials, namely biocomposite plastic. Biocomposite plastics are made from Polylactic Acid (PLA) as a matrix and pineapple leaf fiber (PLF) as reinforcement. PLF was varied by 0, 10, 13, 16 and 19% in PLA matrix. The biocomposite plastic fabrication was solvent casting with chloroform as the PLA solvent. Tensile test and scanning electron microscopy (SEM) were carried out to determine the tensile strength and bond between the matrix/fibers of the biocomposite plastics, respectively. The highest tensile strength was found in the PLA/19%PLF sample for 15.53 MPa. This yield was 242% higher than that of pure PLA. The increase in tensile strength was followed by the modulus of elasticity, which was 54.76 MPa (PLA/19%PLF). This result was also supported by SEM morphological observations which show a good bond between matrix and fiber. This research still needs further exploration in terms of characterization and the fabrication of biocomposite plastics.

*Keywords:* biocomposite plastic, polylactic acid (PLA), pineapple leaf fiber (PLF), tensile strength, SEM morphological observations.

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**INTRODUCTION**

Plastic is one of the most important materials in everyday life. However, most of the plastics on the market are synthetic plastics, which are not environmentally friendly. Plastics are not environmentally friendly can cause environmental pollution such as floods and air pollution. Previous researchers reported that synthetic plastic waste continues to increase every year [1]. One alternative to overcome the problem of plastic waste is the use of natural materials as environmentally friendly plastics. Generally, environmentally friendly plastics are called biocomposite plastics. Biocomposite plastics are made from 100% natural materials such as biopolymer polylactic acid (PLA), starch, cellulose, and chitosan. The advantage of natural biopolymer materials is cheap, abundant availability, and renewable resources [2]. Among several biopolymers mentioned, PLA is the best candidate due to its higher mechanical properties and is competitive with synthetic plastics in tensile strength [3].

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**MATERIALS AND METHODS**

*Materials*

Pineapple leaf fiber (PLF) was obtained from the Mayang area, Jember Regency, East Java, Indonesia. Polylactic Acid (PLA) was purchased from Miyoshi Oil & Fat Co., Ltd; PL-2000 with a density of 1.26 g/cm<sup>3</sup>.

## RESULT AND DISCUSSION

Figure 1 shows the tensile strength of several biocomposite plastic samples from PLA and PLF. It can be seen that the tensile strength of pure polylactic acid (PLA) is lower than biocomposite plastic. From the figure it can be concluded that as the fiber increases the tensile strength value increases, this is indicated by variations of 10%, 13%, 16%, and 19%.

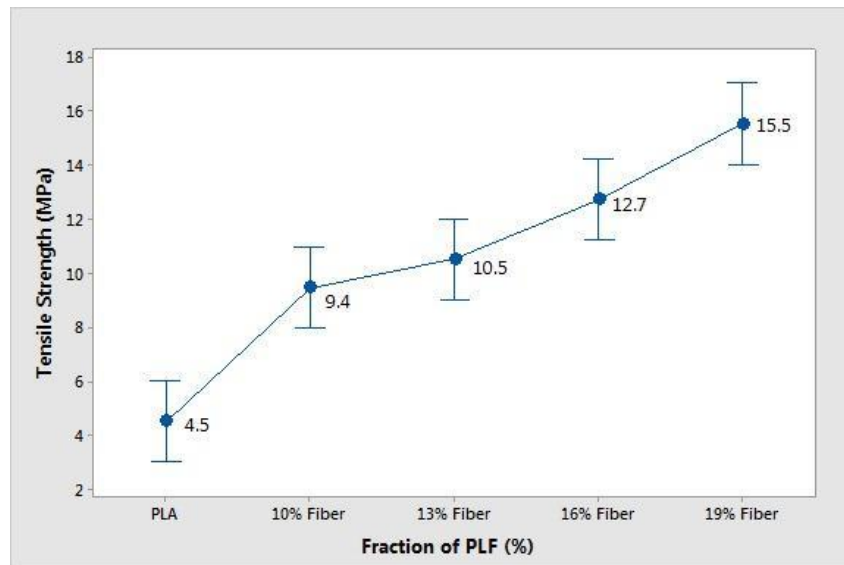


Fig. 1: Tensile strength of all biocomposite plastics tested

## CONCLUSIONS

Tensile strength of biocomposite plastics was improved by addition of PLF in PLA matrix. This is supported by SEM observation indicate good adhesion bonding between matrix and fiber.

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## INVITED SPEAKER 9

SUGAR PALM (*ARENCA PINNATA (WURMB.) MERR*) LIGNOCELLULOSIC FIBRE HIERARCHY: FROM MACRO TO NANO SCALE

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## ABSTRACT

In the past decades, many different resources have been utilised to prepare nanofibre from the macrofibre of lignocellulosic fibre, including sugar palm fibre. There are different methods of disintegration that would lead to different types and sizes of nanofibres materials. These processing methods include mechanical treatments (high pressurized homogenizer (HPH), microfluidisation, ultrafine grinding or refining, ball milling, aqueous counter collision, steam explosion, extrusion, cryocrushing in liquid nitrogen, high-intensity ultrasonication (HIUS), and high-speed blending), chemical treatment (acid hydrolysis, carboxylation, carboxymethylation, quaternization, sulfonation), biological treatment (enzymatic hydrolysis), synthetic and electrospinning treatment, TEMPO-mediated oxidation, and consequent mechanical treatment, or, a combination of two or several of the stated treatments. Sugar palm cellulose fibre can be extracted from sugar palm fibre by using delignification and mercerization treatments. Whereas sugar palm nanocrystalline cellulose (SPNCC) and sugar palm nanofibrillated cellulose (SPNFC) can be isolated from the sugar palm cellulose fibre by using acid hydrolysis and high pressurised homogenisation treatments. Thus, in this review, we hope to impart the readers some of the excitement that currently surrounds sugar palm macrofibre and nanocellulose research, which arise from the green nature of the particles, their fascinating physical and chemical properties.

*Keywords:* sugar palm, cellulose, nanocellulose, nanocrystalline cellulose, nanofibrillated cellulose

## INTRODUCTION

Sugar palm or its scientific name *Arenga pinnata* can be found in Southeast Asia, around the tropical rainforest and dry forest [1]. Sugar palm is a good source for obtaining fibres. Usually, these fibres can be collected from the bases of petioles of mature palms after the palm reaches 5-6 years old, and after that, they can be harvested every two years. The structure of the leaf base is attached with long spines, which acts as protection for the tree. There are three main advantages of sugar palm fibres, firstly

it can withstand a longer life to degrade and has high tensile strength, secondly compared to coir fibres, it is not affected by heat and moisture, and lastly, it has good resistance to seawater and is durable [2]. Besides collecting the fibres from black sugar palm fibre, called *ijuk*, sugar palm fibre can also be found from different parts of sugar palm plant, which are sugar palm bunch (SPB), sugar palm trunk (SPT), and sugar palm frond (SPF) [3]. Sugar palm fibres composed about 37-54% cellulose, 4-8% hemicellulose, 17-25% lignin, 4-30% ash, 0.85-2.5 % extractives, and 5.36-8.7% moisture content. Sugar palm cellulose fibre can be extracted from sugar palm fibre by using delignification and mercerization treatments [4]. Whereas sugar palm nanocrystalline cellulose (SPNCC) and sugar palm nanofibrillated cellulose (SPNFC) can be

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isolated from the sugar palm cellulose fibre by using acid hydrolysis and high pressurized homogenization treatments. Thus, this manuscript will review the methodologies of the extraction of cellulose and nanocellulose from sugar palm fiber as well as their intrinsic properties.

## SUGAR PALM CELLULOSE

Sugar palm cellulose can be isolated from sugar palm fiber. Ilyas et al. [4] had experimented the effects of the delignification ( $\text{NaClO}_2$ ) and mercerization ( $\text{NaOH}$ ) treatments on the physicochemical and thermal properties of sugar palm fibre (SPF). The bleaching and alkali treatments did not only caused changes in the chemical composition of the treated fibres (SPBF and SPC), they also affected the surface structure of the fibres. The pigment of the SPF was altered from black to light brown after the bleaching treatment and became white after the alkali treatment. The longer the reaction time of  $\text{NaClO}_2$ , the smaller the diameter of the fibres [4]. The average diameters of the SPF (Fig 1a), SPBF07 (Fig 1b), and SPC07 (Fig 1c) were approximately  $212.01 \pm 2.17$ ,  $94.49 \pm 0.03$ , and  $8.81 \pm 1.65$   $\mu\text{m}$ , respectively. The cellulose contents for sugar palm cellulose were observed higher compared to raw fibre due to the removal of some extractive components from the raw SPF during the delignification and mercerization treatments, which separated a higher proportion of the insoluble compounds in the treated fibre. The crystallinity index of sugar palm cellulose was higher compared to raw sugar palm fiber. The increase in the crystallinity of the fibres was due to the removal of amorphous substances, such as lignin and hemicellulose, during the delignification and mercerization treatments. The thermal degradation characteristics of the sugar palm cellulose were observed to have two stages: (1) evaporation of the moisture content, and (2) degradation of cellulose.

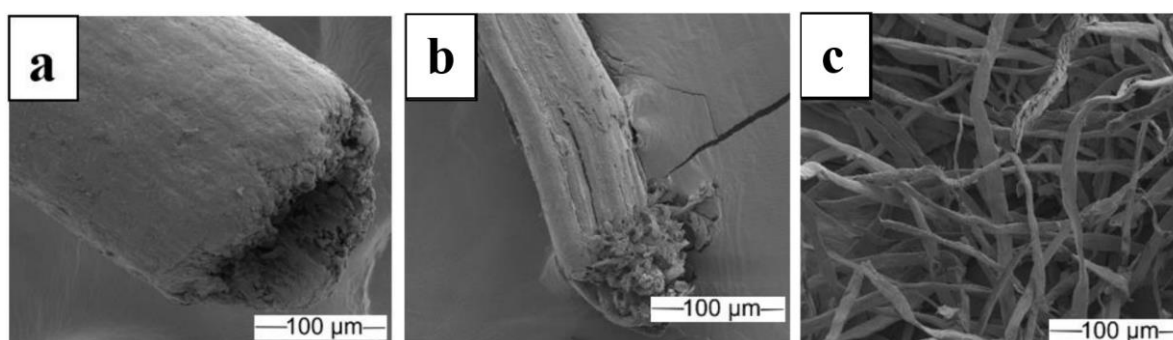


Fig. 1: The morphology structure of (a) SPF, (b) SPBF07, and (c) SPC07.

## SUGAR PALM NANOCRYSTALLINE CELLULOSE

Nanocrystalline celluloses (NCCs) isolated from plant fibers attracted tremendous interest in material science due to its appealing intrinsic properties, including nano-dimension, high surface area ( $100 \text{ m}^2\text{g}^{-1}$ ), high aspect ratio of 100, high crystallinity, low density, high mechanical strength, unique morphology along with availability, renewability, and biodegradability. The natural fibres are composed of three main components such as cellulose, hemicellulose, and lignin. Delignification and mercerization techniques are used to remove lignin and hemicellulose. In order to obtain purified NCC, hydrolysis treatment is utilized to remove amorphous polymers located between two crystalline regions. In the study conducted by Ilyas et. al. [5], acid hydrolysis (60 wt. %) at different reaction times (30, 45, and 60 min, denoted as SPNCC-I, SPNCC-II, and SPNCC-III, respectively) was carried out to investigate the optimum yield of SPNCC. The duration of the hydrolysis process is a crucial single factor in obtaining a suspension of negatively charged isolated NCC in water suspension. The highest yield was recorded for SPNCC-I with 33.5% and decreased to 29.01% and 13.12% for SPNCC-II and SPNCC-III, respectively. The low yield of SPNCC can be explained by the disintegration of amorphous regions and extended degradation of crystalline regions. The results showed that a needle-like shape was observed under transmission electron microscopy (TEM) studies. TEM analysis showed a diameter of  $13 \pm 1.73$  nm (Fig. 2a),  $9 \pm 1.96$  nm (Fig. 2b), and  $8 \pm 1.35$  nm (Fig. 2c) for isolated SPNCC-I, SPNCC-II, and SPNCC-III, respectively. In addition, the optimum aspect ratios of SPNCC were 13.46, 14.44, and 13.13 for isolated SPNCC-I, SPNCC-II, and SPNCC-III, respectively. From thermogravimetric analysis (TGA), the degradation temperature of NCC decreased slightly from 335.15 to 278.50  $^\circ\text{C}$  as the reaction time increased. A shorter hydrolysis time tended to produce SPNCC with higher thermal stability, as proven in thermal analysis by TGA. The optimal isolation time was found to be around 45 min at 1200 rpm during hydrolysis at 45  $^\circ\text{C}$  with 60% sulfuric acid.

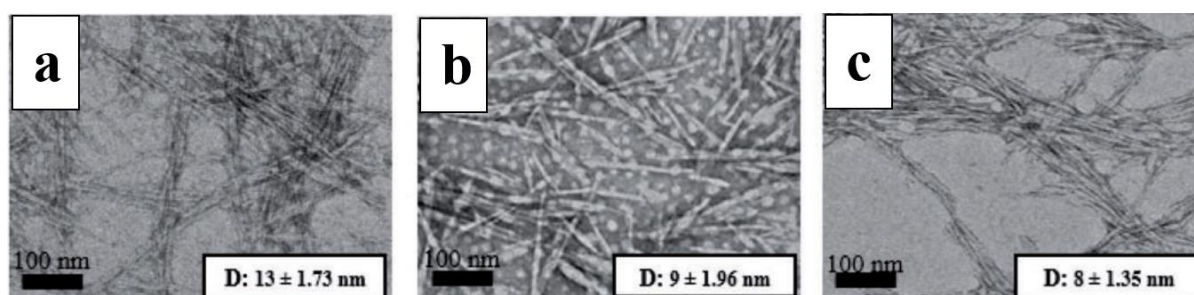


Fig 2: TEM images of (a) SPNCC-I, (b) SPNCC-II, and (c) SPNCC-III.

## SUGAR PALM NANOFIBRILLATED CELLULOSE

There are several methods for producing nanofibrillated cellulose (NFC) that have been utilized by numerous researchers to isolate highly purified nanofibres from treated cellulose [6], [7], including high pressurized homogenizer (HPH), microfluidisation, ultrafine grinding or refining, ball milling, aqueous counter collision, steam explosion, extrusion, cryocrushing in liquid nitrogen, high intensity ultrasonication (HIUS), and high speed blending). The high pressurized homogenization (HPH) process includes passing the cellulose slurry at high pressure into the vessel through very small nozzle purposely for isolating cellulose microfibrils mechanically into NFCs. HPH uses efficient high-pressure energy to break the cellulose present in fluids to the smallest possible size of the fibres from micro to the nanoscale. When fluid is passed through a small nozzle under high-pressure conditions, these microcellulose are broken into nanofibre and become homogenized by the process of cavitation and high shear force. Ilyas et al. [8] conducted a study on the isolation of nanofibrillated cellulose from sugar palm cellulose. Nanofibrillated cellulose (NFC) was extracted from sugar palm fibres (SPS) in two separate stages; delignification and mercerization to remove lignin and hemicellulose, respectively. Subsequently, the obtained cellulose fibres were then mechanically extracted into nanofibres using high pressurized homogenization (HPH). The diameter distribution sizes of the isolated nanofibres were dependent on the cycle number of HPH treatment. TEM micro-images displayed the decreasing trend of sugar palm nanofibrillated cellulose (SPNFC) diameter, from 21.37 to 5.5 nm when the number of cycle of HPH was increased from 5 to 15 cycles, as shown in Fig. 3. Meanwhile, TGA and XRD analysis showed that the degradation temperature and crystallinity of the NFC were slightly increased from 347 to 347.3 °C and 75.38 to 81.19%, respectively when the number of cycles increased.

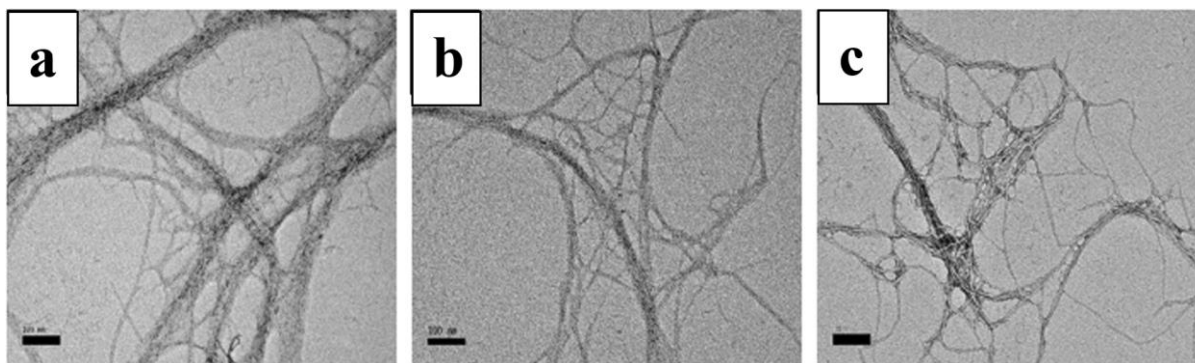


Fig. 3: TEM images of (a) SPNFC-5, (b) SPNFC-10, and (c) SPNFC-15.

## CONCLUSIONS

Sugar palm cellulose can be extracted from the sugar palm fiber using delignification and mercerization treatments, and further treated to yield nanocrystalline cellulose and nanofibrillated cellulose using acid chemical and mechanical treatments, respectively. The sugar palm cellulose possesses advantages compared to raw sugar palm fiber, such as a higher crystallinity, higher purity of cellulose, and better thermal stability due to the removal of lignin and hemicellulose. Besides that, the diameter of sugar palm fibre decreased from  $212.01 \pm 2.17$  (raw sugar palm fibre) to  $94.49 \pm 0.03$  (SPBF),  $8.81 \pm 1.65$   $\mu\text{m}$  (SPC),  $8 \pm 1.35$  nm (SPNCC) and 5.5 nm (SPNFC) when it is treated with delignification, mercerization, acid hydrolysis, and high pressurized homogenization treatments, respectively.

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## INVITED SPEAKER 10

**MECHANICAL AND MORPHOLOGY PROPERTIES OF COMPOSITES FROM *HIBISCUS TILIACEUS* POWDER/POLYESTER: EVALUATION OF THE EFFECT OF ALKALI TREATMENT TIME AND POWDER VOLUME FRACTION**

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**ABSTRACT**

This study aims to improve the mechanical and morphological properties of *hibiscus tiliaceus* powder (HTP) composite by modifying the surface of the HTP with 5% wt. KOH solution. Alkaline immersion times for 2h, 4h, 6h, and 8h and volume fractions of HTP 5%, 10%, and 20% were determined. The tensile, and flexural strengths of the composites were investigated, and the fracture morphology was analysed using SEM. The results showed that the soaking time of the HTP in the KOH solution affected the mechanical properties of composites. At constant HTP, the tensile strength, modulus of elasticity, and bending strength tend to have increased by 6%, 14%, and 12% respectively, after HTP treated for 2 h to 6 h. But the mechanical properties decreased after HTP was treated for 8 h because the HTP were damaged and fractured. The addition of HTP from 5% to 10% tends to increase the mechanical properties of the composite and decreases when HTP is 20%. SEM analysis shows a tighter interface between resin – HTP and a number of HTP pull-outs.

*Keywords:* *Hibiscus tiliaceus* powder (HTP), composites, morphology, mechanical properties, KOH Treatment

**INTRODUCTION**

Best nature has opened up new opportunities for other natural fibers to be investigated and developed in terms of nature and its ability to function more broadly. One of the other natural resources that still need to be developed as a composite filling material is fiber of *hibiscus* plants were plentiful and environmentally friendly. This plant is commonly found on roadsides, rivers, and beaches in Indonesia. The chemical components of HT fibers consist of cellulose, hemicellulose, and non-carbohydrate elements such as lignin [1], [2]. The potential and characterization of HT fibers as reinforcing materials or composite fillers have been investigated by several researchers [1], [3]. Sari and Padang [1] reported that untreated HT fiber had a tensile strength of 650 MPa, and after 8% KOH treatment, the tensile strength reached 5144.9 MPa (an increase of 700%). Furthermore, modification of 49.06% HT fiber and 5.39% rice husk in composite reinforcement is known to provide a bending strength of 99.71 N/mm<sup>2</sup> in composites [4]. Modification of natural resources using a chemical treatment to improve the mechanical properties of composites has been widely studied by industrialists and researchers. Suresha and Hemanth [5] reported that tensile strength and Young's modulus of alkali-treated pineapple fiber reinforced epoxy composite (PF/Ep) with 3 % wt. halloysite nanotubes (HNTs) increased to 16% and 22% respectively. The mechanical properties of the composite depend on the shape and size, orientation, volume fraction of the fiber, aging temperature, and the interaction between the matrix and the fiber. However, to the best of our knowledge, there is not much information and research on the use of HT fiber powders and their modifications. The potential for HT that is abundant and renewable still needs to be developed for a wider application.

Therefore, this paper aims to provide a detailed understanding of the mechanical properties and morphology of KOH treated *Hibiscus Tiliaceus* powder (HTP) composites. The variation of immersion time was 2 h, 4 h, 6 h and 8 h using 5% potassium hydroxide (KOH). The effect of adding HTP volume fraction in composites was also investigated to obtain accurate information related to the bending properties, tensile strength of composites and their fractures analysed by SEM.

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*Materials*

The hibiscus bark fibers are taken from the branches of the hibiscus tiliaceus (HT) tree and dried in the sun to dry. They were mashed using a grinding machine with a capacity of 2 kg. The powder from *Hibiscus Tiliaceus* was then sieved using a 200-mesh sieve.

*Alkali treatment of HTP*

HTP was immersed in 5% wt. potassium hydroxide (KOH) solution with different immersion times, namely 2 h, 4 h, 6 h, and 8 h, then HTP was rinsed with plain water for five times, and dried using an oven at 105°C for 60 min.



Fig. 1 Process of making powder, (a) HT branches, (b) HT fibers, (c) shredded of fibers, (d) fiber milling, (e) fiber sieving, (f) hibiscus tiliaceus powder (PS).

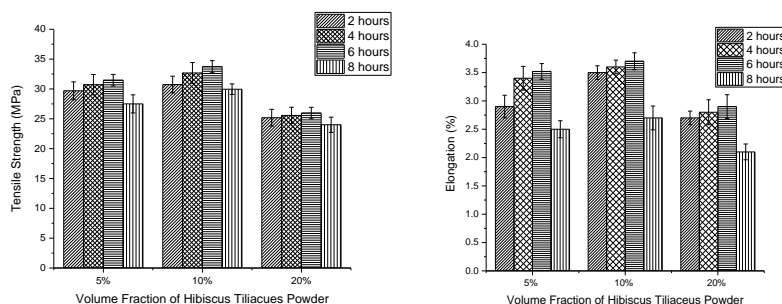
#### Composite fabrication

The hot press technique is used to prepare the composite HTP treated in different time immersion of 2 h, 4 h, 6 h, and 8 h respectively and vol. fraction of HTP namely 10%, 15%, and 20%. Methyl ethyl ketone peroxide (taking 1% of the vol. fraction of polyester) is mixed into the polyester (PE) and stirred; then the dough is poured into a mold that has been filled with HTP. The mold is closed and then given a pressure of  $\pm 5$  MPa for 12 h. The composite is removed from the mold.

## RESULTS AND DISCUSSIONS

#### Tensile strength, elongation, and modulus Young's Analysis

Fig. 6a shows that in composites with an HTP of 5% and 10% (vol. fraction), the tensile strength values of the composites tended to increase by about 28.95% and 34.27%, respectively, after HTP treated for 2 h to 6 h. The interfacial bond between HTP-PE became stronger and ultimately resulted in a higher tensile strength of the composite which was indicated by changes in the HTP surface becoming rough. The mechanism of changes in the surface roughness of natural materials due to KOH treatment was proposed by Sari and Padang [1], which showed that the rough HTP-surface improves interlocking with PE, provides a strong interface, and can transfer loads better. The tensile strength of the composite decreased after the HTP was soaked for 8 h which was related to the HTP being damaged, and a pullout occurred. The maximum tensile strength obtained from the composite with 10% HTP after soaking for 6 h was 33.73 MPa. The tensile strength decreased when HTP above 10% was associated with HTP buildup in the composite; The tensile properties of this composite were higher than *Acacia Arabica* bark composites (15.65 MPa – 18.62 MPa) [6]. Fig. 6b shows there was an increase in the elongation of the composite after HTP immersed in KOH solution for 2, 4, and 6 (h) which was associated with a stronger interfacial bond between HTP and PE. At constant volume fraction of HTP (5%, 10%, and 20%), the MoE value of the composite increased after HTP treated for 2 h - 6 h (Fig. 6c). The most optimal MoE value was obtained from composites with vol. fractions. 10% HTP treated for 6 h reached 1480.63 MPa. On the other hand, more interactions of HTP (20% vol. fraction) creates composite stiffness and causes the MoE to decrease.



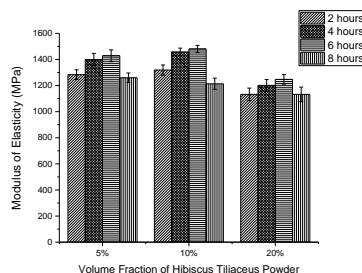


Fig. 6 (a) Tensile strength, and (b). Elongation, (c) modulus of elasticity of composites.

#### SEM analysis

Fig. 9a show that the interface bond between the HTP and PE is dense, the HTP pullout and fracture occurs along with the PE in the composite resulting in low mechanical strength. Fig. 9b compared to other composites, a tighter interfacial bond between HTP and PE in the composite 10%HTP/6h; Fig. 9c shows a fairly dense interface between the HTP–resin, and a small amount of HTP pullout in the 10% HTP/8h composite. Fig. 9d provides a large number of HTP pullouts on composites and a less dense HTP–resin interface; which indicated that most of the HTP were not wetted by the PE resin due to the higher amount of HTP in the composite.

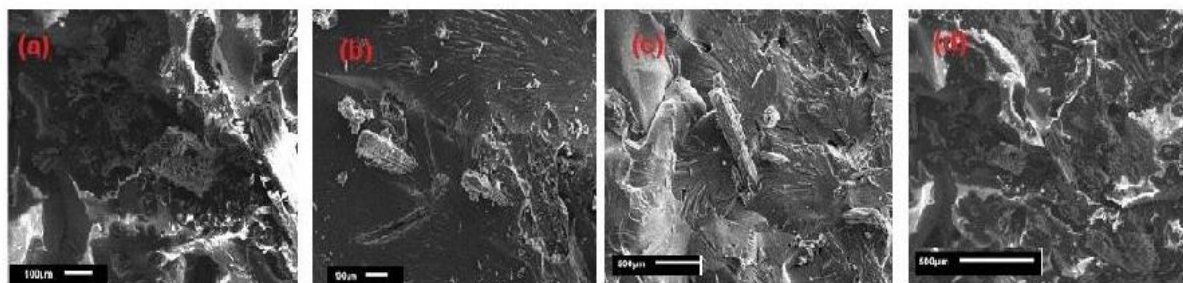


Fig. 9 SEM images of composites, (a). 5%HTP/6h, (100x) (b) 10%HTP/6h, (100x) (c) 10%HTP/8h, (50x), (d). 20%HTP/6h (50x).

#### CONCLUSIONS

The results showed that at the 5% – 10% HTP (vol. fraction), the tensile strength tended to increase by about 34.27% after HTP treated in 5%KOH for 2 h to 6 h, then decreased after HTP treated for 8 h. The highest tensile strength and was obtained from the composite 10% HTP/6h of 58.73 MPa. The increase in strength was associated with a stronger and denser interface between resin and HTP. For composites after HTP was soaked for 8 h in KOH, HTP became damaged and easily broken, causing the composite strength to also be low.

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## INVITED SPEAKER 11

## ANTI UV LIGHT PROPERTIES OF BACTERIAL CELLULOSE/UNCHARIA GAMBIR COMPOSITES TREATED WITH ULTRASONICATION

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**ABSTRACT**

The motivation to develop environmentally friendly materials with good absorption of harmful rays continues to grow. This work aims to characterize the anti-UV properties of bacterial cellulose (BC)/Uncaria gambir (G) composite films made without and with ultrasonication treatment. The results showed that the ability of the sample to absorb UV light increased after the addition of G in the sample. Furthermore, ultrasonication of the BC suspension without and with G increased the transparency of the sample. The results of this study can introduce gambir as an additive for UV absorption.

*Keywords:* Gambir, catechin, nanocellulose, UV absorption

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**INTRODUCTION**

Cellulose is an intermolecular arrangement of hydrogen bonded linear polysaccharides that can form semi- or crystalline microfibril structures [1]. Such a unique structure makes cellulose can withstand high temperatures and is insoluble in conventional organic solvents. Therefore, cellulose has been used as a raw material for the manufacture of environmentally friendly polymers. Transparent polymer of cellulose has been successfully demonstrated [2]. Abrial et al have reported a transparent film made from sonicated ginger nanofibers [3]. Recently, a transparent film of bacterial cellulose nanofiber has been published [4]. However, these transparent films have low UV absorption capabilities.

Gambir (*Uncaria gambir* Roxb.), was commonly found in West Sumatra, Indonesia. The plant has been cultivated for decades by farmers. Gambir leaves and young twigs are plant parts that are processed into gambir sap in the form of blocks or gambir powder. Gambir has a distinctive aroma and tastes bitter on the tongue. This material has long been used as a complement to betel. In addition, the product is also one of the profitable export commodities. Gambir contains several chemical components, one of which is catechins [5]. Catechins are complex flavonoid compounds from the polyphenol group [6]. Research on the use of catechins from gambir is still mostly done in the development of pharmaceutical products [6]. Previous studies have proven that catechins have several antioxidant, antibacterial and pharmacological effects [7]. These polyphenolic catechins have a purple color similar to the color of tannins. Previous research reported that a tannin-based composite film was able to absorb UV rays [8]. To our knowledge, gambir incorporated cellulosic film for packaging materials has not been published so far. Therefore, the aim of this study was to characterize the UV properties of the bacterial cellulose/gambir composite film prepared without and with ultrasonication treatment.

**MATERIALS AND METHODS***Materials*

A wet pellicle was purchased from a local small industry in Padang. TEMPO (Tetramethyl Piperidine-1- Oxyl radical) powder, NaOH, NaBr, NaClO, HCl, and distilled water were obtained from a local shop, Padang.

*Methods*

The pellicle with a size of 6 x 10 cm was soaked for 48 hours in distilled water mixed with 10% NaOH. After neutralization, the clean pellicle was cut into 1 x 1.5 cm size. The 111 g pellicle was then put into a solution with a composition of 0.036 g

TEMPO and 0.4 g NaBr, and 400 ml distilled water and then stirred for 5 minutes. 7.4 g NaClO was added until the solution reached pH 9, and stirred again using a stirrer for 20 minutes at a temperature of 50°C. 5% NaOH was dripped step by step into a beaker containing pellicle to pH 10. The pellicle suspension was stirred at 100 °C for 6 hours at 650 rpm. After cooling for 12 hours, the nata de coco suspension was filtered using a mesh sieve with sizes from 100, 200, 300 and 500 mesh. Then a solution containing 0.05% gambir was prepared. A 200 ml suspension which had passed through a 500 mesh was treated without and with ultrasonic for 15 and 30 minutes. The symbols for treatment of non-gambir samples without and with ultrasonic 15, and 30 minutes are NG-0, NG-15, and NG-30, respectively. The symbols of the mixed

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gambier sample without and with ultrasonic 15, and 30 minutes are G-0, G-15, and G-30, respectively. Furthermore, the suspension was poured into a Teflon to be dried in an oven for 24 hours at a temperature of 50°C. The dry film was then stored in a desiccator before testing at RH 50.

## RESULT AND DISCUSSION

Fig. 1 shows optical photos of the sample without and with gambier and ultrasonication. The sample without ultrasonic has the lowest transparency. For example, samples NG-0 and G-0 display the blurriest words "Universitas Andalas" compared to others. However, after being ultrasonicated, the transparency of the sample improved. This phenomenon can be caused by less light reflected by the ultrasonic film than the non-ultrasonicated film [9]. This result is supported by the transmittance value of the films (Fig. 1b) which shows an increase in the transmittance value after ultrasonication. In addition, films without gambier have a lower ability to absorb UV light (less than 400 nm) compared to gambier cellulose composites. This composite was even able to absorb 100% of UV rays.

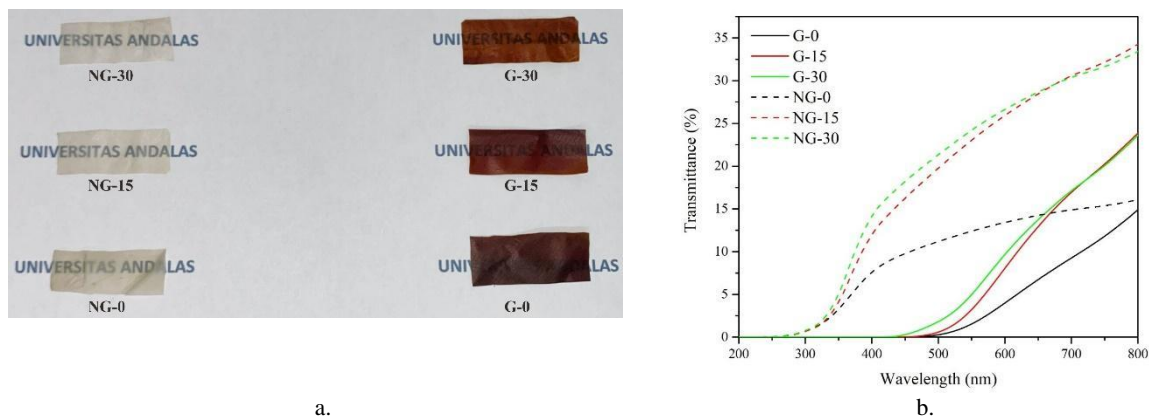


Fig. 1: (a) Optical photo of samples, (b) transmittance value for each film.

## CONCLUSIONS

We have successfully proposed the addition of gambier as a novel material for absorbing UV rays in cellulose-based composites prepared without and with ultrasonication treatment. Ultrasonication has increased the transparency of the cellulose samples. The same phenomenon also occurs in BC/G composites which show more transparency after ultrasonic treatment. Non- and sonicated BC/G composites had the ability to absorb 100% of UV rays. All these findings have clearly demonstrated that our sonicated BC/G composites are transparent, UV protective materials that can be used as protective films for food substances.

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## INVITED SPEAKER 12

## WASTE TO VALUE: APPROACH FOR SUSTAINABLE FUTURE

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## ABSTRACT

The latest research trends focus on the sustainable development of materials, aiming to improve the quality of living, protect the ecosystem, responsible consumption and production. Currently, about 2.01 billion tons of municipal solid waste (MSW) is produced annually worldwide, of which about 15% are plastics. It is estimated that the MSW will increase to 3.40 billion tons by 2050. Only 13.5% of which is recycled and 5.5% is composted. Nowadays to reuse and recycle denim waste is a key challenge towards responsible disposal of textiles waste. So, recycling textile wastes into composites with sustainable method is a crucial issue. To support the circularity, we have to see the waste as potential raw material. The current study focuses on the development of a composite product using textile and polymeric waste. The denim waste was used as reinforcement material, while polyethylene and polycarbonate were used as matrix material. The composites were fabricated using lab scale compression molding machine. The mechanical performance of the composites showed that the acrylic polymeric waste reinforced with denim waste has superior mechanical performance. These composites may find application in numerous areas including automotive interior, structural parts, floor/wall tiles, etc.

*Keywords:* denim waste, polymer waste, composite materials, acrylic, polyethylene

## INTRODUCTION

Sustainability, eco-friendliness, industrial ecology and green chemistry are the key drivers for development of the next generation of materials, processes and products. Natural/Bio fiber composites are evolving as a feasible alternative to glass fiber reinforced composites particularly in building and automotive product applications [1]. The composite produced using nature-based fibers such as flax, jute, kenaf, henequen, hemp, pineapple leaf fiber, and sisal with polymer matrices from both renewable and nonrenewable resources to create composite materials that can compete with synthetic composites needs special attention, i.e., novel processing and bio fiber–matrix interface. Natural fiber–reinforced polypropylene composites have attained commercial attraction in automotive industries [2]. The properties of natural fibers and glass fibers are compared in Table 1 [3].

TABLE 1: Comparison of natural fiber and glass fiber properties

Fiber	Density (g/cm <sup>3</sup> )	Tensile modulus, GPa	Specific tensile modulus, GPa	Tensile strength, MPa	Specific tensile strength, MPa
Flax	1.45-1.55	28-100	19-65	343-1035	237-668
Hemp	1.45-1.55	32-60	22-39	310-900	214-581
Jute	1.35-1.45	25-55	19-38	393-773	291-533
Sisal	1.40-1.45	9-28	6-19	347-700	248-483
Pineapple	1.44-1.56	6-42	4-27	170-727	118-466
Banana	1.30-1.35	8-32	6-24	503-790	387-585
Cotton	1.50-1.60	5-13	3-8	287-597	191-373
Coir	1.10-1.20	4-6	3-5	131-175	119-146
Oil palm	0.70-1.55	3-4	2-4	248	160-354
Bamboo	0.60-1.10	11-30	18-27	140-230	210-233
Glass	2.55	78.5	31	1956	767

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Denim is one of the most worn apparel articles across the globe, both by males and females. The industrial waste and postconsumer waste of denim creates an environmental burden, as it is either burn or landfilled [4]. The most common approach to use this waste include its recycling into fibers that are again spun into coarse yarns, but it requires a lot of processing time. On the other hand, the polyethylene and polycarbonate are one of the most common polymeric wastes. This research focuses on the production of green composites using these waste materials, that would not only be benefiting

environmentally but it would be low cost as well. Such novel green composites can be used in automotive, interiors and other engineering materials as a substitute of traditional composites [5].

### MATERIALS AND METHODS

#### Materials

The textile denim waste and polymer waste (polyethylene and polycarbonate) were used as reinforcement and matrix respectively, for the development of composite materials.

#### Methods

The methodology adopted for the development of composite materials from waste textile and polymer is shown in Figure 1.



Fig. 1: Biodegradable film from sugar palm tree

The composite materials with various fiber volume fractions were fabricated and mechanical properties of the developed composite materials were investigated.

### RESULT AND DISCUSSION

The equipment used for characterization of tensile, flexural and impact properties of developed composite materials are shown in Figure 2. These properties were investigated as per standard test methods ASTM D3039, ASTM D7264 and ISO 179 respectively.

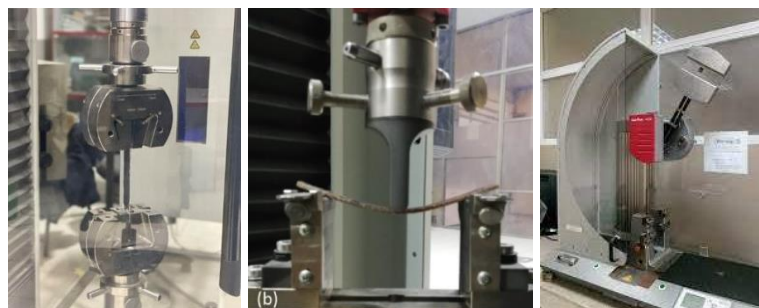


Fig. 2: Equipment used for testing of composites

The stress-strain curves obtained in tensile test showed that the composites having polyethylene and polycarbonate as matrix demonstrate significantly different behavior. The polyethylene composites were characterized by high extension % and less failure strengths, with a reduced elastic part having steeper slope showing low value of modulus. Contrary to that the polycarbonate composites had a low extension %, extended elastic region and slope more inclined towards the y-axis, showing high modulus.

During flexural test the load-deflection curve of polyethylene composites was very steep with deformation % of up to 15% and flexural strength as low as 6.5 MPa. While the polycarbonate composites demonstrated high flexural modulus and strength, owing to low deformation % and higher stress values born by the specimen.

The pendulum impact properties showed a remarkably different trend. The polyethylene composites absorbed higher energy (up to 32 KJ/m<sup>2</sup>), as compared to the polycarbonate composites (up to 16 KJ/m<sup>2</sup>). This can be attributed to the relatively ductile nature of the polyethylene matrix, that resisted more to the deformation and absorbed higher energy during an impact test as compared to the polycarbonate composites.

### CONCLUSIONS

The mechanical properties of the developed composite materials showed that these materials may be used as a substitute of the conventional composite materials. On the basis of energy absorption, bending and tensile properties, it can be concluded that the polycarbonate composites have relatively superior properties as compared to the polyethylene composites. The outcomes of this study conclude that the PIW and PCW can be effectively used to make value added products, e.g., floor tiles, etc. It is also a sustainable approach employing concept of adding value to the waste.

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## INVITED SPEAKER 13

## CELLULOSE NANOFIBRILS FROM TEXTILE WASTES TO BE USED AS REINFORCEMENT IN BIO-NANOCOMPOSITES

Rajesh Mishra<sup>1\*</sup>, Michal Petru<sup>2</sup><sup>1</sup>Department of Material Science and Manufacturing technology, Faculty of Engineering, Czech university of Life Sciences, Prague, Czech Republic<sup>2</sup> Faculty of Mechanical Engineering, Technical University of Liberec, Czech Republic**ABSTRACT**

In the present study, waste jute fibers generated in textile industries, were wet pulverized to the scale of nanofibrils of 50 nm diameter using high energy planetary ball milling for three hours. The rate of refinement of uncleaned jute fibers having non-cellulosic contents was found slower than the cleaned jute fibers. This tendency is attributed to the strong holding of fiber bundles by non-cellulosic contents which offered resistance to the defibrillation during wet milling. In addition, the presence of water during wet pulverization found to reduce the rising temperature of mill, which prevented sticking of nanofibrils on the mill wall and resulted in unimodal size distribution. The goal of present study was to utilize the waste jute fibers in textile industry as a source of cellulose nanofibrils for reinforcement of biodegradable packaging films. The jute nanofibrils were obtained by wet pulverization using high energy planetary ball milling process instead of strong acid hydrolysis due to its simple, economical and environment friendly approach. The extended wet milling for the duration of three hours resulted into unimodal distribution of jute nanofibrils with diameter below 50 nm. In the subsequent step, obtained jute nanofibrils were incorporated at 1 wt %, 5 wt % and 10 wt % loading in PLA matrix and their reinforcement was evaluated based on improvements in mechanical properties.

*Keywords:* nanocellulose, bio-nanocomposite, nanofibrils, food application.

**INTRODUCTION**

Due to limited availability of petroleum resources and increased concerns over disposal from clean environment point of view, research on renewable materials have gained importance in recent years [1,2]. Within the period of 2005 and 2009, global market on the demand of biodegradable polymers was double in size. Among all countries in the World, Europe had the largest growth in the range of 5–10 % on the use of biodegradable polymers in the year 2009. The total consumption of biodegradable polymers has been grown at an average annual rate of nearly 13 % from 2009 to 2014 in North America, Europe and Asia [3]. Nowadays significant amount of research is being carried out to further increase the market potentials of these materials by reducing their higher price and by improving their properties for different applications. The development of biocomposite materials by incorporation of renewable reinforcing elements is considered as one of the favorable solution to meet these requirements [2].

Over the last two decades, reinforcement potentials of lignocellulose fibers have been investigated in numerous studies of biocomposites made from PLA [4]. However, the reinforcement potentials of lignocellulose fibers are found not enough to meet demands of high-performance applications. In addition, there is no clear trend in improvement of mechanical properties after addition of lignocellulosic fibers [5].

**MATERIALS AND METHODS***Materials*

Short waste jute fibers were obtained from India. The fibers were measured to have a density of 1.58 g/cm<sup>3</sup>, modulus of 20 GPa, tensile strength of 440 MPa and elongation of 2 %. The chemical composition of fibers was reported as cellulose (60 %), hemicelluloses (20 %), lignin (10 %) and others (10 %). Poly lactic acid (PLA) was purchased from NatureWorks LLC, USA through local supplier Resinex, Czech Republic. The PLA had a density of 1.25 g/cm<sup>3</sup> and the average molecular weight (Mw) of 200,000. The chloroform which was used as solvent, purchased from Thermofisher, Czech Republic.

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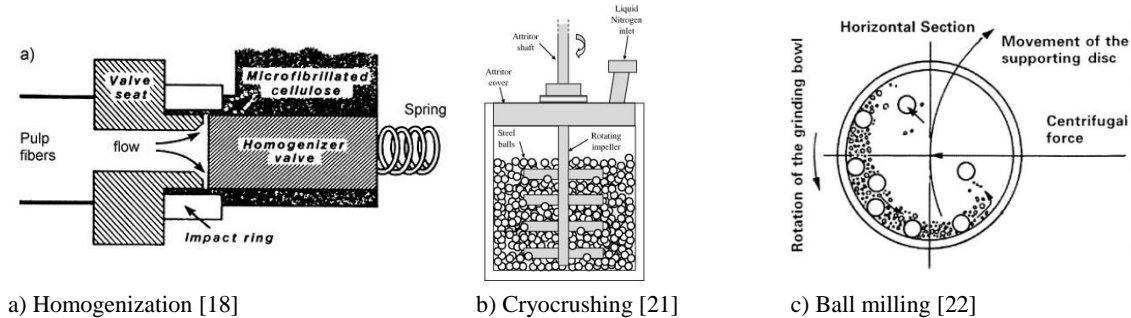
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*Methods**Preparation of nanoscale jute fibers.*

After getting the optimum milling parameters of ball milling process, wet pulverization of waste jute fibers was carried out in distilled water using a high-energy planetary ball mill of Fritsch pulverisette 7. The sintered corundum container of 80 ml capacity and zirconium balls of 3 mm diameter were chosen for 3 hours of wet milling. The ball to material ratio (BMR) was kept at 10:1 and the speed was kept at 850 rpm with reverse rotation of containers. At the end of wet milling, jute particles were separated from water

by centrifugation at 4000 rpm and simultaneously transferred in solvent isopropanol to avoid hornification during drying. On the basis of their dimensions, functions, and preparation methods, nanocelluloses are classified in three main subcategories as nanocrystalline cellulose (NCC), nanofibrillated cellulose (NFC) and bacterial nanocellulose (BNC) as shown in Figure 1.



a) Homogenization [18]

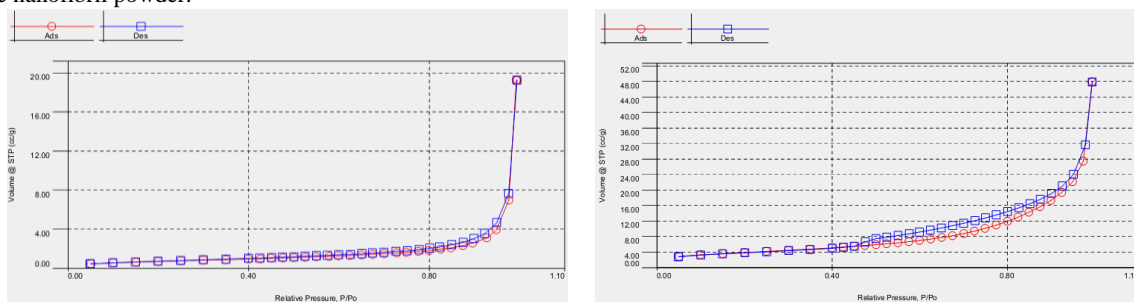
b) Cryocrushing [21]

c) Ball milling [22]

Fig. 1 Extraction methods of cellulose nanofibrils

RESULT AND DISCUSSION

From Figure 2(a) and Figure 7(b), the isotherm was classified to type IV according to IUPAC classification, and the broad adsorption-desorption loop was of type H3. This indicated that the materials comprised of aggregates (loose assemblages) of the plate-like particles forming slit like pores. The sharp nitrogen uptake near P0 indicated a continuity of the pore size distribution between mesopores and macropores and thus the presence of both meso- and macro-porosities in the prepared jute nanofibril powder.



(a) One hour dry milling

(b) One hour wet milling

Fig. 2 Isotherm of jute particles

The surface area that inferred from the N<sub>2</sub> adsorption isotherm of BJH analysis (Figure 3) was found about 15 m<sup>2</sup>/g and 3.3 m<sup>2</sup>/g for wet milled jute powder and dry milled jute powder respectively. This shows that the wet milling of waste fibers is more efficient in refinement of size than the dry milling process.

To further refine the jute particles to smaller size, wet milling was performed for extended duration. The average particle size reached to 443 nm after 3 hours of wet milling and the particle size distribution changed slowly from multimodal nature to unimodal nature as shown. This showed the consistency and homogeneity in milling action on every individual particle as milling continued for longer time. However, the rate of refinement became slower while grinding the smaller particles in addition to the severe damage of milling balls due to direct collision. This could have introduced some inorganic contaminations from mill to the material, so further pulverisation was stopped and jute particles in 500 nm range were used as nano/micro fillers.

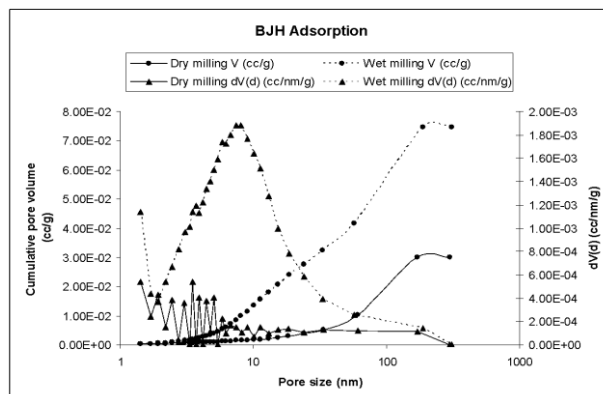


Fig. 3 Surface area of jute particles

**CONCLUSIONS**

The results showed that improvements in mechanical properties are dependent on interaction between nanofibril-matrix as well as on crystallinity of PLA in nanocomposites. Due to the possibility of agglomerations and entanglements of nanofibril at different loading, the surface area of interaction and area of nucleating nanofibril changed in the composites. This resulted in a large difference in the mechanical properties between all samples. The 5 wt % nanocomposite films showed maximum improvements in mechanical properties, while 10 wt % nanocomposite films revealed deterioration in properties. The improvements in storage modulus of PLA nanocomposite films over neat PLA were more evident at 60°C than 35°C. The storage modulus of 5 wt % nanocomposite films found to improve by only 195.79 % at 35°C, whereas huge improvements in storage modulus by 475.00 % observed at 60°C. The restricted mobility of PLA chains by presence of stiff JNF is attributed to the improvements in load bearing capacity of PLA at higher temperature. The initial modulus and tensile strength were increased by 217.30 % and 170.59 % respectively in case of 5 wt % nanocomposite compared to neat PLA film. However, elongation to break was found to reduce in all composite films compared to neat PLA film due to increased brittleness, which was induced by increased crystallinity of PLA in composites. The composite films at higher JNF content of 10 wt % revealed deterioration in mechanical properties due to nonhomogeneous stress transfer from matrix to nanofibrils caused by poor dispersion and agglomerations of nanofibrils. However, 15.70 % improvements in  $T_g$  and 62.15 % improvements in crystallinity are reported at 10 wt % JNF loading in PLA. This suggested that the deterioration in mechanical properties could be prevented by allowing uniform dispersion and less agglomerations of JNF in nanocomposites. Finally, the experimental results of Initial modulus revealed good agreement with predicted elastic modulus of theoretical models up to 5 wt % loading of JNF. The closeness of experimental results with Cox-krenchel theory suggested random orientation of nanofibrils in the composites. In this way, it is possible to say that in order for nanofibrils to reach their full reinforcing potentials, more attention will have to be given into the dispersion and alignment of nanofibrils within PLA matrix.

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## INVITED SPEAKER 14

CITRIC ACID USED AS CROSSLINKING AGENT IN MODIFIED CELLULOSE  
EXTRACTED FROM OAT HULL

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## ABSTRACT

Cellulose is a natural homopolymer that can be obtained from several lignocellulosic residues. The aim of this work was to extract cellulose from oat hull by bleaching with peracetic acid and to modify the cellulose by introduction of crosslinking using an organic acid using reactive extrusion, a totally green process. Citric acid (CA) was employed as a crosslinking agent in different concentrations (5, 12.5 and 20%). The FTIR results proved the esterification process occurred by showing a new band near  $1730\text{ cm}^{-1}$  in all samples. This work proved to be possible to modify cellulose from lignocellulosic residues using an eco-friendly process, with lower effluents generation compared to conventional processes.

*Keywords:* Reactive extrusion, crosslinking agents, cellulose

## INTRODUCTION

Cellulose is a natural glucose homopolymer that can be obtained from several lignocellulosic residues [1], [2]. Modifications in the cellulose structure have been largely studied to enhance its properties. Among all the possible modification types, chemical crosslinking is one of the most used technique [3], [4]. In the crosslinking process, intramolecular bonds are formed between the cellulose chains reinforcing the polymer chain. Citric acid (CA) has been used as a crosslinking agent for cellulose, this renewable source reagent is listed as GRAS (generally recognized as safe) by the FDA in the United States [3]–[7]. CA is an organic acid largely used in pharmaceutical and food industries [5]. According to He et al. [4], cross-linked cellulose has new functionalities and properties, including a more hydrophobic character. The aim of this study was to produce cross-linked cellulose by reactive extrusion using citric acid as a crosslinking agent and to determine its degree of substitution, Fourier transform infrared (FTIR) spectroscopy profile and water and oil absorption capacities of the modified samples.

## MATERIALS AND METHODS

*Materials*

Oat hull was obtained from SL Alimentos e Cereais (Paraná-Brazil). Citric acid (CA) was of analytical grades, like all other chemicals and solvents.

*Methods**Extraction of the Cellulose from Oat Husks*

The extraction of the cellulose was made following the process described by Marim et al. [8]. Oat hulls (10 g) were treated with 250 mL of peracetic acid, the suspensions were maintained on a mechanical stirrer with a controlled temperature of  $60\text{ }^{\circ}\text{C}$  for 24 h. After this procedure, the samples were filtered, and washed with distilled water to reach pH 5-6 and dried at  $60\text{ }^{\circ}\text{C}$  for 24 h in an air circulation oven.

*Modification of Cellulose by Reactive Extrusion*

The citric acid (CA) was used in different concentrations (0, 5, 12.5 and 20 % - g/ 100 g cellulose), it was dissolved in distilled water and mixed with de cellulose (100 g), resulting in the final moisture content of 32% (g/g), and then the solution was slowly added to a sealed bag and equilibrated for 1 h. A control sample was extruded without any reagent other than water, resulting in the final moisture content of 32% (g/g). Then, all samples were extruded in a single screw extruder (AX Plastics, Diadema, SP, Brazil) with a screw diameter of 1.6 cm and a screw length/diameter ratio (L/D) of 40, with four heating zones and a matrix of 0.8 cm in diameter. The temperature in all zones was  $100\text{ }^{\circ}\text{C}$  and the screw speed was 60 rpm. The modified cellulose extrudates were collected, placed in an oven, dried to constant weight at  $45\text{ }^{\circ}\text{C}$ , ground, and sieved through an 80-mesh sieve. The dry mixture was washed three times with absolute ethanol to remove the unreacted citric acid. Finally, the

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washed cellulose was air-dried at  $45\text{ }^{\circ}\text{C}$ .

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*Determination of the Degree of Substitution (DS)*

The DS of the modified cellulose was determined using the method of Karnitz et al. [9].

*Fourier Transform-Infrared Spectroscopy (FTIR)*

The pulverized and dried samples were then mixed with potassium bromide and compressed into tablets. The FT-IR analyses were carried out with a Shimadzu FT-IR —8300 (Kyoto, Japan), which has a spectral resolution of  $4\text{ cm}^{-1}$  and a spectral range of  $4000\text{--}500\text{ cm}^{-1}$ .

*Water Absorption Capacity (WAC) and Oil Absorption Capacity (OAC)*

WAC and OAC were determined following the methodology described by Lu et al. [10].

**RESULT AND DISCUSSION***Fourier Transform Infrared (FTIR) Spectroscopy*

FTIR spectra of cellulose and modified cellulose with citric acid are shown in figure 1. It is possible to see that all modified samples presented a new important band located at  $1730\text{ cm}^{-1}$ . This band can be associated with the C=O stretching of carbonyl in the ester bonds, which confirms that esterification using CA. These results are similar to those presented by other authors [4,6,10,11,12], who also used CA as crosslinking agent, they used the FTIR spectra of the samples between  $1720\text{ to }1750\text{ cm}^{-1}$  to show the success of esterification. The control and unmodified cellulose samples presented the same band near  $1720\text{ to }1750\text{ cm}^{-1}$  lower intensity, and according to Gil Giraldo et al. [10] it can be explained by the thermomechanical treatment that occurs with cellulose during extrusion process, so it could be attributed to the C=O group from the opened terminal glucopyranose rings.

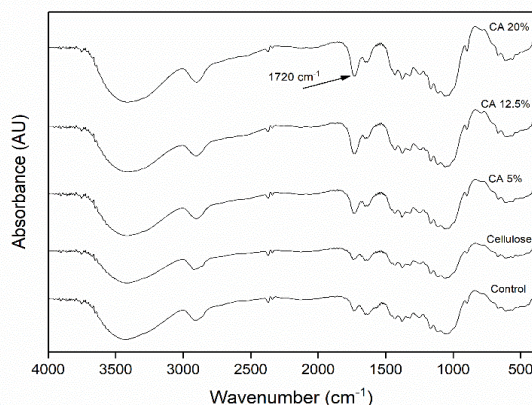


Fig.1: Fourier transform infrared spectroscopy spectra of cellulose, control sample and modified cellulose with citric acid.

The band at  $3500\text{ cm}^{-1}$  can be assigned to O–H stretching groups [6]. The band near  $2900\text{ cm}^{-1}$  is due to the C–H stretching [12]. All the bands between  $1057\text{ and }1162\text{ cm}^{-1}$  are corresponding to C–O and C–O–C stretching vibration in cellulose [6].

*Degree of Substitution (DS)*

The DS results are shown in the table 1. It is possible to see that the DS values increased with increase of citric acid concentration, higher acid concentrations led to a higher DS values. Liu et al. [12] in his study with modified cellulose achieved a DS value ranged from 0.071 to 0.53 while in our study the DS results ranged from 2.3 to 3.

TABLE 1: Degree of substitution of all samples.

Samples	Degree of Substitution
Cellulose	0
Control sample	0
Citric Acid 5%	2.3
Citric Acid 12.5%	2.4
Citric Acid 20%	3.0

*Water Absorption Capacity (WAC) and Oil Absorption Capacity (OAC)*

WAC results are shown in Table 2. For the modified cellulose, WAC values ranged from 6.46 to 7.13 (g/g). When compared to cellulose sample (9.27 g/g), all the modified samples presented a significantly lower value. Gil Giraldo et al. [10] in their study with modified cellulose with citric acid also described these lower WAC results when cellulose was modified with citric acid.

TABLE 3: Water Absorption Capacity (WAC) and Oil Absorption Capacity (OAC) of all samples.

Samples	WAC (g/g)	OAC (g/g)
Cellulose	$9.27^a \pm 0.07$	$1.80^d \pm 0.01$

Control sample	6.55 <sup>bc</sup> ± 0.01	6.25 <sup>c</sup> ± 0.61
Citric Acid 5%	6.46 <sup>bc</sup> ± 0.01	7.26 <sup>bc</sup> ± 1.33
Citric Acid 12.5%	7.13 <sup>b</sup> ± 0.01	9.07 <sup>a</sup> ± 0.55
Citric Acid 20%	6.78 <sup>bc</sup> ± 0.05	9.94 <sup>a</sup> ± 0.32

\*Different letters indicate significant differences ( $p \leq 0.05$ ) between means (Tukey's test).

From the OAC results it is possible to see that the cellulose sample presented the lowest OAC value of 1.80 g/g, while the modified samples CA 12.5% and CA 20% showed the highest values. From these results, we can conclude that the increase of acids in the formulations resulted in samples more hydrophobic. This increase in hydrophobic capacity of modified samples can be due to the decrease of free hydroxyl groups on the cellulose surface by reaction with CA. Adewuyi et al. [14] in their study with modified cellulose with suberic acid also reported the increase in hydrophobicity of modified samples. Gil Geraldo et al. [10] in their study with modified cellulose with citric acid reported the reduction in WAC and increasing in the OAC in all cellulose modified samples.

## CONCLUSIONS

The extraction of cellulose from oat hulls using organic acids was successfully achieved. The FT-IR results proved the esterification with CA in all modified samples with the appearance of the band near 1730  $\text{cm}^{-1}$ . This process can be considered a promising way to obtain polymer modification, being an environmentally friendly process.

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## INVITED SPEAKER 15

## PULP AND PAPERMAKING POTENTIAL OF SUGAR PALM

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## ABSTRACT

*Arenga pinnata* is a kind of sugar palm that suits growing in the tropical countries like Malaysia. It is very popular due to its multipurpose usage that can be produced from parts of the sugar palm. The objective of this paper is to discuss the potential of sugar palm as pulp and papermaking sources referring to many studies of sugar palm in terms of its physical, mechanical, and chemical properties. Other kind of palms that have been tested their pulp and papermaking are also referred as a guidance such as oil palm, coconut and date trees.

**Keywords:** sugar palm, pulp and papermaking, physical properties, mechanical properties, chemical properties.

## INTRODUCTION

In pulp and papermaking manufacture, wood is used as the main raw material due to its good properties in all aspects especially strength. However, paper millers began to find more potential raw materials due to environmental impact of logging to the global warming. The situation is back to the early of pulp and paper history where the beginning of paper used non-woody materials like papyrus, hemp and mulberry bark to make paper [1]. Non-wood materials can be obtained from agro-waste fibres or any biomass. Many studies have proven that non-woody plant such as kenaf, bagasse, corn stalks, cotton stalks, oil palm empty fruit bunches, oil palm frond, oil palm trunk, pineapple leaves, coconut coir, date palm and more are technically suitable to make pulp and later on to make paper [2].

Palm trees are well known as evergreen plant that belong under *Arecaceae* family. There are about 2,600 species of palm trees in the world under over 200 genera that grow at different heights by reaching up to 30 m heights such as coconut palms with branchless stems and huge green leaves. Most palms can grow healthily in warm countries. Palm trees can be identified via the appearance of their heights, leaves and trunk. They have either palmate or pinnate leaves that are feather-like leaves or fan-like fronds accordingly.

Sugar palm, oil palm, date palm and coconut tree are listed among the palm trees that have various usage from their part of plant. Sugar palms are unbranched palm that can grow to more than 12 m and 30 cm in terms of height and diameter respectively having pinnate leaves. It is very popular on its concentrated sugar that have amazing nutritional benefits. It's seera, the sweet liquid from the fruit can be processed into drink, vinegar or sweetener [3] as well palm sap sources, characteristics, and utilization in Indonesia. Besides, one of its important components called ijuk can be made into broom, roof, screener, building material and more. Figure 1 shows the sugar palm tree, sugar palm frond and sugar palm fiber. The fiber has very strong mechanical properties because it has high resistance towards water especially sea water and fungal attack.

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Oil palm is a palm tree that is originated from West Africa can achieve 20 to 30 m heights and is planted all over Malaysia, Indonesia, Thailand, and some other countries in the world. The value of oil palm tree is found from its fruits that contain oil that is beneficial for cooking [4]. Aggressive research has been done for 30 years ago on the other potential part of oil palm tree that can

transform into other precious products. One of the identified products are pulp and paper. Frond, trunk and oil palm empty fruit bunches are 3 main parts of oil palm tree that are proven as suitable raw materials for pulp and papermaking [5].

Date palm appeared as unbranched with a crown of newly formed leaves at the top of stem. It is planted in tropics and subtropics area of the world. The research on date palm on pulp and papermaking are getting higher that mostly part been used in pulp making is the frond and stem parts [6].

Coconut tree is another glamour palm trees because all parts are used as raw materials for various daily products. The trunk can be used to make bridges, the leaves to make broom, cover for 'ketupat nasi', the fruit contain water that are delicious and nutritious to reduce fever, or traditional medication, the coir fibres can be changed into pulp and paper, or fibres to make pots and so many other useful products especially to the villagers in Malaysia or other tropical countries as well [7].



Fig. 1: (a) sugar palm tree, (b) sugar palm frond, and (c) sugar palm fibre

## CHEMICAL AND PHYSICAL PROPERTIES

In pulp and papermaking, chemical and physical properties are the first test that need to be carried out to consider them as potential in pulp and papermaking activities. As shown in Table 1, all raw materials included the sugar palm obtained readings of cellulose content more than 40%. Meanwhile, the holocellulose content for all raw materials shown more than 60%. Lignin content of all raw materials not exceeded 40% except sugar palm trunk. These indicated that sugar palm ijuk, sugar palm bunch and sugar palm frond can be used in pulping process and papermaking as well.

TABLE 1: Chemical constituents of oil palm, date palm, coconut and sugar palm parts.

Raw material	Cellulose Content (%)	Holocellulose Content (%)	Lignin Content (%)	References
Oil palm empty fruit bunch (EFB)	42.7	70.0	17.2	[8]
Oil palm frond (OPF)	56.0	83.5	20.5	[5]
Oil palm trunk (OPT)	41.0	73.1	24.5	[5]
Date palm rachis	45.0	74.8	27.2	[6]
Date palm leaves	33.5	59.5	27.0	[6]
Coconut coir	37.2	69.1	32.7	[7]
Sugar palm ijuk	52.3	65.6	31.5	[9]
Sugar palm bunch (SPB)	61.8	71.8	23.5	[9]
Sugar palm frond (SPF)	66.5	81.2	18.9	[9]
Sugar palm trunk (SPT)	40.6	61.1	46.4	[9]

Based on Table 2, all raw materials have fiber length of more than 0.80 mm which fibers of oil palm frond, oil palm trunk, date palm leaves and sugar palm frond achieved more than 1.00 mm length. Sugar palm frond is found to have thickest fiber than other raw materials which same condition are shown via cell wall thickness readings.

TABLE 2: Physical properties of oil palm, date palm, coconut and sugar palm parts.

Raw material	Fiber length (L), mm	Fiber diameter (d), $\mu\text{m}$	Lumen width (l), $\mu\text{m}$	Cell wall thickness (w), $\mu\text{m}$	References
Oil palm empty fruit bunch (OPEFB)	0.99	19.1	-	3.38	[10]
Oil palm frond (OPF)	1.59	19.7	-	3.95	[11]
Oil palm trunk (OPT)	1.32	35.3	-	4.50	[12]
Date palm leaves	1.18	12.9	8.7	2.10	[13]
Date palm Rachis	0.89	22.3	-	-	[6]
Coconut coir	0.84	20.09	13.59	4.41	[7]
Sugar palm frond (SPF)	1.19	149.3	126.7	22.56	[14]

## CONCLUSIONS

Sugar palm components are expected to provide acceptable characteristics for pulp and papermaking based on its chemical and physical properties after comparing with oil palm, date palm and coconut fibers.

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## INVITED SPEAKER 16

## EXTRACTION AND CHARACTERIZATION OF FIBERS FROM ARGYREIA SPECIOSA CREEPER PLANT

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## ABSTRACT

The growing population has fostered the research on utilization of resources for the value-added products, getting maximum benefit. The petroleum crisis and ever-increasing prices are the other factors contributing to this work. The current study focused to use the plant waste for the extraction of lignocellulosic fibers and their characterization. A creeper plant *Argyreia speciosa* (local name Tezgam) was selected for this purpose. It has a high growth rate and needs trimming on regular basis. The yard cutting waste is either burnt or landfilled. The fibers were extracted from this waste by water retting process. The stems were allowed to remain in water for two weeks. After the given time, the fibers were extracted from the decorticated stems of the plant. These fibers were washed and treated with alkali. The tensile properties, fiber linear density, moisture regain and thermal degradation behavior of these fibers were investigated. The fibers exhibited a mass loss of just 8% at a temperature of 300°C in the thermogravimetric analysis. The thermal and mechanical properties of the extracted fiber mark it a potential resource as reinforcement in composite material applications. These composite materials can be used to fabricate components for automotive interior, packaging, tabletop, etc.

*Keywords:* waste management, natural fibers, composite materials, tezgam

## INTRODUCTION

The growing population has fostered the research on utilization of resources for the value-added products, getting maximum benefit. The petroleum crisis and ever-increasing prices are the other factors contributing to this work [1]. Natural fiber-reinforced polypropylene composites have attained commercial attraction in automotive industries [2]. The current study focused to use the plant waste for the extraction of lignocellulosic fibers and their characterization. A creeper plant *Argyreia speciosa* (local name Tezgam) was selected for this purpose. It has a high growth rate and needs trimming on regular basis. The yard cutting waste is either burnt or landfilled [3]. The fibers were extracted from this waste by water retting process. The stems were allowed to remain in water for two weeks. It would not only be benefiting environmentally but it would be low cost as well. Such novel green composites can be used in automotive, interiors and other engineering materials as a substitute of traditional composites [4].

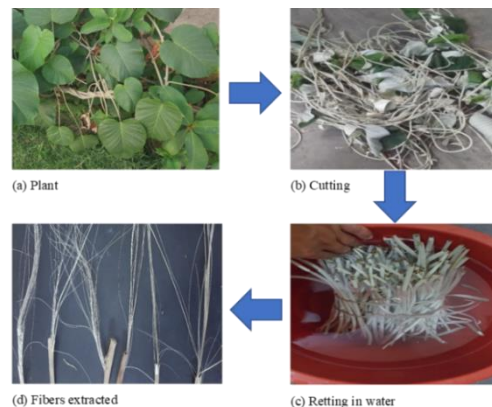


Fig. 1: Different stages of fiber extraction [5]

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## MATERIALS AND METHODS

*Materials*

The trimmings of *Argyreia Speciosa* creeper plant were used for the extraction of fibers.

*Methods*

The methodology adopted for the extraction of fibers from agrowaste is shown in Figure 1. The trimmings were subjected to water retting for a period of 2 weeks and fibers were extracted afterwards. The extracted

fibers were rinsed with water and dried. Later these fibers were treated with alkali solution to remove the waxes, oil and lignin from fiber, thus modifying its surface.

## RESULT AND DISCUSSION

The tensile properties, fiber linear density, moisture regain and thermal degradation behavior of these fibers were investigated, as summarized in Table 1.

TABLE 1. Properties of extracted fibers

Linear density	11.2 tex
Moisture regain	10.0 %
Fiber strength (Untreated)	4.61 cN/Tex
Fiber strength (Treated)	5.91 cN/Tex

The load-elongation curves obtained during tensile test are shown in Fig. 2 (a). It can be observed that there is some variation in the results, which is a characteristic of natural fiber. But the slope of all curves is nearly the same, showing consistency in the modulus. The thermogram obtained in the thermogravimetric analysis is shown in Fig. 2 (b). From thermogram, it can be observed that the fiber is stable upto a temperature of 300°C and shows a mass loss of 21% at a temperature of 350°C. Therefore, this fiber can be processed easily upto a temperature of 250°C, specifically as reinforcement for polymer matrix composites.

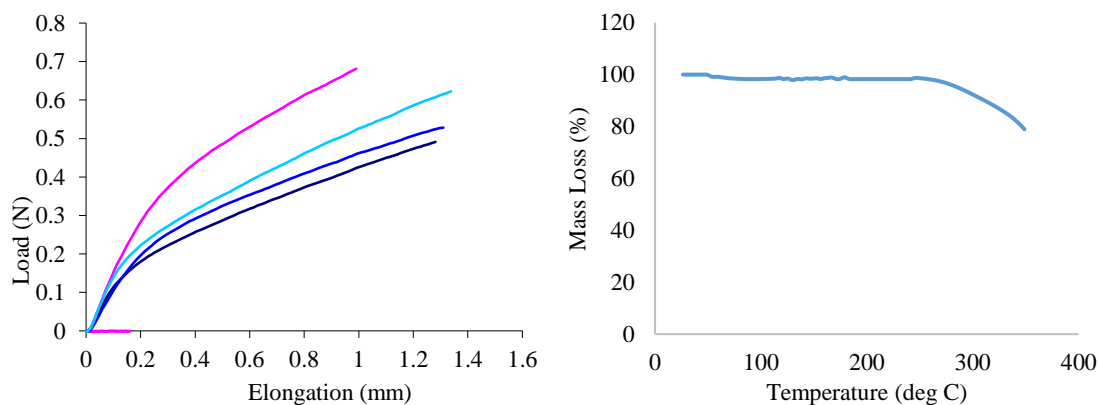


Fig. 2: (a) tensile test curve and (b) thermogram

The properties of extracted fibers and conventional natural fibers are given in Table 2. It can be observed that the extracted fibers have comparable properties with other natural fibers and can be used for composite applications.

TABLE 2. Comparison of properties of extracted fibers and conventional fibers [6]

	Cotton	Flax	Hemp	Jute	Sisal	Tezgam
Linear density [tex]	0.15–0.4	0.2–2.0	2.2–3.0	1.4–3.0	28.6–48.6	11.2
Moisture regain [%]	8.5	12	12	12	11	10
Tenacity [cN/tex]	17–38	40–80	47–80	23.9–27.6	55.5–58.5	4.61

## CONCLUSIONS

The extracted fibers are relatively coarser as compared to the conventional fibers. The moisture regain is comparable with these natural fibers. The breaking strength is also similar to the other natural fibers, while tenacity is lower due to the coarse fibers. The fiber is thermally stable upto 250 °C, and this temperature is higher than the melting temperature of commonly used thermoplastics (polythene, polypropylene, etc.) and glass transition temperature of thermoset matrices (unsaturated polyester, vinyl ester, epoxy, etc.). Therefore, these fibers have the potential to be used as a reinforcement material in polymer composites.

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## NATURAL FIBRE MATERIALS SELECTION USING TOPSIS METHOD FOR DEVELOPING VERTICAL AXIS WIND TURBINE BLADE FROM GREEN COMPOSITES

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### ABSTRACT

This paper discussed on the natural fibre materials selection using the Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS) method, towards developing new vertical axis wind turbine (VAWT) blade using green composites. Eleven (11) lignocellulosic natural fibre were involved as the candidate material for making the new NFC blades, based on four (4) selection criteria for the VAWT blade namely tensile strength and tensile modulus (performance criteria), fibre density (weight criteria) and fibre cost (cost criteria). TOPSIS method was used to analyse the performance score of each candidate materials which best suit the VAWT blade criteria and rank them based on the highest to lowest score. Results from the TOPSIS analysis obtained showed pineapple leaf fibre (PALF) scored the highest value (0.777), while the lowest score was obtained for coir fibre (0.050). The results highlighted that PALF is the best candidate natural fibre to be selected for producing the new green composites VAWT blade based on the design requirements.

*Keywords:* Natural fibre materials selection, Vertical Axis Wind Turbine, TOPSIS method

### INTRODUCTION

Vertical Axis Wind Turbine (VAWT) is among the two type of wind turbine used for renewable energy power generation application. VAWT design are divided into two types, which are Darrieus type and Savonius type. Darrieus type VAWT had two configurations, namely straight blades, and curved blades configurations as shown in Fig. 1 [1]. Improvement efforts are ongoing to further improve the VAWT such as in term of lightweight, structural strength, cost, environmental sustainability [2].

The motivation of the work is to perform materials substitution improvement of existing metal based VAWT blade to using more environmentally friendly green natural fibre composites (NFC). The main challenge to be solve by using TOPSIS method in this work is to perform an accurate decision-making process using scientifically proven method, which able to analyse multiple selection criteria and high order of candidate materials with varying attributes all at once.



Fig. 1: Example of VAWT with straight blades [3]

### METHODOLOGY

Technique for Order of Preference by Similarity to Ideal Solution (TOPSIS) method is a multi-criteria decision making method founded by Hwang and Yoon in 1981 [4]. TOPSIS method is based on the principle that the optimal point should have the shortest distance from the positive ideal solution (PIS) and the farthest from the negative ideal solution (NIS). Therefore, this method is suitable for cautious (risk avoider) decision maker(s) because the decision maker(s) might like to have a decision which not only makes as much profit as possible, but also avoids as much risk as possible [5]. Fig. 2 summarized the overall TOPSIS methodology applied for VAWT blade materials selection in this work.

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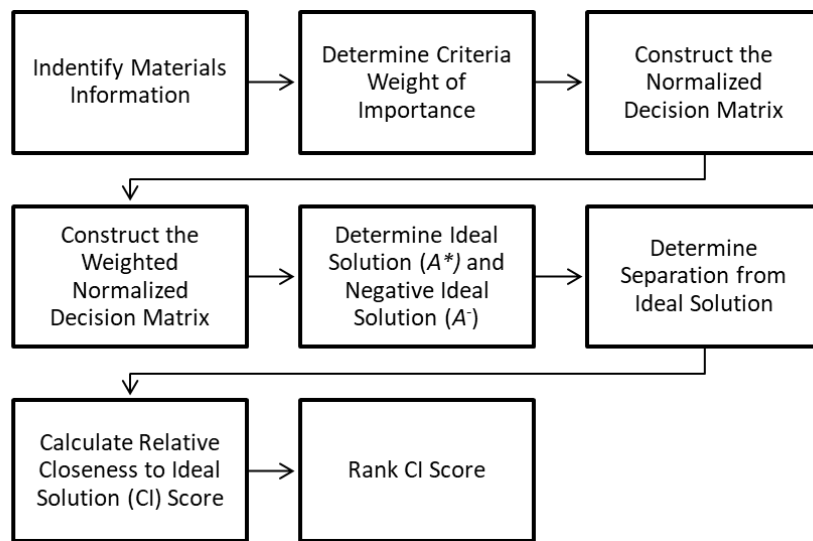


Fig. 2: TOPSIS methodology applied for VAWT blade materials selection.

Eleven (11) lignocellulosic natural fibre were screened and selected as the candidate material for making the new NFC composite blades which includes kenaf fibre, pineapple leaf fibre (PALF), hemp fibre, and coir fibre. The selected was based on literature review conducted. Furthermore, the material selection process is also based on four (4) selection criteria for the VAWT blade namely tensile strength and tensile modulus (performance criteria), fibre density (weight criteria) and fibre cost (cost criteria). Table 1 listed the related material properties and their attributes according to the selection criteria. All materials attributes were also sourced from literature review. Average value for all materials properties were used to run the TOPSIS analysis.

TABLE 1: Material properties of natural fibres candidates for VAWT composites blade [6].

Main Criteria		Weight	Performance			Cost
Sub-criteria		Density (g/cm <sup>3</sup> )	Tensile Strength (MPa)	Tensile Modulus (GPa)	Fibre Cost (USD/kg)	
Candidate materials	Abaca	1.5	430-813	31.1-33.6	0.345	
	Coir	1.20	175	6.0	0.20-0.40	
	Flax	1.38	343-1035	50-70	3.11	
	Hemp	1.35	580-1110	30-60	1.55	
	Jute	1.23	187-773	20-55	0.926	
	Kenaf	1.20	295-930	22-60	0.378	
	Pineapple Leaf	1.50	170-1627	60-82	0.40-0.55	
	Oil Palm	0.7-1.55	248.0	3.2-6.7	0.30	
	Ramie	1.44	400-938	61.4-128	2.00	
	Sisal	1.20	507-855	9-22	0.65	
	Sugar Palm	1.22-1.26	276.6	5.90	1.60-4.00	

## RESULT AND DISCUSSION

Fig. 3 shows the summary of TOPSIS score for each of the natural fibre candidates. Results from the TOPSIS analysis obtained showed pineapple leaf fibre (PALF) scored the highest value (0.777), followed by ramie fibre (0.579) and hemp fibre (0.526). In the other hand, the lowest score was obtained for oil palm fibre (0.050) and coir fibre (0.050). Based on the results, it is shown that PALF is the best type of natural fibre to be selected to make the new VAWT composites blade.

## CONCLUSIONS

Materials substitution effort to improve the existing VAWT blade by changing the existing metal-based component to using green NFC was conducted in this work using TOPSIS method. Final result obtained from the TOPSIS analysis highlighted that PALF is the best candidate natural fibre to be selected for producing the new green composites VAWT blade based on the design requirements, among the eleven (11) candidate materials identified. Similar TOPSIS method may also be extended to other related decision-making situations to further improved on VAWT technologies in other areas such geometrical design, and installation site selection.

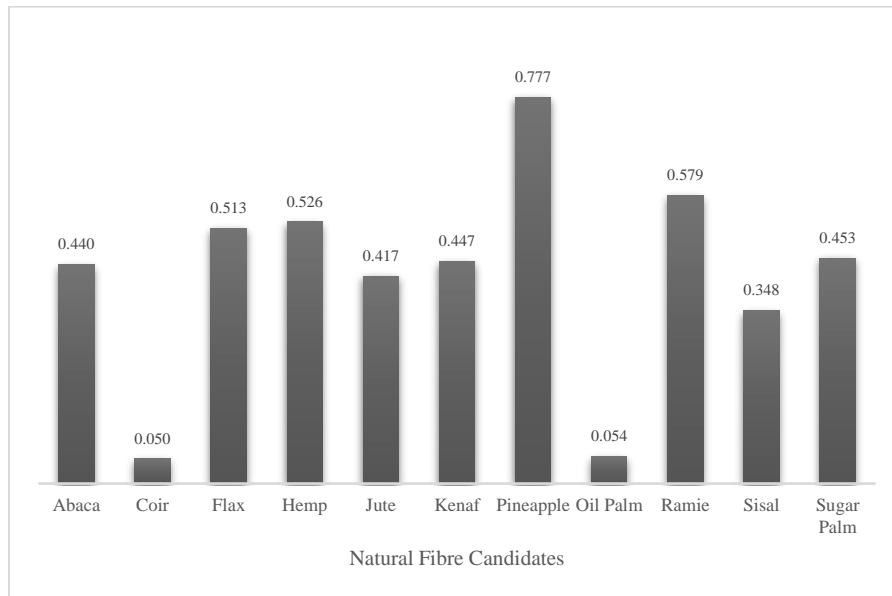


Fig. 3: Topsis score for each of natural fibre candidates.

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## RECENT DEVELOPMENT OF CARBONACEOUS MATERIALS CHAR AS NANOFILLERS IN FUEL BIOCOMPOSITE BRIQUETTES: A REVIEW

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### ABSTRACT

Biomass and plastic wastes which produced from various sources including agricultural, industrial and domestic activities had created a chronic situation towards environmental health. The recycling of these wastes by mean of transforming it into carbon-based product via thermal decomposition approach is one solution for the environmental pollution problem. The production of carbonaceous materials could be utilized into several applications. Plus, eliminate the contaminants. Some applications require carbonaceous materials with high mechanical strength, high porosity degree and better adsorption characteristics. Carbon-based products are the conformed to a procedure involving blending and pressing of char with adhesive materials in order to form char pellets or briquettes. In addition, the impact of adhesive materials on the mechanical and surface properties as well as combustion properties of biocomposite briquettes was studied and compared with different previous works. This review discusses the recent developments of char filled natural starch biocomposite briquettes derived from biomass and plastic waste.

*Keyword:* Biocomposite, carbonaceous char, natural starch, biomass waste, plastic waste.

### INTRODUCTION

Char, including biochar, is a carbonaceous material which is a unique and versatile element which is highly competent enough to form different architectures at the nanoscale regime. The char is a complex material and mainly consists of carbon, ash, and lower molar H/C ratios. Char and biochar were obtained by the thermal decomposition (pyrolysis) of plastic and biomass feedstocks [1]. Enormous interests have been generated to use the intrinsic properties of carbon chars for a plethora of applications such as in composites, automotive, aeronautics, electronics, sensors, and other engineering sectors.

Massive global production of plastic waste and underutilization of agricultural waste have sparked interest from researchers and industrial practitioners. Annual worldwide plastic demand has risen from 1.5 million metric tons in 1950, the beginning of plastic industry, to 3 billion metric tons in 2018. The globe produces 381 million tons of plastic waste per year, which is anticipated to quadruple by 2034 [2]. As stated by Tripathi et al. [3], agricultural and forestry activities generate a lot of waste as a result of harvestable output. The global annual production of biomass waste exceeds 140 Gt, posing considerable management challenges since disposed biomass might have detrimental environmental consequences. Thus, conversion of waste into char briquettes for fuel application is one of the best possible alternative to inhibit the underutilization of the waste, as illustrated in Fig. 1.

From previous years, researchers have concentrated on the study of carbon nanofillers reinforced composites for engineering applications. As far as the author's knowledge is concerned the use of char for fuel briquettes remains a less discovered area. However, several researchers have discovered this briquette manufacture to utilize various source of biomass waste (i.e

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mesocarp fibre [4], *bakau* wood [4], faecal [5], sawdust [6], and agricultural bagasse [7], [8] with different type of natural binders (i.e cassava starch [7], corn starch [8], sugar palm starch [9] etc.), which mainly useful in fuel sectors. Also, the available literature focused on the fuel applications of biochar briquettes with less research on plastic derived char within this spectrum, as shown in Table 1. Thus, the present work is aimed at studying the distinct characteristics possesses by the char nanofillers reinforced composites which is the best possible alternative for the development of fuel energy.

### CARBONACEOUS MATERIALS – CHAR

Carbon-based nanomaterials have drawn the attention of a large section of the scientific community in recent years for high-strength and multifunctional composite materials [9]–[12]. Carbonaceous materials are comprised into two

classes; (i) traditional carbon materials such as char/biochar, carbon blacks (CB), and activated carbons (AC), and (ii) nanostructured carbons, including graphene, graphite, fullerenes and carbon nanotubes (CNT). Char, charcoal, and activated carbons are three carbon compounds with a lot of similarities in structure and processing mechanics [13]. Char quality and properties are determined by pyrolysis parameters including heating duration, maximum temperature, pressure, and oxygen content, which can vary with different feedstock [14]. Ward et al. [5] mentioned that the characteristics and attributes of the char are mainly depend in high extent of the composition of the waste to be pyrolysed. Thus, it will affect its surface area and porosity (see Fig. 2). Plus, different metal ions embedded in the char particles, which make it can acts as catalysts for condensation, cracking volatiles and polymerisation reactions [15].

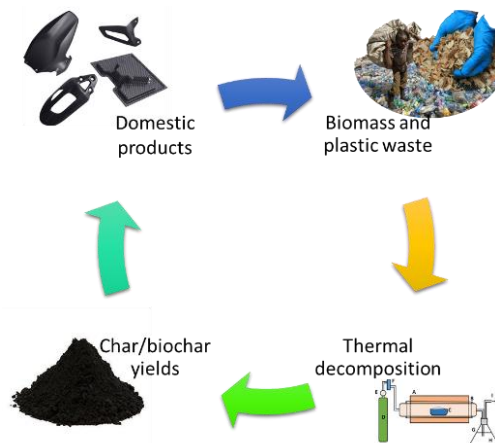


Fig. 1. Conversion of waste into char-based products

### NATURAL STARCH AS MATRIX AND BINDER

Starch is a polysaccharide composed of anhydroglucose units linked by  $\alpha$ -d-(1, 4) glucosidic bonds. Natural starch can be extracted from higher plants' leaves, stems, tubers, seeds, and roots, where it acts as an energy store [16]. In fuel applications, natural starch is widely used as binder, diluent, disintegrant and thickening agent. Table 1 below shows various of natural starch in char briquettes.

### RECENT ADVANCEMENT OF FUEL BRIQUETTES

One of the key avenues of waste conversion and application technologies is briquette fuel technology. After the solid waste was pyrolysed into char, it was moulded into solid briquettes, studies indicated that the combustion characteristics rose by 20% [17]. Furthermore, greenhouse gas, NO<sub>x</sub>, and SO<sub>2</sub> emissions were approximately one-fifth of char emissions. Briquettes made from char offer the advantages of abundant supply of raw ingredients, facile technology, ease of use, and low cost. They are suitable for large-scale use due to their high thermal efficiency, outstanding combustion qualities, convenient delivery features, and simplicity of industrialization of biomass briquette manufacture. Besides that, the mechanical and thermal properties of the briquettes are also significant. Table 1 summarise various char briquettes discovered by various researchers with optimum compressive strength and heating values in energy application. In the sectors of rural cooking, heating, providing clean fuels for cities, supplying energy for greenhouses, and providing fuels for industrial boilers and power plants, biomass briquettes are also appropriate alternatives for coal.

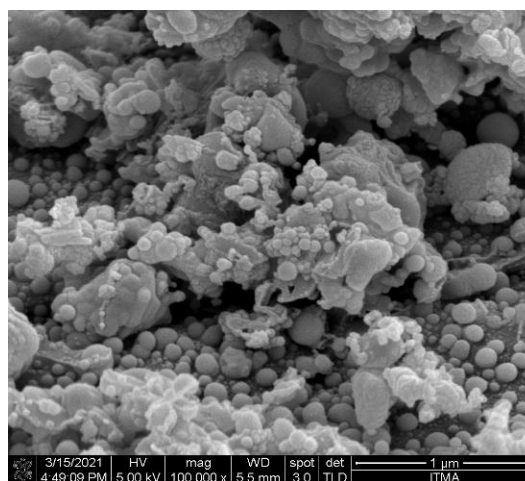


Fig. 2. FESEM images of char particles

TABLE 1: Comparison with other products on their compressive strength and HHV for charcoal applications

Waste Char	Binder (matrix)	Compressive strength (MPa)	HHV (MJ/kg)	Ref.
Mesocarp fibre	Sago starch	4.20	25.41	[4]
Mesocarp fibre	Bio-oil	2.19	29.12	
Sub-bituminous coal	-	-	24.60	
Bakau wood	-	-	25.01	
Faecal (human waste)	Molasses/lime	IRI - 100	23.4	[5]
Faecal (human waste)	Corn starch/wheat starch	IRI - 70	25.1	
Sugarcane bagasse	Cassava starch	5.4	26.67	[16]
Cassava rhizomes	Cassava starch	7.9	26.84	
Water hyacinth	Cassava starch	4.0	16.76	
Durian peel and mushroom compost	Cassava starch	-	22.2	[18]
Municipal waste composting and sawdust	Slop waste	2	22.4	[6]
Sugarcane bagasse	Cassava starch	-	28.32	[7]
Orange bagasse	Corn starch	1.4	26.47	[8]
Waste plastic and coal	Limestone dust, cassava flour and laterite	4	19.27	[19]
PPE isolation gowns (PP)	Sugar palm starch	1.34	17.61	[9]
Rice straw and rice husk ash	Rice husk	3.56	16.92	[20]
Rice husks, coffee husks and	Cassava starch	DS = 86%	16.6	[21]

groundnut shells				
Standard sub-bituminous coal	-	-	16	[22], [23]
Paper and saw dust	Carbonized rice husk	-	13.69	[24]
Waste oil	-	-	14.65	[25]
Sludge	Cassava tubers	-	7.68	[26]

\*IRI = impact resistance index; DS = drop strength; HHV = high heating values

## CONCLUSION

In this review paper, current developments of char/biochar composite briquettes in fuel applications, has been discussed. Char filled natural starch biocomposites have huge potential to be developed to convert waste materials into useful products. As a result of the preceding discussion, it is clear that low-cost, environmentally friendly, green, and simple processing of char briquettes must be considered in order to address current environmental issues such as abundant plastic and agricultural waste and to ensure a sustainable future environment.

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## PRELIMINARY STUDY ON LIGHTWEIGHT AND SUSTAINABLE AIRCRAFT SEAT ARMREST DESIGN

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### ABSTRACT

The demand for lightweight and green design in aerospace has never stopped. In fact, it is always increasing. Today, many aircraft manufacturers and researchers continue to develop lightweight aircraft, inside and out along with the green aviation concept. It is a challenge to obtain both at the same time as it will affect many aspects of aircraft performance, from the scratch up until the disposal. This preliminary study covers review on aerospace interior part design requirement and later, proposed a new Product Design Specifications (PDS) for lightweight and sustainable aircraft seat armrest design. The proposed PDS shall later serve as the primary documented guideline towards the development of new lightweight and sustainable aircraft seat armrest design using biocomposites material. A framework for the conceptual design of the new aircraft seat arm is also discussed in this paper.

*Keywords:* Aircraft seat armrest, conceptual design, product design specifications, lightweight and sustainable design

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### INTRODUCTION

In aerospace industry, the lightweight design act as the most effective method to reduce the fuel consumption and increase the energy efficiency since less mass requires less force during flight. Therefore, to have a weight reduction, there must have improvements to be made. Aircraft seats are among of the important parts in aircraft interior. The introduction to lightweight materials for aircraft seats has resulted airlines in upgrading existing aircraft seats with new ones [1]. One of the main components in the aircraft seat is the armrest. Currently, many of the armrest produced is made up from carbon composite which is classified as synthetic composite. Using the thermoplastic, Reliant Worldwide Plastic has replaced the internal aluminum substructure of the armrest which resulted in more fuel efficiency with greater load bearings compared to the traditional armrest. Nonetheless, the market is still in constant need of reducing aircraft weight without jeopardizing the passengers' safety and comfort. As stated by the Grand View Research (2017), the demand of the lightweight plastic as the alternatives for the metals is expected to reach USD 1.04 billion by 2025 [2]. Carbon Fiber Reinforced Polymer is the common composite material used in the aircraft manufacturing since it has very good strength-to-weight ratio and less sensitivity to fatigue and corrosion [3]. Though the materials contribute many advantages to the modern technologies, according to the National Composites Center, 85% of them are currently not being reused or recycled at the end of their life due to the difficulty to separate the fiber from resin which results in accumulation of plastic waste. Besides, about 95% of them are from raw materials which derived from oil hence causing them unsustainable [4]. Towards green engineering, improvements have to be made to shift the use of non-biodegradable composite to the biodegradable ones. Furthermore, the biodegradability, sustainability, renewability and nontoxicity of natural fibers add more values for the materials to be utilized in the future industry of automotive and aircraft [5]. Changing in material might affects the structural performance. Thus, to achieve the structural performance as good as synthetic composite armrest is by doing the modification on the structural design. On the other hand, the use of biocomposites in aircraft industry is still under rapid investigation due to many challenges in their mechanical, physical and morphological properties and the fabrication method. Therefore, in this paper, an effort to produce new conceptual designs of biocomposite armrest is conducted. This preliminary study covers review on aerospace interior part design requirement and later, proposed a new Product Design Specifications (PDS) for lightweight and sustainable aircraft seat armrest design. The proposed PDS shall later serve as the primary documented guideline towards the development of new lightweight and sustainable aircraft seat armrest design using biocomposites material. A framework for the conceptual design of the new aircraft seat arm is also discussed in this paper.

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### AIRCRAFT SEAT ARMREST CONCEPTUAL DESIGN FRAMEWORK

In this study, the concept of Total design is used. The market demands for lightweight material and green technology to be utilized in order to create more benefits for the product and user. Hence, developing the Product Design Specification (PDS) is prioritized first then followed by the development of conceptual designs using Biomimetic method. The best conceptual design developed is later selected via Analytic Hierarchy Process integrates with TOPSIS (AHP-TOPSIS). As the material required for biocomposites, a natural fiber reinforced polymer material was chosen based on the literature review conducted. Once the

conceptual design and materials selection are completed, evaluation process to testify the structural performance of the new biocomposites aircraft seat armrest is conducted using CAE analysis.

### PRODUCT DESIGN SPECIFICATIONS FOR NEW LIGHTWEIGHT AND SUSTAINABLE AIRCRAFT SEAT ARMREST

Aircraft performance can be improved through advances in aircraft aerodynamics, materials, structures and other parameters including specific fuel consumption [6]. In this study, the features of materials and structures will be the main ones since these are the important aspects to be improved. PDS is used as the guidelines for the whole project. Since that safety is always the main issue in commercial aircraft industry, every design has to meet the standard enforced by the Federal Aviation Authority (FAA). The regulations for that can be referred from Title 14: Aeronautics and Space under Chapter I, Subchapter C, under Part 25. The elements of PDS are elaborated as in Table 1.

TABLE 1: Product design specifications (PDS) of aircraft seat armrest

PDS Element	PDS Details	Reference
Performance	<ul style="list-style-type: none"> <li>Each seat and its supporting structure must met both 9g static and 16g dynamic standard.</li> <li>Provide occupant comfort with the added functionality of housing passenger controls, such as audiovisual and recline controls, among others.</li> <li>It can be moved between a deployed upper position and a retracted position, in particular to facilitate movements by the passenger.</li> </ul>	[6-8]
Size & Weight	<ul style="list-style-type: none"> <li>Minimum height: 16 cm-23 cm</li> <li>Maximum height: 20 cm-25 cm</li> <li>Minimum width: 4 cm</li> <li>Maximum width: 7.62 cm</li> <li>Minimum length: 16 cm-23 cm</li> <li>Maximum length: 27 cm-39 cm</li> <li>Distance between armrest: 39 cm-54 cm</li> <li>Whole seat weight: 8kg</li> </ul>	[9-10]
Life in Service	<ul style="list-style-type: none"> <li>On average seven to ten years</li> </ul>	[11]
Maintenance	<ul style="list-style-type: none"> <li>New materials or structures will be observed frequently</li> </ul>	[12]
Ergonomic	<ul style="list-style-type: none"> <li>The one nearest to the gangway must be capable of moving out of the way to permit clear access to the seat</li> </ul>	[13]
Environment	<ul style="list-style-type: none"> <li>Less carbon emission</li> </ul>	[14]
Material	<ul style="list-style-type: none"> <li>Use of environmental-friendly material</li> <li>Fire Protective Materials: Most materials used in interior compartments which are under Title 14 Code of Federal Regulations (CFR) Part 25 must be self-extinguishing (stop burning after the flame source is removed) as required by FAA</li> </ul>	[15] [16]
Safety	<ul style="list-style-type: none"> <li>Smooth surface</li> <li>Hinge lock to keep the armrest in down position whenever takeoff or landing</li> </ul>	[6]
Process	<ul style="list-style-type: none"> <li>Easy to fabricate for biocomposites</li> </ul>	[17]

### CONCLUSIONS

This paper covers review on aerospace interior part design requirement and later, proposed a new Product Design Specifications (PDS) for lightweight and sustainable aircraft seat armrest design. The proposed PDS served as the primary documented guideline towards the development of new lightweight and sustainable aircraft seat armrest design using biocomposites material. A framework for the conceptual design of the new aircraft seat arm is also proposed for this project.

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**BIOFILM FROM NATURAL FIBER FOR BIOSENSOR: A REVIEW**

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*Institute of Microengineering and Nanoelectronics, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia.***ABSTRACT**

The use of filler or fiber reinforcement from natural fiber for biosensor contributes to reducing the environmental impact of new nanotechnology. In this context, the use of natural fiber is considering sustainable reinforcement for developing countries. Natural fiber substitute in biofilm is biodegradable, renewable, non-toxic and have unique properties. Nevertheless, the limitation properties of natural fiber tend to absorb moisture thus leading the poor mechanical properties and reducing the potential of biofilm performance. Thus, the surface functionalization and modification steps for natural fiber to improve the properties of biofilm. This paper presents a short review of the physical, mechanical, thermal, and electrical properties natural fiber-based biofilms. This review finalizes with concluding remarks and current research trend on film mainly for biosensor applications.

*Keywords:* Biofilm; natural fiber; biosensor; review.

**INTRODUCTION**

Biofilms have emerged as suitable candidates for a variety of applications due to the rapid spread of the internet, as well as the pressing demands in human health monitoring, human-machine interactions, and energy storage, during the previous few decades. Their prospective uses in the preparation of flexible electronic devices like as strain sensors and flexible all-solid-state supercapacitors are based on their inherent high toughness, adjustable mechanical characteristics, ionic conductivity, and biocompatibility. Furthermore, the electronic waste generated by non-biodegradable and nonrecyclable electronic components in flexible electronic devices contributes to the burden on the environment and necessitates the employment of many people to deal with the buildup of electronic trash.

Transient electronics, on the other hand, may be disposed of quickly and easily, sometimes even without causing environmental harm. Numerous researchers have been inspired by the use of biodegradable and biopolymers as substrates for transient electronics to create biodegradable and water-soluble polymers for the creation of multifunctional biofilms to meet the needs of sustainable development. Natural polymer materials often found in nature include cellulose, chitin, alginic acid, starch and other natural polymer compounds that are derived from renewable resources. Starch is commonly used in the creation of environmentally friendly hydrogels because of its benefits in terms of biodegradability, cost effectiveness, availability, and renewability. Nevertheless, starch-based hydrogels are usually fragile due to their rigid macromolecular chains and abundant hydrogen bonds. There have been several attempts to increase the flexibility of starch-based materials, including the construction of composite networks with fillers, the formation of starch-based nanospheres, and the modification of starch-based materials using small molecule plasticizers. Another significant and practical method is to combine starch with other polymers to generate a complex that improves the mechanical characteristics of the starch. A technique for the manufacture of high-strength and recyclable hydrogels is to use natural and synthetic polymers as a matrix material to synergistically alter the network structure of starch. Nowadays, there is a great concern about the decrease in the use of sustainable and environmentally friendly organic compounds. Therefore, researchers have used polymeric films, such as biological origin, for example, biofilm based on cassava starch [1]–[3], sugar palm starch [4]–[6], potato starch [7]–[9], corn starch [10]. They possess a good mechanical properties with the natural fiber reinforcement such as kenaf, sugar palm [10], bamboo [10].

**THERMOPLASTIC STARCH FOR SENSING APPLICATIONS**

A study done by Yadav et al. [11] investigated the PVC film in between potato starch film and silver as the sensing element for humidity monitoring in the environment. The surface plasmon resonances were created using PVC layer fabricated between silver and starch biofilm on a rectangular prism. The proposed sensor has maximum sensitivity 0.45 nm/%RH in wavelength interrogation method and 0.09°/%RH in angular interrogation method. Here the variations in humidity of environment, changes the refractive index of starch biofilm and hence in the surface plasmon resonances. Also, the proposed sensor shows a linear variation in resonance wavelength and resonance angle with respect to the variation in humidity of environment.

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Wang et al. [12] used starch bio film based triboelectric generator which can work stably in high humidity environment with high output performance by forming hydrogen bond with water molecules. In a high humidity environment, the hydroxyl-rich biomaterials, such as starch molecules, and water molecules could form hydrogen bonds spontaneously to fix water molecules on the surface, allowing water molecules to participate in triboelectric charging as a more electropositive material to obtain a higher output of TENG. Unlike the traditional polymer-based TENGs, the output of the starch films-based

TENG increases with the increase of environmental humidity. The output current and voltage could increase from 6.2  $\mu\text{A}$  to 110 V to 16.6  $\mu\text{A}$  and 330 V with the ambient humidity increasing from 15% to 95%, respectively, which is about 12 times larger than the traditional nylon-11 based TENG in the humidity of 95%. It is an essential supplement of TENG family and highly expanded application scopes for energy harvesting and self-powered sensors in high humidity environments, especially in cloudy, foggy days or under water and sweat conditions.

Camargo et al. [13] combined potato starch and carbon nano diamonds and be deposited on glassy carbon electrode in the form of a homogeneous, rough film, with electroanalytical performance tuned by varying the relative potato starch/nanodiamonds concentration. The potato starch/nano diamonds film was used as a matrix to immobilise the enzyme tyrosinase (Tyr), and the resulting biosensor was found to be capable of detecting catechol using differential pulse voltammetry, with a detection limit of  $3.9 \times 10^7 \text{ mol L}^{-1}$  in the range between  $5.0 \times 10^6$  and  $7.4 \times 10^4\%$  of the mol L1 concentration. The potato starch/nano diamonds film was used as a matrix to immobilise the enzyme tyrosinase (Tyr), and the resulting biosensor was found to be capable of detecting catechol using differential pulse voltammetry, with a detection limit of  $3.9 \times 10^7 \text{ mol L}^{-1}$  in the range between  $5.0 \times 10^6$  and  $7.4 \times 10^4\%$  of the mol L1 concentration. Water samples from rivers and wells were also found to contain catechol. Due to the huge surface area and conductivity provided by the tiny nano diamonds (5 nm), the sensor has a high sensitivity that is comparable to biosensors created with more complex processes and materials in the literature. Furthermore, the utilisation of potato starch/nanodiamonds may be expanded to immobilise additional enzymes and biomolecules, hence suggesting a viable biocompatible platform for ubiquitous biosensing applications. Zambianco et al. [14] developed a novel sensor architecture consisting a manioc starch and nanodiamonds particles. It is owing to the remarkable electrical conductivity capabilities of carbon nanoparticles that they are used in this biofilm. The starch extracted from manioc was investigated. The modified electrode displayed linear response when exposed to the herbicide diquat (DQ) at concentrations ranging from 5.0 to 4.6 105 percent by volume in this research, with a detection limit of 1.1 107 percent by volume. This new sensor has been shown to be effective in identifying whether or not DQ is present in rivers and water samples for consumption. Sarkar et al. [15] investigated thermoplastic starch (TPS) as a triboelectric positive material for designing TENGs. According to their findings, the bioplastic-based highly durable TENG (b-TENG) can generate an open-circuit peak-to-peak output voltage of 560 V with an output current density of 120 mA  $\text{m}^{-2}$  and an instantaneous output power density of 17 W  $\text{m}^{-2}$  with an output current density of 120 mA  $\text{m}^{-2}$  and an instantaneous output power density of 17 W  $\text{m}^{-2}$ . We demonstrate b-TENG as a portable power source by powering more than 100 commercial blue LEDs, LED strips, and seven segment LCD screens. Additional functions include being a self-powered pedometer for step counting, as well as a human walking and running speedometer and as a human gait analysis sensor for assessing physical activity, all of which pave the way for future biological applications in healthcare. As a triboelectric positive component, the introduction of environmentally friendly bioplastic TPS as a triboelectric positive component has significant promise for biomedical applications due of its abundance, biodegradability, cheap cost, and simplicity of production.

## CONCLUSION

The advancements in conducting biofilm have a significant influence on the sensing characteristics of the biofilm. Nano-structured conducting polymers are being used to fabricate biofilms for sensor applications, and significant progress is being made in this area on a consistent basis. In this brief study, we briefly discussed the utilization of biofilms, which may be used to create a variety of various synthetic approaches using nano-structured conducting polymers and biofilms. The research also illustrates the importance of biofilm in the rapidly developing area of sensor technology. In order to meet the increasing demands and complexity of the biofilm building using biodegradable and renewable materials, additional synthetic techniques for the production of conducting micro particles will be needed. These technologies will make advantage of new nanotechnological approaches to conducting polymers as well as their applications in sensing systems. Increasing interest in and practical use of nanotechnology, especially, in conducting polymers and polymer composites have led the researchers to the rapid development of nanosensors/biosensors with improved processability and functionality over previously developed biosensors.

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## EVALUATING THE DEVELOPMENT IN SUGAR PALM FIBER (ARENGA PINNATA (WURMB.) MERR) POLYMER HYBRID COMPOSITES: A COMPREHENSIVE REVIEW

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### ABSTRACT

The rapid depletion of petroleum products, together with growing awareness of worldwide environmental concerns associated with the use of petroleum based plastics, are the primary driving reasons behind the broad adoption of natural fibres and biopolymers. Sugar palm fibre (*Arenga pinnata (Wurmb) Merr*) (SPF) is one of the most abundant and renewable fibres in Malaysia. The aim of this article is to examine the development of SPF polymer hybrid composites. SPF are mostly made of cellulose (43.88%), which results in their excellent mechanical properties. According to the literature review, no thorough review paper on SPF polymer hybrid composites has been published. The current study focus on the characteristics of SPFs and polymer matrices, as well as their fabrication. The study also reveals the potential of SPF polymer hybrid composites for industrial applications such as automotive, household items, packaging, bioenergy, and others.

**Keywords:** Sugar palm fibre; Poly (lactic acid); Hybrid composite, Fabrication, Natural fibres.

### INTRODUCTION

As a result of pollution produced by non-biodegradable materials such as synthetic fibers, research on environmentally friendly material development has started. To solve such an issue, researchers moved from synthetic fibers (non-biodegradable material) to natural fibers (biodegradable material). The major benefits of adopting natural fibers are their renewability, ease of availability, biodegradability, non-toxicity, low specific gravity, high toughness, and better strength. [1]. Natural fibers have a low density and a good strength to weight ratio, making them suitable for use as light weight composite and reinforcing materials. The mechanical characteristics of fibers are influenced by their microstructure and chemical composition, with the fiber cross-sectional area being the greatest variable determining fiber strength [2]. Natural fibers absorb water easily attributed to the existence of hemicellulose, which gives them hydrophilic characteristics, making them less suitable in interactions with hydrophobic matrix [3]. Higher cellulose concentration and crystallinity likely to result in higher fiber strength characteristics, whereas lignin is the reverse [4]. Aside from that, fiber anatomical features change across and within species, influencing density and mechanical characteristics [5]. Other elements that influence the size and quality of natural fibers include environmental circumstances, mode of transportation, storage duration and conditions, and fiber extraction [4,6].

Sugar palm fiber (SPF) is black in color, length up to 1.19 m and measures 0.5 mm in average diameter, heat absorb by SPF up to 150°C [9]. SPF has a hollow (lumen) space and random nodes that separate the fiber into different cells. As the lumen size reduces and the thickness of the secondary cell wall improves, due to which the strength and young modulus also increases [16]. The raw surface of sugar palm fiber makes a strong matrix adhesion in the composite structure. Cellulose, hemicellulose, and lignin are the principal constituents of sugar palm fiber derived from sugar palm trees. local name of the sugar palm tree. It is a famous multifunctional tree that is primarily found in tropical areas. It is a member of the Palmae family, which includes 181 genera and around 2600 species [7]. Sugar palm trees may be found in abundance along rivers and bushes in the rural regions of Bruas-Parit, Perak; Raub, Pahang; Jasin, Melaka; Kuala Pilah, and Negeri Sembilan (Malaysia). [8].

### SUGAR PALM FIBRE (SPF) / POLYMER COMPOSITES

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Table 1 shows the different compounding processes and properties of SPF/ Polymer Composites.

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TABLE 1 Compounding Process, Properties of SPF/ Polymer Composites

	SPF/ Polymer composite	Compounding Process	Properties/ Outcomes	Authors/ Year/ References
1.	SPF (S)/ Ramie (R) fiber/ epoxy composites	Hand lay-up, /compression moulding machine	Among the five-layer SPF and SRSRS hybrid composites, RRSRS hybrid composite has higher <i>tensile</i> (52.66 MPa) and <i>flexural</i> (80.70 MPa) strength	Siregar et al. [9]
2.	SPF/PLA composites	Brabender Plastograph/ compression moulding machine	The flexural and tensile strengths of a composite with 30% SPF loading are optimal at 26.65 MPa and 13.70 MPa, respectively.	Sherwani et al. [10]
3.	SPF/GF/thermoplastic polyurethane (TPU) hybrid composites	Melt-mixing compounding/ hot pressing moulding	When compared to untreated SPF, the combination of silane and alkaline treatment produced considerably better results in terms of <i>density</i> , <i>thickness swelling</i> , <i>water absorption</i> , and thermal stability.	Atiqah et al. [11]
4.	SP yarn/GF/unsaturated polyester hybrid composites	Yarning process/ Hot press machine	Dynamic mechanical analyzer (DMA) and Thermogravimetric analysis (TGA). The <i>storage modulus</i> ( $E'$ ), <i>loss modulus</i> ( $E''$ ) and <i>damping factor</i> ( $\tan \delta$ ) were improved after after alkaline treatment of SP yarn and hybridization with GF.	Mohd Nurazzi et al. [12]
5.	SPF/GF/epoxy hybrid composites	Hand lay-up technique	The SPF is benzoylated. The SPF/GF/epoxy hybrid composites' <i>flexural and compressive</i> characteristics were significantly enhanced.	Safri et al. [13]
6.	SPF/GF/TPU hybrid composites	Melt compounding technique/ Compression molding process	Due to the superior hybrid performance of the two fibers, the <i>tensile and impact</i> characteristics of the hybrid composites were enhanced with increasing SPF content (30% /10% SPF/GF) as compared to GF reinforced composites (0% /40% SPF/GF). When a greater amount of GF was added at 40 wt. % , the <i>flexural</i> characteristics improved.	Afzaluddin et al. [14]
7.	Cornhusk/SPF /Cornstarch hybrid composites	Conventional solution casting technique	The polymer matrix and reinforcement fiber had a strong interfacial contact and high biocompatibility, which resulted in increased <i>tensile strength</i> and <i>Young's modulus</i> . In general, the hybridization of CS/CH composites with SP fiber has contributed to the improvement of biocomposites for biomaterials applications, particularly for 6 percent SP fiber loading.	Ibrahim et al. [15]
8.	SPF/ TPU composites	Extruder/ hot press machines	The best tensile strength was 18.42 MPa with a microwave temperature of 70°C and a pre-treatment of 6% NaOH treatment	Mohammed et al. [16]
9.	Roselle RF /SPF/ TPU hybrid composites	Brabender Plastograph/ Hot Press moulding	25%RF/75%SPF Hybrid composites had the lowest <i>water absorption</i> (7.35%) and <i>thickness swelling</i> (7.15%) values. These values increases as the %content of SPF increased.	Radzi et al. [17]
10.	SPF/phenolic (PF) composites	Disk-shaped films/ Alpha Analyzer/ Temperature controller	Dielectric relaxation spectroscopy to examine the effect of treatment on SPF composite. Interfacial bonding is stronger in alkaline treated composites than in untreated and sea water treated composites.	Agrebi et al. [18]
11.	Cassava/SPF/ cassava starch hybrid composites	Casting technique	The addition of SPF caused changes in the film characteristics of cassava starch, potentially compromising the film's performance.	Edhirej et al. [19]
12.	Seaweed SW/SPF/ thermoplastic SP Starch/ Agar hybrid composites	Brabender Plastograph/ Hydraulic thermo-press	The study demonstrated that hybrid composites have enhanced tensile and flexural characteristics while having a reduced impact resistance. SW/ SPF (50/50) hybrid composite had the highest tensile strength (17.74 MPa) and flexural strength (31.24 MPa). <i>Water absorption</i> , <i>thickness swelling</i> , and soil burial tests revealed that the hybrid composites were more resistant to water.	Jumaidin et al. [20]
13.	SPF/ High Impact	Melt compounding technique/	The findings showed that increasing the short SPF loading in the HIPS matrix enhanced the composites' tensile	Sapuan et al. [21]

	Polystyrene HIPS Composites	Compression molding process	strength and modulus.	
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## CONCLUSIONS

The review concludes that sugar palm fiber polymers hybrid composites have greatly expanded their reach in industrial applications. It exhibits good mechanical characteristics after alkaline treatment of SPF, hybridization with glass fiber or any other fibre, and incorporation with various polymer matrices. In summary, the use of SPF polymer hybrid composites can aid in the future development of sugar palm as a new industrial crop, the reduction of reliance on petroleum products, and the reduction of the negative environmental effect of synthetic polymers and fibers.

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## EFFECT OF SLIDING CONDITIONS ON THE WEAR BEHAVIOUR OF CARBON/KENAF HYBRID COMPOSITES

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### ABSTRACT

The potential of hybrid composites from the combination of Kenaf (K) and Carbon (C) fiber as a part of the automotive component has been widely investigated by previous researchers. The attractive attributions such as a comparable mechanical property, low density, and improve biodegradability properties become the main reason for the development carbon/kenaf hybrid composites. Many findings reported on the mechanical behaviors of this hybrid composites based on previous studies. However, the research that discussing on the tribological properties of carbon/kenaf hybrid composite still inadequate. Therefore, the current research focused on the development of carbon/kenaf reinforced epoxy matrix hybrid composite that fabricated using vacuum infusion technique. The ratio of fiber-to-matrix was fixed at 40:60 whereby two different stacking sequences were introduced namely CKCKC and KCKCK. Tribological test was performed using pin-on-disc tester under different applied loads of 10N, 20N, 30N, with different sliding speed of 200 rpm, 350 rpm and 500 rpm. Sliding conditions was fixed under dry conditions with abrasive surface of 400SC. Morphological structure of hybrid composite was also observed to determine the effect of wear towards morphology of hybrid composite. It was found that wear rate decreases with increase in sliding speed. Meanwhile, CKCKC stacking sequence is observed to be more wear resistant than CCKCC due to even distribution of kenaf debris as transfer film.

*Keywords:* Hybrid composites, Tribological properties, wear behavior, pin-on-disc

### INTRODUCTION

The automotive industry is a fast-growing industry with a global market size of USD 13 billion. Steel is a very crucial material in the manufacturing of cars and on average, 900 kg of steel is used per vehicle due to its high strength and excellent energy absorption [1]. In fact, high density of steel caused the reduction in the performance of the vehicle especially in terms of weight and fuel consumptions [2]. Therefore, many manufacturers searched for the lightweight materials to improve the vehicle performances. Carbon fibre reinforced polymer (CFRP) has been proposed as a promising candidate to replace the use of steel due its properties such as lightweight, durable, and has strength [3]. However, the high cost of carbon fiber as well as problem in the recyclability after the end-of-service of CFRP become the major issues that hinder the development of CFRP [4]. Natural fibre reinforced polymer (NFRP) has been introduced as an option to replace the usage of CFRP in automotive applications due to its numerous advantages including reduction of weight, excellent biodegradability, and renewable sources [5]. Despite all its advantages, it is still lacking in strength. This lack of strength makes natural fibres such as kenaf unable to replace conventional materials for the main component in the automotive industry.

The combination of synthetic and natural fibre to form hybrid composite is expected to cope the limitation of CFRP and NFRP composites due to its attractive advantages such as high mechanical properties, improve recyclability, and excellent strength-to-weight ratio [6]. Many researchers have reported on the development of carbon/kenaf hybrid composites from the previous study in which the mechanical behaviors of these hybrid composites have been thoroughly investigated [7]. For instance, Yusuff et al. (2020) has reported the tensile and flexural properties of carbon/kenaf reinforced epoxy hybrid composites where the result shows that mechanical properties of hybrid composites have improved as compared to kenaf reinforced epoxy as well as able to offer comparable mechanical strength as carbon composites [8]. However, the research that discussed regarding the tribological behavior of carbon/kenaf hybrid composite was limited. Thus, it is necessary to have continuous research that

investigate the tribological properties of this hybrid composite so that the potential of carbon/kenaf hybrid composites in the automotive application can be explored. Therefore, the current research is one of the alternatives taken to ensure the distribution of knowledge on the tribological properties of carbon/kenaf hybrid composite for the future research.

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### MATERIALS AND METHODS

#### Materials

Raw materials used in the fabrication of hybrid composite is fine woven kenaf fibre, carbon fibre and epoxy resin. While carbon fibre is supplied by Vestec Technology Services. Epoxy (proset) resin and H10 hardener is provided by Castmech Technologies Sdn Bhd.

*Fabrication and Characterization of Hybrid Composites*

Carbon/kenaf hybrid composites were fabricated using vacuum infusion method with thickness of 3 mm and fiber-to-matrix ratio of 40:60. Stacking sequence of kenaf and carbon fibre were stacked in two forms, namely, CKCKC and CCKCC. Finally, samples were cut with dimension 30mm x 9mm using T-jaw (D511-8) to be used for tribological testing. Tribological behaviour of hybrid composite was analysed using DUCOM pin-on-disc tester machine based on the ASTM G99-05. Tribological test was performed using different applied load (10N, 20N, and 30N) and sliding speed (200 rpm, 350 rpm, and 500 rpm). Sample was first attached on sample holder which is a stationary pin with desired applied load. The pin was then rotated on the rotating disc. Sliding condition and sliding time was fixed with dry sliding conditions and 10 minutes, respectively. Wear rate was calculated based on equation  $W_s = V / (F \times L)$ , where V (volume loss of specimen), F (applied load), and L (sliding distance). Scanning Electron Microscopy (SEM) JSM 5600 was used to analyze morphology of the hybrid composite before and after tribological testing where the specimens were coated with Palladium (Pd) where coating process was performed using Quorum SC7620 Sputter Coater at 10 Kv voltage.

**RESULT AND DISCUSSION**

Fig. 1 (a,b) shows the graph of wear rate versus sliding speed at different applied loads and stacking sequences (CKCKC, refer Fig. 1 (a) and CCKCC (refer Fig. 1 (b)) of carbon/kenaf hybrid composites. From the viewpoint of various sliding speed (200 rpm, 350 rpm, 500 rpm), it can be noted that as the value of sliding speed increased, it tends to reduce the wear rate. Meanwhile, at various loads (10 N, 20 N, 30 N), the value of wear rate increased when the amount of load increased from 10 N to 20 N. However, when the load was applied at 30 N the value of wear rate has decreased. e graph (Fig. 1 (a,b)), when applied load was increased to 30 N, the value of wear rate tends to be reduced. This result due to fiber debris especially kenaf fiber. It indicates, kenaf debris successfully acts as a transfer film between contact that slow down the propagation of wear debris and micro-cracks, thus resulted in the reduction of the wear rate. However, by comparing the range of wear rates for both samples, it is seen that hybrid composite with the CKCKC stacking sequence has range of the wear rate from  $1.27 \times 10^{-6} \text{ mm}^3/\text{Nmm}$  to  $5.0492 \times 10^{-6} \text{ mm}^3/\text{Nmm}$ . Meanwhile, CCKCC hybrid composite exhibits the wear rate value in range of  $1.78 \times 10^{-6} \text{ mm}^3/\text{Nmm}$  to  $7.27 \times 10^{-6} \text{ mm}^3/\text{Nmm}$ . Thus, it signifies that CKCKC has lower and smaller range of wear rate as compared to CCKCC hybrid composite. Therefore, CKCKC able to offer better resistance to wear as compared to CCKCC due the effectiveness role of kenaf debris as transfer film in the CKCKC hybrid composite.

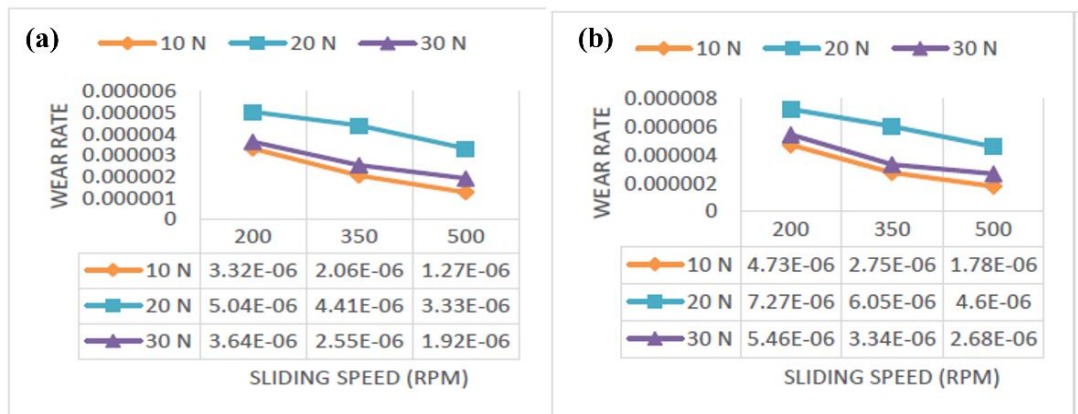


Fig. 1: Wear rate of (a) CKCKC and (b) CCKCC hybrid composites at different applied load and sliding speed

The surfaces of carbon-kenaf hybrid composite with stacking sequence CKCKC after pin-on-disc testing was observed using Scanning Electron Microscopy (SEM) as shown in Fig. 2 (a – c). Based on Fig. 2 (a), the appearance of fibre pull-out and matrix cracking can be perceived on the surface of hybrid composite. As applied load was increased to 20 N, the presence of severe matrix breakage and microcracks able to be seen on the surface of CKCKC as shown in Fig. 2 (b). However, as applied load increased to 30 N, the surface of CKCKC hybrid composite experienced less severe failure in which only matrix cracking can be detected as shown in Fig. 2 (c). The reduction in severity of wear from applied load 20 N to 30 N can be attributed to the abundance of kenaf debris at the surface which effectively acts as transfer film, hence, reducing the wear rate. The appearance of similar failure as CKCKC hybrid composite can be seen on the CCKCC hybrid composite. It is significant to note that, CCKCC hybrid composites showed severe failures as compared to CKCKC hybrid composites. This finding has validated the statement reported in the previous point in which CKCKC stacking configuration able to provide excellent resistance to wear than CCKCC stacking configuration. It should be noted that, the same failure mechanism, namely fiber pull-out, matrix cracking, and microcracks can be detected when the sliding speed has increased from 200 rpm to 350 rpm and 500 rpm.

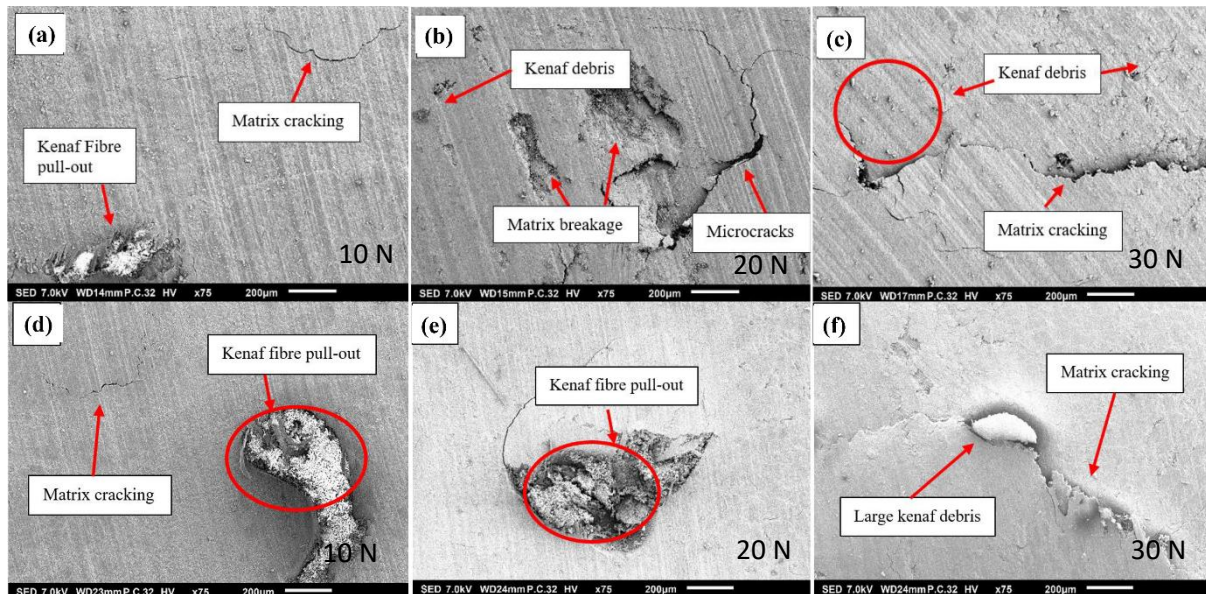


Fig. 2: Micrographs of fabricated CKCKC hybrid composites (a – c) and CCKCC hybrid composites (d – f) at various applied loads (10 N, 20 N, 30 N) at  $\times 75$  magnification

## CONCLUSIONS

The tribological properties of fabricated carbon/kenaf hybrid composites have been studied where the effect of two parameters namely sliding speed and loads towards wear rate were evaluated. Thus, it can be concluded that:

- Increase of sliding speed leads to decrease of wear rate of fabricated carbon/kenaf hybrid composites where it is applicable for both stacking sequences (CKCKC and CCKCC).
- Increase of applied load from 10 N to 20 N has increased the value of wear rate, whereas the value of wear rate reduced when the applied load was further increased to 30 N
- Lastly, CKCKC hybrid composite offered better resistance to wear as compared to CCKCC hybrid composite.

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## EFFECTS OF FIBERS STACKING SEQUENCE AND ITS ORIENTATION ON QSI BEHAVIORS OF CFRP/RAMIE/POLYURETHANE FABRIC INTERPLY HYBRID LAMINATE

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### ABSTRACT

Natural fibers have received major interests in polymers industry due to their cost-effectiveness, excellent strength, and rigidity as well as low specific weight compared to conventional glass fibers. To balance the advantages and shortcomings of natural fibers, hybridization of natural fibers and synthetic fibers with combination of polyurethane (PU) has been investigated. The purpose of this research is to evaluate the energy absorption of hybridization between carbon fibers reinforced polymer (CFRP)/Ramie/PU via QSI test. The effects of stacking sequence of interply hybrid laminate of CFRP/Ramie/PU have been examined. There are four groups of different stacking sequence have been used for QSI test, which are CFR1, CFR2, CFR3, and CFR4. CFR2 displays the highest energy absorption and specific energy absorption capabilities of 10.02 J and 291.90 J/Kg. In conclusion, it can be said that high energy absorption capability can be significantly influenced by the optimum fibers stacking sequence and orientation.

*Keywords:* CFRP; Ramie Fiber, Polyurethane Fabric, Interply Hybrid, Energy Absorption, Quasi-Static Indentation (QSI) Behaviour.

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### INTRODUCTION

Fiber-reinforced polymer (FRP) composites offer exceptional properties such as high durability, rigidity, damping property, flexural strength, and corrosion [1]. The material has high potential to substitute conventional metals in industry due to their behavior that suits the variety of applications. Carbon fibers-reinforced polymers (CFRP) for example, with outstanding properties have been widely used in automotive, civil, aerospace, sport goods etc. It also has been classified as advanced composite materials. Besides, the study of composites has shown significant improvement especially in hybrid composites as mentioned by Padmanabhan (2019) [2], that the weight of roof panel can be minimized and provides a good strength compared to regular panel by using natural fiber hybrid composite sheet material composed of 70% of unsaturated polyester resin and 30% of sugarcane bagasse and stainless-steel wire mesh. Moreover, Mahir et al. (2019) [3] have claimed that natural fibers exhibited many advantages over synthetic fibers including low specific weight, that resulting in greater strength and rigidity compared to glass fibers.

Nowadays, most of the synthetic fibers' utilization have been replaced by natural fibers as reinforcement due to environmental concerns. Moreover, natural fibers possess outstanding properties including low-cost and abundant availability, renewable, biodegradable, low density, high toughness etc. as compared to synthetic fibers. Natural fibers such as Ramie fibers have been widely used in composite materials due to their higher tensile strength and elastic modulus than glass fiber [4]. However, one of the limitations of Ramie is the presence of hydroxyl groups that creating incompatibility problems with many synthetic polymers. This problem can be solved by conducting chemical treatment. Therefore, to balance the benefits and weakness of both synthetic and natural fibers, the hybridization composites between those two types of fibers have been extensively studied. Moreover, it is believed with variety of combination and modification could lead to development of enhanced composite materials that are more sustainable in the future.

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Besides, polyurethane (PU) has shown improvement in tensile properties of coating fabric in textile industry [5]. The ability of the PU fabric will be a great application in hybrid composites study. Due to that, considerably

focused has been paid in the development of PU as additional material in the CFRP/Ramie hybrid composite to add extra strength and flexibility to the composite structures. Up to recently, there is no other studies were extensively discussed on the QSI behavior of CFRP/Ramie/PU laminate composites. The energy absorption of the composite material needs to be determined since the composite often expose to external damage that cause the material failure. The data obtained in QSI experiments can be used to verify numerical attempts to classify damage mechanisms and their interaction during impact events [6]. Therefore, the new combination and sequence of the hybrid composites between CFRP, Ramie fiber and PU fabric should be conducted to address the energy absorption capability of the composite.

## MATERIALS AND METHODS

### Materials

The hybrid composites were prepared by using Cycom 934 prepreg carbon fiber supplied by Cytec Engineered Materials, polyurethane (PU) fabric supplied by Winson Shoes Industries Sdn. Bhd., Johor Bahru, and unidirectional ramie fiber supplied by Konsorsium Ramie Indonesia (KORI). All materials used in this study were cut into 300 x 300 mm each. There is no additional resin was used since the prepreg already contain epoxy. The materials were fabricated into interply laminate composites by using hot-press method using hot-press machine.

### Fabrication of Samples

The sample was hand lay-up one by one according to the desired sequence and orientation (Table 1). Based on the table, there are four samples were prepared with different sequence and combination with variation in Ramie and PU layers. If there is more than one layer of Ramie used, the orientation was varied into 0 and 90° since Ramie is unidirectional fibers. CFR3 and CFR4 is control sample where Ramie position in middle for CFR3 and PU fabric in the middle for CFR4. The samples were pressed in hot press machine for 60 minutes with temperature of 170°C.

### Quasi-static Indentation (QSI) Test

The experimental procedure to determine quasi static indentation response in laminated hybrid composite was referred to ASTM D6264/6264-17 [9]. The test samples with size of 100 mm x 100 mm were labelled accordingly to their variation, which are CFR1, CFR2, CFR3, and CFR4. The samples were tested using QSI test with displacement rate of 1.25 mm/min as suggested in ASTM D6264/6264-17 [9]. The Instron 600Dx- 600 kN Universal Testing Machine were used for the testing. An additional support fixture (Fig. 1(a)) was added to ensure that every laminated composite was tested in the same way. A hemispherical indenter (Fig. 1(b)) has been used for this test since this indenter geometry has produced a larger amount of internal damage than has been observed for similar indenter geometries with sharp tips. Then, the sample was clamped onto the support with a square metal plate that fit snugly into the larger square section of the support. Before the test began, the tip of the indenter was positioned at the center of the sample. The data was recorded at rates of 0.02 seconds for every sample.

TABLE 1: The stacking sequence and orientation of Ramie fiber

Sample group name	Number of ramies fiber layers	Number of carbons fiber layers	Number of Polyurethane Fabric	Fiber stacking sequence (R=ramie, C= carbon, F=Polyurethane Fabric)	Orientation of ramie fibers.
CFR1	1	2	2	C/F/R/F/C	0
CFR2	2	2	1	C/R/F/R/C	0/90
CFR3	1	2	0	C/R/C	0
CFR4	0	2	1	C/F/C	-

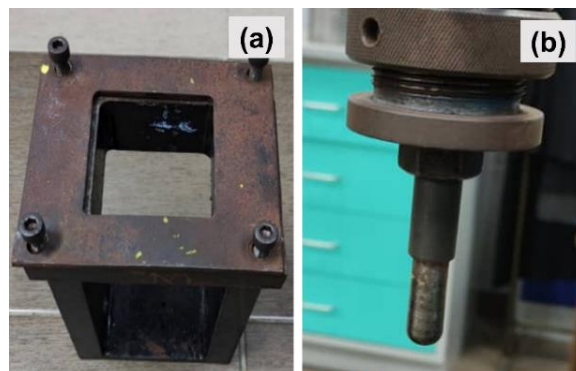


Fig. 1: (a) Support fixture and (b) Hemispherical indenter

## RESULT AND DISCUSSION

From Table 3 and Fig. 2, it can be obviously seen that the absorbed energy and specific energy absorption of CFR2 are the highest as compared to other samples with values of 10.02 J and 291.90 J/Kg, respectively. However, there is insignificant different can be seen between CFR2 and CFR1, which is the absorbed energy of CFR1 is 18% lower than CFR2. In contrast to CFR1 and CFR2 (with thicker structure), CFR3 and CFR4 are the control samples and due to that, the comparison only between the control samples. It can be seen that, the control sample with addition of PU fabric shows better absorbed energy of 3.50 J as compared to CFR3 of 1.67 J. Essentially, the mechanical properties such as tensile strength have the biggest

influence in determining the absorption energy of the composites. However, in this study, the thickness of the composites has shown great impact on the energy absorption. For example, CFR2 with thicker structure than CFR1 possessed higher energy absorbed [11]. Moreover, the variation in orientation of the unidirectional Ramie fibers shows great influence on the energy absorption capacity. As can be seen, CFR2 is the only sample with two plies of Ramies that had advantages in orientation of 0/90°. This orientation will make the punctured of surface difficult as reinforcing fibers are designed to be loaded longitudinally rather than horizontally, the orientation of the fibers imparts highly directional qualities to the composite [12]. Based on literature, when compared to various layup sequences, it is noted that laminates with a (0/90°) layup sequence are the most effective in terms of impact resistance [11].

TABLE 3: Maximum Energy, Absorbed Energy and Specific Energy of Samples

Specimen	Absorbed Energy of Sample, $E_a$ (J)	Maximum Energy of Sample, $E_{max}$ (J)	Average Mass of Sample (g)	Specific Energy Absorption of Sample, (J/Kg)
CFR1	8.21	8.29	32.67	251.19
CFR2	10.02	10.04	34.33	291.90
CFR3	1.67	1.68	20.67	80.93
CFR4	3.50	3.50	17.67	197.87

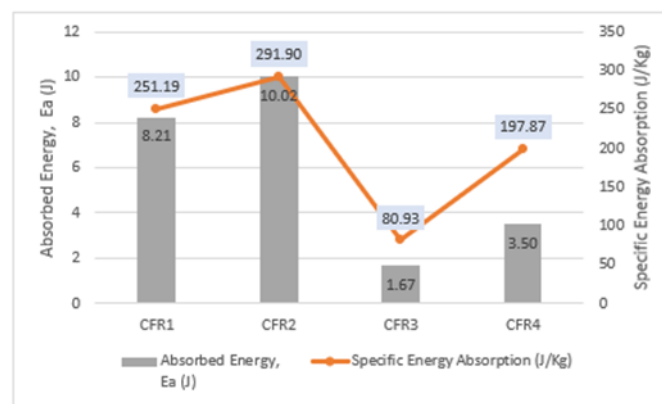


Fig. 2: Bar chart of absorbed energy and graph of specific absorbed energy of the CFRP/Ramie/PU interply hybrid laminates

## CONCLUSIONS

CFR2 exhibits the highest energy absorption and specific energy absorption among other samples with 10.02 J and 291.90 J/Kg, respectively. The control sample shows that CFR4 obtained higher energy absorption and specific energy absorption as compared to CFR3 with percentage different of 70.55% and 83.88%, respectively. Therefore, the samples with three different fibers configuration show that the tensile strength of the raw material can influence the energy absorption. Moreover, the results also revealed that the higher the thickness of the samples, the better the energy absorption capability. However, in control sample, it can be seen that thickness of the sample exhibits opposite behavior, which is the thicker the sample have lower energy absorption capability. In conclusion, energy absorption in this case does not solely influence by mechanical properties of the materials used. The influence of orientation from unidirectional Ramie fibers and thickness of the samples have shown significant impact toward the energy absorption capability.

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## EVALUATION OF QUASI-STATIC INDENTATION BEHAVIOURS ON CFRP/RAMIE FIBERS INTERPLY HYBRID LAMINATE: EFFECTS OF THICKNESS AND INDENTATION SPEED

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### ABSTRACT

This research aims to evaluate the QSI behaviours of carbon fibers-reinforced polymers (CFRP)/Ramie fibers interply hybrid laminate. The behaviors of CFRP/Ramie hybrid laminates were characterized by evaluating the effects of different layers of Ramie fibers (1, 2, 3, 4, and 5 layers) and the different indentation speed (1, 10, 50 mm/min) towards the energy absorption and the indentation force of the samples via QSI test. From the results obtained, hybrid laminates with 5 layers of Ramie (C/R/C/R/C/R/C/R/C/R/C) with 10 mm/min of indentation speed possessed the highest energy absorption and specific energy absorption capabilities of 114.93 J and 1481.94 J/Kg, respectively. It can be stated that high energy absorption capability can be significantly influenced by the thickness of the CFRP/Ramie layers. Meanwhile, the results on indentation speed indicate that the resultant CFRP/Ramie fibers interply hybrid laminate have great potential in low velocity impact applications.

*Keywords:* Natural Fiber, Ramie Fiber, CFRP, Interply Hybrid, Energy Absorption, Quasi-Static Indentation (QSI) Behaviour.

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### INTRODUCTION

Composite structures are widely used in aerospace, marine, transportation, civil and military applications because of their high strength to weight ratio, corrosion resistance, good energy absorption capability, high fatigue life, buoyancy, reduced electromagnetic and acoustic signatures, and lower maintenance cost as compared to traditional metallic structures. Sandwich composites are made of two thin, strong, and stiff face sheets, the core of which is adhesive between the face sheets. The core material is usually a low strength material, but the higher thickness of the sandwich composite gives a high bending stiffness with a low overall density [1]. As a result, sandwich components achieve the same structural performance as conventional low-weight materials. Over the years, through studies, various types of composites were found to have achieved equivalent, or better protection level than steel at a relatively lower density. For example, the breakthrough of synthetic fibers such as aramid and ultra-high molecular weight polyethylene (UHMWPE) has solved the issue of the impracticality of using steel-plated armor especially for soldiers on foot [4].

In the effort of reducing the usage of synthetic fibers, researchers have come up with using natural fibers as substitutes. For example, a study to evaluate the influence of hybridization of glass and ramie fiber hybrid to its dynamic mechanical properties have been conducted [2]. Some research study the hybridization of steels and natural fibers [3]. The study reported that the hybrid gives better ballistic performance compared to individual materials. Despite the vast interest in the research in the performance of hybrid composite material, there is still a lot of room for enhancement and new combination that are yet to be

tested. Due to that, in effort to address the weakness of synthetic fibers composite, the hybridization of synthetic fibers and natural fibers are getting more interest in recent years for composites technology. As compared to synthetic fibers, natural fibers are well-known to have better specific strength and more cost-effective.

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### MATERIALS AND METHODS

#### Materials

The composite prepared using Cycom 94 prepreg carbon fiber and unidirectional ramie fiber mat. The carbon fiber prepreg is a bidirectional woven mat with fiber orientation 0 and 90°. There is no

additional resin or adhesive material is added to the system beside the epoxy resin from the prepreg carbon fiber mat.

#### Preparation of Samples

The unidirectional ramie fiber mat and carbon fiber prepreg were cut into a size of 300 x 300 mm. The excess ramie fiber from the mat was also cut to ensure there is no direct heat contact between the heating plate during the hot-pressing process. All mass of the fiber mats was recorded. The CFRP/Ramie fiber interply hybrid laminate was made by interply configuration. Five groups of samples with different numbers of layers of ramie 1, 2, 3, 4, and 5 layers have been prepared. The ramie fiber mat is placed in between carbon fiber prepreg layers. Hence, creating an alternating stacking sequence of carbon fiber prepreg, which in each sample, there will be one more layer of carbon fiber than ramie fiber. The samples with more than 1 ramie fiber layers, the ramie angle orientation was alternated by 0° and 90°. No changes or alteration of orientation of the carbon fiber prepreg as it is already in bidirectional woven configuration. The configurations of layer are tabulated in Table 1. All samples were arranged according to the stacking sequence as shown in Table 1 prior to hot-pressed. All samples were pressed at 177°C and 1MPa for 65 minutes using the hot-press machine. After that, each sample was cut into 100 x100 mm size for indentation test.

TABLE 1: Number of layers of Ramie fibers, carbon fibers, and its orientation

Sample group name	Number of ramies fiber layers	Number of carbon layer	Fiber of (R=ramie, C= carbon)	stacking sequence	Orientation of ramie fibers.
CR1	1	2		C/R/C	0
CR2	2	3		C/R/ C/R/C	0/90
CR3	3	4		C/R/ C/R/C/R/C	0/90/0
CR4	4	5		C/R/ C/R/C/R/C/R/C	0/90/0/90
CR5	5	6		C/R/ C/R/C/R/C/R/C/R/C	0/90/0/90/0

#### Quasi-static Indentation (QSI) Test

In determining the quasi-static indentation (QSI) behavior of the material system, a QSI test with reference to ASTM D6264/D6264M-17 [5] has been conducted. This test method determines the damage resistance of multidirectional polymer matrix composite laminated plates subjected to a concentrated indentation force. Testing machine used of the test is the Instron 600Dx- 600kN Universal Testing Machine. Additional support fixture with 75 x 75 mm square cut out was added to conduct the test. For this test, a hemispherical indenter has been used. The samples were then fixed onto the support fixture, prior the test and the tip of the indenter was made sure to be on top of the center of the samples. The data was set to be collected every 0.02s until the specimen failed. The different displacement speed of 1, 10 and 50 mm/min has been carried out to test the QSI behaviour of the samples and were tagged as A, B, and C, respectively.

## RESULT AND DISCUSSION

Fig. 1 shows the absorbed energy and specific energy absorbed by all samples. Generally, as the displacement increases, the more energy was required by the indenter. For when the displacement rate was increased from 1mm/min to 10mm/min the Absorbed Energy for CR1, CR2, CR3 and CR5 increased by 5.6%, 26.6%, 6.8 %, and 35.7% respectively. However, in CR4 the Absorbed Energy dropped by 0.24%. By average, when the displacement rate was increased from 1mm/min to 10mm/min, the Absorbed Energy increases by 14.892%. For the same increment of displacement rate, the maximum force on CR2, CR3, CR5 increased by 8.31%, 9.72%, and 16.97%, respectively. Meanwhile, CR1, CR4 dropped by 6.69% and 0.044% respectively.

Next, when the displacement rate was increase from 10mm/min to 50mm/min, Absorbed Energy of CR1, CR2, CR5 dropped by 9.7%, 35.0%, and 24.12% respectively. However, increment was recorded in CR3 and CR4 by 6.7% and 1.87% respectively. By average, a drop of 12.05% was recorded when the displacement rate was increased from 10mm/min to 50mm/min. As for displacement effect on the maximum displacement, no visible pattern can be seen. For the same increment of displacement rate, the maximum force n CR1, CR2 and CR4 increased by 4.69%, 6.70%, and 0.027% respectively. Meanwhile, CR2 and CR5 dropped by -17.28% and -8.51% respectively.

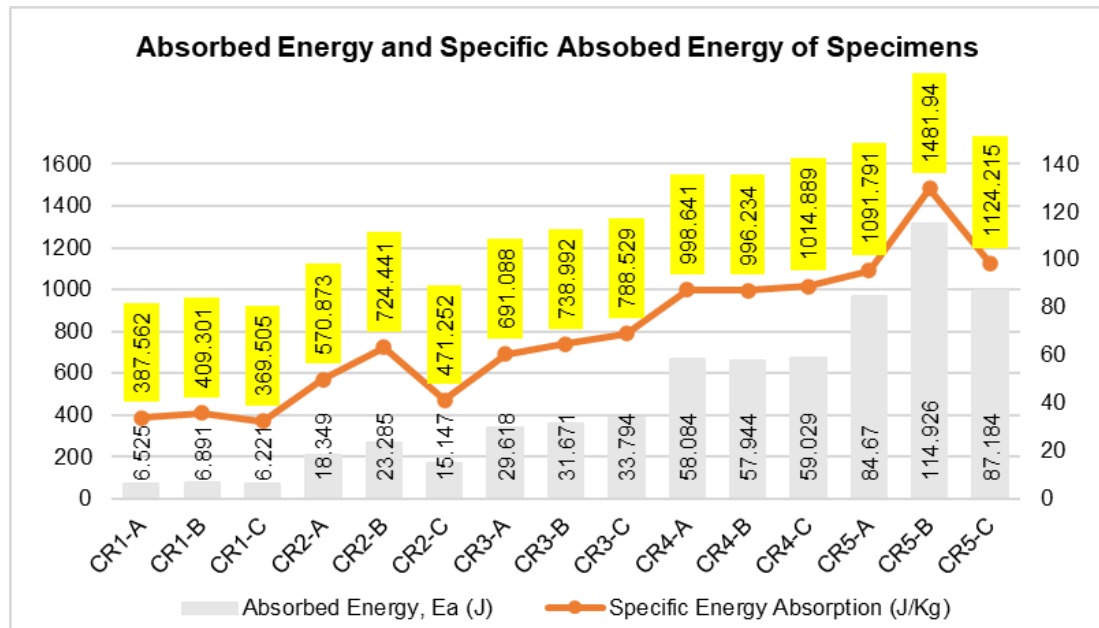


Fig. 1 Absorbed Energy and Specific Absorbed Energy of the Specimens

## CONCLUSIONS

To be concluded, absorbed energy increases as the displacement rate was increased from 1mm/min and 10mm/min. However, the absorbed energy of all materials are generally decreases as the displacement rate was increased from the 1mm/min to 50mm/min. However, when compared to other material system, percentage of increase of energy absorption when the displacement rate was increased from 1mm/min to 10mm/min. Generally, all sample group shown significant increase except for CFRP/ Ramie Fiber Interply Hybrid Laminates 4A. CFRP/ Ramie Fiber Interply Hybrid Laminates 5A also shown 35.7% of increment of Absorbed Energy, when the displacement rate was increased from 1mm/min to 10mm/min.

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## BIODEGRADABLE PACKAGING DERIVED FROM THERMOPLASTIC CASSAVA STARCH: A REVIEW

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### ABSTRACT

Consumption of non-biodegradable materials has had a detrimental effect on humanity and the climate. Non-biodegradable materials are composed of petroleum-based plastic polymers that are harmful to the atmosphere due to their inability to dissolve in landfills. Thermoplastic cassava starch has been recognized as a complete biodegradable material that can be produced by various plants, which is one of the richest resources that is renewable, biodegradable, and readily available at a low cost. Plasticizer, heat, and shear may help convert starch into thermoplastic starch (TPS). This bioplastic is a biodegradable and soluble in water. To be clear, this bioplastic has the restriction that it doesn't have the mechanical characteristics of POM and is also very sensitive to moisture. Hence, this article intends to evaluate the many TPS modifications carried out over the years.

*Keywords:* Thermoplastic starch; biocomposites.

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### INTRODUCTION

Environmental knowledge and interest have grown steadily in recent years around the world. As well as other enforcement of environmental legislation, this helps to maintain the ecosystem, which is critical in this transformation [1]. The use of non-biodegradable material has led to environmental problems such as water waste, ozone contamination, and landfill problems. Both of these challenges also prompted new studies into the production of biodegradable products such as thermoplastic starch derived from natural resources. Due to the widespread usage of plastics in a variety of industries, especially the packaging industry, annual production of petroleum-based plastics exceeded 300 million tons until 2015, with approximately 1% being bioplastic [2], [3]. More recently, 50% of the bioplastic preparation is from starch. It is found to help nature by keeping in mind of carbon dioxide emissions and the use of fossil fuel in processing and can have a significant beneficial effect on global warming.

Due to the environmental impact of traditional thermoplastics, the production of biodegradable thermoplastics has accelerated. Biodegradable materials are both safe for the user and the climate. Thus, it is prudent to minimize the usage of non-biodegradable plastic and to encourage the use of biodegradable plastic. Natural fibers have risen in popularity as reinforcement in composite materials over the years, owing to the growing need for renewable, cost-effective, and eco-friendly materials in a variety of applications. Natural fibers often used as reinforcements include flax, kenaf, hemp, jute, coir, sisal, and abaca. However, sugar palm fiber (SPF), a natural fiber, is gaining recognition as a reinforcement in composites, despite the fact that rural populations have known it for decades for its versatile traditional usage [4].

### THERMOPLASTIC STARCH

#### *Thermoplastic cassava starch*

Cassava starch is a popular choice for making thermoplastic cassava starch (TPCS) since it is inexpensive and processed in large quantities. As a PLA factor for making flexible packaging films, TPCS and updated TPCS have reasonable properties. [5]. Cassava or its specific name is *Manihot esculenta* roots are very plentiful in tropical regions, according to Oladunmoye et al. (2014) [6]. Cassava is a major source of food in the tropics for people who live in lowland Africa. The feature of the granule affects the pace of hydration and capacity swelling process. Additionally, its final aesthetic appearance was affected by color as well as by the ability to inflate, the capacity to absorb water, and the ability to dissolve.

### THERMOPLASTIC STARCH COMPOSITES

Starch is a renewable biopolymer that is widely used in environmentally safe packaging products as a replacement for non-biodegradable petrochemical-based plastic. Although starch in its natural state is not a thermoplastic, it can be transformed into a substance that resembles plastic called thermoplastic starch (TPS). Owing to the hydrophilic nature of the materials, biodegradable composites made of pure thermoplastic starch have inferior mechanical properties and are more hygroscopic than composites made of synthetic polymers [22].

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TABLE 1 Summary of cassava starch from various studies.

Type of polymer/tubers	Product/Application	References
Cassava Starch–Carboxymethyl Cellulose Incorporated with Quercetin and quercetin and tertiary butylhydroquinone(TBHQ).	Active packaging	[7]
Cassava–wheat composite	Bread making	[8]
Cassava starch, glycerol and natural polyphenols extracted from rosemary leaves.	Edible active films	[8]
Konjac glucomannan (KGM)-chitosan-cassava starch-nanosilver films.	Food packaging materials.	[8]
Cassava starch, beeswax, and ethanolic propolis extract	Edible films and coatings	[9]
Cassava starch with ammonium zirconium carbonate(AZC)	Substitute for SBR latex	[10]
Cassava starch-zinc-nanocomposite films	Food packaging	[11]
Cassava starch, essential oil and sodium bentonite nanoclay	Food packaging	[12]
Cassava, potato and sweet potato starch	Food industry	[13]
Cassava starch	Textile Pharmaceutical industries	[14]
Cassava starch	Bioethanol Paper Adhesives	[15]
Cassava starch with carotenoids	Food industry	[16]
Yam and cassava starch with essential oil of oregano	Fruit coating	[17]
Cassava starch, Corn starch and glucose oxidase–peroxidase reagent (GOPOD)	Food processing	[18]
Cassava, corn, potato, sweet potato, glutinous rice, rice, and buckwheat	Ethanol	[19]
Cassava starch with NaOH	Food industry	[20]
Cassava starch hydrolyzed by PoGA15A	Ethanol	[21]

Although TPS has excellent processing capability, it is unsuitable for the majority of typical applications due to a number of drawbacks, including inferior mechanical efficiency, hygroscopic design, and low dimensional stability. TPS's amorphous structure has a propensity to regenerate intermolecular hydrogen bonds when exposed to moisture, resulting in recrystallization (also called retrogradation) and embrittlement. Demonstrated that retrogradation has a detrimental effect on the oxygen and water vapor permeability of TPS films. Enhancing the moisture resistance and mechanical properties of TPS by the use of synthetic polymers has garnered significant interest, allowing for more robust TPS applications. Combining TPS with a hydrophobic polymer increases the total hydrophobicity of the surfaces, where the hydrophobic polymer acts as a barrier, limiting TPS's moisture absorption, and the plasticizer added to TPS may affect the material's mechanical properties [23].

Polymeric nanocomposites including inorganic and organic compounds have been shown to be an important tool for improving the performance of a variety of products and materials, including thermoplastic starch. Due to their superior mechanical properties, huge specific surface region, and high aspect ratio, bio-based cellulose nanomaterials, specifically cellulose nanocrystals (CNCs), cellulose Nano fibrils (CNFs), and micro-fibrillated cellulose (MFC), have been extended in both natural and adapted ways, most notably as an attractive and renewable reinforcement medium for composite materials. Numerous uses of Nano cellulose materials have been identified as cost-effective, including the strengthening of thermoplastic polymers and thermosets by CNCs, the use of MFC in a strongly filled paper in a pilot-scale papermaking system, and the production of highly recyclable superabsorbent [23].

The primary disadvantage of thermoplastic starch composites is the incompatibility of extremely polar thermoplastic starch with nonpolar, resulting in an irreversible decrease in the thermoplastic starch composites' mechanical properties. This incompatibility precludes the forming of strong interfacial hydrogen bonds between thermoplastic starch phases, and numerous studies have been conducted to improve thermoplastic starch compatibility through starch modification, polyethylene modification, and incorporation of a compatibilizer (or coupling agent) into thermoplastic starch composites. The addition of compatibilizers (CA) is a well-established and industrial process for improving the stability and interfacial bonding of immiscible polymer blends [23].

## CONCLUSIONS

To summarize, TPS is a promising material as a replacement for non-biodegradable plastics. Because of its ecologically beneficial properties, this biopolymer is an ideal choice. TPS's mechanical characteristics and moisture sensitivity may be improved by modifying the material with natural fiber reinforcement and various kinds of polymer. Positive outcomes have been recorded as a consequence of the TPS modification work. This suggests that TPS will likely replace petroleum-based polymers in the near future, particularly in packaging applications. The potential use of TPS as a packaging material is regarded as one of the most promising solutions to the world's abundance of plastic trash.

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## A REVIEW ON EMERGENCE, VIABILITY, AND FUTURE OF COMPOSITE MATERIALS IN MARINE SECTORS

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### ABSTRACT

The current global scenario has led to numerous studies that can replace traditional materials in the automotive sector. For instance, the ever-increasing demand for lightweight materials such as polymer composites to achieve better fuel efficiency has attracted a larger community. Moreover, composite materials are considered to be the scientific and practical solutions in several fields, including marine applications. Since, the applications in marine industries are highly challenging compared to other sectors due to the materials susceptibility to damage such as environmental effects, accidental impacts (i.e., grounding of vessels), wear and tear throughout their service life. Also, the marine environment, which is highly saline, also demands specific requirements in material properties such as corrosion resistance, and impermeability etc. The applications of natural and synthetic materials are observed in the construction of marine fields such as vessel building, offshore structures, oil rigs, etc. This has resulted in reliable, durable, and persistent material alternatives. The scope of this article is to review and raise awareness regarding the evolution of composites in marine applications, their current scientific and technological problems, and prospects to support their larger applications in the marine industry. Trends of hybrid composite applications for maritime sectors projected as the future alternative have also been discussed in terms of materials, sustainability, and environmental concerns.

*Keywords:* Composite materials; marine; and hybrid composites.

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### INTRODUCTION

Demand for an alternative material in applications of engineering areas has always been due to the need for minimizing of structural weight. As it is proven that a reduction in structural weight has a tremendous positive impact on energy efficiency. Just like other engineering areas, the major challenge in naval architecture is to achieve a competitive structure as light as possible. This can increase the payload and fuel economy, thereby decreasing environmental emission [1]. Steel is predominantly used in the hull construction of ships, boats, and submarines. Although aluminum is also widely used even in massive floating structures, naval architects prefer lighter materials like polymer-based composites for the construction of structural elements such as bulkhead, deck, mast, propeller, etc.

Also, such replacement is often necessary when steel would make a surface ship top-heavy, and if aluminum is not a viable option. In these cases, for instance, composite materials offer a reasonable alternative [2]. The use of composites for marine structures provides additional advantages such as (i) flatness for stealth requirements, and (ii) increased resistance to corrosion to build reliable and durable vessels. However, it is vital to mention that there are some difficulties in joining composites and metals due to the significant differences in mechanical properties between them. Some of the mechanical properties to be considered are stiffness, coefficient of thermal expansion, etc. between the adherents and the large anisotropy of composites.

### CURRENT SCENARIO

Currently, the percentage of composite materials used in the marine vessels building industry has been around 6% only. However, in a small craft industry that builds boats to a maximum of the overall length of 50 metres, composite materials are higher with a share of 70%. Hence, the share of composite materials varies following the size of the marine vessels ranging from fiber boats to ultra large crude carriers (ULCC). Based on the assumptions concerning penetration of composites, it is expected that the marine composites market will grow from about 135,000 tonnes in 2011 to close to 200,000 tonnes in 2020 at a compound annual growth rate (CAGR) of 5.6%. This corresponds to the overall revenue growth from approximately US \$900 million in 2011 to about \$1500 million in 2018 at a CAGR of 7.1% [3].

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### MARINE – A CHALLENGING ENVIRONMENT

Marine structures are susceptible to damage throughout their design life, which can arise from accidental impacts or explosions like collision, grounding of vessels, etc, fire, wear and tear due to aggressive environments, and a lot more. For example, an important event that occurred recently was the USS Fitzgerald warship's collision with a merchant vessel carrying shipping containers. Several casualties occurred due to severe damage to the hull of the naval vessel resulting in flooding of compartments, whereas the merchant container ship did not suffer any casualties [4].

#### *Damage to marine structures*

Marine structures have a high susceptibility to extreme loads, particularly impact and blast-induced damage. This can significantly reduce their structural performance or cause instantaneous catastrophic failure. Furthermore, environmental effects can cause cyclic fatigue loading on marine structures, as well as damage due to seawater moisture, humidity, and so on. The damage modes that composites undergo are quite complex compared to traditional materials such as isotropic metals, namely matrix cracking, fiber fracture, interface debonding, layer delamination, fiber pull-out, and compound failures including kink bands (buckling delamination due to kinking of fibers under compression) and barely visible impact damage (BVID) cone of fracture[5].

In this regard, composite structures are attractive because they offer significant advantages compared to traditional steel structures, namely high strength to weight ratio, excellent impact properties, low infrared, magnetic and radar signatures, excellent durability, and high resilience to extreme loads[6]. Advancements in damage detection and computational modeling techniques have facilitated accurate predictions of the behavior of marine composite structures under extreme loads and aggressive environments.

### EVOLUTION OF APPLICATIONS OF COMPOSITES IN THE MARINE INDUSTRY

Composites occupy a wide range of marine applications since the early 1950s, including (i) laminated glass-plastic construction in naval boats (ii) reinforced plastic piping and (iii) glass-fiber-reinforced plastics (GFRP) for marine structures and ships. Although the applications of FRP (fibre reinforced polymers) were initially limited to small crafts such as fiber boats, lifeboats, and small fishing vessels, they have also found applications in large-scale structures such as ship hulls, superstructures, submarines, and offshore structures [6].

#### *Metals vs. composites for marine applications*

Given the advances in materials, design process and production procedure, it is now possible to produce high strength, lightweight, and cost-effective composite structures that could compete and outperform their metal counterparts. Unlike metals, the density of a composite can be easily controlled through structural arrangements. In addition, it increases structural buoyancy[7]. Moreover, other salient features inclusive greater material strength, flexibility, environmental resistance, damage tolerance, as well as reductions in weight, size, and cost can also be appreciated [8]. Traditionally, the preferred geometrical arrangement of fiber reinforcement for marine applications has been woven fabric, which is often combined with layers of chopped strand mat. The selection of such currently available materials for fast vessels such as the surface effect ship and hydrofoils, both surface piercing and fully submerged, is quite advanced, as in these applications weight savings are critical and structural optimization is essential[9]. Composite materials and structures for marine applications offer many advantages over traditionally preferred engineering materials. Some of the prominent advantages are being lightweight, corrosion resistive. Composite materials also show excellent behavior under extreme and cyclic fatigue loading, which leads to a significant reduction in maintenance costs[10]

#### *Marine application of fiber reinforced polymers*

Specially, carbon fiber reinforced polymers (CFRP) has high strength-to-weight and stiffness-to-weight ratios compared to steel and aluminum. This significantly improves the performance of marine structures without sacrificing their mobility. Although the effectiveness of GFRP was historically challenged due to fears of its vulnerability under fire, it has been found that GFRP offers an excellent fire barrier because of its low thermal conductivity, as it prevents fire propagation by avoiding flashover into adjacent cabins[12].

A marine tug (tugboat or towboat) is a type of vessel that maneuvers other vessels by pushing or pulling them either by direct contact or using a tow line. Tugboats are powerful for their size and solidly built. Some tugboats serve as icebreakers and salvage boats. In tugboat construction, the resins used in are fiber reinforced plastic structures which are mainly thermosetting (polyester, epoxy, vinylester, etc.). Thermoplastic resins (polyamide, polypropylene, PET, PBT, etc.) have only recently started being used for boat-building or fittings. Bio-based resins have started to appear, but their use is still very limited in the nautical sector.

The Royal Swedish Navy has employed sandwich composites made from carbon, glass, and/or aramid fiber-vinylester skins with a PVC-foam core for constructing large patrol craft (including the 73 m CFRP sandwich Visby class stealth corvette). Using this sandwich composite material, they built a 30 m long surface effect ship, known as the Smyge MPC2000. These composite materials exhibit light-weight, superior corrosion resistance, and excellent resistance to underwater shocks, in addition to stealth properties such as low thermal and magnetic signatures and good noise dampening properties. Despite the superior properties of the Skjold and Smyge MPC2000, large patrol boats are continued to be built from steel and aluminum alloys due to the higher costs associated with composite materials.

Fiber reinforced plastics are still a large part of a wide range of marine structures being developed. Increased range, stealth, stability, and payload are the operational performance indicators that drive the need for the development warships and submarines, as well as the reduction in ownership costs arising from reduced maintenance and fuel consumption. Some of the different material systems used for marine composite structures are explored herein. There has been growing interest in increasing the size of patrol boats up to 55 metres, even though patrol boats are rarely built longer than 20 metres because their hull girder suffers from low stiffness. To this effect, feasibility studies have been conducted for comparing the cost, weight, and structural performance of large patrol boats made from steel, aluminum, and sandwich composites. These studies have shown that patrol boats made from GFRP can exhibit a reduction of weight of up to 10% than that of an aluminum boat and up to 36% than that of a steel boat of similar size.

The disadvantages with fiber reinforced plastics typically stem from the lack of user confidence in plastics outperforming metals in terms of structural integrity. However, in-service monitoring, which embeds sensors in the composite to assess its performance during its service life, may convince sceptics, as well as regulatory authorities of the superiority of fiber reinforced plastics. Another disadvantage with composite structures is their variability between the same types of structural applications due to production methods, which affect their quality, mechanical properties, and additional costs incurred due to the resulting variations in the development of design and manufacturing procedures [12]. This issue had been addressed by suggesting closer collaborations between designers and fabricators build productivity from the beginning of the manufacturing process. In terms of recyclability, thermoset plastics differ from thermoset resins that dominate today in terms of recyclability, but thermoplastics have disadvantages of high energy requirements due to the elevated manufacturing process temperatures, less powerful interfacial resin/fiber bonding, and poor adhesion when secondarily bonded. FRP have significant usage in boat building and marine construction industries in a broader range for decades because it is the optimum choice in terms of durability and workability [13]. The use of composite and sandwich-structured materials in modern engineering applications, such as civil and military aircraft, launch vehicles, wind turbine blades, and assorted marine structures, has grown significantly over the past few decades due to their high strength-to-weight and stiffness-to-weight ratios.

### **NEED FOR MARINE APPLICATIONS OF NATURAL FIBER -REINFORCED COMPOSITES**

A need to find a realistic alternative to glass or carbon-reinforced composites has led to an increased interest in polymer composites filled with natural-organic fibers, derived from renewable and biodegradable sources. When two or more chemically distinct materials are combined, produces a synergistic effect, with a distinct separating interface of the component. The use of natural or plant fiber reinforced composite is increasing with time due to its advantages like low cost, ease of availability, lightweight etc. Natural fiber materials are used as reinforcing the material. Natural fibers are constituting of cellulose, hemi-cellulose, lignin, waxes, and some water-soluble compounds. Regarding fibers, natural fibers are also beginning to enter the composite market, but their use in structural elements is limited. Since they are used mostly for filling functions, research on alternate natural fiber – reinforced composite materials for marine tug boat construction is a current need.

### **HYBRIDIZATION**

The laminated composites can be classified as[15] (i) composite laminates, obtained using a single type of fiber (ii) hybrid composites, obtained from two types of fiber in the same array, where this “hybridization” can offer a more considerable flexibility in searching for the proper material solution with respect to each specific application [16]. Hybridization is a process of incorporating two or more types of fiber of varying properties, usually synthetic fibers with natural fibers, or exclusively natural [17], or synthetic mixed [18], in order to yield better strength, better stiffness, a high strength-to-weight ratio, and other mechanical properties. An exciting application of hybrid composites, including the use of natural fibers, for the fabrication of small boats can be observed, for instance [19]. The hybrid system for improved material or structural performance is, in fact, a well-known concept in engineering design. Furthermore, it can be an extremely valid strategy in the specific case of green composites. Hybridization of natural composites is an effective way to improve a composite’s mechanical properties and dimensional stability (moisture, temperature, etc.). In particular, it permits solving the difficulties in the practical use of natural fibers related to their low stiffness, lower with respect to that of the synthetic ones. The stiffness of biocomposites can, thus, be improved through hybridization, to permit the adoption of this class of composite for structural applications.

### **CONCLUSION**

More recently, bio-based resins have started to appear, but their use is still very limited in the nautical sector. The environmental benefits of bio-based resins and adhesives are mainly to eliminate toxins and to focus on human health and the environment by reducing pollution. Regarding fibers, natural fibers are also beginning to enter the composite market, but their use in structural elements is limited due to their relatively weak physical properties. Currently, they are used predominantly for filling functions. Glass fibers represent 89% of the worldwide volume of fibers used in composites, while only 10% are natural fibers. At the same time, numerous research and development studies for the use of natural fibers are under way and possibly allow for higher industrial applications in the near future.

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## WATER TRANSPORT PROPERTIES ON DIFFERENT PLASTICIZER TYPE AND CONCENTRATION OF BIONANCOMPOSITE FILMS INCORPORATED WITH ESSENTIAL OIL FOR FOOD PACKAGING APPLICATION: A REVIEW

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### ABSTRACT

The escalating environmental devastation caused by the disposal of packaging plastic waste has resulted in an urgent need to produce environmentally friendly packaging materials in order to save our ecosystem. Natural biopolymers have been studied as potential alternatives to conventional plastics in an effort to tackle the ongoing environmental catastrophe created by non-biodegradable plastics. Water barrier resistance of biodegradable packing films has been reported to be low. Such drawbacks severely limit their general utilisation, particularly for food packing. Researchers have performed numerous investigations to increase the water sensitivity of starch-based polymers without affecting their biodegradability. The use of a plasticizer has been proven to improve the water transmission capabilities of biodegradable film. This review focuses on the enhancement of packaging performance of the green materials as well as their water transport properties for food packaging application.

*Keywords:* Water transport, bionanocomposite films, nanocellulose, food application, biodegradability.

### INTRODUCTION

Increased environmental awareness, combined with stringent policies across the board, has spurred the plastic manufacturing industry to develop new materials that can replace synthetic petroleum-based plastic. Food packaging is commonly made of plastic, paper, metal. Plastic, on the other hand, has surpassed metal and paper in prominence due to its superior material qualities and inexpensive cost of production [1]–[3]. The use of conventional petroleum-based polymers poses a number of potential issues due to their non-renewable sources and disposal costs [4], [5]. In order to reduce the amount of synthetic polymers required in packaging application, many types of bionanocomposite films to be used in production of food packaging. It is not to completely replace synthetic plastics, but rather to improve their efficiency.

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Several experiments have been conducted to develop technique to enhance the water-resistant properties of these bio-packaging films. The different strategies device to diminish this water sensitivity include incorporation of nanocellulose within the biodegradable polymers.

### WATER TRANSPORT PROPERTIES ON DIFFERENT PLASTICIZER TYPE AND CONCENTRATION OF BIONANCOMPOSITE FILMS

Water transport properties is one of the most important studies in biocomposite film, which is indirectly related to the properties of water that would act as plasticizer in biocomposite film. Plasticizing hydrophilic polymers with polyols such as glycerol and sorbitol has been reported to be particularly effective [6]–[11]. Natural polymer matrices have a limiting factor linked with water absorption due to a bundle of hydroxyl groups in their structure [1], [4]. Moisture buildup in biocomposites can cause physical degradation such as dimensional changes and thickness swelling, as well as a decrease in the mechanical properties of the film. The adhesion of the fibres to the matrix may be diminished as a result of the thickness swelling. Water absorption modifies the properties of biocomposites in an unfavourable way, hence water absorption tests were performed to assure the quality of bionanocomposite materials. Plasticizers are low-volatility chemicals that are added to biopolymer

materials to change their functional qualities by improving their mechanical properties, barrier properties, flexibility, elasticity, stiffness, and brittleness [8], [12]–[14].

## CONCLUSION

Water vapor barrier properties of biodegradable films were improved by addition of cellulose nanofibers. Plasticizer reinforcement in films may disrupt the continuity of the polymer matrix, resulting in modifications such as films that are more flexible and stretchable. Various studies attested to the growing interest in the utilisation of starch derived from numerous sources incorporated with essential oils to prepare biobased packaging films.

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## EFFECT OF WEIGHT PERCENT GAIN OF TENSILE SPECIMENS ON TENSILE PROPERTIES OF OIL PALM TRUNK (OPT) REINFORCED WITH EPOXY

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### ABSTRACT

Nowadays, the utilization of oil palm trunk (OPT) wastes has been one of the potentials in the composite applications. This research aims to investigate the effect of weight percent gain (WPG) tensile specimens of OPT reinforced with epoxy (OPTE) composites on the tensile properties. The OPTE composites were fabricated by using vacuum-assisted resin transfer moulding (VARTM) technique. The unimpregnated OPT were impregnated with epoxy as resin. The OPTE were divided into three zone which are outer zone, middle zone and inner zone. The tensile test procedure was followed according to ASTM D3039. Three different zones have a significant effect on the tensile performances. The results revealed the tensile specimens in the inner zone of OPTE exhibited the highest percentage of WPG and the lowest tensile properties as compared to the outer zone and middle zone of the OPTE composites.

*Keywords:* Wood impregnation, resin, composite, wood modification, resin infusion.

### INTRODUCTION

Malaysia is very familiar in oil palm industry and one of the top palm oil producers in the world. From the oil production, only 10% of the oil extraction and the rest left as biomass [1]. The massive amount of biomass wastes that are generated during the oil palm fruits harvesting, palm oil processing or during oil palm trees replantation including empty fruit bunches (EFB), palm kernel shell (PKS), mesocarp fibre (MF), palm oil mill effluent (POME), oil palm trunks (OPT), oil palm leaves (OPL) and oil palm fronds (OPF) [2]. About 75% of the wastes of OPT and OPF are left rotten at the plantation area [3]. The OPT are available during replanting process. The OPT is one of the biomasses wastes to convert into value-added products.

There are a few research towards utilization of OPT. Recently, the OPT was modified with formaldehyde by using high vacuum pump [4]. Next, inner part of OPT were modified with nanoparticles and formaldehyde [5]. The impregnation of the OPT and polymer composites were prepared from a combination of the OPT with phenol formaldehyde (PF) and urea formaldehyde (UF) resin in different resin percentages using an impregnation method revealed the dimensional stability of the OPT polymer composites with the highest resin loading being slightly lower when compared to rubberwood [6]. Recently, the vacuum infusion technology promoted excellent performances in fabricating process. One of the vacuum infusion technologies is VARTM technique. From previous study, a poplar solid wood was impregnated by using the vacuum-assisted resin infusion process for manufacturing woody materials and the result showed excellent flexural performances [7]. In this research, the dried OPT were impregnated with epoxy via VARTM technique. The weight percent gain (WPG) of different zone of OPTE composites were evaluated. The effect of WPG on tensile properties were discussed in this paper.

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### MATERIALS AND METHODS

#### Materials Preparation

About 25 years old of OPT provided by a nearby plantation are in Terengganu, Malaysia was used in this study. The OPT were sawn into the OPT panels and dried under open area before further processing. Before fabrication process, the OPT panels were dried under dryer about 40° C until reached the constant weight. The Kinetix R118 epoxy and Kinetix H120 hardener were used in this research. Both epoxy and hardener were mixed in the ratio of 4:1.

#### Fabrication Process

In this research, the OPTE composites were fabricated by using VARTM technique as shown in Figure 1. The OPT was set on a high

tempered glass flat table that act as mould. The mould was wiped out by the acetone and followed by a wax release mould agent. Then the OPT was laid over by peel ply and mesh flow, respectively.

The inlet and outlet of fitting pipe were connected to the laminate specimen. Then a vacuum bag was placed over the mould and sealed carefully by tacky tape. During the drop test, the resin inlet was clamped by a clam while resin outlet was connected to the pressure pot of vacuum pump. The inlet tube was unclamped then immersed in the epoxy pot and the epoxy was allow to infuse to the OPT. After infusion process was completed, the inlet and outlet tube were clamped and let the panel left to be cured under vacuum for about 24 hours. Then the panel was demoulded from the mould.

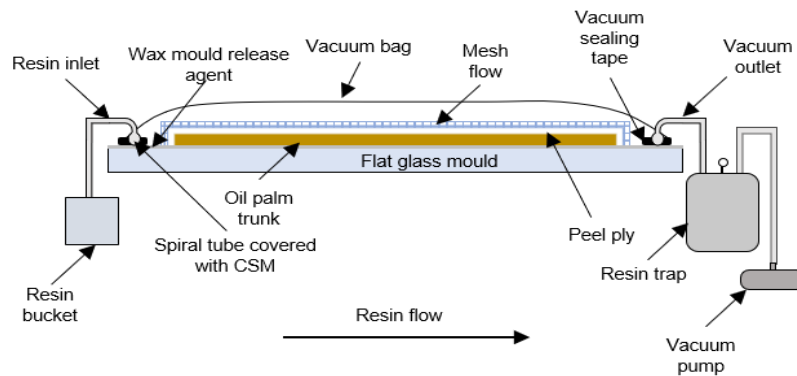


Figure 1: The fabrication process of OPTe composite.

After fabrication process, the OPTe panels were cut into specific dimension of specimens as followed by ASTM D3039 standard to investigate the tensile properties. The OPTe specimens were cut into three zone that parallel to the fibre direction as shown in Figure 2.

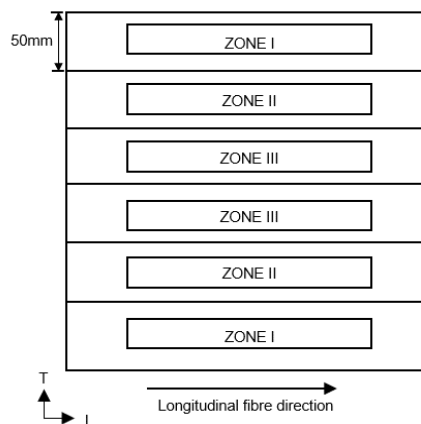


Figure 2: The cutting zone of OPTe specimens

Treatability of OPTe tensile specimens was analysed by weight percent gain (WPG) as follows:

$$WPG (\%) = \frac{(w_f - w_i)}{w_i} \times 100 \tag{1}$$

where  $w_f$  is sample weight of OPTe specimen and  $w_i$  is sample weight of unimpregnated specimen.

**RESULT AND DISCUSSION**

The result of WPG of tensile specimens and tensile strength as shown in Figure 3. It can be observed that the average WPG of OPTe composite specimens increased from outer zone into the middle zone. The highest average of WPG of inner zone was 172.31%. The different of WPG values due to the content of parenchyma tissues in the OPT. The greater content of the parenchyma tissues in the inner zone which tends to absorb water [8], [9].

A similar finding reported by Wahab et al. [10], stated that the WPG inner section of oil palm stem (OPS) veneer was 17% higher WPG as compared the outer zone because of high amount of parenchyma tissues in the inner area of OPS. Therefore, the epoxy filled into the parenchyma tissues of the dried OPT. At the outer zone, the number vascular bundles greater than the parenchyma tissues.

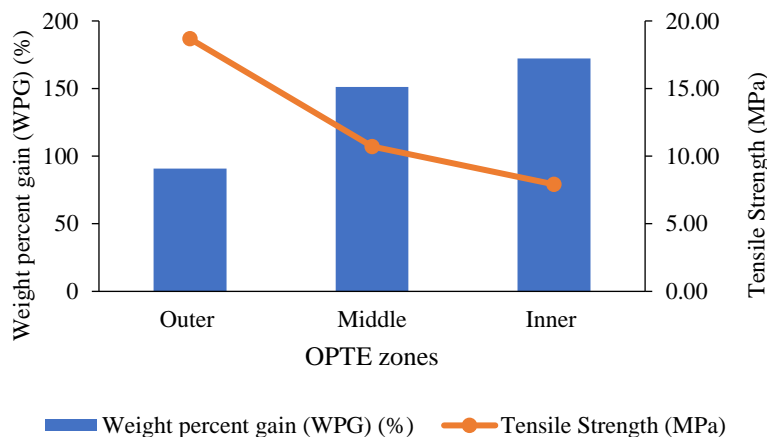


Figure 3: The WPG of tensile specimens and tensile strength in three zones of OPTE composites.

Therefore, the influence of high tensile strength of OPTE composite due to the epoxy content and the number of vascular bundle content at the outer zone. The three zones of the OPTE were enhanced due to the penetration of epoxy into the dried OPT. During curing process, the interaction and bonding between epoxy and OPT caused the improvement of the tensile strength. From previous study by [11], reported that the low flexural properties affected by the excess content of Urea Formaldehyde (UF) in OPT that causes increase in brittleness. Therefore, the low strength of tensile strength at the inner zone because the excess epoxy resin filled into the parenchyma tissues of the OPT.

## CONCLUSIONS

In conclusion, the WPG of tensile specimens and tensile strength of OPTE were investigated. The highest of WPG of tensile specimens at the inner zone of OPTE composites. The WPG attributed to the low of tensile strength in the inner zone of OPTE specimens. The higher the epoxy content the higher the value of WPG of tensile specimens.

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## POTENTIAL FOR IMPLEMENTING FUSED DEPOSITION MODELING TECHNOLOGY USING NATURAL FIBER COMPOSITE FILAMENT

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### ABSTRACT

Implementation of natural fiber in Fused Deposition Modeling (FDM) has attracted many researchers in various application types. This study presents a review of the potential of natural fibers to be employed in FDM technology. The principle of FDM is introduced where it includes the process of thermal extrusion through an FDM machine that consists of an extrusion head, nozzle, and printing platform. Past researchers have studied the various types of natural fibers, and the material selection process of FDM is reviewed to suggest the potential selection process method and selection criteria. Finally, the challenges of the FDM in processing the natural fiber-filled polymer are reviewed.

*Keywords:* natural fiber composite, fused deposition modeling.

### INTRODUCTION

Fused deposition modeling (FDM) is an additive manufacturing technology that is easy to operate, has a low negative impact on the environment, is low cost, is feasible with complex design, and short lead time [1]. This technique has been applied in various product design areas such as medical tools, automotive components, food packaging, and home decoration. The principle of FDM is to fabricate three-dimensional objects by laying up successive layers of thermoplastic material upon one another. Generally, the thermoplastic filament is used as feedstock material will be pulled through rollers and inserted into the heated extruder. The molten filament will be extruded through the heated nozzle and lay on the heated printing platform by following the coordinates provided through digital command [2]. The configuration of the FDM machine could be found as shown in Fig 1. The FDM machine mainly consists of an extrusion head, nozzle, and printing platform. The extrusion head will be moved in X, Y, and Z- direction by following the command created in stereolithography CAD software.

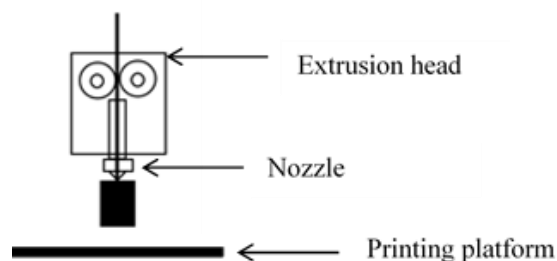


Fig 1. Schematic of FDM

In general, the material used for filament of FDM is thermoplastic polymer where the material can reform through the reheating process. It is an essential characteristic of the filament material where the molten filament will be extruded through a heated nozzle and supposed to solidify when it reached the printing platform. Acrylonitrile Butadiene Styrene (ABS) and Polylactic Acid (PLA) are commonly found as the material of the FDM filament. Both materials are suitable for FDM as they are durable, have good mechanical properties, and have good thermal properties. However, compared to the injection moulding technique, the mechanical properties of the fabricated ABS or PLA parts are better than the printed ABS or PLA parts [3], [4]. Therefore,

there are suggestions from the researchers to add filler in both polymers for composites. The function of the fillers is to enhance the printed part's material properties as the 100% polymer is not strong enough for structural design application. Moreover, the thermoplastic filament FDM must undergo a repeated heating process that could cause degradation of the material properties. Therefore, natural fiber can be found as a filler in thermoplastic composite filament and has already been commercialized. Employment of natural fiber composite filament for FDM has enhanced the advantages of FDM. The reduced composition of polymer in composite filament could improve the environmentally friendly properties of the FDM. Moreover, as a filler in composite filament, natural fiber acts as reinforced materials and

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improves the printed part's mechanical properties. Material properties of the composites significantly depend on their constituent. Characterization of composite is necessary before the fabrication process is conducted in an FDM machine to ensure the correct printing process parameters are used to fabricate good quality printing parts.

### **MATERIAL SELECTION OF NATURAL FIBER COMPOSITE FUSED DEPOSITION MODELING**

Recently, interest in the natural fiber composite FDM has risen significantly as many researchers published their findings and reviews. Aida et al. (2021) reviewed natural fibers' capability to replace synthetic fiber in a composite filament of FDM. They concluded that the natural fiber composite filament would positively impact the environment and the composite industry. Farhan Han et al. (2021) had reviewed the development process of composite filament for FDM and the effects of the printing process on the mechanical properties of the printed composite parts [6]. Mastura et al. (2019) had reviewed essential requirements and challenges in the development process of green composite filament for FDM [7]. According to these studies, many potential natural fibers could be used as fillers for FDM filament. Huang et al. (2021) has studied wood/ABS composite of FDM, where they found that the addition of wood percentage in ABS filament could increase the stiffness of the printed parts [8]. Coppola et al., (2018) used hemp powder as filler in PLA, and they found that treated hemp powder exhibited higher elastic modulus and tensile strength than the neat PLA printed part [9]. Le Duigou et al. (2019) developed a continuous flax fiber composite for structural application using FDM [10]. This innovative method had optimized the mechanical properties of the composite. Anandkumar et al. (2021) had prepared the granulated natural fibers such as sugarcane, jute, ramie, banana, pineapple, and seashell powder using a hybrid additive manufacturing technique [11]. The hybrid additive manufacturing technique consists of FDM and shape deposition modeling. They found that the composites significantly improve the material properties of the fabricated parts.

Many researchers have reviewed the potential of natural fibers in FDM technology, and they had proved the manufacturability of the materials using FDM technology. This situation would risk the design engineer in selecting the suitable natural fiber for FDM as the natural fibers' characteristics are unique and varied. A wide range of the material properties of the natural fibers must be considered in the material selection. It is possible to use several decision-making approaches presented by many researchers in the material selection of natural fibers for various applications. Noryani M. et al. (2018) had used a statistical approach to overcome the unique material properties of the natural fiber [12]. Salwa et al. (2019) used Analytic Hierarchy Process to select the natural fibers for the food packaging industry [13]. Shaharuzaman et al. (2019) selected the suitable natural fiber for an automotive component design using the integrated approach of the Analytic Hierarchy Process and ViseKriterijumska Optimizacija I Kompromisno Resenje (VIKOR) [14], [15]. In the selection process, the requirements are identified initially to avoid misjudgment and caused damages later in the design process. For FDM applications, it is essential to consider the printing process parameter as a requirement in material selection. Agrawal (2021) had identified the important criteria for sustainable material selection for the FDM process [16]. He used the multi-criteria decision-making methods in analyzing and prioritizing the potential materials. Dandagwhal et al. (2020) suggested selecting suitable materials for FDM by considering several factors such as environment, material properties, part orientation, machining parameters, and working parameters [17]. Proper selection of the materials is mandatory in producing functional printed parts. Therefore, consideration in selecting the most suitable natural fibers for FDM should be appropriately performed. The selection criteria should consider the material characteristics and the printing process parameter to ensure the excellent quality of printed parts can be produced..

### **CHALLENGES IN FABRICATING NATURAL FIBER COMPOSITE PARTS USING FUSED DEPOSITION MODELING**

Adding fillers in polymer for a particular manufacturing process can change the properties and quality of the fabricated parts. The flow properties of the polymer are greatly affected by the addition of fillers. In FDM, the flow properties of the filament are important as the material has to undergo the heating and melting phase during the process. An adequate setting of nozzle temperature is highly required to ensure the filament is fully melted in the nozzle. There are some challenges in fabricating the polymer filled with natural fibers using an FDM machine. Inadequate preparation of fiber-filled polymer composite filament of FDM 3D printing can cause nozzle clogging, and the poorly printed part will be produced. The addition of fibers in polymer would increase the brittleness and decrease the toughness of the materials [18]. This condition would make the filament easy to break and blocked the nozzle orifice. Moreover, a weak interfacial bonding condition between the fibers and molecules of the polymer would cause poor fiber dispersion. This would cause the fibers are being pulled out and agglomerate inside the nozzle. As a result, a physical blockage is built by the accumulation of residues at the inner wall of the nozzle. In addition, there is a limitation in FDM where it can well operate with the composite that contains up to 40% of filler, and more than 40% of fillers can cause nozzle clogging [19]. Moreover, insufficient force in the feeding system can cause the nozzle clogging where the FDM machine cannot extrude the composite filament sufficiently. Therefore, understanding the flow properties of the polymer during printing, as well as the influence of the printing parameters on the flow properties, is crucial for improving the quality of the manufactured parts [20].

### **CONCLUSION**

In summary, natural fibers have the potential to be used as material for the filament of FDM. Studies from past researchers show that natural fibers are compatible with the printing process and produce good quality printing parts. Many researchers for the filament of FDM have identified several types of natural fibers. The material selection process is required to identify the most suitable natural fibers for particular printed parts that comply with the principle of FDM. However, the limitation of the FDM and material properties of natural fibers need to be overcome. Further study is required to ensure the manufacturability of the natural fibers is evaluated for all types of natural fibers.

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## DEVELOPMENT OF LOW COST AND ENHANCED DURABILITY OF SACH FOOT: PREDICTION OF MECHANICAL BEHAVIOR OF SACH FOOT USING FINITE ELEMENT ANALYSIS

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### ABSTRACT

Solid Ankle Cushion Heel (SACH) foot is one of most widely use prosthetic foot due to its low cost and easy handling. However, the metatarsophalangeal area of conventional SACH is always broken due to improper design. The objectives of this research is to improve the current design of SACH Foot and to predict the mechanical behavior using Finite Element Analysis (FEA), and compare their mechanical behavior with the current design. The improvised design of SACH foot is designed in a 3D isometric model using Autodesk AutoCAD software. Then, selection of main material and each of its component is continued in Autodesk Inventor Nastran Editor. This software is used to run Finite Element Analysis (FEA) on three different design; Design 1 (current design), Design 2 (improvised design with polyurethane), and Design 3 (Improvised design with latex). At a force of 150kg and a fixed constraint, maximum von Mises Stress for Design 1, 2 and 3 are 0.577262MPa, 3.362630MPa, and 0.578059MPa respectively. The maximum force for 1st principal stress for Design 1,2, and 3 are 0.334278MPa, 0.720254Mpa, and 0.334747MPa. Last but not least, maximum 3rd principal stress for Design 1,2, and 3 are 0.038678MPa, 0.322305MPa, and 0.038722MPa. As a conclusion, Design 2 shows the best results as it is made of polyurethane, 30% of Kevlar reinforcement and belting for stronger base, a wooden keel and a cushion heel to absorb shock.

*Keywords:* solid ankle cushion heel prosthetic foot, finite element analysis, polyurethane, Kevlar/carbon fiber

### INTRODUCTION

Lower limb amputation is an action of removing part of lower limb. Loss of limb can cause a permanent disability that will usually affect the patient's confident, physical health, mental health and mobility. Amputees patient usually have a hard time to live a normal life. In Malaysia, prosthetic foot is imported from other country and can costs up to RM7000 [1]. However, it has low durability and patients often suffers broken tips problem [2]. The common cause of amputation is vascular disease (54%) which includes diabetes, peripheral arterial disease, and cancer [3]. Apart from that, injury due to accident, surgery, and loss of limb since birth also required amputation. Statistic shows that there are 84% of lower limb amputation compared to upper limb amputation which is only 16% [3].

Most of the amputees are using their prosthetic foot for the whole day. Therefore, a very comfortable prosthesis needs to be produced to help them to get through the day. In order to produce a good prosthetic foot, the material used must be chosen wisely. There are still some issues with the current design of prosthetic leg: they are not comfortable and need to be replaced after 6 to 7 months of usage. However, the current market price is ranging from RM 1000 to RM 7,000 for a single unit of SACH which is quite a burden for patients who have a low-income background [4].

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A component mixture is crucial in producing a flexible and comfortable prosthetic foot. Previously, most of the prosthetic foot is made up of carbon fiber which is quite expensive and easily shattered when compressed [5]. In addition, Malaysia are currently relying on imported SACH foot since there are no local SACH foot production [6]. The current design has a high moisture absorption rate which leads to the low durability issues [7]. Most of the patients are having a same problem, which is broken tips of the SACH after some time. This problem happened due to the lack of reinforcement inside the tip of the prosthetic foot [8]. Therefore, this research aims to propose new design for SACH foot with the reinforcement of Kevlar fiber into PU matrix. The new SACH foot design and their mechanical behavior were analyzed by using Finite Element Analysis (FEA) method.

**MATERIALS AND METHODS**

A 3D model of improvised design was sketched in AutoCAD and FEA simulation was run. Each of the component in the SACH foot was assigned with their material, respectively. Auto-contact of material and mesh at small density was set. The test was set at single point for static analysis, at maximum load of 150 kg and a fixed constraint. Results for von Mises stress (VMS), 1<sup>st</sup> principal, 3<sup>rd</sup> principal, Stress XX, XY, XZ, YY, YZ and ZZ were analyzed and recorded.

**RESULT AND DISCUSSION**

The current design (Design 1) of SACH prosthetic foot use polyurethane as the main material. It also has wooden keel and belting as reinforcement. The measured mass of SACH was 0.8078 kg and effective area of 0.5554 m<sup>2</sup>. The belting reinforcement is rather short, which is the main reason for failure at the metatarsophalangeal area. Figure 1 is the improvised design that has a three layer extended belting made of 30 vol% Kevlar fiber belting, a cushion heel to absorbs the shock and a wooden keel to strengthen the structure. For improvised design two rubber matrix were tested; polyurethane rubber matrix (Design 2), and natural rubber matrix (Design 3). The improved design has mass of 1.4051 kg with an area of 0.5554 m<sup>2</sup>.

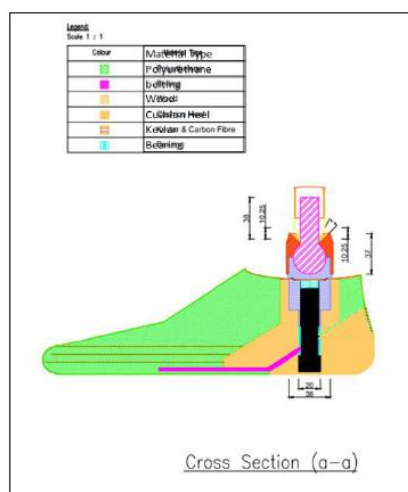


Fig. 1: The cross-section view of the improvised design (Design 2 and Design 3)

Table 1 summarizes the load distribution results for all three designs. All three model were put under the same load of 150kg, and fixed constraint mode. The reinforcement of 30% of Kevlar has improve the mechanical behavior and strength of the SACH Foot. Design 2 which is made up of Polyurethane with 30% of Kevlar Reinforcement, an addition of longer belting, a triangular shape of cushion heel and a wooden keel was proven to be the best formulation and design as it gives the highest value of von Mises Stress, 1st and 3rd Principal, and normal and shear stress when a force of 150 kg acting upon it.

For von Mises Stress, Design 2 has proven to increase the durability of the improvised design of SACH foot where the analysis shows that Design 2 have von Mises stress of 3.36230 MPa compare to Design 1 and Design 3 which is only 0.577262 MPa and 0.578059 MPa respectively.

For 1st Principal, where the maximum tensile stress induced on the particular area is calculated, Design 2 also shows the highest force which is 0.720254 MPa. For 3rd Principal, where the maximum compressive stress is calculated, Design 2 also shows the highest and best value which is 0.322305 MPa compare to other design which are 0.038678 MPa and 0.038722 MPa. In addition, Design 2 is the only design that have a medium load distribution on its model for the maximum compression of 150 kg load.

TABLE 1: Summary of load distribution results

Type of Stress (MPa)	Design 1 (Polyurethane)	Design 2 (Polyurethane-30% Kevlar)	Design 3 (Natural Rubber Latex-30% Kevlar)
1 <sup>st</sup> Principal	0.334278	0.720254	0.334747
3 <sup>rd</sup> Principal	0.038678	0.322305	0.038722
Stress XX	0.137309	0.644751	0.137374
Stress XY	0.112082	0.769137	0.112127
Stress XZ	0.187900	0.822700	0.188154

Stress YY	0.237536	0.643148	0.237847
Stress YZ	0.163987	0.450806	0.164204
Stress ZZ	0.333953	0.442849	0.334422

### CONCLUSIONS

The reinforcement of 30% of Kevlar belting to the polyurethane, a cushion heel, and a wooden keel were shown to enhanced the mechanical strength of the SACH foot. It is highly recommended to reinforce the usage of Kevlar in the polyurethane due to its impressive properties and strength shown in Finite Element Analysis.

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## THE EFFECT OF SILANE TREATMENT OF WATER ABSORPTION TOWARDS KENAF FIBRE REINFORCED POLYLACTIC ACID COMPOSITES

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### ABSTRACT

In this present of study, kenaf fibre reinforced PLA composites were subjected to water absorption tests in order to study the influence of water absorption of its physical properties. Due to pollution issues, rising global warming which mostly come from industries side, the usage of synthetic and carbon fibre has been replaced with natural fibre in way to reduce toxicity. In order to develop sustainability of product, the usage of natural fibre in composites and use biodegradable polymer can directly lead to green concept towards earth. Water absorption samples are been prepared by using 3D printing. To remove impurities, reduce the percentage of water molecule absorption and issues towards fibre surface unevenness can be done by a chemical treatment or known as pre-treatment. Treated fibre with 1% concentration of silane treatment leaving other two samples which are neat polymer and untreated kenaf fibre. Next, the samples have been immersed into water at room temperature for certain time duration. Water absorption tests were handled by followed ASTM standards to evaluate the effect of dimension stability, to prevent swelling and to check the effectiveness of surface treatment. The increasing percentage of water absorption might lead to swelling and instability of composites which clearly leads to negative effect on composites strength.

*Keywords:* Kenaf fibres, polymer matrix composites, silane treatment, surface modification, water absorption

### INTRODUCTION

Hydrophilic property is existed in natural fibre such as kenaf. This property stated due to presence of hydroxyl group (-OH) in their cellulose's structure, as (-OH) is exist in natural fibre's structure, thus the moisture content might increase gradually [1]–[5]. This moisture effect can cause swelling in structure, instability of dimension and also can lead to crack [6]. This characteristic also was disadvantage which can affects adhesion bonding between fibre and polymer matrix [2], [7].

Some research's experiment towards surface modification will endure the alkaline treatment and silane coupling agent. In order to improve the wettability of natural fibre by polymer and promotes interfacial bonding, methods such as coupling agent is used [8]. Silane is an example of coupling agent in surface modification that shows the excellent treatment and important which can improve interlocking adhesion between fibre and polymer matrix better than other treatment [5], [9], [10]. It also interacts with chemical bonds of natural fibre and polymer matrix.

Asim et. al. (2016) conducted an experiment regarding surface treatment between kenaf fibre and PALF composites. In this experiment, data has been collected among 4 different parameters which are untreated fibre, alkaline treated fibre, alkaline-silane treated fibre and silane treated fibre. From researcher's observation, stated that by endure alkaline treatment, all the impurities in fibre can be removed completely depends on alkali concentration and soaking time. By doing surface treatment, enhancement of strength in composites occur [4].

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In this paper we studied the treatment of kenaf fibre with NaOH concentration 6% for 24h followed with next chemical treatment 1% silane coupling for 3h in way to modified the surface characterization of natural fibre. This paper aims to investigate the effect of chemical treated kenaf fibres on water absorption of kenaf fibre reinforced PLA composites.

### MATERIALS AND METHODS

#### Materials

Kenaf fibre powder (un-sieve) was supplied locally from Lembaga Kenaf dan Tembakau Negara (LKTN) before being treated and mixed with Poly Lactic Acid (PLA) pellets. Poly Lactic Acid (PLA), Silane (Aminopropyltriethoxysilane Agent) was obtained from Mechsoft Sdn. Bhd.

#### Surface treatment

In this experiment, kenaf powder was treated with alkaline treatment. The kenaf fibre were immersed in sodium hydroxide solution with concentration is fixed, 6% for 24 hours [11]. After alkaline treatment, the fibre was washed thoroughly with running water and been dried in oven at 110°C 24h. Surface treatment is then followed with silane coupling agent method. In this treatment 1% was dissolved in solution which contain 70% of methanol and 30% of water. Next the solution was stirred for 30 minutes. Then the kenaf fibre which already endure the alkaline treatment was then soaked in silane solution for 3 hours and been dried in oven for 110°C 24h to remove all the moisture content in fibres.

#### Composites mixture

The fibre and matrix were prepared using the law of mixture formula. The weight of elements is being calculated using equation (1).

$$\text{Weight Percentage of Element, } W_e \times \text{Weight of composites} = \text{Weight of elements} \quad (1)$$

#### Extrusion filament

Twin screw extruder has been used to produce filament composites with different of parameter. With constant pulling speed (25.5 rpm) and constant filament size which is 1.75mm [12].



Fig. 1 (a) Kenaf fibre PLA composites filament (b) Neat polymer filament

## RESULTS AND DISCUSSIONS

During chemical treatment process, many voids have been remove including the chemical content of natural fibre such as hemicellulose, lignin, cellulose and any other impurities. This process led to enhancement of bonding between fibre and matrix, fibre wetting characteristic and mechanical interlocking [13].

The most critical issue that natural fibre needs to overcome is its hydrophilic nature. The hydrophilic characteristic of natural fibre can cause the absorption of water in fibre, the growth of fungi on natural fibre surface, the swelling of natural fibre, the change of dimension, and rotting due to fungi attacks. The hydrophilicity comes from lignocellulose that strongly polarised with hydroxyl groups. If the wetting of the fibre-matrix occurs, the weak interfaces between the two phases will occur. The swelling that occurs due to the absorption of water will lead to poor mechanical properties of the composites. Many surface modifications are available, such as alkaline treatment.

During the water absorption test, neat polymer, untreated composites and treated composites are immersed in water for 24 hours and the reading was taken every 2 hours by following ASTM D570-98. The final mass indicates the penetration of water that enter the composites and swell. The swelling that occurs will change the dimension of the composites and micro cracks will start visible. The longer the time of immersion, the larger amount of water will penetrate the interphase of the composites through the micro cracks and the detachment between fibre and matrix will occur. As can conclude in water absorption of composites issue, it is very important especially for outdoor application [14]. Akil (2011) stated that water absorption may occur due to fibre dispersion in matrix, permeability of the fibres, void content temperature and etc. [15].

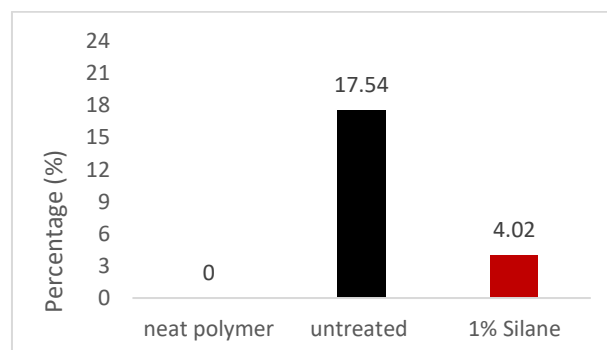


Fig. 2 Percentage of water absorption of composites and neat polymer at 24 h

The water absorption of fibre reinforced polymer composites has been investigated. As seen in Fig. 2, there is no percentage increment for neat polymer as the polymer can resist water molecules better compared to the fibre due to the hydrophobic

properties of polymer plastic. Unmodified kenaf fibre has a wetting problem compared to modified fibre thus it might have higher cellulose content, which attracts more water molecules as we can see in figure untreated obtained the highest percentage of water absorption compare with treated kenaf fibre. More cellulose content allows more water to penetrate the fibre-matrix interphase, which can lead cracking in microstructure and swells. The longer the immersion time, the larger amount of water will penetrate the interphase of the composites through the microcracks and the detachment between the fibre and the matrix will occur. If there were small cracks such as porosity, as more water that penetrated will enter the small cracking and make the small crack become larger. The water absorption behaviour of the fibre reinforced composites needs to be properly analysed, especially for outdoor products [15]. Akil (2011) stated that water absorption might occur due to the fibre dispersion in the matrix, fibre permeability, and void content temperature [15]. Neat polymer can resist water well because the polymer is hydrophobic, hence the polymer cannot react with hydroxyl groups, in contrast to natural [14], as illustrated in Fig. 3. The modified composites can resist water well due to the surface treatment that roughens the surface and enhances the bonding between the fibre and the polymer; therefore, the composites can resist water molecules in the long term. Logically that we can conclude, the layer of surface modification can resist the water molecules from absorption process occur. Neat polymer can resist water because of hydrophobic characteristic where they cannot react with hydroxyl groups contrast with natural fibre [14].

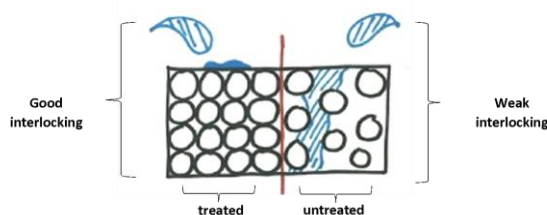


Fig. 3 Different penetration of water between treated and untreated composites

## CONCLUSIONS

The effect of surface modification on natural fibre towards water absorption has been investigated thoroughly in this paper. Silane treatment on natural fibre were studied in way to reduce the hydroxyl group that naturally contain in natural fibre and enhance the adhesion bonding between natural fibre and polymer matrix. Untreated fibre composites have highest percentage of water absorption compare with treated composites. In results and discussion which proves that treated fibre can prevent water molecules better due to surface modification. Silane treatment with 1.0% of concentrated can reduce the chemical content that naturally exist in natural fibres such as cellulose and hemicellulose. As can conclusion, surface modification is a success method in reduce the hydrophilic properties which leads to major failure in order to replace synthetic fibre with natural fibre in application industries.

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## FLAME RETARDANCY EVALUATION OF NANOCELLULOSE BASED POLYMER REINFORCED COMPOSITES

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### ABSTRACT

The rapid development and widespread usage of polymer materials have brought attention from researchers to bring new applications in our daily lives. However, most polymeric materials are characterized as having low flammability properties as they are easily ignited and release heat and even toxic gases during the combustion process. The enhancement of polymeric-based materials via the incorporation of flame-retardants has been widely reported. The flame retardant materials include halogenated and halogen-free flame retardants, phosphorus-containing compounds, and metal hydroxides have been widely used to be incorporated into polymer materials but were not considered to meet the requirements of high performance and eco-friendly polymer applications. Nanomaterials are a new class of material that can offer multi-functional applications, including flame-retardant fillers. Nanoscale flame retardant materials can offer a marked reduction in flammability of polymer at a very low loading level (5 wt. %) such as a decrease in heat-released rate (HRR) and mass loss rate (MLR). To date, inorganic materials from natural fibers, viz. nanocellulose, have attracted growing interest in the reduction of flammability of polymers over conventional flame retardant materials with better flame retardancy properties.

*Keyword:* Flame retardant, natural fibers, nanocellulose, polymer nanocomposites

### INTRODUCTION

Nanocellulose is a renewable and biodegradable nanomaterial that possesses high strength, low density, high surface area, and tunable surface chemistry which allow controlled interaction with the polymer. Nanocellulose is known as a natural biopolymer mainly derived from lignocellulosic materials. The cellulose constituents are usually extracted chemically and mechanically to remove the undesirable constituents viz. lignin and hemicellulose [1]. Nanocellulose has a nanometer range with one of its dimensions (<100 nm) and it can be differed based on its class includes cellulose nanocrystals (CNCs), cellulose nanofibrils (CNFs), and bacterial nanocellulose (BNC). In general, nanocellulose has attractive mechanical properties including tensile strength (10 GPa), stiffness, Young's modulus (150 GPa), and low density (1.6 g/cm<sup>3</sup>). with superior barrier properties, flexibility, transparency, low thermal expansion coefficient which made this material an incredible candidate for various applications for instance for biomedical and biosensing, food packaging, water filtration membranes, electrical and electronics materials, and also for paint and coating materials [1], [2]. However, despite the excellent properties, nanocellulose suffers from certain conditions associated with highly flammable properties, moisture absorption, and the high tendency of agglomerations which have always been the major concern that considered for improvements.

Until now, scientists and researchers are working on developing nanocellulose based materials with enhanced properties while at the same time maintaining their inherent excellent attributes to achieve high value-added applications. One of the attributes that have been the focus includes the flammability properties. The fact that nanocellulose has gained so much attention to be used as bio-reinforcement for polymeric materials for many applications, the important characteristics like high inflammability properties are required as one of the important features for the application of high-end products. Tong et al. [3] highlight that the nanocellulose imparts low properties in flame retardancy thus restricts its applications in some fields with safety concern such as thermal insulations for building materials [4], supercapacitors [5], [6], and separators for lithium-ion battery [7]. Thus, upgrading nanocellulose via the addition of flame retardant materials or chemically grafting the nanocellulose with flame retardant-based materials are necessary to increase the value of the end-products especially those that required high flame retardancy and fire safety. This factor is very important especially for designing the building materials, electronics, furniture,

textile, etc. particularly during the event of a fire that the flame retardant materials will delay or block the flame propagation which is directly related to the survival of humans [8]. A composites design strategy using embedded nanocellulose can demonstrate a variety of advantageous material properties. Therefore, the development of nanocellulose reinforced composites with desirable flame-retardant properties are considered crucial to satisfy the ever-increasing demands.

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### FLAME RETARDANCY AND FLAMMABILITY CHARACTERIZATION

Fire hazard is a combination of different factors including ignitability, amount of heat release, ease of extinction, weight loss, flame spread, smoke index, and toxicity [9]. There are a variety of readily combustible

materials, for instance, cellulosic materials and made-made polymers which contribute to the factors of ignition and rapid spread of fire in vehicles, residential buildings, and other facilities [10]. In order to achieve fire safety requirements, different solutions have been developed including the through chemical and physical strategies to prevent one material from burning or to lower the heat release amount [11]. Therefore, flame retardant materials have been introduced which refer to various groups of chemicals that are applied to synthesis materials such as polymer to impede or slow down the combustion process. Flame retardants may be based on phosphorus compounds (e.g. inorganic and red phosphorus), halogen compounds, silicon (e.g. silicones, silica), minerals (e.g. hydrocarbonates, metal hydroxides, and borates), and nanometric particle materials (e.g. layered (nanoclays), fibrous (nanotubes), and particulate materials) [12], [13]. The addition of these flame retardants to polymers, fibers, and papers can help to protect the sample from burning. Therefore, the role of flame retardants was seen very important especially for many products available in the industry.

Flammability characterization is a method used to evaluate the flame retardancy of one sample either in gas or solid phase, by studying the morphology and composition of the char layer. The common characterization methods used are limited oxygen index (LOI), UL-94 (Underwriters Laboratories) test, and cone calorimetry test. LOI is the most popular method used in many years to investigate the relative flammability of materials [14], [15]. It measures the minimum concentration of oxygen that supports materials combustion. Materials with less LOI value of 21 % are known to burn easily, whilst materials with higher than 21% LOI value are indicated as good flame retardant as it exhibits reduced flammability after removal from the ignition source. Meanwhile, UL-94 tests were used to evaluate the burning properties of the sample. It can be divided into several types of testing according to the types and dimensions of the samples. UL-94 vertical tests are widely used to determine the ignitability and flame spread rate of materials, where the samples are burnt using specific flame conditions according to a certain period. The time required for fire to be extinguished becomes the indication of the fire retardancy level of the sample [15]–[17]. Another important characterization method to evaluate the flammability properties is cone calorimetry which measures the reducing oxygen concentration in the combustion gasses of materials subjected to a given heat flux. The sample was exposed to the conical radiant electrical heater, creating an electrical spark to trigger the combustion. Through this evaluation, important data can be reported such as heat release rate (HRR) as a function of time, peak of heat release rate (pHRR), total heat release rate (THR), and mass loss rate (MLR) [10], [16].

#### **NANOCELLULOSE FOR FLAME RETARDANT APPLICATIONS**

Nanocellulose is considered as a natural biopolymer derived from cellulose materials which exist abundantly in nature as a constituent of plants and microorganisms [18]. It was known to possess a variety of remarkable properties, however, the flammability properties of nanocellulose, nonetheless, hinder its practical applications. Being organic, cellulose-based fibers and their products thereof pose considerable fire risk attributed to their high cellulose content. This phenomenon can be ascribed to the weaknesses of cellulose and most of its derivatives which tend to burn with a limited amount of char and led to the high flammability of the materials [18], [19]. In general, cellulose material decomposed in the vicinity of 260 – 350 °C and produced by-products of flammable volatiles and gasses, non-combustible gasses, tars, and some chars [20]. Therefore, researchers have been focused on developing flame retardant nanocellulose while maintaining its unique properties for varying technological purposes.

To date, nanocellulose has been developed in the application associated with fire-resistant materials. However, due to its high flammable characteristics, nanocellulose is commonly hybridized with fire retardant agents to impart flame retardancy properties [21]. A rare combination of good thermal stability and low flammability can be provided of nanocellulose with flame retardants-based materials. Halogen, phosphorus, nitrogen, metal ions, and nanofiller-based flame retardants are usually utilized to be combined their function with nanocellulose to impart inflammability properties, with halogen-based flame retardants have been reported to be the most effective [22]. Nevertheless, despite remarkable improvement in flame retardancy, high loading of reinforcement is needed to achieve the satisfactory flame retardant effect through the formation of layers of barriers which then cause unfavorable optical transparency and mechanical flexibility of the nanocellulose based materials. In addition, the applications of most flame retardants especially halogen-based led to a negative effect on both ecology and health as the halogen possesses toxic compounds produced during the process as it reacts with polymer matrix and reaches out to the environment and accumulates in living beings. These occurrences are usually associated with the formation of hazardous smoke which can cause death under practical situations [23]–[25]. Therefore, nowadays there are attempts of constructing nanocellulose based materials with chemical grafting with the flame retardant groups for example by attaching phosphor-containing groups with flame retardant function with CNF during the pre-treatment of cellulose fibers. Salmeia et al., [18] in their study mentioned the suitability of cellulose to impart flame retardant properties owing to the structure containing hydroxyl groups. The mechanism of flame retardancy using phosphorus-based flame retardant is by dehydrating the cellulose constituent as the carbon source during the thermal decomposition process, thus leading to the formation of char instead of volatile species. The materials are then insulated by the layer of char, retarding the spread of fire [22]. The effectiveness of nanocellulose modified by phosphorylation method to increase the flame retardancy was also reported by Ghanadpour et al. [23] in their study. The nanocellulose was produced from sulfite dissolving pulp fibers, chemically treated with phosphorus compound to produce cellulose nanofibers with a width of approximately 3 nm. The phosphorylation process was done using diammonium hydrogen phosphate, (NH<sub>4</sub>) HPO<sub>4</sub> in the presence of urea. The finding was found the improvement in the inflammability properties of CNF with the presence of phosphate group in the structure of CNF. In addition, the nanopaper sheets prepared from phosphorylated CNF also showed self-extinguished properties during horizontal burning test using methane flame subjected for 3 seconds and cone calorimetry test subjected to cone calorimeter heat flux. Meanwhile, Wicklein et al. [27] also study the effect of crosslinking boric acid with nanocellulose to improve flame retardancy and ignition resistance. The mechanism of the flame retardancy was attributed to the crosslinking at the alkaline condition with the formation of thermally

stable borate ester bonds. The boric acid crosslinking modifies the thermal degradation properties of nanocellulose by transforming the cellulose structure into stable aromatic char during thermal annealing and the evolution of flammable volatile levoglucosan is small. The authors also reported the effectiveness of treated nanocellulose – borate to impart ignition retardancy when reinforced together with sepiolite nanoclay when tested with radiant heat exposure in cone calorimetry testing, with maintained integrity even after prolonged heat exposure. These results prove the concept, the potential of nanocellulose materials to be used as sustainable and comparatively environmentally friendly flame retardant materials.

## NANOCELLULOSE BASED POLYMER REINFORCED COMPOSITES

Polymeric materials have been widely applied in many areas of industries including automotive and aerospace interiors, electronic devices, and insulation building materials. However, most polymers are highly flammable which becomes a threat to the safety of life and property [28]. A combustible polymer with the presence of oxygen and heat generates combustible volatiles causing mass loss. Then, the char layer was formed as a by-product of the process in the condensed phase during the decomposition. Intrusion or absence of one or more of these steps will consequently terminate the fire spread [17]. Therefore, a different solution has been developed to meet fire safety and diminish fire hazards, particularly in polymer-based products. The utilization of flame retardant materials has been recognized as a fire safety tool capable of lowering the possibilities of fire injuries and death. Flame retardant materials are commonly introduced to polymers, fibers, and papers to impart the fire retardancy of its final product. Thus, the use of flame retardant materials is considered a crucial part of polymer composite formulations [10].

The application of flame retardant materials in nanocellulose based polymer reinforced composites have been widely reported with the aim to achieve suitable inflammability properties while maintaining other important properties for them to be applied for practical applications. For example, Guo et al. [29] used cellulose nanofiber (CNF) in the form of composites foam or aerogel and combine it with hydroxyapatite (HAP) to produce alternative materials for common traditional petroleum-derived insulating materials like polyurethane foam (RPUF) and expanded polystyrene (EPS). The resultant HAP-CNF composite foam was found to impart a very low heat release rate (20.4 kW/m<sup>2</sup>) and total heat release (1.21 MJ/m<sup>2</sup>) with thermal conductivity value in the range of 38.5 – 29.1 mW/(m.K). Besides, the vertical burning test was also observed to have excellent fire-resistant and self-extinguishing behaviors. This finding proves the capability of nanocellulose based materials to be significantly applied for fire safety materials for building insulation materials. In another separate study, Guo et al. [26] prepared CNF aerogel via freeze-drying and post crosslinking method with N-methylol di-methylphosphonopropionamide (MDPA) and 1,2,3,4-butanetetracarboxylic acid (BTCA) as the co-additives. Through the evaluation, the CNF/BTCA/MDPA aerogel showed better flame retardant performance with outstanding self-extinguishing behavior and higher char yield compared to that neat CNF aerogel. This finding proved the prospective of nanocellulose based material to be used for thermal protective equipment with promising insulation properties.

Other than that, nanocellulose was also commonly reported to be incorporated with phosphorus compound, however, it usually resulted in deterioration to the mechanical strength and thermal stability. To date, researchers are working on improvements particularly on the types of flame retardant materials to be reinforced in the composite system. For example, Wang et al. [28] have developed an improved version of reinforcement using an interlayer-confined synthesis of multilayer zirconium phosphate-reduced graphene oxide (ZrP-RGO) nanoplates to be incorporated into cellulose nanofibers to fabricate the hierarchical nanocellulose composites through a structural inspiration of nacre. The bio-inspired lamellar barriers consist of highly aligned ZrP-RGO nanoplates as well as planar orientation contribute to lowering the heat and mass transfer between the flame zone and underlying matrix which attribute to the 75.1 %, 71.4 %, and 54.6 % reduction in the peak of heat release rate, peak of smoke release rate and peak CO production rate in nanocellulose composites, respectively. These results presented the enhancement of hierarchical nanocellulose composites with better thermal stability, mechanical strength, and toughness compared to conventional nanocellulose composite.

## CONCLUSION

To date, polymer reinforced in bio-based composites seems to become a huge trend attributed to the environmentally friendly of their synthesis and applications. Despite its remarkable importance in the industry and irreplaceability in human being's life, the application of polymer bio-composites has nonetheless created concerns to the need to reduce as much possible the risk of loss of possession, health, and life, imparting from the threat of the flammability of polymer materials. Fact that the flammability of polymer bio-composites remains a very complex problem, different strategies have been initiated to overcome their flammability. Nowadays, nanocellulose based polymer composites for flame retardant application have been continuously reported and become one of the popular topics among researchers for the development of high value-added products derived from green materials. The combination of nanocellulose and flame retardant materials for reinforcement in polymer composites was able to replace the application of conventional flame retardants and impart better effects towards the environment and living beings by reducing the formation of hazardous by-products such as toxic gases, at the same time enable to save a life during the event of a fire.

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**WOOD DUST FIBRE COMPOSITE TOWARD SUSTAINABLE FUSED DEPOSITION MODELING (FDM) FILAMENT**Nafis Syahmi Z.A.<sup>1</sup>, M. Nuzaimah<sup>2\*</sup>, Syahibudil Ikhwan A.K.<sup>1</sup><sup>1</sup>Faculty of Mechanical Engineering, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.<sup>2</sup>Faculty of Mechanical and Manufacturing Engineering Technology, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.**ABSTRACT**

Currently, research and development of materials, particularly from the bio composites industry, is being pushed forward by the increased demand for sustainable materials. Bio-composite based on natural fibre reinforcement has been used widely in many applications, such as body panels on transportation vehicles, X-ray couches on medical devices, performance footwear on sporting goods, and other related applications, including applications as fused deposition modeling (FDM) filament. The selection of natural fibres that are suitable to be incorporated into the matrix to develop the FDM filament bio-composite is crucial to meet the existing FDM filament quality. Next, configuring process parameters for natural fibre composite on extruder machines is important to get the size, suitable temperature, time, and speed of the extruder to get a good filament and consistent size. Past researchers have studied the various suitable natural fibres, and the process parameters of the natural fibre composite on extruder machines for the FDM filament fabrication process (FFP).

*Keyword:* natural fibre composite, biodegradable, fused deposition modelling

**INTRODUCTION**

Several advantages of natural fibres such as wood and sugar palm fibre include that they are renewable, recyclable, have a low specific gravity, and have a high specific strength. Modern life necessitates the use of environmentally friendly, cost-effective, and healthy items that are more environmentally friendly, cost-effective, and healthy. Forestry dust is essentially the remnants of tiny particles found in the furniture business, pulp and paper industry, and other industries [1]. It can form pile shapes and, in most cases, burn, causing environmental contamination. Among the efforts that may be used for a range of applications, including 3D printing technology, is the manufacture of bio-composites from waste polymers and natural fibres such as recovered polypropylene (PP) and wood dust [2], [3]. When it comes to fused deposition modelling, the thermal and rheological properties of the filament materials are essential, as the method is dependent on the extrusion of a heated feedstock filament during extrusion. High viscosity materials and polymers with low glass temperatures, excellent melt properties, and excellent melt qualities are appropriate for this application. In other words, there are several ways to do sustainable work, like making treatment. Treatment with NaOH is common for natural fibres because it is generally easy, inexpensive, and provides excellent results in improving adhesion between the filler and the matrix [4]. NaOH alters the surface of the fibre by mechanically removing it, resulting in increased surface roughness and, as a result, improved matrix coverage on the fibre.

As many academics published their findings and reviews, interest in natural fibre composite FDM increased substantially. Aida et al. (2021) reviewed the possibility of natural fibres to substitute synthetic fibre in FDM composite filament [5]. In addition to being simple to use, fused deposition modelling (FDM) has a minimal environmental effect, is inexpensive, is possible with complicated designs, and has a short lead time. FDM is an additive manufacturing technique [6]. This method has been used in a variety of product design fields, including medical devices, automobile components, food packaging, and home décor. A thermoplastic filament has traditionally been used in FDM to produce three-dimensional things by stacking materials such as acrylonitrile butadiene styrene (ABS), polylactic acid (PLA), and polycarbonate (PC), with bio-composite filament being the most recent material to be introduced. Researchers have increased their efforts in recent years to develop alternative bio-composite filaments for FDM by mixing thermoplastic materials with natural fibres, which they hope will be more environmentally friendly. Locally available natural fibres such as kenaf and maize, as well as oil-palm empty fruit bunches and wood, are examples of materials that can be used because of their low cost, availability, biodegradability, and possibility for future use. In recent years, a large number of researchers have discovered a variety of ways to decrease the cost of FDM filament for use in FDM printers.[7]–[9].

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Raw materials are pellets in which a feed zone transfers hopper pellets to the barrel to start the longitudinal movement of plastic through the rotating action. Rod heaters are used to melt materials and screws are utilised to feed the input raw materials along the barrel longitudinally. The screw is made up of three areas, namely feeding, melting, and transition. Heaters assist the plastic to create contact with the chamber wall to accelerate its rubbing. Due to the compression, shearing and heating created in the barrel, the plastic granules are liquefied [10]. The melted plastic then passes through the measuring area. In this zone, the vibration diameter stays constant, and the molten plastic poured into the extruder dies under high pressure. The filament size is usually 1.75 mm or 3 mm in diameter. Since the filament is in hot

condition when extruded through the die, it is cooled using a cooling system such as water or a fan. A puller is also employed in some circumstances. [11]. The puller is coupled to a filament winder that rolls the filaments onto a spool. The simpler filament extruder machine is shown in Figure 1.

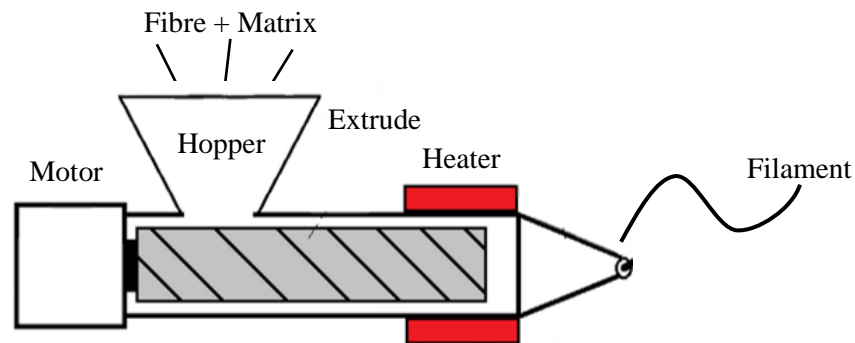


Figure 1: Schematic of extruder filament [10]

### SELECTION OF FIBER MATRIX COMPOSITE FUSED DEPOSITION MODELING

The natural fibre sources that existed in the past are discussed in detail in the section on bio composites, which is the most relevant portion. Bio composites are defined as materials that are created by combining a matrix, which is frequently resin, with a reinforcing element, which is generally comprised of natural fibres obtained mostly from plants or cellulose in the case of cellulose composites. It is the word cellulose that is used to describe the fundamental properties of fibre and matrix materials such as wood dust and polypropylene [12].

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Chosen of polymer thermoplastics because can reaching a specific temperature, plastic material melts into a soft, malleable form, which solidifies when the temperature is reduced. Thermoplastics can be melted and reshaped an unlimited number of times [16]. To facilitate the moulding process, they are typically stored in the shape of pellets. Acrylic, polyester, polypropylene, polystyrene, nylon, and Teflon are just a few of the thermoplastics that are commonly used. These materials are used in the production of a wide range of items, ranging from garments and non-stick cookware to carpets and laboratory equipment. Most of the time, the colour of this material is transparent and extremely bright. Regarding the product that may be manufactured with this material, it is coincident and appropriate. Thus, consideration should be given to selecting the most suitable fiber matrix composite for FDM. In order to guarantee the good quality of the printed components, the selection criteria must take into account the material properties and printing process parameters.

### DIFFICULTIES OR STRUGGLES IN CONFIGURING PROCESS PARAMETER NATURAL FIBER COMPOSITE ON EXTRUDERS MACHINE FOR FUSED DEPOSITION MODELING

Finding process parameters that are suitable for the extruder machine is complicated because they are related to various types of other money attributes depending on the mixture of additional materials. The mixture of additional materials is a mixture of natural fibres and matrix materials that require tuning because formal characteristics are not designated in general as usual materials frequently used by FDM, such as The important process parameters include the size, suitable temperature, time and speed of the extruder, all of which are significantly different from the material typically utilised, necessitating a bit more time and expense to determine the right parameters for the bio composites produced. Additionally, the issue with FDM is magnified if the filament size is not constant, since this results in nozzle blockage and the production of components that do not print [17]. The addition of fibres to the polymer increases its brittleness and decreases its strength. This situation will easily break the filament and obstruct the nozzle hole. Additionally, insufficient interfacial interaction between fibres and polymer molecules results in poor fibre dispersion. This results in the fibres being drawn out and accumulating in the nozzle. Apart from that, Other issues will manifest themselves on the machine, which must undergo maintenance owing to congested physical circumstances. As a result, the machine must be re-calibrated in terms of platform, temperature, printing axis nozzles that are X, Y, and Z, and others [18].

As a result, selecting a suitable filament has a significant impact on the process settings configured on the extruder machine to prevent this issue. By adding fillers to a polymer during a manufacturing process, the characteristics and quality of the

produced components may be altered. Fillers have a significant effect on the flow characteristics of the polymer. The filament's flow characteristics are critical in FDM since the material must endure a heating and melting phase throughout the process. Appropriate nozzle temperature setting is critical to ensure the filament is completely melted in the nozzle. Fabricating the polymer filled with natural fibres using an FDM machine presents several difficulties. Inadequate preparation of the fiber-filled polymer composite filament used in FDM 3D printing may result in nozzle blockage, resulting in a badly manufactured object. The addition of fibres to polymers increases their brittleness and decreases their toughness. This situation weakened the filament and clogged the nozzle aperture. Additionally, a weak interfacial bonding state between the fibres and the polymer molecules would result in poor fibre dispersion. This results in the fibres being drawn out and aggregating within the nozzle. As a consequence of the buildup of residues on the nozzle's inner wall, a physical obstruction is formed. Additionally, FDM has a restriction in that it can only work with composites containing up to 40% filler, and more than 40% filler may clog the nozzle [19]. Additionally, inadequate force in the feeding system may clog the nozzle, preventing the FDM machine from adequately extruding the composite filament. Thus, it is critical to understand the flow characteristics of the polymer during printing, as well as the effect of printing settings on the flow properties, in order to improve the quality of produced components.

## CONCLUSION

In conclusion, natural fibers such as wood dust, sugar palm and others have great potential through the production of filaments for FDM, which is important through the production method of the extruder machine to produce good quality printing parts. The selection of fiber matrix composite fused deposition modeling is important to ensure that industrial sectors such as timber can be sustainable. Furthermore, the finding of the right process parameters is very helpful in facilitating printing on FDM that needs to get the material properties for this fiber matrix composite. The usage of bio composites in automotive technology, such as insulator bolting in autos, and power energy are being researched. The use of thermoplastic resins in wind turbine designs, automation to reduce costs and labor content, and joinable pultruded wind turbine components are all being researched as potential uses.

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## FIBRE MODIFICATION/TREATMENT FOR BIO-COMPOSITES: A SHORT REVIEW

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### ABSTRACT

Sustainable development and increased environmental concerns have diverted researchers focus towards natural fibre-based composites from various bioresources as well as agricultural wastes. Natural fibres are renewable, bio-degradable, requires low energy consumption, lightweight and abundant in nature which makes them a promising material to be utilized for the development of bio-composites. However, there are some limitations of natural fibres that need to be overcome such as its high hydrophilicity that would cause incompatibility with a hydrophobic polymer matrix. Therefore, fibre treatment is essential prior to fabrication of bio-composites in order to ensure good interaction between fibre-matrix, which would create strong interfacial adhesion and subsequently improves the properties of the bio-composites. Various surface modifications of fibres have been proposed in past research works and the findings have been proven to be successful. This review covers various types of fibre surface modifications and application of bio-composites.

*Keywords:* natural fibre, surface modification, bio-composites, polymers.

### INTRODUCTION

Natural fibres (animal based and plant based) are classified based on their origin and specific applications. It has been widely utilised as the reinforcing filler in polymer composites attributing to its low cost, eco-friendliness, renewability and environmental sustainability [1]. The main constituents of fibres are cellulose, hemicellulose, lignin, pectin and wax, which vary depending on the climatic condition, type of soil as well as extraction method [2]. The chemical composition of selected natural fibres is shown in Table 1.

Table 1. Chemical composition of selected natural fibres [3]

Fibres	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Flax	56.0-71.0	15.0-20.6	2.2-6.0
Sisal	47.0-78.0	10.0-24.0	7.0-12.0
Kenaf	31.0-57.0	15.0-21.5	8.9-21.5
Pineapple	70.0-82.0	-	5.0-12.0

Cellulose, the major constituent of natural fibre originates from plant polymer which consists of D-anhydro glucose (C<sub>6</sub>H<sub>11</sub>O<sub>5</sub>) and other different hydroxyl groups [4-5]. Cellulose is highly crystalline in structure and provides mechanical strength to the fibre meanwhile hemicellulose has random amorphous structure with lower strength [6]. Various natural fibres have been used as a reinforcement in polymer composites namely flax [7], kenaf [8], oil palm [9], banana [10], sisal [11] and etc. The common problem faced by all types of natural fibres are its hydrophilic nature that leads to high moisture absorption and subsequently decrease dimensional stability of the bio-composites. Therefore, fibre treatment or fibre surface modification is found necessary to be carried out prior to incorporation of natural fibre reinforcement into the polymer matrix.

### BIO-COMPOSITES

Bio-composites are one of the materials that provide easy processability, productivity and cost reduction [12]. Incorporation of natural fibres into the polymer matrix resulted in a lightweight material since the density of natural fibre is ~1.2-1.6 g/cm<sup>3</sup> while glass fibre is 2.4 g/cm<sup>3</sup>. Reduction in the composite weight has been one of the factors for its increasing demand in various industrial sectors such as automotive, interior linings (roof, side panel lining, wall), furniture, packaging, construction, and etc. [4], [13].

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A great number of research have been conducted to investigate the effectiveness of fibre surface treatment on the physical and mechanical properties of bio-composites. The properties of fibre-reinforced composite materials are dependent on a number of factors i.e., magnitude and proportion of the fibre-matrix elasticity, type and properties of the matrix (ductile or brittle), fibre condition (content, length, and orientation) and interfacial bond strength in the fibre-matrix [14], [15]. However, the bond strength of composite is highly depending on the effectiveness of natural

fibre as reinforcing filler in the composite, which resulted from good fibre-matrix interlocking. Therefore, it has been of great interest to modify the fibre surface by various treatments in order to improve the properties of bio-composites aimed for a wide number of applications and commercialization.

## NATURAL FIBRE TREATMENTS

Fibre treatments are classified into physical, chemical and biological (enzymatic) techniques. Each technique possesses its own merits and demerits that should be considered prior to being applied for the targeted fibre. Plasma treatment is a physical technique that is utilised to modify the surface of various natural fibre by introducing various functional groups onto the natural fibre surface. The functional groups can form strong covalent bonds with the matrix leading to strong fibre-matrix interface. In addition, surface etching due to plasma treatment may improve the surface roughness and results in better interface with the matrices through mechanical interlocking [16]. However, it is rarely used for the surface modification of fibre for composite reinforcement due to high cost of machinery and helium gas that is mostly preferred as carrier gas [1]. Chemical treatment such as alkaline, silane, acetylation, peroxide, potassium permanganate and etc. involves the removal of impurities, substitution of hydroxyl groups presents on the fibre surface and simultaneously increases its surface roughness. This would enhance the interaction between fibre and matrix, thus improving the mechanical properties of bio-composites. A study by [17] treated *Carica papaya* fibres with 5% concentration of NaOH by varying the soaking time from 15 to 90 min at room temperature. They found that the fibres treated at 60 min with 5% alkaline solution showed the optimum results which showed that complete elimination of hemicelluloses and lignin from the fibre and simultaneously improved the fibre's surface roughness. Another work reported that composites with silanized jute fibres showed about 20% lowered moisture at equilibrium. Silane treatment reduces the amount of hydroxyl groups available on the jute fibres and the composites resulted in increased (~30%) static characteristic values compared with unmodified composites at standard humidity [18]. Enzymatic treatment is an eco-friendly method of fibre surface modification as it does not discharge harsh effluents to the environment and mild treatment condition compared to chemical techniques. Seghini and co-workers treated flax yarn by using commercially available enzymes. The results showed that the enzymatic treatment resulted in a cleaning effect on the fibre surface, with an increase in thermal stability [19]. Besides effectiveness in the fibre treatment, utilization of enzyme has also been associated with high material cost and restricted its usage. Therefore, extensive works need to be carried out to cater the need and fill the lapses on this issue. Based on the previous works reviewed, it can be observed that fibre surface modification has successfully improved the interfacial adhesion between fibre and matrix, thus improving the properties of the bio-composites.

## CONCLUSIONS

Physical, chemical and biological treatments of natural fibres have been explored in many published works mainly emphasising the need to develop better composite materials. Based on the past works reviewed, it has been proven that surface treatments of natural fibres have achieved substantial progress in the treatment of reinforcing fibre in the composites. As technology emerges, efforts should be sustained to broaden research studies on sustainable treatment approaches with the point of convergence on the environmentally friendly options.

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## DEVELOPMENT OF 3D PRINTING FILAMENT MATERIAL USING RECYCLED POLYETHYLENE TEREPHTHALATE (PET) REINFORCED WITH COCONUT FIBER

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### ABSTRACT

Material savings in manufacturing are being driven by additive manufacturing (AM) and 3D printing. Due to the continuous growth of 3D printing, assisted by low-cost entry-level material extrusion printers, the sustainability of this popular method of additive manufacturing is crucial. As a result, recycling 3D printed waste and 3D parts at the end of their useful life is crucial. The objective of this research is to boost the biodegradability of 3D printing filaments and to replace oil-based feedstock with bio-based biodegradable plastic. The researchers created coconut Fiber/polyester composites using an unsaturated polyester resin generated from glycolized polyethylene terephthalate (PET) waste as a matrix. The recycling of PET waste was accomplished through a glycolysis and poly esterification reaction, culminating in the synthesis of an unsaturated polyester resin composition (UPR). To optimise the adherence of coconut Fiber to polyester resin, we applied varied concentrations of alkali, silane, and silane to alkalized Fiber. We studied the effect of water uptake on the sorption properties of composites by immersing them in distilled water at room temperature.

*Keywords:* 3D printing, coconut fiber, polyethylene terephthalate (PET).

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### INTRODUCTION

Numerous environmental issues have arisen as a result of the rising usage of plastics and industrial waste. This has caught the attention of scholars who are looking for novel solutions to the problem. PET is a flexible plastic used in the manufacture of packaging films and bottles. PET is not a direct environmental threat, but it may be considered a pollutant substance as a result of the growing amount of plastic rubbish and its high resistance to atmospheric and biological degradation. As a result, not only would PET recycling reduce solid waste, but it will also conserve raw petrochemical resources and energy. Several studies were undertaken to determine the compatibility of polyester with coconut fibre in order to create coconut composites. However, there is no research on the usage of coconut fibre as a polyester reinforcement made from recycled PET [1]. Coconut Fiber has a number of advantages over other natural fibres, including enhanced failure strain and weather resistance due to the presence of lignin. Due to the lower cellulose concentration in coconut fibre, it absorbs less water. Coconut Fiber, like other natural fibres, has been shown to be a poor reinforcement material due to its broad and variable diameter, high microfibril angle, and high lignin and hemicellulose content [1]. 3D printing is a word that refers to a process for rapidly and simply generating three-dimensional items using digital computer-aided design (CAD) data. Today's 3D printers are capable of processing a broad variety of materials and generating completely functional components. 3D printing technology have been used to research robotics, vehicle components, weapons, medicine, and space exploration, among other applications [2]. The purpose of this research is to produce a 3D printing filament material made from recycled polyethylene terephthalate and reinforced with coconut fibre.

### FILAMENT FOR 3D PRINTING

The filament chosen is not only determined by the printer's specifications; it is also determined by the aesthetic, mechanical, and functional features of the prints you desire to generate. To begin, aesthetic features will undoubtedly influence the 3D printing filament selection procedure. There are presently a variety of hybrid materials available, the bulk of which are constructed of PLA and another material like as wood, bamboo, cork, brick, or cement, and which give the filament a unique appearance and feel, whether in terms of colour or texture. Along with colour and texture, it must decide on the level of detail and surface quality required. For instance, the finer the printing layers, the less noticeable they are [3]. When sturdy, impact-resistant objects are required, polycarbonate or ABS filaments are recommended. On the other hand, when a flexible or semi-rigid product is required, Tensor Processing Unit (TPU) or TPE-based materials give easy-to-form parts. As a result, it is critical to consider the intended use of the printed 3D item while selecting the filament. If the filament will come into touch with food, for example, pick a PET-based filament [4]. Finally, the qualities of the environment in which printing will take place have an effect on the printing fibre employed. Certain polymers emit toxic Volatile Organic Compounds (VOCs) during fusion, which can build over time and become poisonous in a restricted area. It is recommended that you print with such filaments either inside or outside a 3D printer. This is especially true in the case of ABS. Determine whether the filament is susceptible to humidity and should be stored in a dry environment. Consider its resistance to water as well. Finally, ascertain whether the filament emits scents when utilised in a living space; the best options are PLA, PHA, or PET [5].

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3D printing filaments have a high tensile strength and a low weight-to-strength ratio, which is advantageous. This makes it an excellent material for applications requiring strength and longevity in 3D printing. When compared to more conventional materials such as wood and steel, it is recognised for its higher rigidity and durability while remaining relatively lightweight. Carbon fibre filaments are resistant to heat and corrosion due to their low thermal expansion coefficient. By preventing items from shrinking as they cool, carbon fibre filament simplifies 3D printing. This improves the print's quality. Carbon fibre filaments offer a number of benefits and can be utilised to create prints for a wide variety of businesses. Carbon fibres are stronger than metal, lighter than plastic, and heat resistant, making them ideal for 3D printing practical prototypes [6].

Meanwhile, a downside of 3D printing is its high energy consumption. According to a study conducted by Loughborough University, 3D printers consume between 50 and 100 times the energy required for injection moulding when melting plastic with heat or lasers. According to studies conducted by The Environmentally Benign Manufacture, a research organisation dedicated to examining the environmental repercussions of product manufacturing, direct laser metal deposition consumes 100 times the amount of electrical energy required for traditional manufacturing. Due to the high energy consumption associated with bulk production, 3D printers are best suited for small batch manufacturing runs [7].

### **COCONUT FIBER**

Coconut Fiber is a type of natural fibre derived from the husk of an unripe coconut. After steeping the coconut in hot seawater, the fibres are extracted from the shell in a manner similar to how jute fibre is extracted. Each fibre cell is approximately 1 mm in length and 10–20 μm in diameter, with strong cellulose walls. Raw coconut fibres range in length from 15 to 35 cm and have a diameter of 50 to 300 μm. When they are young, their walls develop a covering of lignin, stiffening and yellowing them. Coconut fibre is utilised in a variety of products due to its rigidity, including floor mats, doormats, brushes, mattresses with a coarse stuffing, and upholstery [8].

The advantage of using coconut fibre is that it is extracted from the exocarp and endocarp layers of the fruit. Coconut Fiber has a number of advantages over other natural fibres, including a high failure strain and an increased resilience to weathering due to the presence of lignin. Additionally, because coconut fibre contains less cellulose, it absorbs less water. Coconut fibre, like other natural fibres, has been shown to be an ineffective reinforcing material due to its wide and variable diameter, high microfibril angle, and high lignin and hemicellulose content. These disadvantages, however, can be mitigated by chemically modifying the fiber's surface. It is very robust, chemically resistant, and recyclable. The ability of thermoplastic polymers to be rapidly heated and cooled without changing their microstructure is one of their advantages. The hybrid composite was chosen for this study because it exhibits superior mechanical and thermal properties that a single fibre reinforced composite cannot match [9].

### **POLYETHYLENE TEREPHTHALATE (PET)**

PET is a thermoplastic aromatic semicrystalline polymer having a high glass transition temperature, outstanding mechanical characteristics, and chemical resistance. Additionally, PET has a number of advantages, including low cost, great transparency, and moderate recyclability. PET is used in industrial applications such as fibres, films, bottles, and engineering polymers because to these qualities. PET bottles have been used to hold a variety of beverages, snacks, and other consumer goods [10].

The majority of PET (more than 60%) is used to manufacture synthetic fibres, with bottle manufacturing accounting for around 30% of global demand. PET's ability to produce a wide variety of grades with a wide range of molecular weights in a single polymerization unit is a fundamental reason for its extensive use. PET is a thermoplastic polymer that comes in two forms: amorphous (transparent) and semi-crystalline (opaque and white). It is available in the form of a resin, a film, or a fibre. PET in semi-crystalline form is sturdy, ductile, stiff, and hard, but amorphous PET is more ductile. PET is extremely easy to recycle and may be separated into its monomers for usage in a variety of applications [11].

The advantage PET is an excellent 3D printing material. It is a strong and flexible material that has a high success rate when used to create 3D printed prototypes. It is an excellent material for items that require both flexibility and durability, such as mechanical components or electrical equipment casings. While many businesses prefer the latter materials for 3D printing, PET is known for emitting fewer odours than other commonly used 3D printing materials like as ABS or PLA. PET injection moulding is simple and is commonly available in pellet form for this purpose. Due to the hygroscopic nature of PET, it must be dried before using it in a moulding machine. PET shrinkage is extremely low (less than 1%), although it varies significantly depending on a variety of parameters, including holding pressure, holding duration, melt temperature, mould wall thickness, and mould temperature, as well as the amount and kind of additives [12].

Polyethylene terephthalate has the disadvantage of being prone to oxidation. For example, it is not typically used to store beer or wine since the shelf life of these beverages is regarded to be sufficiently long that some flavour degradation may occur prior to consumption. PET is a non-biodegradable substance that can be advantageous or detrimental, depending on your perspective and intended use. In general, polyethylene terephthalate is a great material. It possesses a unique set of qualities that make it ideal for consumer goods, most notably textiles [12].

The recycling of PET unsaturated polyester resin was developed as a matrix for the manufacture of coconut fiber/polyester composites. After glycolysis and polyesterisation of PET waste, an unsaturated resin composition was formed (UPR). A Fourier Transform Infrared (FTIR) study of the glycolysis product and resin created demonstrated the formation of linkages at saturated sites and the formation of a cross-link network between an unsaturated polyester chain and styrene monomer. To improve the adhesion of coconut fibre to polyester resin, we applied various concentrations of alkali, silane, and silane to

alkalized fibre and identified the optimal treatment concentration. We studied the effect of water absorption on the sorption properties of composites by immersing them in distilled water at room temperature. The optimal surface treatment for coconut fibre was 0.5 percent silane on a 5 percent alkalinized coconut fiber/polyester composite, which significantly improved tensile characteristics. Additionally, it was demonstrated that treated fibre composites have decreased water absorption properties when compared to untreated fibre composites [1].

### CONCLUSION

In conclusion, it is acceptable to conclude that recycled polyethylene terephthalate has a variety of advantages and can be used as a valuable material when properly handled. Additionally, academic and industrial research has established that PET may be successfully recycled in the production of a wide variety of items. By mixing the matrices and fibres, a biodegradable and recyclable material is developed by constructing a three-dimensional filament from PET reinforced with coconut fibre.

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## DEVELOPMENT OF 3D PRINTING FILAMENT MATERIAL USING RECYCLED POLYPROPYLENE REINFORCED WITH SUGAR PALM FIBER

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### ABSTRACT

Additive manufacturing (AM) which also known as 3D printing has been growing and new advancements are being introduced such as the application of natural fiber composite as printing filament. Plastic is the suitable material for 3D printing material because it is extremely stable, lightweight, and low price, but plastic waste causing environmental issue because it is difficult to recycled. These issues should be resolve by producing a biodegradable product made from renewable materials such as recycled polypropylene and natural fiber. Natural fiber such as sugar palm fiber (SPF) can potentially be used as reinforcement as the fiber helps in improving strength of the composite. This study presents a review of development of 3D printing filament material using recycled polypropylene reinforced with sugar palm fiber.

**Keywords:** 3D printing, sugar palm fiber, recycled polypropylene, natural fiber composite

### INTRODUCTION

After 30 years of 3D printing was invented, additive manufacturing (AM) has progressively outgrown its specialty uses and is helping develop a wide range of manufacturing methods [2]. AM is utilized in a variety of manufacturing industries, including automotive, biomedical, and aerospace. There are three types of 3D printing technologies exist based on the materials utilized in the additive manufacturing process such as liquid-, solid-, and powder-based additive manufacturing. Out of all technology, fused deposition modelling (FDM) is the most often used technology for printing fiber-reinforced polymer composites. Wang et al. (2017) reported that FDM printers run by the controlled extrusion of thermoplastic filaments as shown in Figure 1. Filaments in FDM machine melt into a semi-liquid form at the extrusion nozzle and are extruded layer by layer onto the print bed, where layers join together and form into end product [4]. Printing parameters, including layer thickness, printing orientation, raster width, raster angle, and air gap may all be modified to enhance the performance of printed components.

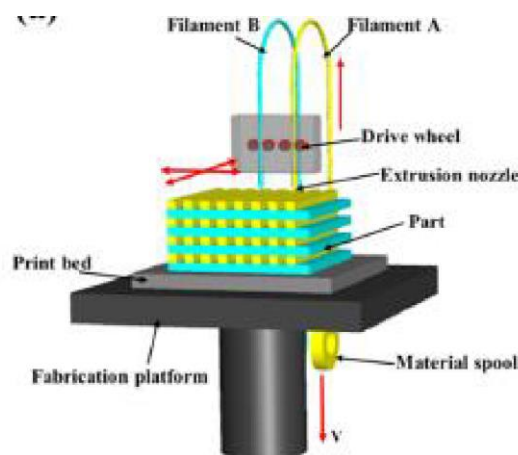


Figure 1 FDM setup [3].

Generally, there are a lot of 3D printing filament material with different properties and need different temperature to print. Filament of 3D printing is made from thermoplastics feedstock, which plastics or polymer that melt, can be shaped and mold into shape layer by layer, and solidify when cooled. Some examples of most often used in 3D printing filament material are polylactic acid (PLA), acrylonitrile butadiene styrene (ABS), polyvinyl alcohol plastic (PVA), polycarbonate (PC), pure composite such as polypropylene (PP), polyethylene terephthalate (PET), and other several materials. However, 3D printing innovations have created an opportunity to recycle thermoplastics as 3D printing feedstock material. Recently, researchers have focused on the possibility of employing recycled plastic fibers as a matrix for the 3D printing filament. Nevertheless, this matrix alone is insufficient for the structure of printed parts. Thus, adding natural fiber as filler in polymer can enhance the material properties. Natural fiber

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such as SPF is potential to foster advantages such as renewability, sustainability, non-toxicity, biodegradability, outstanding mechanical properties, and low-cost commodity has attracted a lot of interest to the manufacturing industry. Past researchers have studied the various type of natural fiber as reinforcement material and polymer matrix were used in 3D printing filament material as the advantage of using FDM technology be able to insert many materials at the same time [3].

#### POTENTIAL OF USING NATURAL FIBER AS REINFORCEMENT AND THERMOPLASTIC AS MATRIX POLYMER

Natural fibers as reinforcement material, along with thermoplastics, have gained popularity in 3D printing market. Natural fibers are highly convenient to use because they are readily available, cost-effective, environmentally safe, and biodegradable. Natural fiber may be utilised in three industries: textile, paper, and fabrics, as well as a reinforcing material for composites. Natural fiber can also replace fiber glass in some application such as in composite part in the automotive industry, construction, and plastic industry. Previous study has reviewed natural fibers as reinforcement material with polymer matrix. Sugar palm fiber (SPF) was extracted from sugar palm tree (*Arenga Pinnata*) which were usually found in Southeast Asia like Malaysia and Indonesia. Other name for this tree that familiar to local Malaysian is *enau* or *kabung* [5]. Ilyas et al., (2018) have reported that SPF offers some benefits over traditional reinforcement fibre materials such as cost, recyclability, non-toxicity, abrasiveness, density, and biodegradability. Hence, SPF which also known as *ijuk* is one of the most common fiber used among researchers. Siregar, (2015) reported that *ijuk* can withstand temperatures of up to 150°C and has a flash point of around 200°C. Other than SPF, jute fiber is by far the most common natural fiber used to improve composite materials [8]. Jute is a *corchorus capsularis* bast fiber extracted from *corchorus* tree. Jute is the most affordable natural fiber and is produced in large quantities [9]. Jute has several clear advantages as a natural fiber, including shine and glitter, high tensile strength, low stretchability, medium heat and fire resistance, and long staple lengths. Some of the drawback in jute fiber include thermal stability, crease resistance, stiffness, fiber shedding, and fading when exposed to sunlight. Besides natural fibers, previous researchers also reviewed on polymer matrix such as PP. Generally, polymers have been used in the industry due to their ease of fabrication and usability. Thermoplastic like polypropylene is a multifunctional and hard material with many advantageous physical properties, and it is also able to be recycled. The ability to recycle a portion of this plastic waste into a matrix that can be utilised for the 3D printing filament opens an entirely new channel for recycling plastic waste and decreasing global plastic pollution. PP offers several benefits such as low cost, high melting point, sustainable and most important thing is, it is 100% recyclable. Some research has been done using recycled material such as rPP reinforced with natural fiber composite.

A research conducted by Stooft & Pickering, (2017), mentioned that a set of 3mm composite 3D printing filaments with different weight ratios of hemp, harakeke, and recycled gypsum were successfully extruded. As they get the result of 3mm composite filament, the strongest composite filament obtained from the study was composed of 30% harakeke fiber in a recycled polypropylene matrix. The result that they obtained shows that the mechanical properties of recycled polypropylene as a matrix material was better compared to pure PP because it strong, stiff, affordable, and recyclable 3D printing filament. Stooft & Pickering, (2017) reviewed thermo-mechanical, degradation, and interfacial properties of jute fiber-reinforced PET-based composite, where they found that jute fiber-reinforced PET matrix composites gained higher mechanical properties over the matrix PET, thus indicating good fiber–matrix adhesion. Bachtiar et al., (2014) studied tensile properties of hybrid sugar palm/kenaf fiber reinforced polypropylene composites. They found that the tensile strength of hybrid composites containing more kenaf fiber is higher that composites that SPF. Chestee et al., (2017) used short jute fiber reinforced PP. A few test has been done to study the thermal and mechanical properties. F. P. Brito, (2020) reviewed PLA composites reinforced with flax and jute fiber. This paper examines the current state of PLA composites reinforced with flax and jute, which are two high-strength natural fibers suitable for this purpose. Mastura et al., (2020) reviewed mechanical properties of treated kenaf/ABS composites in fused filament fabrication (FFF). The fibers are treated to improve their strength properties with the polymer matrix and are ready to be used as FFF feedstock. Based on these studies conducted by the previous researchers, the addition of natural fiber to polymer composites as reinforcement has created more “green” composites and can replaced conventional glass fiber and other synthetic fiber composites. Nevertheless, there is no study that have been conduct using rPP reinforced with SPF. Thus, this study is to develop 3D printing filament material using rPP reinforced with SPF.

#### THERMAL, PHYSICAL, MORPHOLOGICAL AND RHEOLOGICAL PROPERTIES OF NATURAL FIBER AND POLYMER MATRIX

To get a good quality of filament, SPF and rPP must undergo some treatment to enhance its properties and quality of 3D printing filament material. Properties of SPF can be determined through TGA testing for thermal properties and SEM analysis for morphological properties. Alkaline treatment using NaOH is a chemical treatment which is it commonly used to improve the fiber properties such as thermal properties in order to enhance the addition between fiber and matrix and it used to remove impurities on the fiber surface [14]. The previous study conducted by were demonstrate the TGA curve for pure TPU and TPU-SPF composites. In their study, the composites 250 µm TPU-SPF (428 °C) and 425 µm TPU-SPF (425 °C) separated at slightly higher temperature than the 160 µm TPU-SPF (417 °C), proving that the connection between the fiber and the matrix expanded as fiber size increased. For physical properties of SPF, Mohammed et al., (2016) have obtained the result from SEM analysis where SEM images of pure TPU, SPF fiber, and TPU-SPF composites with fiber sizes of 160 µm, 250 µm, and 425 µm. They found that fiber sizes of 160 µm and 425 µm had bad impacts on fiber-matrix bonding, due to low physical behaviour in these composites, possibly due to even more defects on the fiber surface (untreated fiber). However, the composite with 250 µm had the best bonding to the TPU matrix. This could be due to the appropriate particle size of 250 µm fiber size for bonding to the TPU matrix, which gives the proper opportunity to boost interfacial bonding and create a smooth texture with greater physical properties. Besides that, properties of rPP can be determined from thermal and rheological properties. A research were conducted by Morales et al., (2021) on thermal analysis of rice husk (RH) and recycled polypropylene (rPP). From their findings, TGA was used to measure the thermal stability of RH fiber, neat rPP, rPP/RH (5 wt.%), and rPP/RH (10 wt.%).

Furthermore, a study has been done by Al-Mulla et al., (2013) where rheological analysis was performed on a rheometer with a parallel plate shape with a 25 mm of diameter plates at 175 °C. These analyses are important as it can determine the properties of natural fiber and polymer matrix for developing 3D printing filament material.

## CONCLUSION

In summary, PP is a good polymer matrix in composite because polypropylene can be recycled to produce new sustainable biodegradable polymer. Plus, the addition of natural fiber as filler to the polymer composites can enhance the printing parts and it can replace fiber glass. Both materials can work together to develop the better composite's properties in 3D printing technology such as better strength and stiffness of the printed product. Last but not least, this study is to develop 3D printing filament material using rPP reinforced SPF which comes from natural resources to solve the problem of an environmental issue and develop biodegradable polymer that can be safely disposed to the environment.

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**DEVELOPMENT OF 3D PRINTING FILAMENT MATERIAL USING RECYCLED POLYPROPYLENE(PP) REINFORCED WITH WOOD DUST**Muhammad Zulqarnain M.Z.<sup>1</sup>, M. Nuzaimah<sup>1\*</sup>, Yusliza Yusuf<sup>1</sup> and R. Nadlene<sup>2</sup><sup>1</sup>Fakulti Teknologi Kejuruteraan Mekanikal dan Pembuatan, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.<sup>2</sup>Fakulti Kejuruteraan Mekanikal, Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.**ABSTRACT**

In recent years, the usage of natural fibre composites has gained significant interest due to their low density, high availability, and low cost. An investigation into the development of sustainable 3D printing filaments based on wood dust and recycled polypropylene (rPP) and the effect of fibre weight ratio on thermal, mechanical, and morphological properties of 3D printing filaments are being conducted in this study. The abilities of combination between wood dust and recycle polypropylene as new composite material will be evaluated whether it is suitable to be used in the 3D printing field as a composite filler or filaments. This study will be conducted based on the review from the other related research article to identify the characteristics and the properties of wood dust and recycle polypropylene when it was combined with the others matrix or natural fibre.

*Keywords:* natural fibre (wood dust), 3D printing filaments, recycle polypropylene

**INTRODUCTION**

Additive manufacturing (AM) and 3D printing have been used widely worldwide. With this technology, it gives an advantage such as advanced healthcare, environmentally friendly, minimising waste and cost-effective which can be utilized by many sectors such as aerospace, automotive, medical, architectural, education, and fashion industries. In 3D printing, there were a few techniques that have been used such as Fusion deposition modelling (FDM), stereolithography apparatus (SLA), electron beam melting (EBM), laminated object manufacturing (LOM), selective laser sintering (SLS), digital light projection (DLP) to produce a product [1]. 3D printing used a filament to print the desired product. Plastic filaments, synthetic fibre composite and natural fibre composite was the material that has been used for 3D machine filaments. Example for plastic filaments that was commonly used was ABS and PLA [2]. The usage of natural fibre composite such as rice husk and recycle polypropylene composite filaments and bamboo filled PLA composite material for 3D printing filaments also was a priority and has been used widely due to the significant benefit such as low cost, high elasticity, high strength, eco-friendly material, and the others compared with synthetic fibre composite filaments [2]. Examples of other natural fibre that can be used for composite material for 3D printing filament was wood dust, sugar bagasse, jute, and sugar palm [3]. Fig 1 show the example of natural fibre that can be used for 3D printing material.

**POTENTIAL OF RECYCLE POLYPROPYLENE AND WOOD DUST TO BE USED AS 3D PRINTING FILAMENTS MATERIALS.**

In 3D printing industry, there was many types of filaments that can be used such as plastic and composite filaments. The natural fibre composite filament was better compared with other synthetic fibre composite material due to the environmental impact such as recyclability, renewability and biodegradability, as well as lower raw material costs and light weight [2]. Chawla et al.(2020) has studied the composite filaments using wood dust reinforced with recycle acrylonitrile butadiene styrene (ABS) plastic. They concluded that the reinforcement between wood dust and recycle ABS matrix in form of composite filaments has successfully achieve the desire properties for non-structural application. For thermal properties, it has low capacity for heat that caused the composite cannot be used for high temperature application. Da Silva et al.(2021) had reviewed on manufacturing disposable cup using polylactide acid PLA reinforced with wood dust. Referring to this studied, by reinforcing wood dust with PLA can increased the hardness properties, elongation at break and greater performance in impact strength. The result from SEM test has shown that there was a low interfacial adhesion between PLA and wood dust, generating loses in tensile properties. Correa-Aguirre et al.(2020) studied on effect of fibre treatment on polypropylene reinforced with sugarcane bagasse bio composite. A few tests have been done on to study the thermal characterization, mechanical properties, and the others. The result of the study has shown that the thermal characterization of the fibre has increased the crystallization temperature and the thermal stability of PP phase for all extrusion cycle without disturbing the melting process of PP matrix. The treated fibre using silane has the highest thermal stability in the study. Evidence from previous study, they have proven that the usage of natural fibre composite especially recycles polypropylene and wood dust as 3D printing material increased the characteristic, properties of the printed product, also reduce cost and waste [2], [6].

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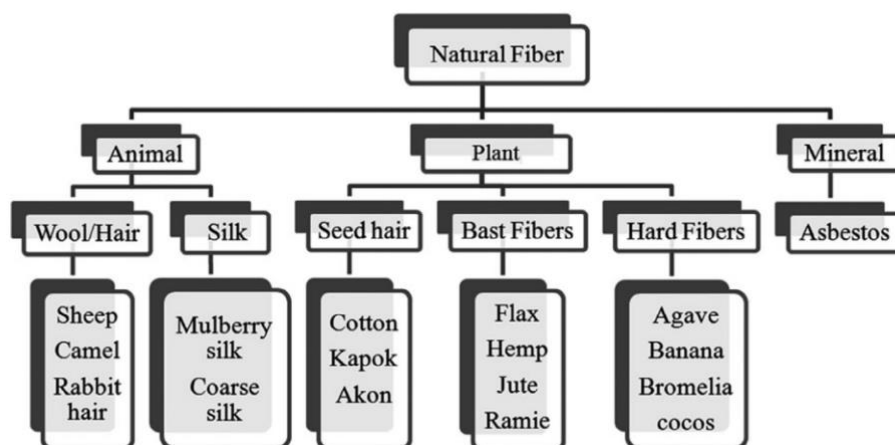


Fig 1. Example of natural fiber material [7].

### FIBER TREATMENT AND CHARACTERIZATION.

In producing a natural fibre composite for 3D printing used, the material needs to be through a few processes and testing to make sure that the composite material is suitable to be used as filaments. Firstly, the particle analyzer method will be used to get a suitable particle size of wood dust for producing natural fibre composite. Hossain et al. (2014) has used this method in his research to classify the particle size of wood dust and its apparent and true density for his research on the thermal conductivity of wood dust reinforced composite. Sodium hydroxide (NaOH) will be used on wood dust to treat the fibre to remove the impurities of the material. Karthikeyan et al. (2014) has used this treatment method on coir fibre at room temperature (27 °C - 29 °C) at different concentrations of 2%, 4%, 6%, 8% and 10%. Then, both materials will be tested using the thermogravimetric analysis (TGA) method to determine the thermal stability of the materials. Correa-Aguirre et al. (2020) in his research on the effect of reprocessing and fibre treatment on the properties of polypropylene – sugar bagasse biocomposite also used this thermal analysis to evaluate the thermal characterization of the material. Abdul Hamid et al. (2020) in his review on rheological and dimensional properties of polypropylene on the end of life vehicle has used the rheological analysis to study the shear rate and shear viscosity of the material. So, rheological analysis should be included to ensure that the recycled polypropylene was suitable to be used as composite material for 3D printing filaments. And, the morphological analysis will be done using scanning electron microscopy (SEM) machine to observe the surfaced composite material in a high magnification view. S. Kumar et al. (2019) in his study also using an SEM machine to make an observation on the surface structure and failure analysis of the composite material. This is important to identify weakness structure of the material that will be used for 3D printing filaments material.

### CONCLUSION

In summary, the natural fibre filaments that was made from recycle polypropylene reinforced with wood dust might have a good potential to be used as 3D printing filler. Studies from the past researcher on both materials has shown that it has an advantage such as high strength, high thermal characterization and have a good quality as 3D printing filaments when combined with the other matrix and natural fibre. However, a further study need to be done to identify and overcome any weakness of this natural fibre composite for 3D printing purpose.

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**DEVELOPMENT OF 3D PRINTING FILAMENT MATERIAL USING RECYCLED POLYETHYLENE TEREPHTHALATE (rPET) REINFORCED WOOD DUST**Muhammad Khairul A. S<sup>1</sup>, M. Nuzaimah<sup>1\*</sup>, Yusliza Yusuf<sup>1</sup> and R. Jumaidin<sup>1</sup><sup>1</sup>Fakulti Teknologi Kejuruteraan Mekanikal dan Pembuatan, Universiti Teknikal Malaysia Melaka, Taman Tasik Utama, 75450 Ayer Keroh, Melaka, Malaysia.<sup>2</sup>Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia.**ABSTRACT**

Implementation of natural fibers in 3D printing filament materials has attracted many researchers in various applications. This study presents a review of the potential of natural fibers to be employed as 3D printing filament material. This review aimed to review the development of 3D printing filament materials using rPET reinforced with wood dust. Wood dust were characterised in terms of the physical, thermal and rheology properties and then will be treated using sodium hydroxide (NaOH). Previous studies have also shown that composites developed from wood dust reinforcement and polymers matrix such as polylactic acid (PLA) and acrylonitrile butadiene styrene (ABS) can produce filaments for 3D printing. Finally, the challenges of the 3D printing filament materials in processing the natural fiber-filled polymer are also reviewed.

*Keywords:* 3D printing, rPET, natural fiber, wood dust.

**INTRODUCTION**

3D printing is a relatively new technology that has gained a lot of popularity in recent years. Because of its simplicity and low cost, it is mostly utilized for prototyping and small-scale manufacturing. 3D printing is becoming more popular in a variety of industries, including aerospace, military, automotive, medical, and construction. Like any manufacturing process, 3D printing needs a mixture of high quality materials to produce a device according to the specifications and quality to be used to the maximum [8]. rPET stands for recycled polyethylene terephthalate, which is the most prevalent polyester resin. rPET is a colourless and transparent material in its natural form, however it loses its transparency when heated or cooled. Instead of polluting our landfills, rPET may be recycled into new products, thereby turning trash into wealth. rPET may therefore be turned to powder and utilized to print machine components because of its great tensile and mechanical strength, toughness, and hardness. Mechanical characteristics of the rPET powder must be evaluated to determine its compatibility for 3D printing in order to ensure that standard components are produced [9]. The structure and build-up of fibers, consisting of lignin and long chains of cellulose, give wood its compressive and tensile strength. Wood is a natural resource, organic material that is readily accessible in the form of wood wastes, which may be processed into smaller fractions or fine wood powder and utilized in 3D printing with conventional plastics, natural adhesives, or cement as a binder [10]. rPET waste of postconsumer plastic is among the least recycled plastics, meanwhile wood dust is abundant in woodworking industries such as furniture factories. Therefore, the recycling of waste materials is one of the promising solutions to minimize the environmental impacts of this waste while minimizing the depletion of natural resources.

**PROPERTIES AND BENEFITS OF WOOD DUST THAT CAN BE FUSED WITH rPET TO PRODUCE GOOD 3D PRINTING FILAMENT**

3D printing is rapidly grown in industrial and household environments because they have a variety of interesting features that can be used. For example, if production is required on a small scale, parts can easily be obtained with limited scrap production and energy consumption, and without the need for expensive tools or complicated installation [11]. Thermoplastics are used in 3D printing as filament material. According to a study by Mikula et al. (2020), polylactic acid (PLA) and acrylonitrile butadiene styrene (ABS) are the most popular materials used in 3D printing [12]. PET is one of the most widely available thermoplastic polymers on the market. PET is used primarily for the production of containers and bottles for beverages such as water and carbonated soft drinks, in industrial food, as well as nonfood uses. It can be processed at high temperatures easily and recycled easily [13]. A study has shown that virgin low-density polyethylene (LDPE) and rPET are optimal materials as an alternative filament for 3D printing. The outcome showed that rPET is better contrasted with virgin PET. The recycling of PET likewise showed potential as an elective filament to 3D printers [14]. Toughness, hardness and high ductile and mechanical strength are the benefits of PET which can be changed over into powder and used to print machine parts. In addition, comparisons were also made on 3D printing plastic materials such as HDPE, Nylon, PVA, PLA and ABS showing the results that PET powder is very suitable to use as 3D printing materials [9]. Wood dust is a tiny object that results from cutting wood using a sharp tool such as a saw. Wood dust is considered as an industrial waste that pollutes the environment but can be reuse with three methods including manufacturing, energy and agricultural utilization [15]. For example, wood dust is able to produce something valuable as raw material for wood boards, light construction material such as cupboards, notice boards, wall and roof

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sheeting [16]. Studies show that using materials from wood elements such as wood dust has its own special features. Wood dust which is wood based materials is a biomass and low cost alternative that has shown great promise. Next, good tensile strength and stiffness are also shown on wood materials and wood types tested for 3D printing purposes including softwood and hardwood types [17]. NaOH is an inorganic compound that is also known as sodium hydroxide and caustic soda and Iye. The highly alkaline and basic NaOH is capable of causing severe chemical burns and breaking down proteins at normal ambient temperatures [18].

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### THE EFFECT OF NAOH TREATMENT ON THE NATURAL FIBER

The use of alkaline NaOH can improve the properties of natural fibers by increasing cellulose content, waterproof properties of fibers and mechanical properties. High specific modulus, lightweight, non-toxic and absorbs CO<sub>2</sub> during its growth and easy to process are the advantages of using NaOH. The use of NaOH makes the fiber surface cleaner and is able to change the molecular structure of cellulose. NaOH is also able to increase the aspect ratio or length by reducing the fiber diameter and good adhesion to the matrix [19]. Chemical treatment for wood dust becomes popular because it achieves the adhesion of the filler matrix and the mechanical properties of the composite. Dust on the surface will be removed by washing and drying at a temperature of 65°C after being processed in alkali treatment. The wood dust is dried up in the oven at 70°C for 72 hours. 1%, 3%, 5% and 7% are concentrations to be immersed in NaOH solution with a dry substrate at room temperature for 2 hours. The acetic acid solution is diluted for a few minutes and being washed again with distilled water to neutralize the filler by being soaked. On the other hand, Rexen et al. (1976) found that treating wood dust or straw with up to 7% of NaOH will produce the best result [20].

### PROPERTIES OF MATERIALS IN TERMS OF THE PHYSICAL, THERMAL AND RHEOLOGY OF WOOD DUST AND rPET

To create a new filament combination, the mechanical properties of wood dust are an important factor. The purpose is to ensure that mixing between rPET with wood dust will produce good filaments for the 3D printing process. Wood is considered one of the most sustainable materials in the world due to its high value and benefits such as its low cost, low weight and recyclability and biodegradability [21]. Wood dust was tested for porosity, water retention, and water drainage. Scanning electron microscopy (SEM) was used to examine the morphology of raw wood dust. This is in order to detect changes in the structure and other characteristics of wood dust after usage. Various measurements in the physical properties of wood dust can be modified by combining wood dust particles [22]. Wood dust has a positive effect on the thermal insulation quality of building materials. Low-cost efficient unfired brick can be developed by using wood dust instead of bricks. Wood dust has excellent thermal conductivity, with 20.1% thermal diffusion, 30% earth building blocks, and 22.7% density [23]. This is because the filler material includes thermal releasing elements that are conducting elements, and the wood being used in the composites is conductive when compared to polyester [24]. Understanding the relationship between structures and properties of polymer nanocomposites is important in polymer processing for the analysis and design of processing operations. Rheological properties of carbon nanotubes reinforced polymer nanocomposites are closely related to the microstructure of the material, and the condition of nanotube dispersion and dispersion in the matrix. Melt rheological measurements were carried out at 270 °C in this experiment [25].

### CONCLUSION

In summary, the possibility of recycling waste material to become a commercial product that can be utilized in different sectors as a component of their production materials may be described in the conclusion of this literature study. Waste material such as wood dust may be developed as natural fibers that can be reinforced with other recycled plastic materials like polymers. Due to its physical and mechanical properties, reinforcing this recyclable material can enhance its strength. The research found that adding this recycled waste material can improve mechanical characteristics when wood dust interacts with polymer in the correct formula and ratio during the mixing process. The ability of wood dust when being combined with other types of polymers to produce a new filament that will be used as materials for 3D printing may be successful.

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## EFFECTS OF SONICATION IN THE DISPERSION PERFORMANCE OF BACTERIAL CELLULOSE NANOFIBER MODIFIED WITH TEMPO AND SILANE AS WATER-BASED LUBRICANTS

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### ABSTRACT

The stability tests of Bacterial Cellulose (BC) suspensions modified with Silane and 2,2,6,6-tetramethyl-1-piperidinyloxy (TEMPO) with various sonication time were performed. Zeta potential, transmittance, and photo-capturing method were investigated. It was found that modified the BC with silane improved the stability of the sample. Sonication in 30 minutes resulting in the highest transmittance for the TEMPO-Oxidized sample. The effect of sonication was also decreasing, but not significant, the zeta potential value. This was mainly due to the lot of BC particle in the suspension. Excessive BC particle will increase the van der Waals interactions between BC particles that will increase the sedimentation velocity. The photo capturing results showed that there were no sedimentation in the sample.

*Keywords: Stability, TEMPO, Lubricant, Bacterial Cellulose*

### INTRODUCTION

Lubrication is an effective way to reduce friction and regulate wear. Material waste and mechanical performance loss are mostly caused by friction and wear. One-third of the world's energy resources are projected to be used to overcome friction in some form or another. As a result, any reduction in friction and wear can save a lot of energy. One of a way is improving the lubricants properties by adding additives [1],[2].

Additives like viscosity improvers, antiwear agents, thermal conductivity enhancers, detergents, and other additives with varied functions have been used and validated [3],[4]. Despite the technical requirements, lubricants' contamination and environmental health issues have garnered increased attention in recent years [5],[6]. Considering this, it is important for now to perform such research with environmental friendly materials.

BC has a considerable amount of hydroxyl groups and is hydrophilic in nature [7]. The hydrophilic BC is dissolved and agglomerated in a hydrophobic synthetic lubricant, lowering its tribology performance. It's crucial to have individual cellulose nanofibers distributed in a lubricant. A common way for reducing cellulose agglomeration is to use 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO). Furthermore, due to the nature of the BC that is hydrophilic, another modification is also needed to enable the BC dispersed in an non-aqueous solution. It has been proved that Methyltrimethoxysilane successfully hydrophobize BC in the form of composite [8].

For this basis, in this study, the modification of TEMPO-Oxidized BC (TOBC) with Silane was carried out to find out how effective it was in the suspension form. The effect of sonication time was also investigated. The success of this research will prove that silane-treated BC is a potential additive which can be added into lubricants, or in the form of water-based lubricants.

### MATERIALS AND METHODS

BC which source is from nata de coco was purchased from a small local market in Padang, Indonesia. TEMPO (98% Pure), Methyltrimethoxysilane, Sodium Hypochlorite (NaClO), and Sodium Bromide (NaBr) used came from Sigma-Aldrich, Indonesia, United States.

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#### Sample Preparation

The BC was first processed into paper form and the method is the same with our previous work [3]. About 0.1wt% of BC was solved into 250 ml of distilled water. For the TEMPO treatment, the process is also similar with our work [3]. The modified BC with TEMPO (TOBC) was then sonicated with various duration. Further, the silane modification was done by adding 1 ml of Methyltrimethoxysilane into 150 ml of TOBC solution. The sonication was also applied in these samples.

#### Stability Investigation

Numerous stability investigation was conducted in this research. A photo-capturing method was conducted for detecting sedimentation by visual. The photo at the first day at preparation and 2 weeks after was taken. At

the same time, the zeta-potential (Horiba-SZ 100z) and UV-Transmittance (Shimadzu UV-1800) were used. The zeta-potential measurement also analyze the sample's particle size.

## RESULT AND DISCUSSION

On visual observation, it can be seen in Fig.1 that there is no sedimentation after two weeks of observation. This is because the properties of BC and water have the same hydrophilic properties. TEMPO's effect on BC also reduces agglomeration, resulting in excellent wettability or surface contact of BC fiber with water. Furthermore, the UV-transmittance showed results at a wavelength of 450 nm. The tests were carried out on the first day, second, and one week after preparation. It can be seen in Table 1, the transmittance value in TOBC becomes more stable when compared to pure BC. This is due to the effect of TEMPO, which can make BC in suspension more stable [3],[9],[10]. However, samples with modified silane (TOBC-S) obtained a lower transmittance and more unstable transmittance results than TOBC.

This proves the effect of silane that hydrophobize BC [8]. Hydrophobic BC cannot absorb water [11]. Therefore this will affect the light that can be passed. Visual observations also proved this, and it can be seen that the TOBC-S sample is more opaque than TOBC. Then, on observing the particle size diameter, shown in Fig. 2f, BC particles experienced a significant decrease after sonication. This result is shown by previous work that has been done [12],[13]. For the zeta potential results, the TOBC-0 sample obtained the best stability with an average value of 66 mV [14].

The literature disclosed that a zeta potential value of more than 60 indicates a very good sample stability [14]. However, ultrasonic effects reduce the zeta potential value. This is probably due to the effect of ultrasonic, which makes more BC particles in the suspension. This then increases the van der Waals attraction that occurs. Studies reveal that the ultrasonic effect makes the agglomerated BC particles have a larger surface area, and the fibers become longer [14]. In the TOBC-S sample, it can be seen that the particle size also becomes smaller due to the ultrasonic effect. Then, the zeta-potential value is smaller than TOBC. This could be due to the influence of silane, which makes the presence of BC particles hydrophobic so that the wettability will be reduced.

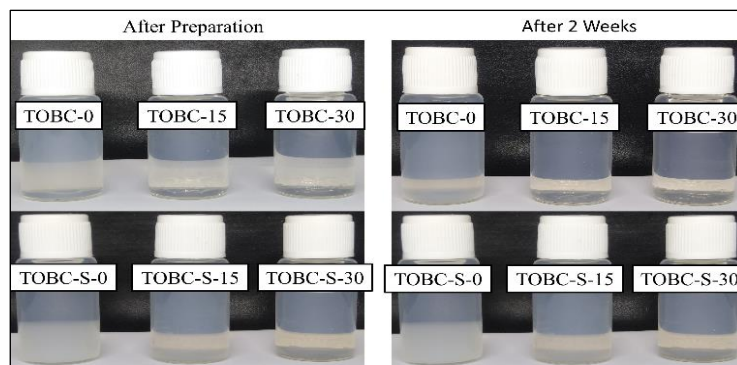


Fig. 1: Photo-capturing stability at the first day and after 2 weeks

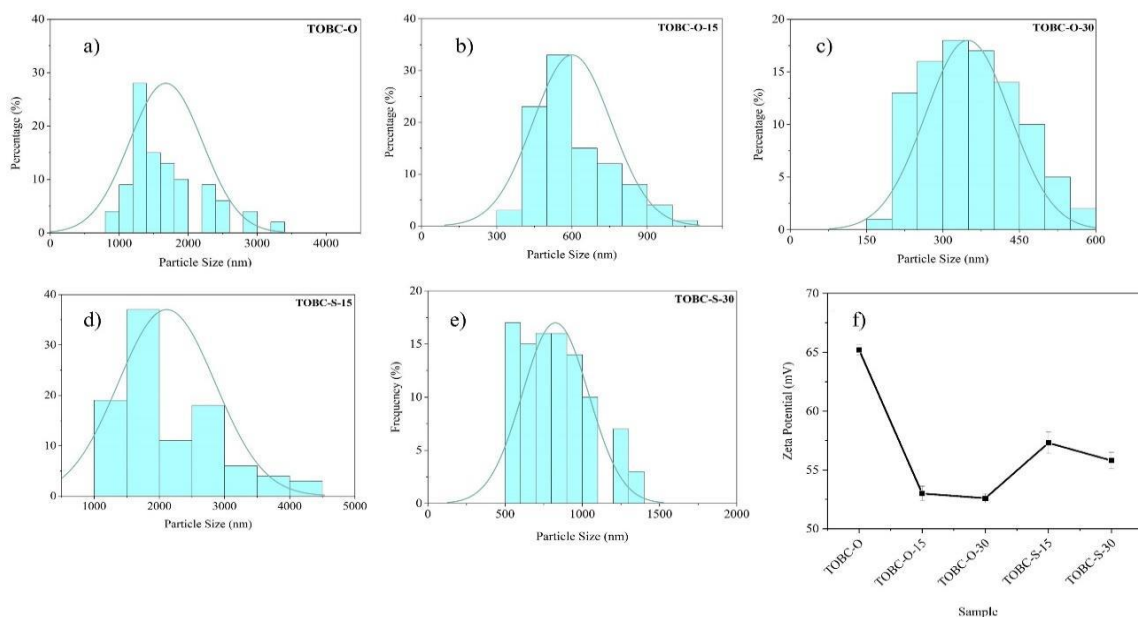


Fig. 2: Particle size (a-d), and zeta potential value (f) of TOBC and TOBC-S.

TABLE 1 : Result of UV-Transmittance at wavelength of 450 nm.

Samples	Peak Wavelength (nm)	Transmittance (%)
TOBC-O	3419	97
TOBC-O-15	3490.65	77.1
TOBC-O-30	3446.30	80.3
TOBC-S-15	3451.31	85.5
TOBC-S-30	3459.19	91.7

## CONCLUSIONS

This study reports the effect of silane on BC suspension. Ultrasonic variations were also carried out to determine how it affects TOBC-S. The results of the zeta potential test prove that silane treatment still makes BC hydrophilic. However, a decrease in value compared to samples without silane proves that silane affects BC. The decrease in the zeta potential value was probably a change in one of the BC particles to become hydrophobic. These results can potentially be further tested by using oil or lubricant as the base fluid.

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## FIRE-RETARDANT AGENT REINFORCED BAMBOO KRAFT PAPER

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## ABSTRACT

For hundreds of years, humans have been relying on wood as the primary source for cellulose fibers. Unfortunately, with the exploding global pollution, wood suffers to meet the market demand causing natural forests to decrease at an alarming rate. In an effort to save the earth, finding a more sustainable alternative should be approached. Since the age of time, bamboo has been utilized in multiple ways and proven to be a great alternative compared to wood. Bamboo fibers have gotten a lot of attention as a viable alternative to wood fibers for pulp and paper because of their mechanical properties, which are comparable to wood. However, lack of researches have been done on the fire-resistance of bamboo paper, causing its practicality to impede. Although the thermal properties of bamboo paper might be overlooked when compared to its mechanical properties such as tear, tensile and folding, increasing the fire-resistance of bamboo can broaden its usability, safety and increases its durability. Bamboo paper making can be widely used in packaging, construction, agriculture, and for daily necessities, thus increasing the demand for bamboo paper.

**Keywords:** Bamboo; fire retardant; kraft paper; cellulose; natural fiber; impregnation.

## INTRODUCTION

Bamboo is a type of cellulose fiber, grows abundantly in subtropical and tropical countries. Known to be low-cost, flexible, lightweight, and tough material, for these reasons bamboo has been considered as a replacement for timber [4]. For decades, timber has been the main source of cellulose fiber to produce everyday essential items such as paper, furniture, flooring, and construction material. Due to its chemical composition, bamboo is a better raw material for pulp and paper making compared with other non-wood fibers such as rice straw, reed and bagasse [1]. Bamboo pulp fibers have been used for papermaking as a substitute for wood pulp fibers because of their long fiber length and the high length-to-diameter ratio [2]. A study shows that bamboo paper made from *Gigantochloa scortechinii* (Semantan bamboo) fiber can be good material for the paper industry, especially for those paper strength-oriented industry [3]. Although bamboo paper has been widely used since ancient China. Its versatility is still limited due to its high flammability. Thus, developing flame-resistant bamboo paper is crucial as more companies are moving towards a sustainable and environmental conscious approach in replacing wood.

Adding fire retardant to bamboo materials is a popular form of fire retardant treatment [3]. Generally, fire-retardant can be classified into two categories, reactive fire-retardants and added fire-retardants, depending on whether a chemical reaction is involved in the material. To make fire-retardant paper, additive-type fire-retardants are usually applied directly to the papermaking pulp [4]. In added fire retardant, it can further be classified into inorganic fire-retardant and organic fire-retardant. The main types of inorganic fire retardants include metal hydroxides, metal oxides, and alkali metal salts, ammonium salts and molybdenum compounds. Halogen flame retardants, phosphorus flame retardants, nitrogen flame retardants, and other are examples of organic fire-retardant. The manuscript overviews the recent advancements on fire-retardant agent reinforced bamboo kraft paper.

## BAMBOO AND ITS NATURAL FIBER

Bamboo is a perennial evergreen grass belonging to the Poaceae family, subfamily Bambusoideae, and tribe Bambuseae [5]. Bamboo comes in over 1500 different species [6]. In Malaysia, there are 50 species of bamboo, 25 of which are indigenous and 25 of which are exotic. *Bambusa*, *Dendrocalamus*, *Dinochloa*, *Gigantochloa*, *Racemobambos*, *Schizostachyum*, *Thyrsostachys*, *Chusqua*, *Phyllostachys*, and *Yushania* are the genera found in Malaysia [7]. *Gigantochloa scortechinii* (Semantan bamboo) is the most common and abundant bamboo species found wild in Peninsular Malaysia forests and worldwide [8].

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The basic anatomy of bamboo is consisting of rhizome, roots, bud, shoots, culm sheath, culm, node, and internodes. The fact that the bamboo culm is growing on the rhizome is the most important fact [9]. Some regarded the culm as the most important part of a bamboo shoot. The culm's anatomy can be broken down into nodes and internodes. The internodes are hollow tubes with

axially oriented cells at their core. A diaphragm is formed on the inside of the node, and the culm-sheath and branches are formed on the outside, as can be observed in Figure 2 [10].

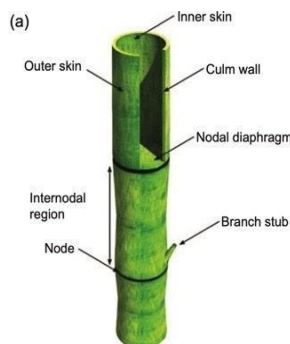


Fig. 1: Schematic View of a Culm Segment [10]

The bamboo culm or stem can be processed to produce bamboo fiber. Bamboo fibers are comparable to hardwood fibers in terms of fiber length, aspect ratio, and fibrous cell wall cavity ratio, making it an essential non-wood fibre raw material for pulp and paper production.[1]. In 2017, China's total bamboo pulp production capacity was 2 400 000 tonnes, with the majority of the bamboo pulps (about 80%) going into the production of domestic paper grades [1].

### FIRE RETARDANT AGENTS

Combustion occurs when heat from an ignition source combines with volatile combustible compounds in a concentration within the flammability limits, and at a temperature above the ignition point, [13]. The combustion will continue as long as the heat applied to the polymer is adequate to keep it thermally degrading at a rate faster than the rate required to feed the flame. Otherwise, the flame will go out. If the following heat requirements are met, a self-sustaining process occurs through thermal oxidation in the gas phase or condensed phase, if the heat input from the ignition source is ceased or is insignificant. The goal of fire retardant systems is to keep the amount of heat delivered to the polymer below the crucial threshold for flame stability [13]. There are two methods to modified paper with fire retardant, (1) additive flame retardant where the flame retardant is added (additive flame retardant) directly to pulp or paper and (2) reactive flame retardant, introducing flame retardant group on paper fibers through chemical reaction [11]. In practice, additive flame retardants are more popular since selecting a reactive flame retardant is more complicated. However, fire retardant additives may require relatively high loadings to be effective (usually 10–40 wt %, unless in the case of nanoparticles) and may cause undesirable modifications to the polymer's physical and mechanical properties. However, when compared to reactive fire retardants, fire retardant additive offers a fair balance of cost and functionality. Halogenated compounds, phosphorus compounds, inorganic compounds, and so on are used as additive-type flame retardants.

The most effective and widely used commercial fire retardant system currently available is halogenated compound [12]. However, it is found that halogen-based fire retardant to be toxic and can be harmful to the environment. When heated, such as during production, a fire, recycling, or exposure to the sun, halogenated compounds containing aromatic rings can breakdown into dioxin derivatives [13]. Phosphorus compound fire-retardant such as ammonium polyphosphate (APP) may hydrolyse to phosphoric acid in with moisture and lose its flame retardant efficacy over time [14]. For inorganic fire retardant, it can act as a filler and improve the mechanical properties of the material, but it has some major disadvantages, such as a large amount of inorganic flame-retardant requirement and an adverse effect on the physical properties of paper [15], [16]. It can be concluded that each type of fire retardant has its own advantages and disadvantages. Therefore, a new type fire retardant has emerge known as synergistic fire retardant. Synergism occurs when the combined effect of two or more components outweighs the total of their individual effects. Various types of synergistic fire retardants have been produced, such as antimony-halogen synergism, phosphorus-halogen synergism, nitrogen-phosphorus synergism, and more.

### CELLULOSE IMPREGNATED FIRE-RESISTANCE AGENTS

Open flames can easily ignite paper, which has an ignition temperature of roughly 232 °C. Therefore, several studies have related the effect of cellulose impregnated fire-resistance agents on its properties, especially paper. For example, the effect inorganic fire-retardant salt such as sodium silicate and sodium on paper was investigated. It was found that addition of both fire retardant improves the retardancy of paper [18]. However, sodium silicate gave the best result. Another study also compared two kinds of fire-retardant agents, namely guanidine salts such as guanidine sulfamate and guanidine phosphate [17]. Based on the result, guanidine phosphate possessed much better retardancy than guanidine sulfamate on the wood pulp paper at 23.5% loading. The same researchers also utilized both sodium salts and guanidine salts to produce acid-base synergistic flame retardant using a pair of acid-base fire retardants, guanidine phosphate and sodium borate [17]. The fire retardant used on the wood pulp paper was much lower at 7% loading than using guanidine sulfamate alone. In a large scale, a low loading of fire-retardant agent is beneficial as it will bring down the cost to manufacture fire-retardant paper. Thus, producing synergistic fire-retardant agents had captured the interest of many researchers not only in pulp and paper but other polymers as well.

It was found that boron compound and ammonium polyphosphate (APP) had an obvious synergistic effect as boron formed a glassy substance that covers the surface of the paper when it was heated and promoted carbonization of cellulose fiber [14]. While reported combining inorganic fire retardant with phosphate or halogenated fire retardant showed a strong synergy effect as it improves the flame retardancy of the composite [16]. Another synergistic fire retardant using APP and melamine cyanurate (MCA) was investigated on bamboo pulp paper [18]. In this research, MCA was firstly decomposed into ammonia and CO<sub>2</sub>, which would dilute the oxygen in the air. Then, APP took the dominant role in the synergistic fire-retardant system and decomposed into phosphoric acid. The phosphoric acid formed a compact carbide layer on the fiber surface to isolate oxygen and inhibit pyrolysis. Thus, increased the fire retardancy of bamboo paper.

## CONCLUSIONS

Malaysia is known to have one of the fastest rates for disappearing forest due to deforestation at 14.4% of forest loss from 2000-2012. Therefore, finding an alternative for wood fibers has only become more significant. Based on the properties of bamboo fibers, such as high strength and fast-growing characteristics, it has been shown from numerous studies that bamboo has the potential to replace wood fibers as the main source of raw material for the pulp and paper industry. Unfortunately, to date, relatively limited efforts have been devoted to investigating the thermal performance and flame retardancy of impregnated fire-retardant of bamboo paper. This will impede the versatility of bamboo paper for various applications because of its high flammability and low thermal stability. Out of all fire retardant available, synergistic fire retardant had shown to be the best option as a fire-retardant agent to reinforced bamboo kraft paper.

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**POLYHEXAMETHYLENE GUANIDINE HYDROCHLORIDE (PHGH) AS  
ANTIMICROBIAL AGENT**A.M. Hazzawanni<sup>1,2</sup>, R.A. Ilyas<sup>1,3\*</sup>, R. Ibrahim<sup>2</sup><sup>1</sup>Department of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 Johor Bahru, Johor, Malaysia<sup>2</sup>Pulp and Paper Laboratory, Forest Research Institute Malaysia (FRIM), 52109 Kepong, Selangor, Malaysia<sup>3</sup>Centre for Advanced Composite Materials (CACM), Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia**ABSTRACT**

Research development on the production of biodegradable materials from renewable sources is increasing as environmental awareness rises over the year. Biopolymers, which are less expensive and abundant in nature, have recently received a lot of attention. However, cellulosic materials can experience bacterial attachment due to its characteristic. Many studies have developed a new and effective antibacterial material to overcome the infection. Therefore, Guanidine has been introduced and widely used for sterilization since it exhibits good antimicrobial properties. A lot of research has been carried out to study the efficiency of antimicrobial agents since various antimicrobial materials play an important part in many sectors. Therefore, this manuscript overviews the guanidine derivative polymer, which is polyhexamethylene guanidine hydrochloride (PHGH).

*Keywords:* Polyhexamethylene guanidine hydrochloride, polymer, PHGH, antimicrobial agent

**INTRODUCTION**

Antimicrobial films were implemented in various sectors or industries to give protection against a variety of microbes [1]–[3]. This is because most microbes are likely to cause many diseases infections, and cause harm to humans [4], [5]. Along with the improvement in human living standard, the demand for antibacterial materials are high. Therefore, antimicrobial agents are required to overcome microbial infection. Nowadays, antimicrobial agents are used in paper manufacturing, food packaging industry, wound dressing in medical equipment, clothing in the textile and footwear industries [6], [7]. Generally, antimicrobial agents can be classified into three types: organic, inorganic, and natural [8]. There are several types of chemicals that can be used as antimicrobial agents. Antimicrobial agents that are available for the prevention of microbial infection are the inorganic type such as noble metals (silver, copper, and zinc) and natural products (essential oil, biopolymer, and organic acid) [6]. Guanidine derivative polymer is mainly used as an antimicrobial agent as it possesses several unique structures with much higher antimicrobial efficiency and lower minimum inhibitory concentration (MIC) [9]. Therefore, this paper review polyhexamethylene guanidine hydrochloride (PHGH) as an antimicrobial agent.

**POLYHEXAMETHYLENE GUANIDINE HYDROCHLORIDE (PHGH)**

Guanidine has been widely used for sterilization since it exhibits good antimicrobial properties and has been used in medical, fiber, textile, plastic industries. Guanidine polymers synthesized from hexamethylene and guanidine salt have been the most extensively investigated due to good properties. Among various types of guanidine polymers, polyhexamethylene guanidine hydrochloride (PHGH) exhibits good properties such as high-water solubility, wide spectrum antimicrobial activity, excellent biocide efficiency, and nontoxicity [10]. Guanidine polymer effectively inhibits the growth of bacteria by destroying the membrane of bacteria cells and cause leakage of intracellular contents. Hence, the growth inhibition increased with the increasing of the concentration of PHGH in modified paper. Moreover, guanidine polymer is a water-soluble polycation type with broad antibacterial action against gram-positive and gram-negative bacteria. In addition, guanidine derivative polymers are mainly used as antimicrobial agents as they possess several unique structures with much higher antimicrobial efficiency and lower minimum inhibitory concentration (MIC) [9].

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**MECHANISM OF ANTIMICROBIAL AGENTS**

There are a lot of works that has been done to reveal the mechanism of antimicrobial agents against gram-positive and gram-negative bacteria. For the antimicrobial mechanism of guanidine polymer, the destruction of bacteria's cell membrane of bacteria which cause the leakage of intracellular components from bacterial cells, has been shown by UV260 absorption and Atomic Force Microscopy (AFM) images. AFM has been known as the preferred technique to investigating antimicrobial mechanisms by visualizing bacterial cells. Besides, morphology of bacterial cells exposed to guanidine polymer was also visualized using

AFM to reveal the transformation of the cell membrane and antimicrobial mechanism of guanidine polymer [11]. Therefore, AFM was applied to reveal the morphology of fresh *Escherichia coli* (*E. coli*) and the *E. coli* treated with modified guanidine polymer. By comparing the AFM images for both fresh and treated *E. coli*, it could be seen that the surface membrane was structured and integrated for fresh *E. coli* and resulting in a different pattern at low and high concentrations after being treated with guanidine polymer [11].

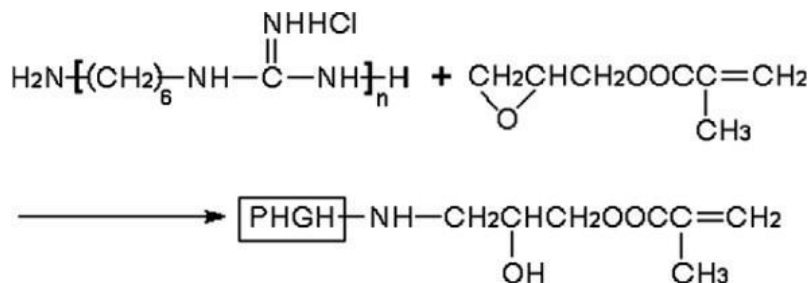


Fig. 1: Reaction between PHGH and GMA

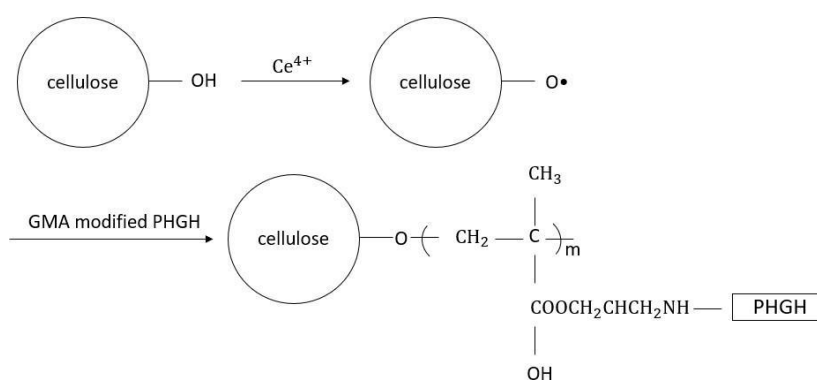


Fig. 2: In situ copolymerization of PHGH onto cellulose.

## CONCLUSIONS

Polyhexamethylene guanidine hydrochloride (PHGH) antimicrobial agent was used in many sectors or industries to provide protection against a variety of microbes. Moreover, PHGH exhibits good properties such as high-water solubility, wide spectrum antimicrobial activity, excellent biocide efficiency and nontoxicity. There were many studies and analysis shows that PHGH can prevent and inhibit the growth of bacteria. Therefore, the demand for PHGH as antimicrobial agents is high as it can overcome microbial infection and give more benefits. Plus, it can prevent harm to humans, such as a diseases infection.

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## ALKYL KETENE DIMER (AKD) REINFORCED BAMBOO KRAFT PULP PAPER

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## ABSTRACT

The presence of hydroxyl group in the cellulose fibre leads to disadvantages of cellulosic fibre where it is hydrophilic to water. Bamboo has a higher amount of cellulose fibre where it is easily to absorb water. It is estimated 90% of the main composition in bamboo is cellulose fibre. Over 1200 species is found in global evaluation and used fibre composite materials especially in paper production. The main method to produce paper used in the industry is pulping process. Pulping process can be done by mechanical or chemical method. Kraft pulping process is a chemical method to produce paper where it can give high yield of pulp from the original source. Since the presence of cellulose in bamboo composition is high, sizing agent is needed in the paper industry to give the hydrophobic element to the paper. One of the popular agent used is Alkyl Ketene Dimer (AKD), where it has many advantages and high demand in industry.

*Keywords:* Alkyl Ketene Dimer (AKD), Reinforced Bamboo, Kraft Pulping, Paper

## INTRODUCTION

Paper is a useful need in daily life. Education, food and parcel packaging, restaurant sector, building sector, and office works are using paper as their main needs in any works. Production of paper involve many steps and stages where the main two stages are the converting of raw material to pulp by pulping process and the converting of pulp into paper. The common material used to produce is wood, where the main compound in the paper structure is cellulose. Cellulose is known majorly contain in natural fibre structure where it also can be found in bamboo. Bamboo is considered as second valued non-timber forest by Malaysia government [2] Cellulose fibre has a great potential in bio composite material where it is a green material that easily degrade and has an expenditure resources especially in a tropical country likes Malaysia.

Nevertheless, the presence of hydroxyl group in the cellulose fibre leads to disadvantages of cellulosic fibre where it is hydrophilic to water. Waterproof paper would be beneficial in a variety of sectors if it is water repellent. In the recent research, superhydrophobic coatings have been investigated to give a great water repellence on paper. A superhydrophobic paper surface has a water contact angle (CA) > 150° and a water slide angle (SA) 10°, where the water will easily flow like a sphere on the surface [3]. Alkyl Ketene Dimer (AKD) is a common chemical used as the additive in sizing agent. It is classified as non-hazardous chemicals under OSHA regulations [4]. Recent research has proved that AKD is effective as sizing agent where most of the result obtain a contact angle >90°.

## BAMBOO

Bamboo is well known as a plant resource used in building industry. It is also considered as natural composite which grow rapidly in tropical country. In global evaluation, over 1200 species of bamboo was found and use as composite material [5]. In ancient time, bamboo is used as a memory book to record history and building material in China. In Malaysia, it has been recognised as second authority in non-timber forest and the researchers in Malaysia have found over 50 species of bamboo in this tropical country [6], [7]. It has been widely used as chopsticks, handcraft, papers production and baskets.

The main component in bamboo is cellulose, hemicellulose, and lignin. It is estimated that 90% of the bamboo composition is cellulose, hemicellulose, and lignin content while the 10% is other element [5]. It is important to know the chemical composition in bamboo as their characteristic also contribute to physical and mechanical properties. Since the green campaign have rapidly been promoted now, bamboo and others green resources are used to replace the unrennewable resources to produce a better environmental atmosphere.

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To utilize bamboo better in its application, physical and mechanical properties need to be understood first. In chemical composition view, bamboo consist of cellulose, hemi-cellulose, and lignin with minor element such as tannins, resins, wax and inorganic salt. Researchers [5] have develop a study on chemical composition in bamboo with different layers. It was concluded that cellulose content is increased from down to top section of bamboo, lignin content is constant, and the outer layer contain the most amount of holocellulose, alpha cellulose and Klason lignin.

## PULPING PROCESS

The production of paper or paperboard can be categorized into various stages. Paper is made in a two-step process that begins with the conversion of a fibrous raw material into pulp, followed by the conversion of pulp into paper [8]. Pulping process objective to remove the amount lignin in the fibre. The harvested wood is first treated to remove the fibres from the lignin, which is a useless component of the wood. Depending on the type and grade of paper to be produced, the pulp is bleached and further treated. To make paper sheets, the pulp is dried and pressed in a paper factory. After use, a growing percentage of paper and paper products are recycled. Non-recyclable paper is either buried or burned [9]. Pulp can be produced either mechanically or chemically [8]. This paper is focused on chemical pulping (kraft pulping). Chemical pulping is the process that undergoing the process of removal cellulose by dissolving the lignin that hold the cellulose fibre together. The kraft (sulphate) and sulphate processes are used to heat or digest the raw ingredients and produce chemical pulps [8].

The kraft (sulphate) process is the world's most widely used chemical pulping method. The term "sulphate" comes from the makeup ingredient sodium sulphate, which is used to compensate for chemical losses during the recovery process. Sodium hydroxide and sodium sulphide are the active cooking chemicals (white liquor) in the kraft pulp process. Although the Kraft procedure is applicable to all types of wood species, its chemistry has the potential to produce odorous chemicals. In comparison to sulphate pulp, kraft pulp has better pulp strength qualities [8]. The sulphate method attacks and removes lignin using a variety of chemicals. In comparison to the kraft process, which is a very homogeneous approach that can only be carried out with strongly alkaline cooking liquid, the sulphate process is characterized by its considerable flexibility. The sulphate process can be divided into distinct types of pulping based on the pH adjusted. Acid (bi)sulphate, bisulfite (Magnesite), neutral sulphate (NSSC), and alkaline sulphate are the four main sulphate pulping procedures [8].

## WATERPROOF AGENT

In paper production, internal agent commonly known as sizing agent is widely used to produced waterproof paper. Sizing agents, which are functional compounds, are the second biggest category of specialty chemicals used in paper production, after coating binder. In addition, the depletion of free energy surface on paper can prevent the water from absorbed into the paper. The reason why fluids easily impregnate in paper is because of the ability of water to form hydrogen bond with surface contain hydroxyl group as cellulose. Meanwhile, a hydrophobic surface is achieved if the contact angle produced by the water droplets less than  $90^\circ$ . The most common materials of this category are rosin size, alkyl ketene dimer (AKD), alkenyl succinic anhydride (ASA), and polymeric sizing agents (PSA) [10].

For providing water resistance to paper and paperboard products, alkyl ketene dimer (AKD) has become the most used internal sizing agent in the world. It is not only effective, but it's also simple to use. Since its growing popularity, it is more essential than ever to understand the impacts of AKD treatment on paper characteristics [11]. Many different types of paper and board goods are sized using AKD emulsions. The usage of short stay coaters has resulted in decreased dosing rates of wet end sizes and a continuous need to improve hydrophobic sizing systems as paper machine speeds have increased. Even when very high temperatures are employed in the papermaking process, it has been found that AKD size is relatively unreactive towards both water and hydroxyl-containing chemicals, such as cellulosic fibres and starch products [11]. The molecular structure of AKD is varies from  $C_{28}H_{25}O_2$  to  $C_{36}H_{68}O_2$  and molecular structure is shown in figure 2.4.

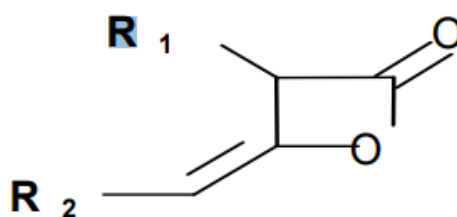


Fig. 1: Molecular Structure of Alkyl Ketene Dimer

A research conduct by Bildik et al., (2016) it shows that AKD is a good additive or sizing agent to be use. The experiment was conducted by using standard commercial paper as the sample. The result shows that the contact angle gain is  $>90$  on the highest concentration of AKD. The sample also being test with others test likes tensile strength burst index. The data shows an increasing tensile and burst index proportional with the concentration of AKD. Others experiment conduct by Chen et al., (2019), the result obtain where the highest concentration give highest contact angle. The morphology test by using Scanning Electron Microscope (SEM) also shows the presence of hydrophobic structure in the sample.

## CONCLUSION

Bamboo kraft pulp is suitable to impregnated with sizing agent. Kraft pulping process used the chemical to break the structure in high temperatures. Many researchers suggest that kraft pup bamboo have high yield where it is ranged from 60-70 %. Alkyl Ketene Dimer (AKD) can sustain in high temperature and good index reading for tensile and burst index. Recent research

shows a significant result on the test for the effectiveness of AKD on pulp. AKD give an average of contact angle result with  $>90^\circ$ . This method can give a new approach to invest in the paper production where the hydrophilic cellulosic structure in bamboo can be solved.

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## DESIGN IDEATION OF A TWO-STROKE MARINE DIESEL ENGINE CROSSHEAD BEARING DESIGN USING TRIZ AND BIOMIMETICS

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### ABSTRACT

The current design of the crosshead bearing of a two-stroke marine diesel engine is similar to that of conventional tin-based journal bearings. To consider natural fibre composites (NFC) as replacement material for journal bearings is somewhat unsuitable due to its' limited mechanical properties. For such a feat to be achieved, suitable design improvements would be necessary. By employing the innovation tool, TRIZ (Theory of Inventive Problem Solving) the preliminary step in this design process began by pointing in the right general direction. With the aid of biomimetics, several improved ideas were visualized from the initial thought ideated from TRIZ. Three potential designs inspired by nature have been selected which are the Amazon water lily, the hedgehog spine and the macadamia nut shell.

Keywords: two-stroke marine diesel engine, TRIZ, biomimetics

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### INTRODUCTION

In two-stroke marine diesel engines, the crosshead assembly plays a vital role in its efficient operations. Compared to trunk type engines, the crosshead assembly takes up the sideway forces and acts to maintain alignment of the piston and liner assembly. The crosshead assembly is essentially made up of the crosshead pin, bearing, and guides. The crosshead pin functions in an oscillating manner, centered at the crosshead pin. When the engine is at rest, the static load of the piston and piston rod assembly rests on the crosshead assembly. For a 60 cm bore sized piston, such as those on a MAN B&W S60MC, the piston and piston rod assembly would weigh around 1381 kg [5]. This load is transferred to the crosshead pin which is in direct contact with the journal bearing. Lubricating oil ceases to flow once the pumps are stopped, hence causing the oil film between the pin and bearing to get squeezed out [1]. Current marine journal bearings are made of tin based white metal lined onto a steel back, which are more than capable of withstanding this vertical load with plastic deformation.

With increasing pressure from environmental policies, more emphasis has been placed on the use of environmentally sustainable technology. The use of natural fibre composites (NFC) has already been included in some studies [2]. Concerns over the lower mechanical properties of NFC has been raised despite its natural abundance. For NFC to be considered for the manufacture of more critical parts in machineries, certain advancements would be necessary to accommodate its use. In context, journal bearing made from NFC would require some alterations to improve its worthiness in workings of a two-stroke marine diesel engine. To realize such an engineering endeavor, the authors have considered addressing the concern of compressive strength of NFC as bearing material from a geometrical standpoint. In this initial stage, the conceptual idea of the crosshead journal bearing is scrutinized for improvements to accommodate both the existing load with the use of NFC. For this stage, TRIZ and biomimetics were employed to aid in the conception of a possible design.

### TRIZ AND BIOMIMETICS

TRIZ is the Russian acronym for "Teoriya Resheniya Izobretatelskikh Zadatch", translated to mean Theory of Inventive Problem Solving. Developed by Genrikh Altshuller, TRIZ is an approach that provides systematic processes to innovative ideation. The general idea behind TRIZ is the formulation of a technical contradiction of the problem at hand. By breaking down the present condition into a contradictory statement involving two engineering parameters, a set of possible solutions can be found in the Contradiction matrix. These solutions, however, are often general statements that "point" in the general direction of the solution and not the exact answer in itself [3]. Often, employing TRIZ provides guidance to the answer by suggesting general ideas without any clear conclusion. Biomimetics on the other hand, provides solutions closer to the answer by copying from the natural world [6]. In biomimetics, scientists and researchers look towards nature in understanding how the natural world has solved similar problems are faced in the human world.

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### DESIGN IDEATION

In the case of the crosshead bearing assembly using NFC, the contradicting statement was "improve the durability of the journal bearing (moving object) without increasing the strength of the bearing material". Normally, when a

component of a system needs to be made more durable, one common means is to change it to a stronger material. However, in the case presented, the desired bearing material is NFC, which is key to the new design, thus, the situation presents itself as self-contradicting. The parameters were then put into the TRIZ Contradiction Matrix, to obtain the generic solutions. Figure 1 shows the ideation flow process.

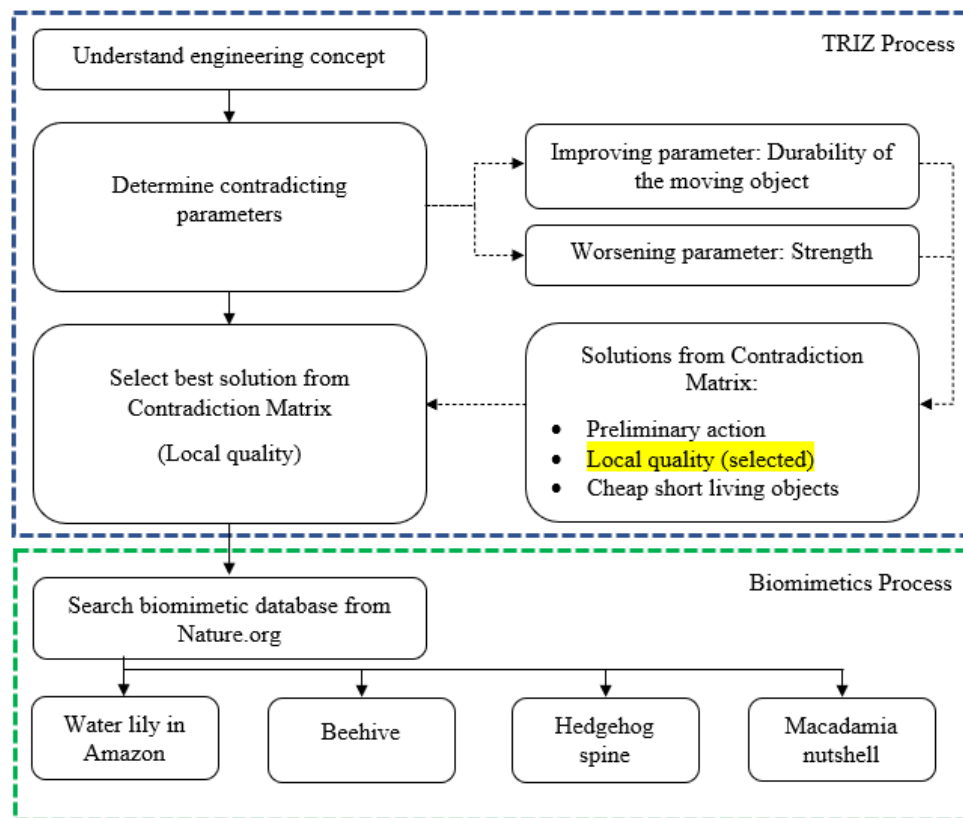
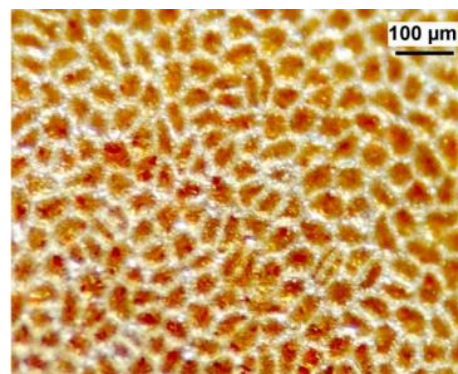


Fig. 1: Design ideation using TRIZ coupled with Biomimetics

Based on the selection of “Local quality”, the follow up action was then to find ways to innovate the crosshead bearing design that improved its capability while retaining the material strength. Here, a search was made by inserting key words in the search box of Asknature.org [6] to provide examples of solutions that nature has come up with to improve durability with a similar strength of the member part.

## RESULTS AND DISCUSSION

From the search, several ideas were generated for the design of the crosshead bearing assembly. The narrowed down ideas included the Amazon water lily, hedgehog spines and macadamia nut shells. The Amazon water lily has a ribbed structure build into its large leaves allowing it grow up to a few meters wide. These structures also provide durability in keeping the leaves intact when animals use them as means of crossing the wide river. The hedgehog spines are structured in such a way that allows it to dampen the impact of experienced by the animal when falling from heights. Macadamia nuts have shells that contain fullerene structures, making them resistant to cracks.



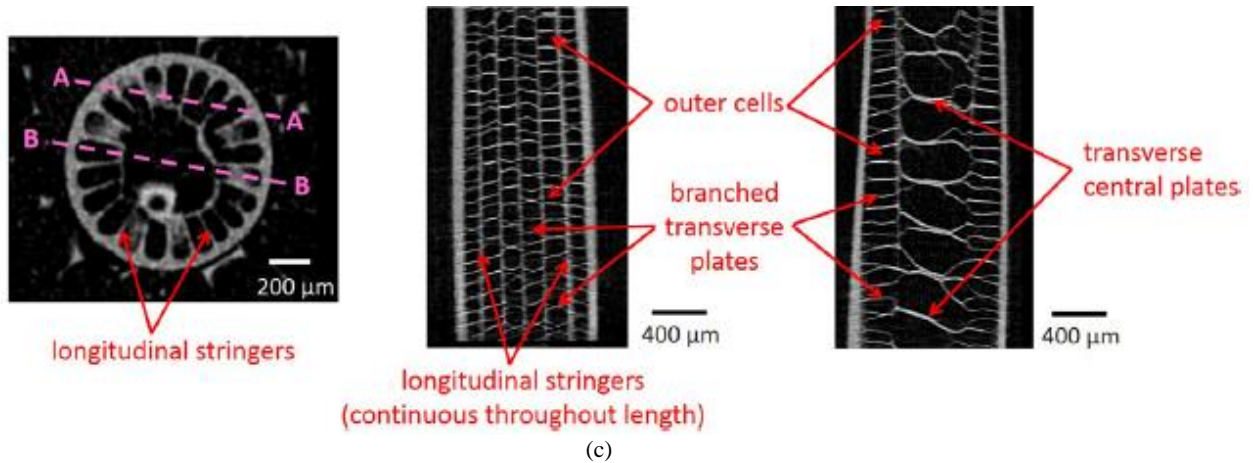


Fig. 2: (a) Ribbed structure of Amazon water lily (top left) [6], (b) fullerene structure of macadamia nut shell (top right) [6], (c) hedgehog spine internal structure [4]

## CONCLUSION

Innovative design ideation framework using the synergetic combination advantages between TRIZ and Biomimetics is presented in this paper. Further investigation will be carried out to analyze the feasibility of the proposed ideas and formulate specific geometric design solutions. The geometric designs will also be visualized with computer aided drawing (CAD) motivated by each of the following examples followed by simulation to determine the feasibility of the structures in satisfying the problem at hand.

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**TRANSFORMATION OF COFFEE HULL IN A FUNCTIONAL INGREDIENT RICH IN FIBERS**J. B. M. D. Silva<sup>1\*</sup>, M.T.P. Paiva<sup>1</sup>, S. Mali<sup>1</sup><sup>1</sup> Department of Biochemistry and Biotechnology, CCE, State University of Londrina (UEL), Londrina, PR, Brazil**ABSTRACT**

The objective of this work was to evaluate a combination of chemical (alkaline peroxide) and physical (hydrothermal) treatments in a single step to increase the cellulose content and to reduce the material's recalcitrance, increasing its fiber content, as well as to characterize the material according to its chemical composition and functional properties. As a result, there was a significant increase in insoluble fibers by 106%, with cellulose as the major component. The water absorption and swelling capacity suffered were affected by the combined treatments; it can be observed an increase in the hydration capacity due to structural and chemical changes in the material. Thus, the modification of the coffee hull through chemical and physical combined treatments was promising and resulted in a new material with potential to be used as a functional ingredient rich in dietary fibers.

*Keywords:* lignocellulosic residue, cellulose, hydrothermal treatment, alkaline peroxide.

**INTRODUCTION**

Food supply is a basic daily need for human beings. The food production chain can generate a large amount of lignocellulosic residue. In the search for technological solutions for the recovery and conversion of these lignocellulosic residues, the coffee hull can be considered an important raw material, as coffee is the second largest commodity in the world, and coffee processing can generate coffee hull corresponding to 30 - 50% of the grain total weight [1]. The objective of the work was to evaluate a combination of chemical (alkaline peroxide) and physical (hydrothermal) treatments in one-step to increase the cellulose content and to reduce the recalcitrance of the material, increasing its fiber content, as well as to characterize the material according to its chemical composition and functional properties.

**MATERIALS AND METHODS***Materials*

The coffee hulls were supplied by Instituto de Desenvolvimento Rural do Paraná - IAPAR (Londrina - PR). The residue was washed with distilled water, dried in an air-circulating oven (Marconi MA 035, São Paulo, Brazil) for 12 h at 45 °C, and then ground and sieved (180 to 300 µm) to ensure homogeneity in terms of particle size.

*Treatment of the residue*

Coffee hulls were subjected to combined treatment in a one-step as described by Debiagi et al. [2], 20g of sample was dispersed in 200 mL of alkaline peroxide solution (2%, pH 11.5), the solution was subjected to the hydrothermal treatment in an autoclave (Primatec, CS, São Paulo, Brazil) (1.5atm, 121 °C, 30 min). Then, the sample was filtered, and the solid fraction of the sample was washed (pH 5.5-6.5), and dried at 60 °C for 12 to 24 h.

*Chemical composition*

The chemical composition was performed according to the official AOAC methodologies - Official methods of analysis of AOAC (2012) [3].

*Cellulose, Hemicellulose and Lignin Contents*

Cellulose and hemicellulose were determined by the Van Soest [4] method, and the lignin content was determined according to a standard method of the Technical Association of Pulp and Paper Industry (TAPPI T222 om-88) [5].

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*Scanning Electron Microscopy (SEM)*

The morphology of the raw coffee hull and the treated coffee hull was observed by SEM. The SEM analyzes were performed with a FEI Quanta 200 microscope (Oregon, USA). The dried samples were mounted for visualization on bronze stubs using double-sided tape. The surfaces were then coated with a thin layer of gold layer (40–50 nm). All samples were examined using an accelerating voltage of 30 kV.

*Water absorption capacity*

The water absorption capacity was performed following the methodology described by Fernández-Lopez et al. [6] and the water holding capacity was expressed in g H<sub>2</sub>O/g of residue.

*Swelling capacity*

The swelling capacity was performed as described by Mateos-Aparicio et al. [7]. The residue was mixed with distilled water and then the system was stirred, and the samples were left to rest for 20h. The volume (mL) occupied by the sample was measured and divided by the mass (g) of the sample, obtaining the swelling capacity (mL/g).

*Statistical analysis*

The data were analyzed using R software (R Foundation for Statistical Computing, Vienna, Austria) and Tukey's test was employed to evaluate differences between means ( $p \leq 0.05$ ).

**RESULT AND DISCUSSION**

Table 1 describes the results of the chemical composition and functional properties of the coffee hulls before and after treatment. In general terms, the moisture, ash, lipids and proteins content decreased after the treatment, this can be explained by the structural modification resulting from the treatment, which changes the chemical composition of the lignocellulosic material, and macromolecules may be lost in the process of washing [8]. However, the content of insoluble fibers had a significant increase, differing statistically, there was an increase in the percentage 106.38% after treatment of the coffee hull. The high content of insoluble fibers is due to the presence of cellulose, hemicellulose and lignin fractions, which naturally make up the matrix of these agro-industrial residues [9].

TABLE 1: Chemical composition and functional properties of raw coffee hull and treated coffee hull

Parameter	Raw coffee hull	Coffee hull after treatment
Moisture (%)	15.09 ± 1.46 <sup>a</sup>	8.15 ± 0.13 <sup>b</sup>
Ash (%)	5.77 ± 0.02 <sup>a</sup>	2.39 ± 0.05 <sup>b</sup>
Lipids (%)	2.47 ± 0.27 <sup>a</sup>	1.62 ± 0.30 <sup>b</sup>
Proteins (%)	8.09 ± 0.24 <sup>a</sup>	4.11 ± 0.18 <sup>b</sup>
Carbohydrates		
Insoluble fibers (%)	40.92 ± 0.40 <sup>b</sup>	84.45 ± 0.21 <sup>a</sup>
Soluble fibers (%)	9.37 ± 0.03 <sup>a</sup>	2.64 ± 0.22 <sup>b</sup>
Total fibers (%)	50.30 ± 0.42 <sup>b</sup>	86.87 ± 0.10 <sup>a</sup>
Cellulose (%)	16.51 ± 2.60 <sup>b</sup>	50.40 ± 1.45 <sup>a</sup>
Hemicellulose (%)	2.90 ± 1.23 <sup>b</sup>	12.51 ± 2.74 <sup>a</sup>
Lignin (%)	27.01 ± 0.11 <sup>a</sup>	26.82 ± 0.64 <sup>a</sup>
Functional properties		
Water absorption capacity (g/g)	4.46 ± 0.52 <sup>b</sup>	6.56 ± 0.29 <sup>a</sup>
Swelling capacity (mL/g)	4.26 ± 0.23 <sup>b</sup>	5.58 ± 0.40 <sup>a</sup>

Mean ± standard deviation. Means in the same line followed by different letters are significantly different ( $p > 0.05$ ).

The percentage of cellulose in the treated residue increased from 16.5% (raw coffee hull) to 50.4% (treated coffee hull) corresponding to an increase of 205%, indicating that the combination of treatments in a one-step was efficient in increasing the content of cellulose in the coffee hull. Lignin is a component that generates recalcitrance in the residue, as a result, after modification, there was no decrease in its content, however, Fig. 1 it can be observed that the disruption of the lignocellulosic complex occurred resulting in more exposed and less compacted fiber bundles, decreasing your recalcitrance. However, lignin degradation and hemicellulose solubilization can be more expressive with the use of two or more combined treatments [10]. For the functional properties, the coffee hulls after treatment presented increased water absorption and swelling capacities. Materials that contain higher contents of soluble fibers tend to have a better hydration capacity than cellulosic fibers and lignin due to its hydrophobic capacity, depresses the water binding capacity [11]. However, in the treated material, with the alteration of the fiber structure (Fig. 1), there was an increase in its porosity, which increased the absorption of water by the residue matrix.

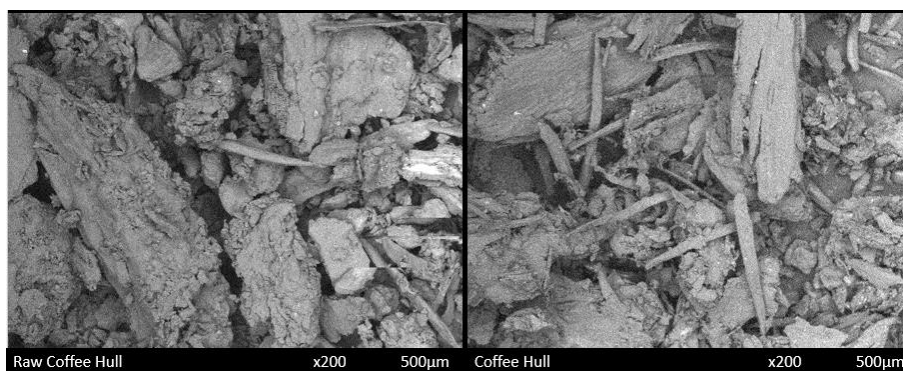


Fig. 1: Scanning electron micrographs (SEM) of raw coffee hull and coffee hull modified by chemical and physical treatment in a one-step.

### CONCLUSIONS

The combination of chemical and physical treatments in one-step process to obtain new material rich in fibers was successfully performed and resulted in materials with increased availability of cellulose and decreased lignin, content. Which is an important and significant role in health, acting mainly in the intestine regulation. Moreover, the functional properties showed an increase in the hydration capacity, obtaining a promising residue for use as a food additive.

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## CELLULOSE FROM AGROINDUSTRIAL RESIDUES: EXTRACTION AND SURFACE MODIFICATION

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## ABSTRACT

The valorization of agroindustrial residues have grown substantially, as they are interesting alternatives for cellulose extraction, because they have a low production costs and high availability. The objective of this study was to extract cellulose from oat hulls using peracetic acid and modify the cellulose with vegetable oils from different sources (soybean, sunflower and coconut) in an ultrasonication assisted-process to produce modified cellulose (MC) with increased hydrophobicity. Peracetic acid was effective as a bleaching agent, while raw oat hulls presented 26% cellulose, 30% hemicellulose and 22% lignin, after bleaching with peracetic acid, the obtained sample presented 81% cellulose, 7% hemicellulose and 3% lignin. The increased hydrophobicity of modified samples was evidenced by the wettability parameter, indicating that after modification with vegetable oils the modified samples presented higher affinity by a non-polar solvent.

*Keywords:* lignocellulosic residue; hydrophobicity, oil absorption capacity.

## INTRODUCTION

The use of agroindustrial residues to obtain new products is inserted into the concept of biorefineries, which can be described as the evolution of technologies, consisting of integrated systems of sustainable processes. The biorefinery concept meets the vision of a sustainable economy using biological resources, maximizing benefits and profits through strategies to add value to the plant biomass chain [1]. Oat hull is a byproduct of oat grain milling, and represent 25 to 30% of the oat grain weight, with approximately 90% insoluble fibers, a cellulose content of approximately 28 - 35%, hemicellulose of 18 - 28% and lignin of 18 - 22% [2].

The transformation and valorization of agroindustrial residues have grown substantially, as they are interesting alternatives for cellulose extraction, as they have a low production costs, high availability, low density, good thermal and mechanical resistance [2]. In order to extract cellulose from lignocellulosic materials, the structural arrangement of the lignocellulosic complex has to be disrupted to remove hemicellulose and lignin. Peracetic acid (PA) is a strong oxidizing bleaching agent suitable for the extraction of cellulose from agroindustrial residues, as it has low environmental impact when compared to the chlorinated reagents employed in conventional processes [2,3].

Regardless of the origin of the cellulose, the chemical structure is the same: glucose units connected by  $\beta$  (1-4) glycosidic bonds between positions 1C and 4C [2, 3]. Due to the richness of hydroxyl groups on its surface, the cellulose molecule has a polar character and chemical modification by esterification promotes the replacement of hydroxyl groups by nonpolar molecules, resulting in more hydrophobic surfaces. A large proportion of the chemicals used for the chemical modification of cellulose and other biopolymers are toxic and derived from nonrenewable sources. Thus, in the last few years some less aggressive organic reagents from renewable sources have been studied for cellulose modification [4]. Thus, the objective of this study was to extract cellulose from oat hulls using peracetic acid and modify the cellulose with vegetable oils from different sources (soybean, sunflower and coconut) in an ultrasonication assisted-process to produce modified cellulose (MC) with increased hydrophobicity.

## MATERIALS AND METHODS

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*Materials*

Oat hulls were donated by a local oat processing industry (SL Alimentos e Cereais, Paraná, Brazil). Soybean (Cocamar, Maringá – PR, Brazil), sunflower (Cocamar, Maringá – PR, Brazil) and coconut (Natural Life, São José dos Campos – SP, Brazil) oils were used without further purification.

*Methods**Cellulose Extraction from Oat Hull*

Cellulose was extracted from oat hull by bleaching with peracetic acid according to Marim *et al.* [3].

*Cellulose Modification with Vegetable Oils*

Cellulose modification was performed based on the protocol described by Dong *et al.* [4] (2013), with some modifications. Three different vegetable oils, soybean, sunflower or coconut oil, were used. About 1.0 g of each oil was dispersed in 38 g of ethanol and 2.5 g of cellulose, the mixture was submitted to ultrasound treatment (Fisher Scientific Sonicator model 505, Pittsburgh-PA, USA) coupled with a probe with a tip diameter of 1.27 cm (Fisher Scientific model FB 4219, Pittsburgh-PA, USA), employing 40% amplitude for 1 or 2 min (MCA401min and MCA402min samples) and 80% amplitude for 1 or 2 min (MCA801min and MCA802min samples). The samples were placed in an oven (Tecnal, São Paulo, Brazil) at 110 °C for 2 h, and the material was washed by centrifugation (Hettich centrifuge, universal model 320R, Germany) for 30 min at 9000 rpm

#### Cellulose, Hemicellulose and Lignin contents

Cellulose and hemicellulose contents were determined by Van Soest method [5], and lignin contents were determined by TAPPIT222 om-88 method [6].

#### Wettability

Samples (cellulose and MC) were mixed with two immiscible solvents, water (density = 1.000 g cm<sup>-3</sup>) and dichloromethane (d: 1.335 g cm<sup>-3</sup>), with different polarities and densities to observe affinity between samples and solvents, according to the protocol described by Namazi and Dadkhah [7].

## RESULT AND DISCUSSION

Oat hull bleaching was performed with peracetic acid to obtain pure cellulose. Raw oat hulls presented 26% cellulose, 30% hemicellulose and 22% lignin, and after bleaching with peracetic acid, the obtained sample presented 81% cellulose, 7% hemicellulose and 3% lignin, indicating that the bleaching with peracetic acid was effective in the lignin and hemicellulose removal, resulting in a material composed mostly by cellulose. Marim *et al.* [3] reported that peracetic acid is a non-toxic and efficient bleaching agent to obtain cellulose from agroindustrial residues.

In Fig. 1 we can observe the dispersion of the samples in a immiscible solvent system prepared with water and dichloromethane. It can be observed that after stirring unmodified cellulose migrated to the water fraction, which are located on the top of the container, indicating its higher affinity for water, which can be attributed to the presence of the hydroxyl groups located on its surface [8].

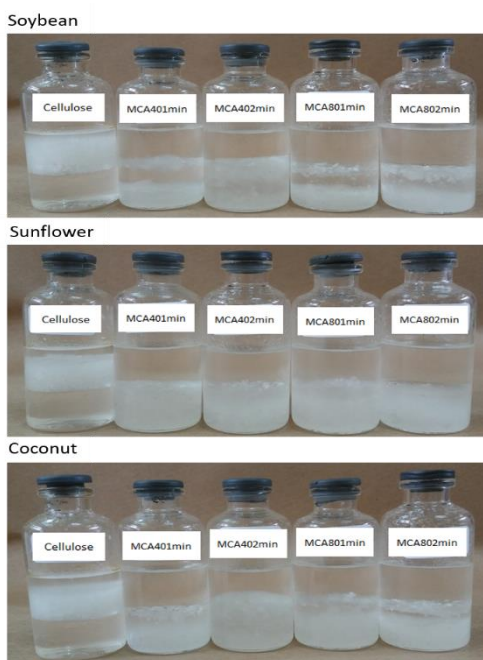


Fig. 1: Dispersions of cellulose and modified cellulose (MC) in a water/ dichloromethane system.

All MC samples were able to migrate into the dichloromethane after stirring (Fig. 1) because non-polar groups from oils replaced the hydroxyl groups located on the cellulose surface, suggesting an increase in their hydrophobicity. These results are similar to those presented by Dong *et al.* [4] and Sai *et al.* [6]. These authors reported that cellulose hydrophobization can be observed by changes in its wettability pattern. Regardless of the treatment employed in this study, an increased affinity for dichloromethane was observed.

## CONCLUSIONS

The different operational conditions employed in the modification and the different oil sources did not affect the main properties of the modified samples. After modification, all samples presented higher affinity for the non-polar solvent, indicating its higher hydrophobicity.

### ACKNOWLEDGEMENTS

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**TENSILE PROPERTIES OF POLYPROPYLENE COMPOSITES REINFORCED BY BIO-NATURAL FIBER (SANSEVIERIA LEAF FIBER)**S. R. Ahmad<sup>1\*</sup>, M. F. Hamid<sup>2</sup> & S. S. M. Saleh<sup>3,4</sup>

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**ABSTRACT**

Currently, the reinforcement used in the polymer composite is more expensive due to the complexity of the preparation process. The additional reinforcement in thermoplastics, especially bio-natural fibre, is to reduce the application of plastic that is hard to dispose of for certain applications. The combination of reinforcement in polymers is to increase certain mechanical properties of pure polymers as well as to reduce the material cost. Nowadays, Sansevieria has become a viral plant that can be the perfect houseplant. This plant requires less water and is easy to grow either outdoors or indoors. In this research, the effect of fibre loading on tensile test of a polypropylene (PP) filled bio-natural fibre called Sansevieria leaf fibre (SLF) was investigated for bumper fascia application. The additional SLF as filler is to reduce plastic usage, besides to overcome the limitation of pure PP. The PP/SLF were mixed at 0, 10, 20, 30 and 40wt% fibre loadings in a Haake PolyLab QC mixer machine at 180°C with a rotor speed of 50rpm for 10 min. Then, the dumbbell shape was obtained from the Hydraulic hot-press Machine (GT 7014) before the tensile test of the composites were analyzed. The tensile strength and the elongation at break of the PP/SLF composite decreased by 53% and 76%, respectively. However, the Young's Modulus of the composites increased (27%) as well as increasing the SLF loading.

*Keywords:* bio-natural fiber, sansevieria leaf fiber, polypropylene, polymer composite, tensile properties

**INTRODUCTION**

The biodegradable-natural fibre (Bio-NF) can be used as a component of a composite material, where the orientation of fibres has a significant impact on the properties. Natural fibre reinforced composites are an emerging area in polymer science and the natural fibres are low-cost fibres with low density and high specific properties. Bio-NF is made from natural fibre that can be obtained from either crops or animals, such as hemp, kenaf, wool, and others. Those materials can be easily disposed of, which makes them commonly used as reinforcement in polymer composites. After blending with polymers, waste materials are transformed into engineering materials suitable for the manufacturing, automotive, and aerospace industries today. Previously, the presence of bio-natural fibre was used to blend with thermoset. In the new era of novel materials development, the widespread use of thermoplastics makes the combination of bio-natural fibre and thermoplastics one of the most interesting research topics.

Bio-NF thermoplastic composites offer several advantages. For instance, lowering the application of host polymer that is hard to dispose of towards the environment and sustainability, reducing the cost of host polymer from petroleum-based products, with comparable properties and suitable for specific applications, Sansevieria Leaf is the viral or trending indoor plant currently in Malaysia. It is easy to plant, take care of, and grow with minimum effort. The leaves of Sansevieria were typically arranged in a rosette around the growing point, although some species were distichous. There was great variation in foliage from within the genus. Besides the ability of absorption as well as reducing the radiation from electronic devices such as computers, laptops, televisions, and smartphones, the fibre of this plant is very useful. Sansevieria Leaf Fiber is one of the stronger fibres among other plant fibres. The fibre is extracted from the leaves to make coarse fabrics, ropes, and tail-ropes [1]. More importantly, nowadays, almost all parts of the plant are used for medicinal purposes.

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Polypropylene (PP) is a petroleum-based material. In many aspects, it is similar to polyethylene, especially in solution behaviour and electrical properties. The additionally present methyl group improves mechanical properties and thermal resistance, while the chemical resistance decreases. PP composites are widely used as automotive components and parts. The low density (lightweight), ability to be recycled, non-toxic and non-hazardous properties. The relatively low cost of PP makes these composites very significant engineering materials today. The addition of SLF to PP is expected to enhance the tensile properties of the polymer composites. In this study, the effect of filler loading on tensile properties for PP filled SLF composites were investigated.

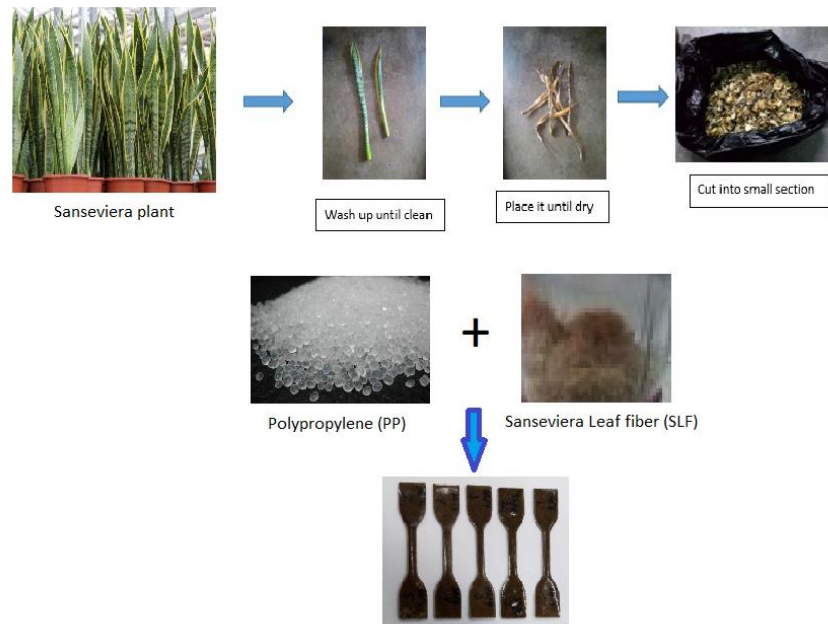


Fig. 1: Biodegradable filler preparation from Sansevieria plant and Polypropylene/Bio-NF(SLF) composites

TABLE 1: Mechanical properties of Bio-NF (Sansevieria leaf fiber) filled Polypropylene composites

Authors	Polymer	Source of nanocellulose	Description
Abral & Kennedy [2]	Sansevieria-trifasciata/ Polypropylene	Sansevieria-trifasciata	• Additional of 2 % of ST content caused slight increase of tensile strength in the PP/ST composite.
Apang et al., [3]	Sansevieria-trifasciata/ Polypropylene	Sansevieria-trifasciata	The untreated and unidirectional <i>sansevieria trifasciata</i> fiber/Polypropylene with fiber volume fraction of 15%, which was 48.092kJ/m <sup>2</sup> for impact resistant.

## MATERIALS AND METHODS

### Materials

A Sansevieria plant in Kulim (Kedah Darulaman, Malaysia) were used in this study. Polypropylene was obtained from Polypropylene Malaysia Sdn Bhd in the form of paletlets with a grade of S12232 G112. Table 2 show the formulation of PP/SLF composites at different SLF loading.

TABLE 2: Formulation of PP/SLF composites at different filler loadings.

Material	PP/SLF composites (wt%)
Polypropylene (PP)	100, 90,80,70,60
Sansevieria fiber (SLF)	0, 10, 20,30,40

### Preparation of Sansevieria Leaf Fibre

Firstly, the leaves were dipped and rubbed by hand in clean water to remove the dirt and soil. After that, the leaves were placed on the tray for the drying process. After the leaf is completely dry, it is cut into small pieces by shortening the fibre. Then the material was blended in a rough blender. After that, the product was again blended by using a fine blender to get into the tiniest particles. The product was filtered using a sieve to obtain 1 mm short fibers.

### Mixing Process

The mixing of composites was prepared in the Haake Polylab QC Mixer Machine at a temperature of 180 °C and a rotor speed of 50 rpm. Polypropylene was discharged into the mixing chamber. It will melt completely into the mixer chamber after 5 minutes. Then, the SL fibre is inserted. The mixing continues for another 5 minutes until the 10-minute mark is reached. The mixing It took 10 minutes for each mix to be completed. The previous steps were repeated five times for each composition. Based on table 2, the composite mixtures are prepared with different loadings of SL fiber.

### Compression Molding.

The composite mixtures are then compressed in a hydraulic hot press machine (GT 7014) to produce a 1.0 mm sheet of composite using the moulding of a dumbbell shape to comply with the ASTM D638 standard. The force was set at 10 kN and the temperature at 180 °C. The mould was placed on the machine to pre-heat it for five minutes. After that, the sample was pressed for 4 min. After completing the process, the sample was removed and blown with air to make it cool faster. The process was completed after it was removed from the mould.

#### Measurement of Tensile Properties.

The tensile test is carried out according to ASTM D638 using a Universal Testing Machine (UTM): GALDABINI. A dumbbell-shaped (Type IV) specimen is needed for reinforced composite testing. The testing was done in a standard laboratory atmosphere of 25 °C ± 3 °C. The tensile testing machine was used with a crosshead speed of 50 mm/minute and the gauge length was set at 50 mm. The test is carried out on five specimens for each formulation.

## RESULT AND DISCUSSION

Figure 2 shows the tensile strength, Young's modulus, and elongation at break of SLF filled PP at 0, 10%, 20%, 30%, and 40% of SLF loading. The tensile strength has decreased by about 53% due to the size and random orientation of the fibers. However, the Young's modulus of the composites increased when the sansevieria leaf fibre content was increased (27%). This happened due to the increased stiffness of the composites [4]. The elongation of the sansevieria composite gradually decreased as SLF loading increased from 10% to 40%. It might be owing to the hard contact between the filler and the PP matrix's limited deformability. The drop is proportional to the amount of fibre present. According to Supri et al. (2009), the elongation at break is reduced because the fillers have hardened the composites and reduced their ductility [5].

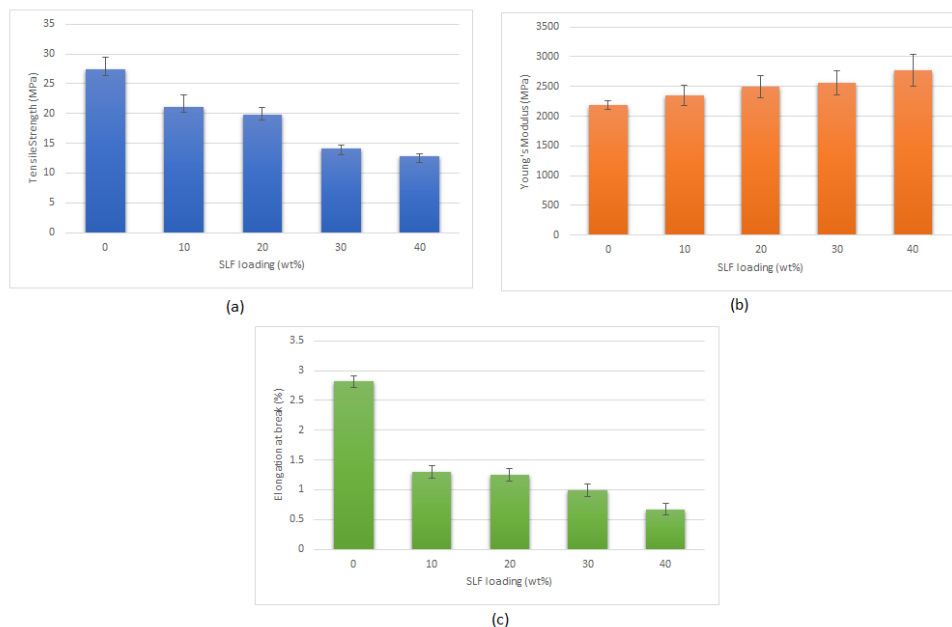


Fig.2 The tensile properties of PP/SLF composites at different SLF loading; (a) Tensile strength, (b) Young's modulus and (c) Elongation at break

## CONCLUSIONS

The addition of SLF enhanced the Young's modulus of PP/SLF composites. However, the tensile strength and elongation at break were decreased as the SLF loading increased. The PP/SLF composites were expected to be used in automotive applications, for example as bumpers.

## ACKNOWLEDGEMENTS

The authors would like to thank Universiti Kuala Lumpur Malaysian Spanish Institute, Universiti Malaysia Perlis, TAJ International college for the collaboration towards the development of new materials for automotive application.

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## BIODEGRADABLE-NATURAL FIBER (COCONUT MESOCARP) FILLED POLYPROPYLENE COMPOSITES: EFFECTS OF THE COMPATIBILIZER AND COUPLING AGENT ON TENSILE PROPERTIES.

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### ABSTRACT

Malaysia is in Southeast Asia, a region rich in natural resources. One of the remarkable things is the biodegradable-natural fiber such as Coconut Mesocarp (CM). It is also known as fibrous husk and coir. It has an outstanding potential as reinforcement in thermoplastic composites. This CM thermoplastic composite is high in demand and widely used in automotive, construction and other industries. In this study, Coconut Mesocarp is used as a filler to enhance the mechanical properties of the composite that offers several advantages. For instance, they are lightweight, relatively low-cost, non-toxic, and non-hazardous, making them suitable for specific applications. Polypropylene (PP) is used as a matrix polymer. Maleic Anhydride grafted Polypropylene (MAPP) as a compatibilizer and 3-Aminopropyltriethoxysilane (3-APE) as a coupling agent to improve the bonding between PP and CM. The composites were mixed at 0, 10, 20, 30 and 40wt% fiber loadings in a Haake PolyLab QC mixer machine at 180°C with a rotor speed of 50rpm for 20min. Then, the dumbbell shape was obtained from the Hot-Press Machine (GT 7014) before analyzing its tensile properties. Young's modulus increased, but tensile strength and elongation at break of composites decreased by increasing the filler loading. The PP/CM with coupling agent composites show higher tensile strength and Young's modulus, but lower elongations at break compared to PP/CM with compatibilizer and PP/CM composites. Therefore, the additional 10wt% of CM composition brings an improvement to the composite properties.

*Keywords:* biodegradable-natural fiber, coconut mesocarp, polypropylene, compatibilizer, coupling agent, composite

### INTRODUCTION

Biodegradable-natural fiber (Bio-NF) is a fibre made from natural fiber that can be obtained either from crops and animals such as hemp, kenaf, wool and others. Those materials can be easily disposed makes them commonly used as filler in polymer composites. From the waste materials, they become engineering materials after blend with polymer that suitable to manufacturing, automotive and aerospace industries nowadays. Petrochemical based fibers are expensive due to the increasing of oil price, the use of Bio-NF as substitute is seen to be feasible in terms of cost saving. Previously, the presence of bio-natural fiber is used to blend with thermoset. However, the widely used of thermoplastic makes the combination of bio-natural fiber and thermoplastic gain the interest of researchers toward the development of novel materials.

Bio-NF thermoplastic composites offers several advantages. For instance, lower the application of host polymer that hard to dispose towards the environment and sustainability, reduce cost of host polymer from petroleum based product, comparable properties and suitable for specific application. Coconut Mesocarp (CM) is well known as coir and coconut fibrous husk is one of bio-natural fibre. The application of CM in polymer such as Polypropylene is increased drastically and become trending in global market.

Polypropylene composites are widely used as automotive components and parts. The low density (lightweight), can be recycle, non-toxic, non-hazardous, relatively low cost makes PP composites very significant engineering materials today. However, the addition of CM in PP reduce the mechanical properties such as strength of PP as host polymer itself. To overcome this problems, the compatibilizer to treat the host polymer and coupling agent to treat a filler are applied.

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In this study, MAPP is use as compatibilizer and 3-APE as silane coupling agent that act as a booster to the mechanical properties of PP/CM composites. The comparison effect of both filler and matrix treatment compared to untreated PP/CM composites towards their properties were discussed in this paper.

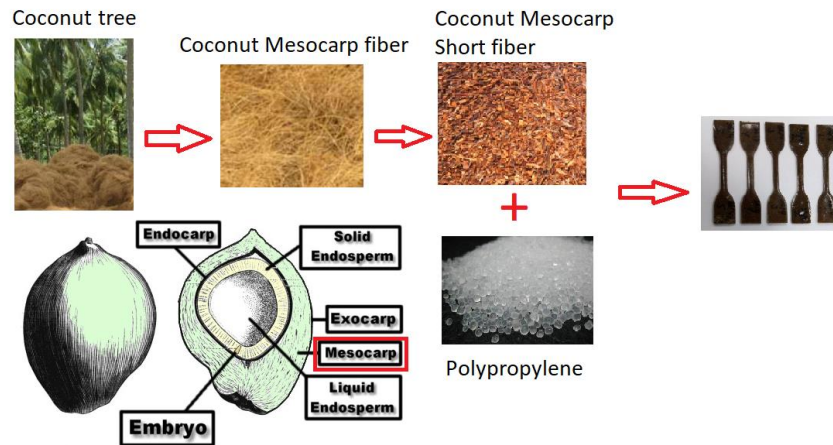


Fig. 1: Biodegradable-natural Fiber (Coconut Mesocarp short fibre) from the coconut tree to the PP/CM composites process.

TABLE 1: Water permeability properties of biodegradable films reinforced with nanocellulose for food packaging application

Authors	Polymer	Source of nanocellulose	Description
Bettini et al., [1]	Polypropylene/Coir fiber	Coir fibre	• The presence of PP-g-MA increased tensile properties
Samia et al., [2]		Coir fiber	• The performance of coir fiber composites in industrial application can be improved by chemical treatment.
Sabri et al., [3]	Polypropylene/Coconut fiber/MAPP	Coconut fiber	• By increasing of filler loading from 10% to 40%, elastic modulus was increased while the tensile strength and elongation at brake was decreased. The present of MAPP increased tensile properties of the composites.
Sabri et al., [4]	Polypropylene/Coir fiber/3-APE	Coconut fiber	• The additional 3% of 3-APE increases the tensile properties of the treated composites compared to untreated composites. The Young's modulus was increased. However, tensile strength and elongation at break decrease as filler content increases.

### Materials

The coconut fiber is supplied by Avasia Agro Sdn. Bhd. in a form of coconut husk and Polypropylene grade S12232 G112 is supplied by Polypropylene Malaysia Sdn Bhd in a form of pellet. The coconut fiber is extracted from the fiber of coconut husk and cut into tiny pieces with average length of 1 mm to 3 mm. Table 1 show the formulation of PP/CM composites at different filler loading. The Maleic Anhydride grafted Polypropylene (MAPP) with a grade 426512 is supplied by ExxonMobil Chemical. The 3-(Aminopropyl) triethoxysilane (3-APE) is supplied by ZARM Scientific & Supplies Sdn Bhd from Sigma Aldrich.

TABLE 2: Formulation of PP/SLF composites at different filler loadings.

Material	PP/CM composites (wt%)	PP/CM/MAPP composites (wt%)	PP/CM/3-APE composites (wt%)
Polypropylene (PP)	100, 90,80,70,60	100, 90,80,70,60	100, 90,80,70,60
Sansevieria fiber (SLF)	0, 10, 20,30,40	0, 10, 20,30,40	0, 10, 20,30,40
Maleic Anhydride grafted Polypropylene (MAPP)*	-	3	-
3-(Aminopropyl) triethoxysilane (3-APE)*	-	-	3

\*wt% of filler

### Preparation of Coconut Mesocarp Fibre

Firstly, the leaves were dipped and rubbed by hand in clean water to remove the dirt and soil. After that, the leaves were placed on the tray for the drying process. After the leaf is completely dry, it is cut into small pieces by shortening the fibre. Then the material was blended in a rough blender. After that, the product was again blended by using a fine blender to get into the tiniest fiber. It was filtered using a sieve to obtain 1-3 mm short fibers.

### Filler Treatment.

The filler treatment is not required for MAPP. It was added directly into the mixing chamber. However, for 3-APE, the filler treatment is required. Firstly, 3-APE was diluted in ethanol using a stirrer. The amount of 3-APE used is 3 wt.% of the filler

weight. The filler is charged into the chamber mixer with the solution slowly to ensure uniform distribution of 3-APE. The filler was continuously mixed for another 1 hour. After an hour, the filler is filtered out and then dried in a forced-convection oven at 80 °C for 24 hours to allow complete evaporation of the ethanol.

#### Mixing Process

At a temperature of 180 °C and a rotor speed of 50 rpm, composites were mixed in the Haake PolyLab QC Mixer Machine. Polypropylene was discharged into the mixing chamber. It will melt completely into the mixer chamber after 5 minutes. Then the fibre is inserted. The mixing continues for another 5 minutes until the 10-minute mark is reached. The mixing It took 10 minutes for each mix to be completed. For compatibilized and treated composites, after 3 min of the fibre being inserted into the mixing chamber, the MAPP or 3-APE (after filler treatment) was added before the process continued to complete at 10 min. Based on table 2, the composite mixtures are prepared with different loadings of SL fiber.

#### Compression Molding.

The composite mixtures are then compressed in a hydraulic hot press machine (GT 7014) to produce a 1.0 mm sheet of composite using the moulding of a dumbbell shape to comply with the ASTM D638 standard. The force was set at 10 kN and the temperature at 180 °C. The mould was placed on the machine to pre-heat it for five minutes. After that, the sample was pressed for 4 min. After completing the process, the sample was removed and blown with air to make it cool faster. The process was completed after it was removed from the mould.

#### Measurement of Tensile Properties.

The tensile test is carried out according to ASTM D638 using a Universal Testing Machine (UTM): GALDABINI. A dumbbell-shaped (Type IV) specimen is needed for reinforced composite testing. The testing was done in a standard laboratory atmosphere of 25 °C ± 3 °C. The tensile testing machine was used with a crosshead speed of 50 mm/minute and the gauge length was set at 50 mm. The test is carried out on five samples for each formulation.

## RESULT AND DISCUSSION

Figure 2 shows the tensile strength, Young's modulus, and elongation at break of PP/CM composites at 0, 10%, 20%, 30%, and 40% of SLF loading.

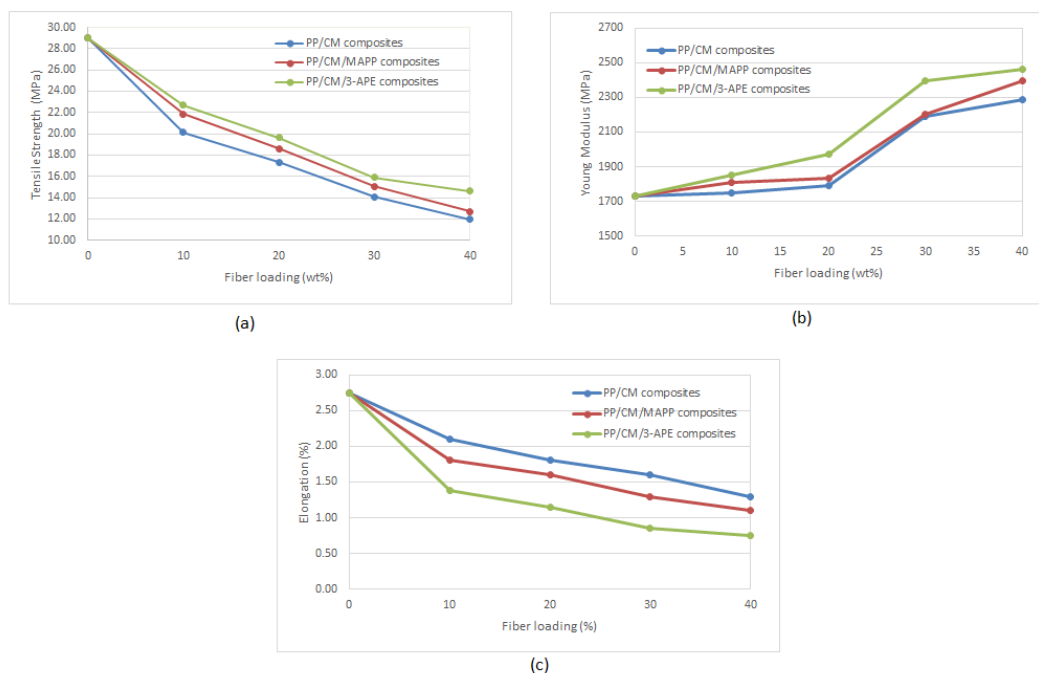


Fig.2 The tensile properties of PP/CM, PP/CM/MAPP and PP/CM/3-APE composites at different fiber loading; (a) Tensile strength, (b) Young's modulus and (c) Elongation at break

The tensile strength has decreased by about 53% Due to the polarity difference between the phases, composites without compatibilizers have poor fibre attachment to the matrix, resulting in a loss in tensile strength. [1]. But the Young's modulus of the composites increased when the CM fibre content was increased (27%). This happened due to the increased stiffness of the composites [4]. The elongation of the composite gradually decreased as CM loading increased from 10% to 40%. It might be owing to the hard contact between the filler and the PP matrix's limited deformability. The drop is proportional to the amount of fibre present. According to Supri et al. (2009), the elongation at break is reduced because the fillers have hardened the composites and their brittleness [5]. The present of 3-APE as silane coupling agent shows the composites reached near 2500MPa of Yong's modulus.

### CONCLUSIONS

The Young's modulus of the composites enhanced by present of CM as the filler. Nevertheless, the tensile strength and elongation at break were decreased as the CM loading increased. The PP/CM composites were expected to be used in automotive applications, for example as bumpers. The additional of 3-APE in PP/CM composites indicates the improvement of tensile properties compared to MAPP and without treatment (control) composites.

### ACKNOWLEDGEMENTS

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## THE ENERGY-ABSORBING CHARACTERISTIC OF CELLULAR CORE STRUCTURES MADE OF FLAX-BASED COMPOSITES

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### ABSTRACT

The mechanical behavior of flax fibre reinforced polylactide (PLA) composites of sandwich structure with double cell wall square interlocking and honeycomb cores are investigated experimentally under the quasi-static compression loading. The square interlocking core is fabricated through a slotting technique, whereas the honeycomb core made using a corrugated mould that initially used to create the corrugated core composite profile, which then cut into corrugated webs and assembled to form the honeycomb core. For each type of core, four specimens with dimension of 87 x 87 mm were created (length x width). The sandwich structures are tested at a crosshead displacement rate of 2 mm/min. The experimental results showed that honeycomb outperform the square interlocking core in terms of their loading capability and SEA by around 14%, and 34% respectively.

*Keywords:* Compressive strength, energy absorption, flax, honeycomb core, interlocking core.

### INTRODUCTION

The usage of natural fibre composites in engineering applications has expanded in recent years. Because natural fibre composites are a more environmentally, low cost, low density, have good specific strength and specific energy absorptions less hazard and easy to handle compare to synthetic fibre composites. Several natural fiber-reinforced polymer composites have recently been investigated for usage in sandwich structure can be found in [1-5]. In this paper, square interlocking of double cell walls and honeycomb core structure made of flax/PLA composites were investigated to study their compression properties.

### EXPERIMENTAL PROCEDURES

Initially, flax/PLA composites were manufactured using a hot press machine under the pressure of 10 bar and temperature of 180°C with three plies of flax fibre and 20 plies of PLA film. Then, the double cell walls of core interlocking structures was fabricated using the slotting technique. The dimensions of the sample produced are approximately 87 mm x 87 mm x 20 mm (length x width x height). Similarly, a corrugated mould is used to create the corrugated core composite profile, which later cut into corrugated webs and assembled to form the honeycomb core with a dimension of 87 mm x 87 mm x 20 mm (length x width x height). Figure 1 shows the photograph image of square interlocking core and honeycomb core of flax/PLA.



Fig. 1: Photograph of (a) square interlocking core and (b) honeycomb core of flax/PLA

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### RESULTS AND DISCUSSION

Figure 2 show the results of the quasi-static tests for the flax/PLA composites of interlocking square honeycomb core structures. As seen in Figure 2, the loading gradually rises until it reaches around 29 kN for honeycomb core and 25kN for interlocking core structure at

displacements of around 2.5 mm and 1.1 mm, respectively. Local densification of the interlocking core occurred simultaneously with core experiencing local buckling of the cell walls when displacement reached 11 mm, resulting in higher load with additional displacement rise. In contrast to the honeycomb structure, the collapsed core components made contact with the skins at 12 mm. The average SEA values for interlocking and honeycomb core structures are around 5.4kJ/kg and 8.2kJ/kg, respectively. The difference in SEA between the two core structures was around 34%.

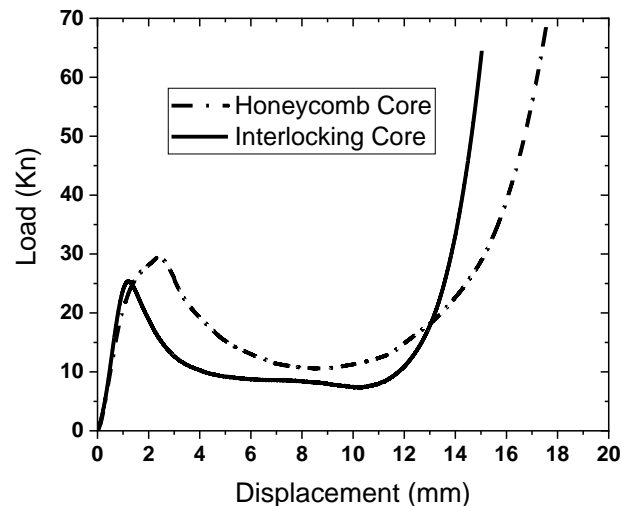


Fig. 2: Typical load-displacement of honeycomb and interlocking core structure

## CONCLUSIONS

The compressive properties of interlocking square and honeycomb core structures that made of flax/PLA composite have been studied. According to the results, the honeycomb core structure outperformed the interlocking core structure in terms of loading capability and SEA by around 14% and 34%, which might be attributed to the fact that the honeycomb core structure has lesser mass than its counterpart. It is also worth noting that although there is no significant difference in their densifications displacements.

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**CELLULOSE NANOFIBRILS FROM TEXTILE WASTES TO BE USED AS REINFORCEMENT IN BIO-NANOCOMPOSITES**Rajesh Mishra<sup>1\*</sup>, Michal Petru<sup>2</sup>

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**ABSTRACT**

Textile industries generate significant amount of waste fibers in form of short lengths during mechanical processing. However, these short fibers possess excellent properties suitable for many other applications. The objective of this work was to use them for the preparation of nanoparticles/nanofibrils, as fillers in biodegradable composite applications such as food packaging, agriculture mulch films, automotive plastics, etc. In the present study, waste jute fibers generated in textile industries, were wet pulverized to the scale of nanofibrils of 50 nm diameter using high energy planetary ball milling for three hours. The rate of refinement of uncleaned jute fibers having non-cellulosic contents was found slower than the cleaned jute fibers. This tendency is attributed to the strong holding of fiber bundles by non-cellulosic contents which offered resistance to the defibrillation during wet milling. In addition, the presence of water during wet pulverization found to reduce the rising temperature of mill, which prevented sticking of nanofibrils on the mill wall and resulted in unimodal size distribution. In the subsequent stage, 1 wt %, 5 wt % and 10 wt % of jute nanofibrils were incorporated in poly lactic acid (PLA) matrix to prepare nanocomposite films by solvent casting. The reinforcement of nanofibrils was investigated from the improvements in mechanical properties based on tensile tests, dynamic mechanical analysis and differential scanning calorimetry. The maximum improvement was observed in case of 5 wt % nanocomposite film where initial modulus and tensile strength increased by 217.30 % and 170.59 % respectively compared to neat PLA film. These improvements are attributed to the increased interaction between nanofibrils and matrix as well as to the increased crystallinity of PLA in composites. The improvements in load bearing capacity of nanocomposite films were significant at 60°C than 35°C, which showed ability of jute nanofibrils to improve the softening temperature of PLA matrix. In the end, experimental results of Initial modulus were compared with predicted modulus of mechanical models. A good level of agreement was observed up to 5 wt % loading of jute nanofibrils.

*Keywords:* nanocellulose, bio-nanocomposite, nanofibrils, food application.

**INTRODUCTION**

The nanostructures of cellulose have gained significant amount of importance due to its higher mechanical properties. The crystalline segments in cellulose have a greater axial elastic modulus than the synthetic fiber Kevlar, and their mechanical properties are within the same range as those of other reinforcement materials such as carbon fibers, steel wires and carbon nanotubes [1,2]. The nanostructures of cellulose are considered as bundles of molecules which are elongated and stabilized through hydrogen bonding. The remarkable improvements in mechanical properties of cellulose nanostructures, in range of 130-170 GPa, are considered due to this parallel arrangement of molecular chains which are present without folding [3]. In a unique manner, these nanocelluloses combine important cellulose properties such as hydrophilicity, broad chemical-modification capacity, and the formation of versatile semicrystalline fiber morphologies due to the large surface area of these materials. Previous work on composites made from cellulose nanostructures showed improved strength and stiffness with a little sacrifice of toughness, reduced gas/water vapor permeability, lower coefficient of thermal expansion, and increased heat, deflection temperature [4]. These properties thus could promise in replacement of conventional petroleum-based composites by new, high performance, -and lightweight green nanocomposite materials [5].

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*Materials*

Short waste jute fibers were obtained from India. The fibers were measured to have a density of 1.58 g/cm<sup>3</sup>, modulus of 20 GPa, tensile strength of 440 MPa and elongation of 2 %. The chemical composition of fibers was reported as cellulose (60 %), hemicelluloses (20 %), lignin (10 %) and others (10 %). Poly lactic acid (PLA) was purchased from NatureWorks LLC, USA through local supplier Resinex, Czech Republic. The PLA had a density of 1.25 g/cm<sup>3</sup> and the average molecular weight (M<sub>w</sub>) of 200,000. The chloroform which was used as solvent, purchased from Thermofisher, Czech Republic.

*Methods*

**Preparation of nanoscale jute fibers.** After getting the optimum milling parameters of ball milling process, wet pulverization of waste jute fibers was carried out in distilled water using a high-energy planetary ball mill of Fritsch pulverisette 7. The sintered corundum container of 80 ml capacity and zirconium balls of 3 mm diameter were chosen for 3 hours of wet milling. The ball to material ratio (BMR) was kept at 10:1 and the speed was kept at 850 rpm with reverse rotation of containers. At the end of wet milling, jute particles were separated from water by centrifugation at 4000 rpm and simultaneously transferred in solvent isopropanol to avoid hornification during drying. On the basis of their dimensions, functions, and preparation methods, nanocelluloses are classified in three main subcategories as nanocrystalline cellulose (NCC), nanofibrillated cellulose (NFC) and bacterial nanocellulose (BNC) as shown in Figure 1.

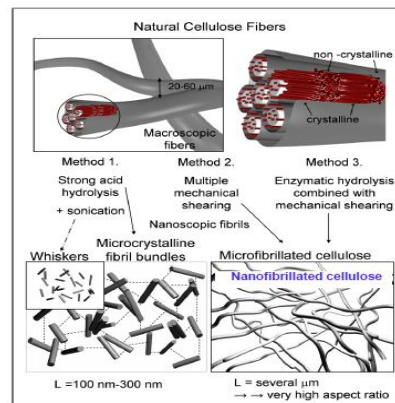


Fig. 1 Types of cellulose nanostructures [5]

## RESULT AND DISCUSSION

### Effect of milling condition on particle size reduction of jute fibers

Under one-hour dry milling, jute fibers were pulverised to microparticles with average size of 1480 nm in wider particle size distribution as shown in Figure 2(a) and Figure 3(a). The reason behind multimodal distribution of particles was due to increase in temperature within the mill because of continuous impact of balls. The increased temperature of mill rendered the jute particles to undergo cold welding and deposited a layer on the surface of container and balls as milling progressed. The growth of deposited layer on the milling media changed the impact force of balls on the material with least impact on particles at bottom of layer. In case of wet milling, the increase in temperature was slowed down by deionised water which consequently resulted in narrow particle size distribution with significant reduction in average particle size to 640 nm after one hour of wet milling as shown in Figure 2(b) and Figure 3(b). This can be attributed to uniformity in impact action of balls on every individual particle in wet condition.

### Dynamic mechanical analysis (DMA) of PLA/JNF composites

The load bearing capacity of neat PLA and PLA composite films was studied from the storage modulus results shown in the Figure 4. The storage modulus of the PLA composite films improved over the entire temperature span compared to neat PLA. The maximum improvement was observed in case of 5 wt % nanocomposite where storage modulus was increased from 3.0 GPa to 9.0 GPa at 35°C. This increase in storage modulus value is attributed to the higher stiffness of JNF during the transfer of stress from the matrix to nanofibrils. However, with further increase in loadings of JNF to 10 wt %, storage modulus reduced to 5.0 GPa because of poor dispersion and agglomerations of nanofibrils.

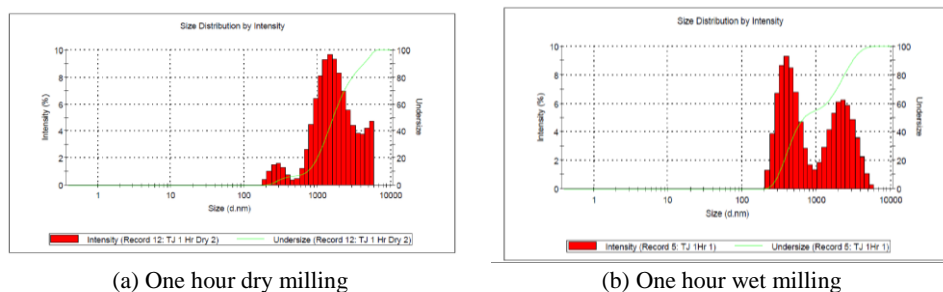


Fig. 2 Particle size distribution of jute particles

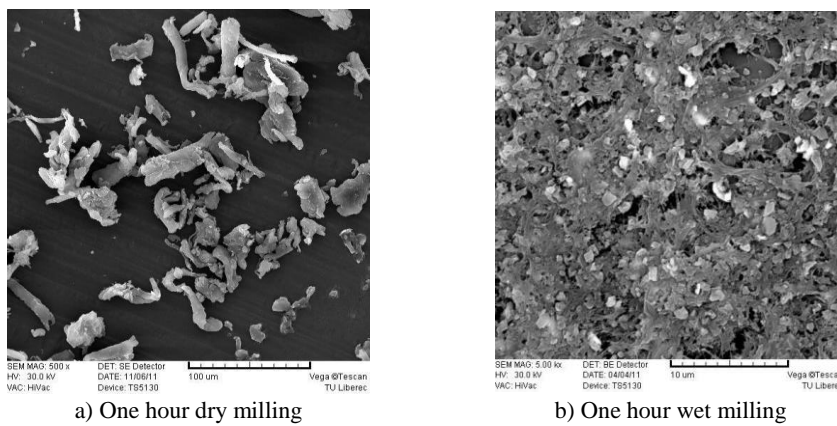


Fig. 3 SEM image of jute particles

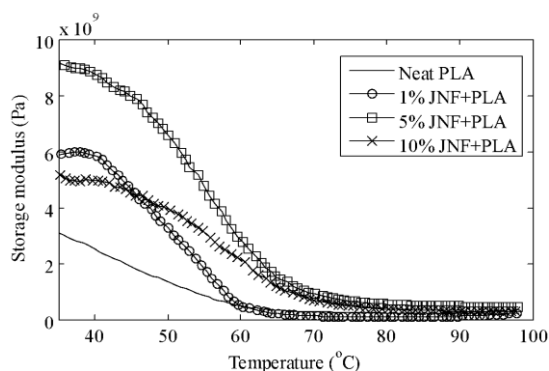


Fig. 4(a) Storage modulus of neat and JNF/PLA composite films

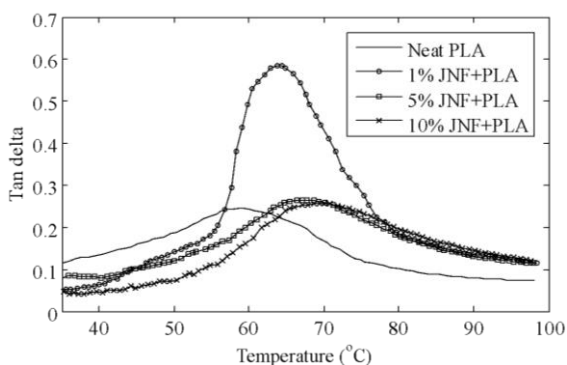


Fig. 4(b) Damping factor of neat and JNF/PLA composite films

## CONCLUSIONS

Water vapor barrier properties of biodegradable films were improved by addition of cellulose nanofibers. The goal of present study was to utilize the waste jute fibers in textile industry as a source of cellulose nanofibrils for reinforcement of biodegradable packaging films. The jute nanofibrils were obtained by wet pulverization using high energy planetary ball milling process instead of strong acid hydrolysis due to its simple, economical and environment friendly approach. The extended wet milling for the duration of three hours resulted into unimodal distribution of jute nanofibrils with diameter below 50 nm. In the subsequent step, obtained jute nanofibrils were incorporated at 1 wt %, 5 wt % and 10 wt % loading in PLA matrix and their reinforcement was evaluated based on improvements in mechanical properties. The results showed that improvements in mechanical properties are dependent on interaction between nanofibril-matrix as well as on crystallinity of PLA in nanocomposites.

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## MECHANICAL PROPERTIES OF GREEN COMPOSITE BASED ON POLYVINYL ALCOHOL (PVA) AND CASSAVA STARCH FILLED EDAMAME SOYBEANS PEEL FIBER

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### ABSTRACT

The effects of using synthetic plastics cause problems for the environment such as environmental pollution. A new breakthrough was made to replace the synthetic plastic material, namely the use of green composite material from a combination of Polyvinyl Alcohol (PVA), cassava starch (CS) and edamame soybeans peel fiber (ESPF). Green composites were manufactured using a solution casting process. Tensile test and morphological imaging by scanning electron microscopy (SEM) were carried out to determine the mechanical properties of green composite and bonding condition between matrix and fiber in fracture surface, respectively. The results showed that the highest tensile strength was found in pure PVA film for 7.25 MPa. The addition of CS and ESPF decreased the mechanical properties of green composites. This was triggered by poor adhesion bonding between the matrix/fiber and agglomerated fiber.

*Keywords:* green composite, polyvinyl Alcohol (PVA), cassava starch (CS), edamame soybeans peel fiber (ESPF), mechanical properties

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### INTRODUCTION

Plastics for packaging are usually made from petroleum-based polymers that cause environmental pollution. In an effort to reduce petroleum-based polymers and its use, many researchers have finally turned to biopolymers. Biopolymer is one of the best solutions to overcome current problems due to its properties such as easily decomposed [1]. The use of biopolymer as a matrix has several weaknesses, one of which is low mechanical properties and thermal stability. To overcome this case, other components are needed to improve into biopolymer matrix, such as natural fibers or fillers. Polyvinyl Alcohol (PVA) is a candidate biopolymer which has good biodegradable properties and good film formation [2]. However, the weakness of PVA lies in the production price which is quite expensive. Therefore, the addition of starch and natural fiber is an alternative to cover these weaknesses.

### MATERIALS AND METHODS

#### Materials

Edamame soybean peel fiber (ESPF) was obtained from edamame soybean sellers in Jember Regency, East Java, Indonesia. Polyvinyl Alcohol (PVA) technical type was purchased from CV. Aneka Kimia Jember (full hydrolysis). The cassava starch (CS) under "Pak Tani" brand was purchased from a local shop in Jember.

#### Methods

Tensile test was carried out using a Hung Ta type 2328 tensile testing machine with a capacity of 2 kN. All green composite samples were molded according to ASTM D882. The test was conducted at room temperature and at 1 mm/minute of speed test.

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### RESULT AND DISCUSSION

In this study, it can be seen in Figure 1 that the highest tensile strength was shown in the pure PVA for 7.25 MPa, which was followed by a decrease with the addition of starch and fiber variations. The addition of fiber did not increase the tensile strength of the PVA biocomposite with cassava starch. The decrease in the tensile strength value was strengthened by the results of the Scanning Electronic Microscope (SEM) test, which

showed the amount of accumulation or clumping and gaps in the green composite from PVA/cassava starch/edamame soybean peel fiber.

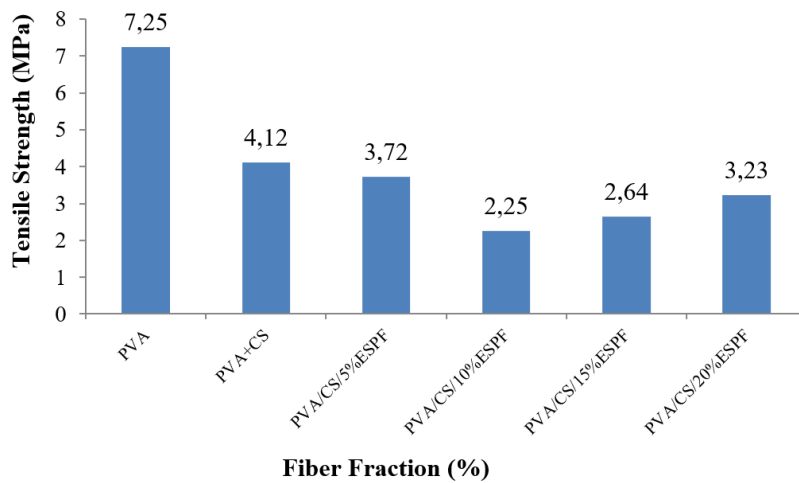


Fig. 1: Tensile strength of all green composite tested

## CONCLUSIONS

The highest tensile strength value is found in the sample of pure PVA film, which is 7.25 MPa. The addition of starch and fiber reduces the tensile strength of the green composite. This was due to poor bonding between PVA, starch and edamame soybean peel fibers.

## ACKNOWLEDGEMENTS

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**GRAPHENE OXIDE/ CELLULOSE AEROGELS  
NANOCOMPOSITE**Chong Li Hui<sup>1</sup>, R.A. Ilyas<sup>1,2\*</sup><sup>1</sup> School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia (UTM), Johor Bahru 81310, Johor, Malaysia<sup>2</sup> Centre for Advanced Composite Materials (CACM), Universiti Teknologi Malaysia, 81310 UTM Johor Bahru, Johor, Malaysia**ABSTRACT**

The porous nanomaterials have drawn much attention and rapid development due to their superb properties. Graphene oxide/cellulose aerogel nanocomposite is one of the popular materials with potential applications and environment remediation. Graphene oxide is a good nanofiller with long-range  $\pi$ -conjugation and abundant oxygen containing functional group. This allows it to have extraordinary thermal, mechanical and electrical performance as well as strong interactive ability with polar polymer and solvents. Besides, cellulose aerogel is also widely used in many fields due to its biocompatibility, biodegradability, and hydrophilicity. Cellulose aerogel has also contributed to the advantages of high specific surface area, low cost, and good chemical stability properties. This review focuses on the studies and discussion from different literature of graphene oxide/ cellulose aerogels as well as performance and properties as nanocomposite.

*Keywords:* graphene oxide; cellulose; aerogels; nanocomposite.

**INTRODUCTION**

Coating is a process of enhancing material and protecting them from mechanical and chemical damages by offering layers on any exposure structure. There are many properties that coatings can offer, such as enhancing surface hardness, providing corrosion or wear resistance, and modifying the surface texture [1]. Composite coatings are the result of co-deposition of fine inert particles into a metal matrix from an electrolytic or electroless bath [2]. The purpose of adding co-deposition additives is to improve the performance of coatings. These composites especially metal matrix nanocomposite exhibit excellent performance such as having unique mechanical, magnetic and optical properties. However, most of the synthetic are heavy metals that will cause harm to human health and the environment. Besides, these applications will cause damages to organs, disturb the enzyme system of the human body and cause environmental toxicity even at low-level exposures [3]. Hence, organic additives received great attention nowadays as an alternative to improve the performance of coatings.

Cellulose aerogels can be prepared using nanocrystalline cellulose, bacterial cellulose or various plant cellulose [4]. Nanocrystalline cellulose technique produces cellulose aerogels with brilliant mechanical properties comparable to other reinforcement materials while bacterial cellulose has high crystalline structure, purity, and aspect ratio [5]–[9]. Ultra-light aerogels can be obtained by incorporating graphene oxide with bacterial cellulose suspension [10]. Cellulose aerogels derived from plant fibers have been widely used as reinforcements in polymers for packaging and biomedical applications [11]. The potential plant cellulose nanofiber (CNF)-based aerogels, including composites and coatings, have become extensive review due to their properties, especially biodegradability potential. There are many plants materials employed in the fabrication of cellulose aerogel such as wheat straw, cotton, filter paper and bamboo fiber [12].

The porous nanomaterials have drawn much attention and rapid development due to their superb properties. Graphene oxide/cellulose aerogel nanocomposite is one of the popular materials with potential applications and environmental remediation. Graphene oxide is a good nanofiller with long-range  $\pi$ -conjugation and an abundant oxygen-containing functional group. This allows it to have extraordinary thermal, mechanical and electrical performance as well as strong interactive ability with polar polymer and solvents [13]. Besides, cellulose aerogel is also widely used in many fields due to its biocompatibility, biodegradability, and hydrophilicity. Cellulose aerogel has also contributed to the advantages of high specific surface area, low cost, and good chemical stability properties [14]. This manuscript overviews the recent advanced graphene oxide/ cellulose aerogels nanocomposite.

**GRAPHENE**

Graphene, the new nanocarbon, is the thinnest yet strongest material in the world. Graphene is a single layer atomic plane of graphite, which is sufficiently separated from its bulk to reach free-standing. It is a two-dimensional material composed of

layers of carbon atoms forming six-membered rings [15]. It is impossible to grow a 2D crystals naturally as it will transform into a stable 3D structures under high temperature growing condition. The thermal fluctuation prohibits the macroscopic 2D object to remain stable and causes the phonon density to

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integrate over the 3D space available for thermal vibrations when their lateral size increase (Geim, 2009).

There are two artificial approaches in graphene isolation which are mechanical split and chemical cleavage. The handcraft manual technique splits strongly layered materials into the individual atomic plane. This provides high structural and electronic quality graphene, but the whole process is very time-consuming. Besides, chemical cleavage such as chemical exfoliation and ultrasonic cleavage are also some automated processes in graphene isolation. In these processes, graphite is chemically detached by intercalation and produced through sonification on industrial scale. The alternative routes to grow graphene epitaxially are by chemical vapor decomposition of hydrocarbons on metal substances and by thermal decomposition of SiC [17].

Graphene is widely used in the laboratory and industrially as it has many superior properties. It performs giant intrinsic mobility with electron mobility of  $2.5 \times 10^5 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ , has Young's modulus of 1TPa, intrinsic strength of 130GPa, very high thermal conductivity which is above  $3000 \text{ W mK}^{-1}$ , optical absorption of exactly  $\pi\alpha = 2.3\%$ , completely impermeability to any gases and the ability to sustain extremely high densities of electric current (Geim & Novoselov, 2009). Hence, graphene is widely used as composite material for painting and coating, energy generation and storage, sensors and metrology, and bio-applications.

Graphite exfoliation have been primally focused on producing graphene oxide (GO), a nonconductive hydrophilic carbon material. The most common graphite used for oxidation is flake graphite, a naturally occurring purified mineral that is readily dispersed in water and suitable for large-scale preparation. The process increased the interlayer spacing characteristic and improved its overall performance. Hummer's method is generally utilized, but the treatment with  $\text{KMnO}_4$ ,  $\text{NaNO}_3$  in concentrated  $\text{H}_2\text{SO}_4$  will generate toxic and explosive gases such as  $\text{NO}_2$ ,  $\text{N}_2\text{O}_4$  and  $\text{ClO}_2$ . Hence, an improved synthesis method is introduced by the usage of  $\text{KMnO}_4$  and a 9:1 mixture of concentrated  $\text{H}_2\text{SO}_4 / \text{H}_3\text{PO}_4$  and it is obtained that the improved method shows more regular structure with a greater amount of basal plane framework retained, higher yield, no hazardous gases evaluation during preparation and possess equivalent conductivity upon simpler protocol [18].

### AEROGEL

Aerogel is a microporous solid material derived from a gel, in which the pore liquid is replaced by air without significant shrinkage of the typical solid structure. In conceptual example, we can say that aerogel has structure similar to the solid networks of a gel with gas, or vacuum in-between the skeletons [19]. This unusual microstructure results in aerogel exhibit many unique properties such as having the ultralow thermal conductivity, modulus, dielectric constant, refractive index, sound velocity, relative density with the ultrahigh porosity and high specific surface area compared to all solid ever tested. Besides, aerogel can be classified into two categories, which are single-component aerogels and aerogel composites.

There are many recent developments for aerogels production. Ambient pressure drying is one of the developments for cheaply produced aerogels. The alkoxide sol-gel method is used by aging the wet gels in water/alcohol solution and followed with alkoxide/alcohol solution. This increases the Young's modulus of the wet gel sufficiently for it to overcome the capillary pressures from surface tension. The next approach is the rapid supercritical extraction process which is a rapid heating process of a non-constrained gel. Solid and liquid phases of the wet gel show different thermal expansion coefficients and results in the rise of stresses. Besides, we can also produce aerogel through aerogel beads. The production of aerogel beads is carried out with spraying an acid and water-glass solution from a mixing jet into a flask, where the beads are solvent exchanged and then super-critically dried with respect to  $\text{CO}_2$  [20].

The applications of aerogels are unlimited. To mention a few of those, aerogel can reduce the acoustic wave by amplitude and velocity due to its general texture. The part in which energy is transferred from gas to solid phase resulted in the lost to the acoustic propagation. Next, the homogeneous porous texture adaption of aerogels resulted in its application for the optical field. Its inhomogeneities in the nanometer are responsible for Rayleigh scattering and it can entrap all kinds of molecules with special fluorescent properties [21]. Moreover, aerogels can be utilized as filler. It can provide paints and varnishes some thickening characteristics, act as a carrier for diverse components, store or transport hazardous liquids or any other tasks consists of adsorbing and extracting some chemical components.

### GRAPHENE REINFORCED CELLULOSE AEROGEL

Graphene oxide is a bulk scalability substance rich in oxygen functioning group in its two-dimensional carbon layered structure such as carboxylic, epoxy, and hydroxyl groups. These characteristics of graphene oxide improve their biocompatibility and absorbance capacity as matrix, building blocks and supporting materials to nanocomposite. At the same time, cellulosic materials also consist of the abundance of hydroxyl group and are appeared to be a very good supporting material in metallic nanoparticle immobilization. Figure 1 shows the schematic illustration of hydrogen bond interactions between graphene oxide sheets and cellulose molecules. To prepare graphene reinforced aerogel, graphene oxide aqueous dispersion was added into the cellulose solution according to desired mass ratio [13]. The mixed solution is then stirred and frozen to undergo the regeneration and freeze-drying process. It is observed that the preparation techniques for cellulose aerogel, bamboo aerogel, and graphene reinforced bamboo aerogel are the same despite the changes of raw materials. The fabrication of graphene oxide/cellulose aerogel nanocomposite through the mixing-regeneration-freeze drying process shows excellent electromagnetic shielding ability, good electrical conductivity, hydrophobicity and flame retardancy [13].

## CONCLUSIONS

Nanocomposite properties of biodegradable cellulose aerogels were improved by the addition of graphene oxide. From the literature we can summarize graphene oxide characteristics, which are rich in oxygen functioning groups such as carboxylic, epoxy and hydroxyl group have promoted high hydrophilicity and interaction with the cellulose aerogels. The obtained performance can be explained by the strong interaction bond formed between graphene oxide and cellulose. The interactive bond restricted the mobility of cellulose polymer and induced carbon yield at the interface of graphene oxide and cellulose. The performance of the nanocomposite also shows improved properties from different literature to investigate the additional of metal oxide into the natural fiber- PP composites.

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## ULTRASONICATION-ASSISTED PROCESS FOR FUNCTIONALIZATION OF CELLULOSE EXTRACTED FROM OAT HULLS

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## ABSTRACT

Lignocellulosic fibers are complex materials composed essentially of cellulose, hemicellulose and lignin. The agribusiness is responsible for bring forth numerous lignocellulosic residues, such as oat hulls, the main by-product of oat milling, which is often used for animal feed and even unduly discarded. In the biorefinery concept, the use of this residue for the extraction and modification of cellulose provides a product with higher added value. Therefore, the present study aimed to modify cellulose from oat hulls by combination of ultrasonication and citric acid and to analyze the consequences of the modification in terms of hydrophobicity and by Fourier transform infrared spectroscopy (FTIR). The FTIR spectrograms of the modified cellulose exhibit an expressive new band at 1740 cm<sup>-1</sup>, a band not presented in the cellulose *in natura* spectrum, associated with ester carbonyl groups (C=O) that were introduced in cellulose after modification, which also presented a higher hydrophobic character observed by a decrease in its the water holding capacity. The new properties of modified cellulose expand its use in the industry.

*Keywords:* agroindustrial residue, cellulose, new materials.

## INTRODUCTION

Brazil is one of the largest producers and exporters of agricultural products in the world. In 2021, Brazil will produce around 1028 thousand tons of oats, which have multiple uses, such as grain production (human and animal food), forage (grazing, hay, silage), ground cover, green manure, furthermore, inhibit invasive plant infestations and improve soil health. For the processing of grain production, the mainly use, the hull and other residues are removed, representing about 50% of the total weight, in which these hulls are most often inadequately discarded and even burned, causing negative environmental impacts. One of the ways to give an adequate destination to this agroindustrial residue is its use for cellulose extraction [1-3].

Cellulose is a linear and unbranched homopolymer, composed of D-glucose molecules linked by  $\beta$ -1,4-glycosidic bonds. The hydroxyls present in its structure allow the formation of intra and intermolecular hydrogen bonds, which lead to form crystalline regions. However, beyond the possibility of interaction with neighboring glucose molecules, the hydroxyls can also interact with water molecules, an instability factor to the polymer. The polarity and richness of hydroxyl groups enable the chemical modification of cellulose, a technological alternative to increase its applicability [4].

There are several physical and chemical methods for the derivatization of biomaterials [5]. Nonetheless, currently only a few studies describe the chemical modification of cellulose with products from a renewable source, through simple, fast and low-cost methods. Therefore, the objective of this study was to chemically modify cellulose extracted from oat hulls by combination of ultrasonication and citric acid treatment and to analyze the consequences of the modification in terms of hydrophobicity and by Fourier transform infrared spectroscopy.

## MATERIALS AND METHODS

*Materials*

Oat hull was kindly provided by the company SL Cereais e Alimentos (Mauá da Serra, Paraná, Brazil). Citric acid (purchased from Acros Organics) and all reagents used in this study were of analytical grade (PA).

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*Methods**Cellulose modifications*

Cellulose was extracted from oat hulls according to Paschoal *et al.* [7] protocol employing peracetic acid as a bleaching agent. Cellulose modification was performed using an adapted protocol from Dong *et al.* [6], where 2,5 g of cellulose and 1 g of citric acid was dispersed in 38 g of ethanol under continuous stirring, and this mixture was submitted to an ultrasound apparatus (Fischer Scientific-600W) at different times (1 and 2 min) and with different amplitudes (40% and 80%). The samples were labeled according the operational conditions as MC-A40-1min, MC-A40-2min, MC-A80-1min and MC-A80-2min, for samples subjected to amplitudes of 40 and 80% for 1 and 2 min, respectively. The obtained material was dried at room temperature for ethanol evaporation. Then, each sample was taken to the oven at 110 °C for 2 h and washed by centrifugation with ethanol three times and dried at room temperature.

*Water holding capacity (WHC)*

About 0.5 g (M) of each sample was dispersed in 10 ml of distilled water in a centrifuge tube, previously weighed (M1), which was placed in a water bath at 37 °C for 30 min. Afterwards, the tubes were centrifuged for 15 min at 4000 rpm and the supernatant was removed and weighed (M2). WHC was estimated as described by Zhang, Zhang and Shresta [8]. as follows:

$$\text{WHC} = (M_2 - (M + M_1)) / M_1$$

*Fourier Transform Infrared Spectroscopy (FTIR)*

Dried samples were incorporated into potassium bromide and subjected to high pressure to produce tablets. Infrared spectra were recorded on a Shimadzu FTIR Prestige-spectrometer (Japan) in the range of 4000–500 cm<sup>-1</sup>, which has a spectral resolution of 4 cm<sup>-1</sup>.

*Statistical analysis*

The Tukey's test employed to evaluate difference between means ( $p \leq 0.05$ ) were performed using R software (R Foundation for Statistical Computing, Vienna, Austria).

**RESULT AND DISCUSSION***Water holding capacity (WHC)*

Water holding capacity is the ability of cellulose fibers to hold water in their structure per unit weight of cellulose, especially when subjected to external forces, like centrifugation. The results presented in Table 1, show a higher WHC for the unmodified cellulose (10.93 g/g), which was significantly higher from the modified celluloses, which did not present significant difference between themselves, ranging from 3.32 to 3.37 (g/g), due to the probable reaction of the hydroxyls on the surface of the cellulose with carboxyl groups, forming esters, causing an increase in hydrophobicity [9].

TABLE 1: WHC of cellulose and modified celluloses (MC-A40-1min, MC-A40-2min, MC-A80-1min e MC-A80-2min).

Sample	WHC (g H <sub>2</sub> O/g Sample)
Cellulose	10.93 ± 0.14 a
MC-A40-1min	3.37 ± 0.14 b
MC-A40-2min	3.35 ± 0.13 b
MC-A80-1min	3.34 ± 0.13 b
MC-A80-2min	3.32 ± 0.14 b

<sup>a, b</sup> Different letters in the same column indicate significant differences ( $p \leq 0.05$ ) between means (Tukey's test).

*Fourier Transform Infrared Spectroscopy (FTIR)*

Figure 1 shows the FTIR spectra of cellulose *in natura* and modified celluloses (MC-A40-1min, MC-A40-2min, MC-A80-1min e MC-A80-2min). At 1740 cm<sup>-1</sup> a difference is observed between the spectrogram signals of the modified cellulose and the cellulose *in natura*, since it is attributed to the stretching of the C=O bond, indicating the probable substitution of the hydroxyls of the cellulose by ester radicals [10]. All other peaks presented on all samples are related to the cellulose peaks and are consistent to those presented by other authors [3,7,11].

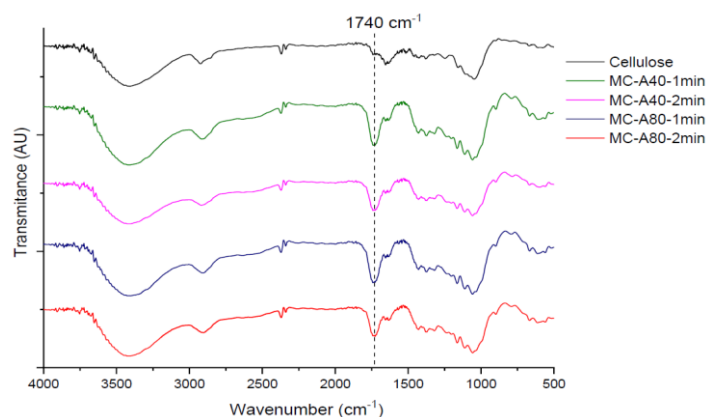


Fig. 1: FTIR Spectra of cellulose and modified celluloses (MC-A40-1min, MC-A40-2min, MC-A80-1min e MC-A80-2min).

### CONCLUSIONS

Chemically modified celluloses were obtained through treatment with citric acid assisted by ultrasonication. The chemical substitution of the surface was evidenced by the increase of hydrophobicity and the new characteristic band of ester shown on FT-IR of the modified celluloses at 1740 cm<sup>-1</sup>. Therefore, due to the use of products from renewable sources and the low waste generation, the treatment used was considered promising for the chemical modification of cellulose.

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## CROSSLINKING OF OAT HULL CELULOSE WITH TARTARIC ACID BY REACTIVE EXTRUSION

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### ABSTRACT

The current study aimed to crosslink cellulose extracted from oat hull employing a simple and eco-friendly process based on reactive extrusion, using tartaric acid (TA) as a crosslinking agent. TA used at different concentrations (0, 5, 12.5, and 20% - g/100 g cellulose). The degree of substitution (DS) of crosslinked cellulose ranged from 1.58 to 2.93, which was confirmed by Fourier transform infrared spectroscopy (FT-IR) with a new band appearing at 1735 cm<sup>-1</sup> associated with ester carbonyl groups. The samples crosslinked with TA 20% were more efficient as a crosslinking agent, resulting in samples with higher DS. This study proves that reactive extrusion can be an effective technology for the crosslinking of cellulose, as the process is eco-friendly, simple, with short reaction times, and is easy to adapt to an industrial scale.

*Keywords:* cellulose, organic acids, crosslinking

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### INTRODUCTION

The use of agroindustrial residues to obtain cellulose is an interesting alternative to transform these materials, which generally contain about 20-50% cellulose, 20-30% hemicellulose and 20-30% lignin [1]–[3]. Cellulose is a biopolymer that regardless its origin, its chemical structure is the same; D-glucose units linked by  $\beta$ -(1 → 4)-glycosidic bonds between the hydroxyl group of C4 and the C1 carbon atom. Cellulose has a linear polymeric chain with a large number of hydroxy groups [3]–[5]. Hydroxyl groups can participate in several chemical reactions, which makes cellulose a versatile platform for surface chemical modifications, including crosslinking. Cellulose is a material from a renewable source, biodegradable and non-toxic that has attracted a lot of attention due to its advantageous properties, such as its wide applicability in various fields. However, some limitations of cellulose may be related to the dissolution of this biopolymer, cellulose is hardly dissolved in water and most organic solvents, and the introduction of crosslinking through the esterification reaction of hydroxyl groups gives a hydrophobic character to cellulose, making it more compatible with nonpolar polymers and solvents [5].

Tartaric acid (C<sub>4</sub>H<sub>6</sub>O<sub>6</sub>) is a dicarboxylic acid and its reaction with cellulose occurs due to the attachment of the carboxylic group via esterification with a cellulosic hydroxyl group, and also a further reaction via esterification with another cellulosic hydroxyl group can produce a crosslink between cellulose chains [6]. The reactive extrusion process is an efficient method to modify cellulose without generating polluting residues [3]. Reactive extrusion combines the thermo-mechanical energy needed to catalyze the reaction between cellulose and organic acids, in a single process without the use of other solvent reagents [7]. Therefore, this study aimed to extract cellulose from oat hulls and then obtain crosslinked cellulose by reactive extrusion using tartaric acid as crosslinking agent.

### MATERIALS AND METHODS

#### Materials

The oat hull sample used in this study was supplied by a local oat processing industry (SL Cereais e Alimentos, Paraná - Brazil). Cellulose was extracted from oat hulls using peracetic acid as bleaching agent by the methodology described by Marim et al. [2]. The cellulose and hemicellulose contents of the raw and bleached oat hulls were determined by the Van Soest method [8] and the lignin content was determined according to a standard method of the Technical Association of Pulp and Paper Industry [9].

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#### Methods

Tartaric acid was dissolved in water in different concentrations (5, 12.5, and 20 g / 100g of cellulose), and then the solution was slowly added to the cellulose (100 g) in a sealed bag, resulting in final moisture content of 32% (g/g), and each the mixture was manually stirred intermittently for 1 h before being processed. Then, all samples were extruded in a single screw extruder (AX Plastics, Diadema, SP, Brazil) with a screw diameter of 1.6 cm and a length/screw diameter (L / D) ratio of 40, with four heating zones and a 0.8 cm diameter matrix. The temperature in all zones was 100 °C and the screw speed was 60 rpm. The crosslinked cellulose extrudates were collected, placed in an oven, dried to constant

weight at 45 °C, ground and sieved (80 mesh). The dry mixture was washed three times with absolute ethanol to remove

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unreacted tartaric acid. Finally, the washed samples of crosslinked cellulose were air dried at 45 °C, and they were labeled as TA5, TA12.5 and TA20, throughout the study.

#### Fourier transform infrared spectroscopy (FTIR)

The dry samples were mixed with potassium bromide and compressed into tablets. FTIR analyzes were performed with a Shimadzu FT-IR-8300 (Kyoto, Japan), which has a spectral resolution of 4 cm<sup>-1</sup> and a spectral range of 4000-500 cm<sup>-1</sup>.

#### Degree of substitution

The degree of substitution was determined using the method described by Volkert et al. [10].

#### Crystallinity index

The analysis was performed using a Panalytical X Pert PRO MPD diffractometer (Almelo, The Netherlands) with copper K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) under the operational conditions of 40 kV and 30 mA. All of the assays were performed with a ramp rate of 1° / min. The Segal et al. method [11] was employed to determine the relative crystallinity index (CI) of samples: CI (%) =  $[(I_{002} - I_{am}) / I_{002}] * 100$ , where  $I_{002}$  is the intensity of the 002 peak (at  $2\theta = 20^\circ - 22^\circ$ ) and  $I_{am}$  is the intensity corresponding to the peak at  $2\theta = 18^\circ$ .

## RESULT AND DISCUSSION

The non-cellulosic fraction present in the oat hull was removed by the bleaching with peracetic acid, resulting in a cellulose rich material. Raw oat hull had 26% cellulose, 30% hemicellulose, and 21% lignin, and the bleached material presented 78% cellulose, 8% hemicellulose, and 3% lignin.

The FTIR spectra of the polymers are shown in Fig. 1. All samples showed similar spectra, however, some differences observed in the FTIR spectra can be used to assess the success of crosslinking using tartaric acid. In all spectra it was observed a band at 1735 cm<sup>-1</sup>, since the cellulose was extracted from the oat hull, there is a trace of hemicellulose, originating from the lignocellulosic residue, this band corresponds to the acetyl or uronic groups of the hemicelluloses [2]. However, in the samples crosslinked with TA this band at 1735 cm<sup>-1</sup> appeared with higher intensity, which can be assigned to the stretching vibration of ester carbonyl groups [6]. This most evident band indicated that the TA molecules were successfully grafted onto the cellulose chains and crosslinked with the side chains that constitute the tartaric acid.

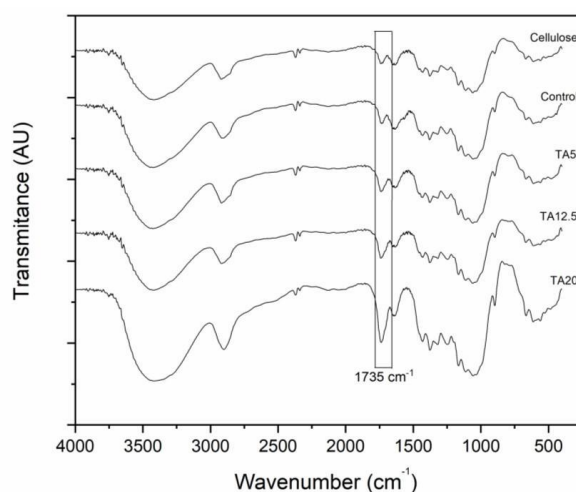


Fig. 1: FT-IR spectra of cellulose, the control sample (extruded without reagent) and crosslinked cellulose obtained by reactive extrusion through reaction with TA.

The degree of substitution (DS) is an important parameter to ascertain the extent of extension of modification, and it corresponds to the average number of substituents linked to the free hydroxyls on glucose units in cellulose chains [12]. DS values of crosslinked cellulose were presented in Table 1, and when the level of tartaric acid increased from 5 to 20%, the DS increased from 1.58 to 2.93 (Table 1). According to Ding et al. [14], the process conditions of the modification determine the DS and the properties of the obtained materials, and crosslinked cellulose obtained by reactive extrusion with tartaric acid had DS ranging from 0.77 to 1.32, which are lower than the values obtained in this study.

Crystallinity index (CI) values observed for crosslinked cellulose samples ranged from 35 to 37%, lower values than the values observed for unmodified cellulose (42%). The higher concentration of the crosslinking agent resulted in lower CI values (Table 1). Reactive extrusion is a physical method that combines thermo-mechanical and chemical treatments, which can affect the proportion of crystalline and amorphous fraction in cellulose samples, and possibly the higher concentrations of the crosslinking agent resulted in a lower amount of free hydroxyls, which are responsible to the formation of hydrogen bonding in the crystalline structure of cellulose.

TABLE 1: Degree of substitution and crystallinity index of crosslinked cellulose subjected to reactive extrusion with tartaric acid.

Samples	Degree of substitution (%)	Crystallinity index (%)
Cellulose	-	42
Control sample	-	37
TA5	1.58	37
TA12.5	1.79	35
TA20	2.93	35

## CONCLUSIONS

Crosslinked cellulose was obtained by reaction with tartaric acid using reactive extrusion obtaining samples with degrees of substitution ranging 1.58 to 2.93. The occurrence of chemical modification was confirmed by FTIR spectroscopy by the appearance of a band at  $1735\text{ cm}^{-1}$ . There was no difference between the crystallinity index. Reactive extrusion can be considered an efficient technology to obtain crosslinked cellulose, with the advantages of reducing the processing times with low generation of effluents when compared to the conventional processes employed to obtain modified celluloses, being considered a green alternative for this purpose.

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## CELLULOSE-BASED MATERIALS FROM ORGANGE BAGASSE OBTAINED BY REACTIVE EXTRUSION

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### ABSTRACT

Orange bagasse (OB) is considered a sustainable, renewable and lignocellulosic residue for extraction of cellulose. Thus, the present study aimed to obtain a cellulose-based material by reactive extrusion process. Combinations with water and sodium hydroxide in one-step processes were tested. The cellulose content of materials obtained from OB ranged from 18.8 to 21.2%, with a process yield of 43.5 to 79.2 %. Extrusion process was considered an efficient and sustainable process for cellulose obtainment from orange bagasse. Materials produced in this study can be used as cellulose fiber source for various products and processes, such as in food industry, fermentation substrates or refined applications after subsequent treatments.

*Keywords:* one-step processes, alkaline, sustainable, fiber source.

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### INTRODUCTION

Orange fruits (*Citrus × sinensis* (L.) currently plays an important role in Brazil's export economy. After the extraction of the juice (the main product) and essential oil, the orange bagasse (OB) represents around 50-60% of total mass of the fruit. Most of this residue is used as animal feed [1]. However, this important residue of agroindustry can be used as adsorbent biomaterials for pollutants [2], biogas and bioethanol obtaining [3], hesperidin extraction [1], source of pectin and soluble sugar. In addition, OB is a potential source of cellulose fibers when properly treated [4].

Cellulose is a linear homopolymer formed by D-glycopyranose units linked by β-1,4 glycosidic bonds, resulting in units that are repeated along the chain, called cellobiose. There are intramolecular hydrogen bonds, responsible for the rigidity of the chains, and intermolecular bonds, responsible for the formation of the fibrillated supramolecular structure. It is a fibrous substance, resistant and insoluble in water. In the plant wall, cellulose has a supporting function and is associated with hemicellulose and lignin. These three components are physically and chemically linked [1], [4].

As a lignocellulosic matrix, the use of this residue requires pretreatment that can be chemical, physical or combinations. Extrusion is a versatile processing technique that has been successfully applied to promote the modification of lignocellulosic biomass; several studies with cereal and grain residues, such as soy and oats, were developed in order to obtain fiber enriched materials. Advantages include the possibility of combinations with different chemical reagents, high productivity, lower effluents generation and lowest overall processing cost. Furthermore, the development of totally chlorine-free technologies collaborates with the sustainability of the lignocellulosic biorefinery [5]. However, the treatment of fruit residues is still little explored, especially orange bagasse [6].

To increase the economic viability of OB residue, the present study proposes an eco-friendly approach to obtain cellulose-rich materials based on reactive extrusion in combination with water and alkaline medium in one-step. Chemical composition (cellulose, hemicellulose and lignin) was analyzed. The field of application of produced cellulose-based materials can be extended to areas such as food, health and the environment, thus favoring a circular economy.

### MATERIALS AND METHODS

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#### Materials

Orange bagasse (OB) was provided by an orange-juice processing industry from Paraná, Brazil (Integrada Cooperativa), dried at 50 °C in an air-circulating oven (Marconi MA 415, Piracicaba, Brazil) and milled to yield particles < 0.3 mm (IKA-A 11 Basic Mill, Germany).

#### Methods

The reactive extrusion steps were performed using a single-screw extruder constructed from special grade of corrosion-resistant stainless-steel (AX Plastics, Diadema, SP, Brazil) with a diameter of 1.6 cm and a screw length/diameter ratio (L/D) of 40, with four heating zones and a matrix of 0.8 cm in diameter. The temperature in all zones was 120 °C, and the screw speed was 50 rpm. One hour before extrusion of the sample, the

reagent (NaOH 10% w/v) were dissolved in distilled water and homogenized with the raw OB in sealed plastic bags. Sample (120 g) were extruded with an initial moisture content of 40% (w/w). After, the sample were washed with water and neutralized to pH 5–6, dried in a ventilated oven at 40 °C (035 Marconi MA, Brazil) and milled (particles 0.3 mm). The chemical composition of raw OB and cellulose-based materials was analyzed for cellulose, hemicellulose and lignin contents. Cellulose and hemicellulose were measured by the Van Soest method [7], and lignin content was measured by the standard method of the Technical Association of Pulp and Paper Industry [8]. Analyses of Tukey's mean comparison test ( $p \leq 0.05$ ) were performed using R software (S R Core Team. R, Austria, 2016).

## RESULT AND DISCUSSION

In this study, the raw OB had  $12.4 \pm 0.6\%$  of cellulose,  $7.5 \pm 0.1\%$  of hemicellulose,  $8.9 \pm 0.5\%$  of lignin, with a total of 28.8% of the content of insoluble dietary fiber (cellulose, hemicellulose and lignin). The cellulose, hemicellulose and lignin contents related in literature are approximately 18-20, 6-14, and 4-6%, respectively [1, 9]. The cellulosic fraction of the orange bagasse has great potential for exploration, as the residue has high availability in Brazil.

TABLE 1: Cellulose, hemicellulose, lignin contents, process yield, cellulose yield and crystallinity index (CI) of raw OB and cellulose-based materials from OB obtained by extrusion

Treatment	Cellulose content (%)	Hemicellulose content (%)	Lignin content (%)	Process Yield (%)*	Cellulose yield (%)**
OB	$12.4 \pm 0.4^c$	$7.5 \pm 0.1^c$	$8.5 \pm 0.4^c$	-	-
EXH2O	$18.8 \pm 1.6^b$	$10.6 \pm 0.6^a$	$22.5 \pm 1.2^a$	79.2	100
EXNaOH	$21.2 \pm 0.3^a$	$8.8 \pm 0.9^b$	$20.1 \pm 0.2^b$	43.5	73.4

a-e- Different letters in the same column indicate significant differences ( $p \leq 0.05$ ) between means (Tukey's test). \*Process yield: the amount of cellulose-based material obtained for each 100 g of raw OB or raw OB-P (dry basis). \*\*Cellulose recovery: cellulose content in relation to the original cellulose content of raw OB (dry basis)

Orange bagasse (OB) was modified by water and alkaline extrusion process to obtain cellulose based-materials, EXH2O and EXNaOH, respectively. Table 1 shows the modifications in the resulting lignocellulose fractions. Extrusion processing significantly increased the cellulose content in samples tested, including water treatment. Reactive extrusion is considered an effective method for deconstructing the three-dimensional structure of lignocellulose, resulting in a cellulose-rich material [5,6]. EXNaOH shows the highest cellulose content compared to the water treatment. The alkaline treatment promotes solubilization and dissolution of lignin, and the removal of hemicellulose and pectin by breaking the ester linkages between carboxyl groups of pectin and hydroxyl groups of hemicellulose without significantly affecting the cellulose structure, also NaOH has advantages in terms of cost and environmental impact [11, 12].

The alkaline treatment was also more efficient in removing hemicelluloses and lignin; the contents ranged from 7.5 and 8.5% in raw OB to 8.8 and 20.1% to EXNaOH, respectively. Complete hemicellulose and lignin removal was not observed for both samples. The total elimination of these components requires subsequent or more aggressive treatments [4]. Table 1 also shows the cellulose yield (% of cellulose in relation to the original cellulose content of raw OB): in one-step water treatment 100% of cellulose was recovered, while in alkaline treatment there was a decrease (73.4%), indicating that there was little loss of the original cellulose during the employed processes. The process yield decreased in the two treatments tested (79.2 and 43.5%), indicating that the non-cellulosic fraction was partially eliminated (Table 1). Bicu and Mustafa reported that maximum cellulose contents result in minimum yields [11].

## CONCLUSIONS

In this study, reactive extrusion was used as a green route to produce cellulose-based materials from orange bagasse, resulting in materials with 18.8 to 58.4% of cellulose. Combining extrusion and chemical (alkaline) processes was considered an excellent approach to reduce energy consumption and environmental impacts. One-step treatments were crucial to determine the economic viability and sustainable character of this study. Cellulosic fibers fraction can be used for several applications, which contributes to a proposal for circular economy.

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**PHYSICAL AND BARRIER PROPERTIES OF ARROWROOT (*MARANTA ARUNDINACEA*) STARCH BASED COMPOSITES: A REVIEW**

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**ABSTRACT**

In recent years, biopolymers have received a lot of attention intending to develop high-performance biocomposites with low environmental impact due to their unique and useful characteristics such as renewability, abundant availability, eco-friendliness and lightweight. As investment and research into biopolymer composites raise, composite materials are projected to replace many conventional materials in optical, biological, and packaging applications. In order to produce the desired characteristics of biopolymer composites, it is necessary to blend a suitable biopolymer with additives that facilitate the interaction between the polymer and the filler. Arrowroot (*Maranta arundinacea*) belongs to the *Marantaceae* family and is mostly found in tropical forests. Arrowroot has a high content of amylose (~35.20%) and cellulose (~45.97%) which makes it suitable for better composite film production. Hydrophobicity is an important factor to consider when selecting materials for food packaging applications because it can affect the performance of the final product irrespective of whether it is water-sensitive or not. Therefore, films with low water vapour permeability are appropriate for food packaging applications, such as reducing and inhibiting moisture transfer between the surrounding environment and the food. The present review focuses on recent works related to the physical and water barrier properties of arrowroot starch-based composite films and their effects on bio packaging applications. To the best of our knowledge, no review paper was published on the physical and water barrier properties of arrowroot starch-based composites.

**Keywords:** arrowroot, biopolymer composite, physical properties, water barrier properties, bio packaging.

**INTRODUCTION**

The numerous types of plastic items currently available in the market, including from household, personal goods, food packaging, and packaging to the manufacture of construction products, are responsible for the rapid growth of plastic usage. The widespread usage of plastics has resulted in an abundance of plastic garbage in the environment. Therefore, to overcome this problem, a change must be made to replace frequently consumed petroleum-based plastics with biodegradable plastics to maintain a better environmental condition for coming generations while also reducing the amount of plastic disposed [2], [3]. Because starch is completely biodegradable and abundant in nature, it is one of the most promising biopolymers for replacing petroleum-based plastics [4], [5]. It is widely available in plants such as corn, cassava, potato, arrowroot tuber, and many others. In this way, the rhizomes of arrowroot (*Maranta arundinacea*) have emerged as powerful and effective sources of starch (amylose 35.20%) [6], and fiber (cellulose 45.97%) [7]. Arrowroot belongs to the *Marantaceae* family, which is typically found in tropical forests. Biopolymer composites have become an important research topic in recent years, as evidenced by the increased number of publications every year [2], [8]–[11].

**PROPERTIES ENHANCEMENTS OF STARCH-BASED FILMS USING ARROWROOT**

Numerous researchers examined the potential of films made from native starches derived from various sources, as well as starches that had been chemically or physically modified to form a variety of applications. Nogueira et al. [12] investigated the physical properties of arrowroot starch/blackberry particles films with the concentration (0 to 40%) of blackberry particles in the polymeric matrix. They observed an increase in moisture content in films from 9.66 to 11.84% as blackberry content increased (0 to 40%). Besides, water solubility and water vapor permeability of films increased significantly with increasing blackberry powder content. This characteristic could be attributed to blackberry powder, which is highly hygroscopic and hydrophilic, allowing the film to interact with water molecules more easily, resulting in increased solubility and permeability. Numerous researchers examined the potential of films made from native starches derived from various sources, as well as starches that had been chemically or physically modified to form a variety of applications. Nogueira et al. [12] investigated the physical properties of arrowroot starch/blackberry

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particles films with the concentration (0 to 40%) of blackberry particles in the polymeric matrix. They observed an increase in moisture content in films from 9.66 to 11.84% as blackberry content increased (0 to 40%). Besides, water solubility and water vapor permeability of films increased significantly with increasing blackberry powder content. This characteristic could be attributed to blackberry powder, which is highly hygroscopic and hydrophilic, allowing the film to interact with water molecules more easily, resulting in increased solubility and permeability.

Similar behavior was observed by Nogueira et al. [13], who investigated the influence of the incorporation of blackberry pulp on the physical and water vapor permeability of arrowroot starch-based films. They noticed that the incorporation of blackberry pulp (from 0 to 40% wt.%) in the thermoplastic film resulted in increased solubility in water and water vapor permeability from 14.18 to 25.46% and 3.62 to 4.60 g.mm/m<sup>2</sup>.day.kPa, respectively. Thus, the incorporation of blackberry particle and blackberry pulp into arrowroot starch-based film improve the physical and water barrier properties. Therefore, many researchers are concentrating their efforts on reducing the water vapor permeability of starch-based biocomposite films. Fig.1 shows the arrowroot fibre reinforced arrowroot starch biopolymer composites film.

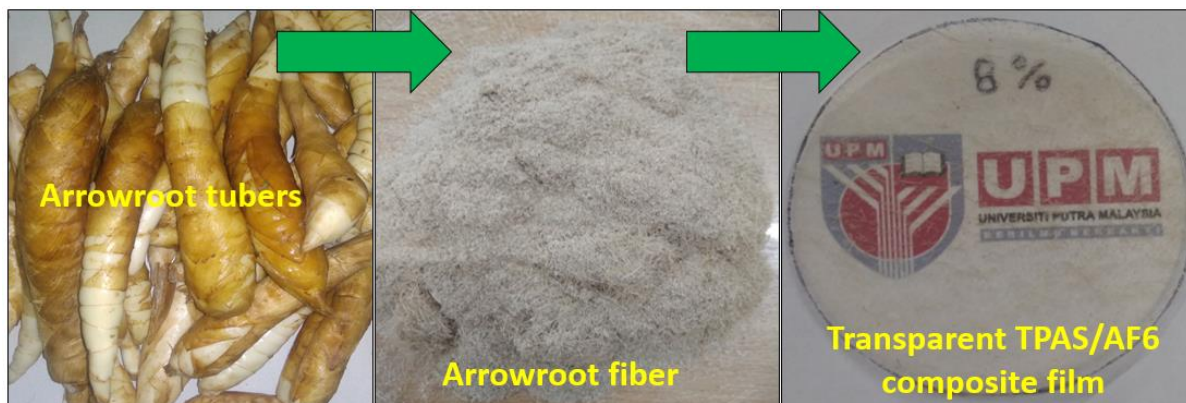


Fig 1: The arrowroot tubers, the fibre obtained from starch extraction, and transparent biocomposite film with arrowroot fibre loading.

## CONCLUSIONS

This paper presents an overview of biopolymers as well as the potential of starch-derived thermoplastics as replacements for existing petroleum-based plastic products. Blending starch with other biopolymers was proposed as a practical solution for overcoming the deficiencies of native starch. Nevertheless, the degree of compatibility between starch and other biopolymers varies greatly depending on the biopolymer. In terms of the global environmental issue, biodegradable material properties are crucial and should be taken into account. Even though starch/biodegradable blends are an excellent solution for environmental problems, their mechanical properties frequently have an inverse relationship with their degradability. Another option is to use natural fibers as fillers in the starch matrix. As a result, optimizing their properties necessitates additional research. Overall, we can conclude that biopolymer composites are rapidly growing in popularity and are expected to become a future material for a variety of uses.

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## TENSILE PROPERTIES AND CORRELATION OF FIBRE CONTENTS TOWARDS MANUFACTURING DEFECTS AND INTERFACIAL ADHESION OF ARENGA PINNATA FIBRE REINFORCED FIBREGLASS/KEVLAR HYBRID COMPOSITE IN BOAT APPLICATION

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### ABSTRACT

Recently, natural fiber-enhanced hybrid compounds that incorporate one or more types of natural enhancement are gaining scientific interest. Arenga Pinnata are one of the famous natural fibre in the hybrid composite due to eco-friendly, light weight, high stiffness, inexpensive and it have similar specific properties to synthetic fibre. Hybridization of Arenga Pinnata fibre with fibreglass/ Kevlar very recommended in any applications and it is also new in the marine field. The mechanical properties which is tensile test was determined in this research according to ASTM D 3039. Arenga Pinnata fibre fabricated in the specimen is based on volume fraction as 30%, 45%, 60% and 75% respectively. Besides, the surface morphology analysis also determined manufacturing defects and interfacial adhesion by using the Scanning Electron Microscopy (SEM) to verify the results of tensile properties. As conclusion, 60% of the fibre content in the specimen exhibited good interfacial adhesion and less manufacturing defects that showed better result of tensile.

*Keywords:* hybrid composite, natural fibre composite, mechanical properties, tensile test.

### INTRODUCTION

Nowadays, synthetic fibre is the main materials that have widely used in the marine field. However, some researcher has found the problem which is high maintenance costs. The natural fibre utilization as the alternative replacement for the synthetic fibre give great advantages due to low weight, low density, low cost, biodegradable, and good thermal characteristics [1]. Even though the fact that natural fibre have more advantages but they are not free from the issues. Hybridization of natural fibre with fibreglass/Kevlar was introduced in polymer composites to overcome the problems. Hybridization of *Arenga Pinnata* with fibreglass/Kevlar usually give balanced strength to mechanical properties such as tensile test, impact test and flexural test. Besides, the utilization of *Arenga Pinnata* in this research give high durability and resistance to sea water [2]. However, the failure of the hybrid composite was also depending on the fabrication process. The manufacturing defects that occurred during fabrication process such as gap, resin-rich zone, undispersed crosslinker pocket, misaligned fibres, and area where resin has been improperly wetted fibre usually always affect to the poor performance of mechanical properties of natural fibre hybrid composites [3]. Hence, this research study the relation of fibre contents toward manufacturing defects and interfacial adhesion of *Arenga Pinnata* reinforced hybrid composites fibreglass/Kevlar in boat construction.

### MATERIALS AND METHODS

Arenga Pinnata fibres gathered in Negeri Sembilan, Malaysia were used in this study. The polyester was used as a resin for reinforced composites. Fibreglass and Kevlar were supplied by MSET Shipbuilding Sdn. Bhd, Terengganu *Arenga Pinnata* fibres were combed and cut into 20cm. The moulding was prepared with the 150mm (length) x 150mm (width) x 30mm (thickness) dimension of stainless steel. The fabrication of composites involved of four different fibre volume content which are 30%, 45%, 60 and 75%. Hand lay-up method was used for this hybrid composite preparation. Preparation of polyester is based on the calculation of volume of matrix. Before used the matrix which is polyester, catalyst must be added into the matrix to cure or harden the matrix. After the mixture of *Arenga Pinnata* fibre, fibreglass, Kevlar and polyester, this moulding of specimen was left 24 hours in the room temperature. Tensile test was conducted to determine the strength of the hybrid composites. The testing machine was used is Geotech to test the specimen. This test was conducted according to ASTM D 3039 and the maximum load capacity that applied was 50kN. The data such as tensile strength, tensile modulus and elongation at break were obtained from the raw data of the test. For morphological

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test, the Scanning Electron Microscopy (SEM) was used to observe the shapes of fibres, fracture surfaces and the information the bonding between the matrix and fibres

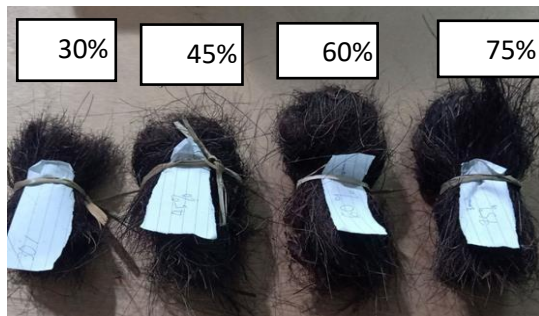


Fig. 1: Different composition of fibres



Fig. 2: Sample of hybrid composites material

RESULT AND DISCUSSION

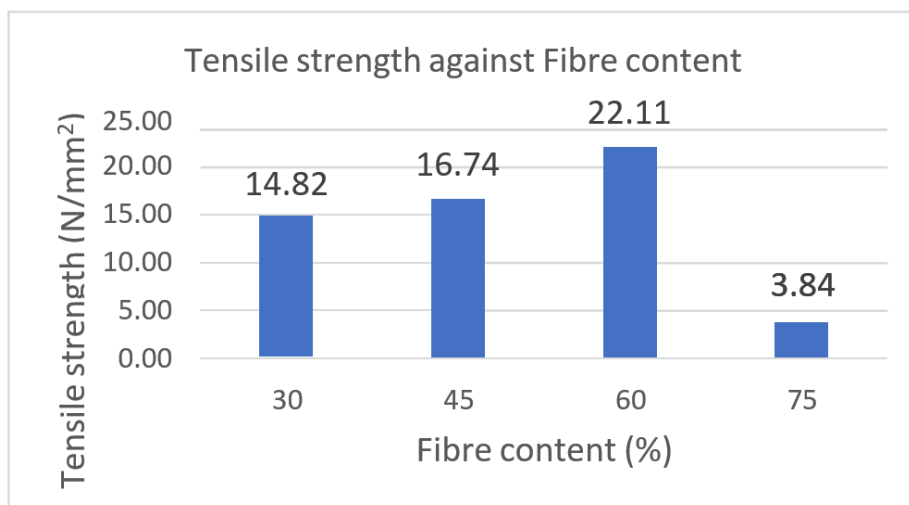


Fig. 3: Tensile strength graph

From the figure 3 show that Arenga Pinnata fibres reinforced hybrid fibreglass/Kevlar with different fibre content which are 30%, 45%, 60% and 75% showed the tensile strength 14.82N/mm<sup>2</sup>, 16.74N/mm<sup>2</sup>, 22.11 N/mm<sup>2</sup> and 3.84 N/mm<sup>2</sup> respectively. The result shows that the 60% of fibre content gives the highest result of hybrid composites due to an increase in the weight of Arenga Pinnata fibre hybrid fibreglass/Kevlar in the composite. The fibre loading in these hybrid composites plays an important role in the increment of tensile strength. Usually, there must be an increment on the fibre loading to achieve the maximum strength in the hybrid composite. The properties of hybrid composites were mainly dependent on several factors such as fibre content, fibre length and fibre orientation [4]. For the 75% of fibre content, the lowest result of tensile strength is observed because the Arenga Pinnata fibres are not able to support the stress transferred from the polyester matrix successfully, resulting in the failure of Arenga Pinnata fibre composites at a lower load as compared to the glass fibre reinforced composite [4].

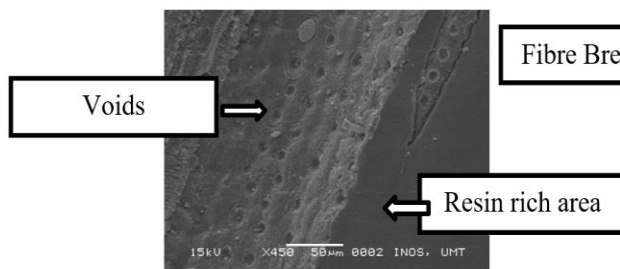


Fig. 4: 60% of Arenga Pinnata fibres

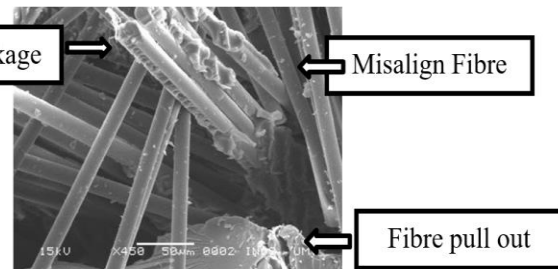


Fig. 5: 75% of Arenga Pinnata fibres

From figure 4, the 60% of fibre content shows that the voids in the specimen exhibit due to the fabrication process. The air bubbles caught between the framework influence from several factors such as curing pressure, resin system and environmental condition. The resin-rich zone always occurs during the fabrication process and this will give poor interfaces near those zones and a tendency to matrix crack. Figure 5 shows the 75% of fibre content and it shows fibre breakage, misalign fibre and fibre pull out. The misalign fibre might occur due to poor dispersion fibre-matrix. The fibre pull out always occurs caused by poor interfacial adhesion between fibre and matrix [5]. These defects during the fabrication process affected the performance of the mechanical properties for hybrid composites.

### CONCLUSIONS

The performances of mechanical properties *Arenga Pinnata* hybrid fibreglass/Kevlar depends on the defects that occur during fabrication process. The fibre loading in the hybrid composites also give the important role in the increment of the tensile strength. The study shows 60% of fibre content was the highest tensile test compared to others.

### ACKNOWLEDGEMENTS

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**VALORIZATION OF OAT HULLS TO OBTAIN NANOCELLULOSE USING REACTIVE EXTRUSION**F. Debiagi<sup>1\*</sup>, S. Mali<sup>2</sup><sup>1</sup> State University of Northern Paraná - UENP/CLM, Center of Agrarian Sciences, Bandeirantes, Brazil;<sup>2</sup> Department of Biochemistry and Biotechnology, CCE, State University of Londrina (UEL), Londrina, PR, Brazil Malaysia**ABSTRACT**

The objectives of this study were to produce nanocellulose from oat hulls (OH) employing a simple method based on reactive extrusion and to characterize the obtained materials according to their microstructure, morphology and composition in cellulose, hemicellulose and lignin. OH extruded in three sequential steps with NaOH and H<sub>2</sub>SO<sub>4</sub> (twice) resulted in nanofibers with diameters around  $100 \pm 25$  nm and lengths of several micrometers. Nanocellulose produced in this work presented diameters of nanometric scale and lengths of several micrometers, being classified as nanofibrillated cellulose. Reactive extrusion was effective in production nanocellulose from OH, and it has the advantages of simplicity and was less polluting than the conventional methods.

*Keywords:* Nanofibrillated cellulose, oat hulls, reactive extrusion.

**INTRODUCTION**

Cellulose can be obtained from several residues using different extraction processes, and in the last few years that are an increased interest in nanocellulose obtained from lignocellulosic materials. Several methods have been reported in the literature for obtaining nanocellulose, such as high-pressure homogenization, milling, microfluidization and ultrasonication. For the obtainment of nanocellulose from lignocellulosic residues, hemicelluloses and lignin have to be removed. The resulting cellulose has to be hydrolysed, and higher amounts of reagents are required in the usual processes, including several steps of bleaching and acid hydrolysis of the residue, resulting in long process and higher amounts of effluents. Merci et al. [1] reported the production of microcrystalline cellulose from soybean hulls employing reactive extrusion, they reported that reactive extrusion is an efficient method because provides a combination of thermo-mechanical and chemical treatment for the extraction of cellulose from lignocellulosic residues, resulting in lower amounts of effluents and higher yields. Thus, the objectives of this study were to produce nanofibrillated cellulose from oat hulls employing a simple method based on reactive extrusion, and to characterize the obtained material according to its microstructure, morphology and composition in cellulose, hemicellulose and lignin.

**MATERIALS AND METHODS***Material*

Oat hulls (OH) were kindly provided by SL Alimentos (Mauá da Serra, Paraná, Brazil), dried (45 °C), and milled to yield particles < 0.30 mm.

*Methods**Reactive extrusion for production of nanocellulose*

Single screw extruder (AX Plastics, Diadema, SP, Brazil) with a screw diameter of 1.6 cm and a screw length/diameter ratio (L/D) of 40, with four heating zones and a matrix of 0.8 cm in diameter was employed to obtain nanocellulose from OH. The temperature in all extruder zones was fixed at 110 °C and the screw speed was 100 rpm. OH were extruded based on previous study [1], with some modifications, and in all cases the samples were prepared and allowed to stand for 1 h in sealed plastic bags before extrusion (Table 1). A control sample called EOH (Table 1) was extruded without any reagent other than water, resulting in a final moisture content of 32 %.

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The EOH1 sample (Table 1) was obtained from the OH after a two-step extrusion process, in the first step, the OH was extruded with NaOH, followed by extrusion with H<sub>2</sub>SO<sub>4</sub> in the second step. The EOH2 sample (Table 1) was obtained from the OH after a three-step extrusion processes, in the first step, the OH was extruded with NaOH, followed by two sequential extrusions steps with H<sub>2</sub>SO<sub>4</sub>. For EOH1 and EOH2, the reagents were dissolved in distilled water and mixed with the OH, resulting in a final moisture content of 32 %. After each extrusion step, all samples were washed with distilled and neutralized until pH 5-6. Then, the samples were dried in a ventilated oven at 40 °C (035 Marconi MA — São Paulo, SP, Brazil) and milled to yield particles < 0.30 mm.

Characterization of OH and OH submitted to reactive extrusion. Cellulose, hemicellulose and lignin contents

The cell walls constituents (cellulose and hemicelluloses) were determined employing the Van Soest (1965) method [2]. The lignin content was determined according to a standard method of Technical Association of Pulp and Paper Industry TAPPIT222 om-88 [3].

TABLE 1: Processing conditions to obtain nanocelulose from oat hulls.

Sample	Bleaching with Peracetic acid	NaOH (%)	H <sub>2</sub> SO <sub>4</sub> (%)	Moisture (%)
EOH	-	-	-	32
EOH1	-	10	2	32
EOH2	-	10	2(2X)	32

Scanning electron microscopy (SEM) and atomic force microscopy (AFM)

SEM analyses were carried out with a FEI Quanta 200 microscope (Oregon, USA). Firstly, the dried samples were mounted for visualization on bronze stubs using double-sided tape. The surfaces were then coated with a thin gold layer (40–50 nm), and it was utilized an accelerating voltage of 30 kV for all samples. AFM images were recorded with a NanoSurf Flex-AFM instrument (AG, Switzerland) using silicon AFM probes as reported by Debiagi *et al.* [4]

Statistical analysis

The data were performed with Statistica software version 7.0 using analyses of variance (ANOVA) and Tukey's mean comparison test ( $p \leq 0.05$ ) (Statsoft, OK, USA).

## RESULT AND DISCUSSION

As seen in Table 2, all extruded samples (EOH1 and EOH2) presented cellulose, hemicelluloses and lignin values significant different ( $p \leq 0.05$ ) from raw oat hulls (OH). The hemicelluloses and lignin contents reduced in all samples. Cellulose increased when compared to raw OH, showing that the processes employed in this work were effective in removing lignin and hemicelluloses. According to Ng *et al.* [4], the most critical stage of cellulose extraction from lignocellulosic residues corresponds to the pre-treatment, and many techniques have been studied to remove the amorphous matrix consisting of lignin and hemicelluloses.

TABLE 2: Cellulose, hemicelluloses and lignin contents of oat hulls samples submitted to reactive extrusion and/or to bleaching

Samples	Cellulose (%)	Hemicelluloses (%)	Lignin (%)
OH	31.16 ± 1.15 e	28.72 ± 0.25 a	18.12 ± 0.63 a
EOH	34.32 ± 2.06 e	26.40 ± 0.53 a	15.00 ± 1.30 b
EOH1	53.14 ± 1.95 d	18.38 ± 0.35 b	12.76 ± 0.98 c
EOH2	61.21 ± 0.92 c	16.64 ± 0.42 b	9.62 ± 0.75 d

\*Different letters in the same column indicate significant differences ( $p \leq 0.05$ ) between means (Tukey test).

Merci *et al.* [1] reported that soybean hulls extruded with NaOH followed by an extrusion step with H<sub>2</sub>SO<sub>4</sub> resulted in microcrystalline cellulose with 83.97 % of cellulose, however the initial lignin content of soybean hulls was 1.54 %, much lower than the initial lignin content of OH employed in this work, and this higher lignin content to be removed could affect the process efficiency.

Scanning electron microscopy (SEM) and atomic force microscopy (AFM)

Fig.1 presents SEM images of OH, EOH, EOH1 morphology surfaces. OH and EOH presented a well-organized and compact structure composed by cellulose embedded in a noncellulosic matrix composed mainly of hemicelluloses and lignin, which is characteristic of lignocellulosic materials. In EOH1 sample, the extrusion with NaOH followed by H<sub>2</sub>SO<sub>4</sub> resulted in the removal of most hemicellulose and lignin present in raw OH, which permits the defibrillation of cellulose resulting in individualized cellulose microfibrils.

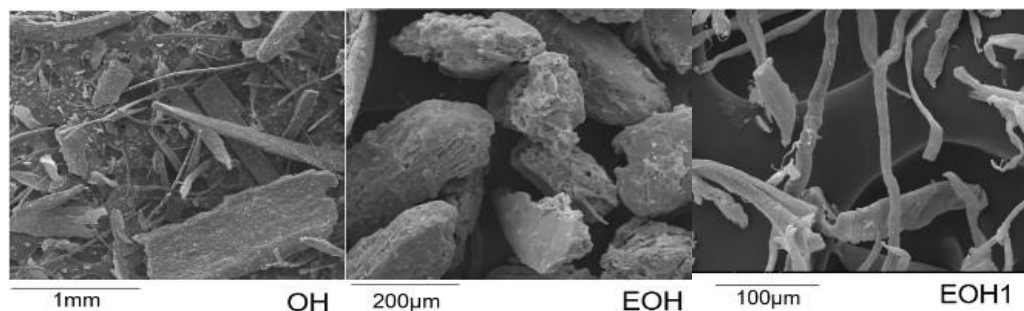


Fig.1 - Micrographs obtained using SEM: OH, EOH and EOH1

Cellulose is the core of lignocellulosic complex, and according to Ng *et al.* [3], during the bleaching the amorphous matrix of hemicellulose and lignina will be removed. It can be observed from the AFM images of EOH2 sample (Fig. 2) that this sample can be classified as nanofibrillated cellulose, with the aspect of interconnected webs of tiny nanofibers with diameters of  $100 \pm 25$  nm and lengths of several micrometers. It can be observed by AFM images of EOH2 (Fig. 2) that these samples can be classified as NFC, with an aspect of interconnected webs of tiny nanofibers with diameters of  $100 \pm 25$  nm and  $12 \pm 2$  nm respectively, and lengths of several micrometers.

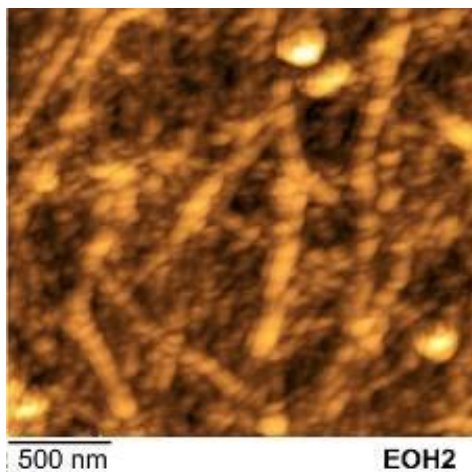


Fig.2 - AFM pictures for cellulose nanocrystals: EOH2

In addition, the aqueous suspensions of these samples (EOH2) were stable, and there was no sedimentation when they were stored at room temperature during 24 h.

## CONCLUSIONS

In this work, nanocellulose was produced from oat hulls employing reactive extrusion. Considering the nanofibers morphology, it was obtained nanofibrillated cellulose. Reactive extrusion was effective in the production of nanocellulose from oat hulls, with the advantage of reducing the process time and low generation of effluents when compared to the conventional processes employed to the obtaining of nanocellulose.

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## A BRIEF REVIEW ON NATURAL FIBERS AND ITS COMPOSITES: LATEST DEVELOPMENTS

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### ABSTRACT

In the concern of sustainability and environmental issues, this decade has witnessed the usage of green materials in material science technology through the growth of bio-composites. The potential engineering applications utilizing natural fibers worldwide because of environmentally friendly aspects and abundance. Natural fibers are bio-degradable, low cost, renewable sources, recyclable in nature and possess moderate mechanical properties. The major challenges in using natural fibers are poor water resistance, less fire-resistant, restricted processing temperature and being greatly influenced by weather conditions. These natural fibers are reinforced with different polymer matrices and results in natural fiber reinforced polymer composites (NFRCS) for high performance applications. These NFRCS are popular in various sectors like automotive, construction, defense, packaging, aerospace and domestic applications. Furthermore, these natural fibers are reinforced with various synthetic materials to increase their applicability and performance in different potential sectors. This review briefly describes the introduction to natural fibers, natural fiber composites, the broad classification of natural fibers, recent developments in natural fiber composites with their characterization and applications.

*Keywords:* Composite materials, natural fibers, epoxy, hybrid composites, mechanical properties, thermal properties.

### INTRODUCTION

The composite materials are built using two or more constituents with distinct physical or chemical properties and the developed composites possess unique properties which are different from individual constituents. In present days, advanced composite materials are broadly utilized in various engineering sectors due to their acceptable mechanical properties [1]. There are various things which are a burden for the worldwide environment, such as the huge amount of plastics disposal, elevation in global temperature, fast consumption of petroleum products, and decrement in polar ice caps. These are the reasons for the development of sustainable and green materials which are continuously supportive for defensible environments [2, 3]. In the previous decades, glass fibers are preferred as reinforcements and then followed by carbon and kevlar fibers. These fibers are highly expensive and they cause pollution in the environment. Hence researchers are motivated towards relatively cheap and non-polluting natural fibers as compared to synthetic fibers besides optimum mechanical properties [4, 5]. Normally the green composites are divided into two groups, namely partial and fully bio-degradable composites. In the partial biodegradable composites, the first case is one of the reinforcing elements is natural resources like natural fibers with epoxy matrix phase, while in other cases reinforcements are synthetic fibers with PLA as matrix material [6]. The second category of fully biodegradable composites involves the use of natural fibers for the reinforcement and PVA, PLA, etc. are used as a natural polymer matrix material. The composite materials which are fully biodegradable interns of both reinforcement and matrix materials obtained from natural sources are called bio-composites or green composites. The essential composites are prepared with thermosetting resins, which are non-recyclable or can't be reproducible [7, 8]. By keeping these points, researcher's attention going towards the development of green composites which has no negative effects on the environment and are popular due to their eco-friendly nature, bio-degradability, and fully sustainable nature. There exist various types of naturally occurring

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resins and polymers that are used for developing green composites, they are poly-glycolate (PGA), poly-hydroxyvalerate (PHV), poly (butylene succinate), polycaprolactone (PCL), lingo phenolic resin, poly (ethylene succinate) and other soy oil resins, etc. [9, 10]. These biodegradable matrices are compatible with different natural fibers and their combinations are suitable for potential applications. The natural fibers applications in various sectors are illustrated in Fig. 1. Natural fibers possess unique advantages over synthetic fibers, such as lower density, lightweight, non-abrasive nature, less energy consumption, optimum specific tensile properties, and less harmful to human beings [11, 12]. The applications of natural fiber reinforced polymer composites are growing continuously in different sectors like the automotive industry, sports, aerospace, construction and building industries. Even though there are some drawbacks in using natural fibers with polymer matrices, they are hydrophilicity, less heat resistant, lesser durability and less fire-resistant [13]. Many research works are found in the literature regarding usage and applicability of natural fibers with their potential applications in different

sectors. This review briefs the various types of natural fibers, characterization of natural fiber composites and their applications in various domains.

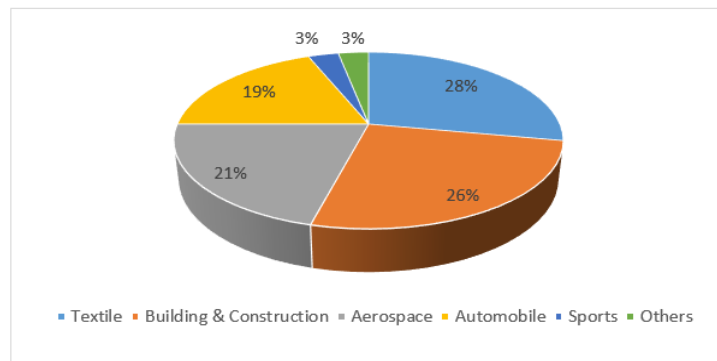


Fig. 1: Natural fibers applications in various sectors [14]

## NATURAL FIBERS

The “natural fiber” term covers a wide range of plant, animal, and mineral-based fibers. Usually, in composite industries these are referred to as wood fibers and agro-based seed, stem, bast and leaf fibers. The detailed classification of natural fibers is shown in Fig. 2. These fiber contents are helpful in the plant for strength and structural performance, also these are preferred as reinforcements in polymer composites [15, 16]. In the previous decades before use in composites, these fibers are used for the manufacturing of papers (wood), cigarette and linen papers (flax), and burlap, ropes are prepared using jute fiber. The majority of the natural fibers are annual crops and hence issues are raised in variability and storage during growing seasons. The fiber extraction methods and procedures depend on the plant type and area of the extraction in the plant, also it depends on the economic conditions and fiber performance [17, 18]. The natural fibers are having a three-dimensional complex structure with cellulose, hemicellulose and lignin as primary constituent. It also contains smaller amounts of extra components, includes organic components of lower molecular weights (extractives) and inorganic components (ash). Even though they are in small quantities, these extractives are highly influenced on decay resistance, odor and, color of the plant/fibers. The higher ash contents in the fiber contribute to good abrasion resistance [19, 20].

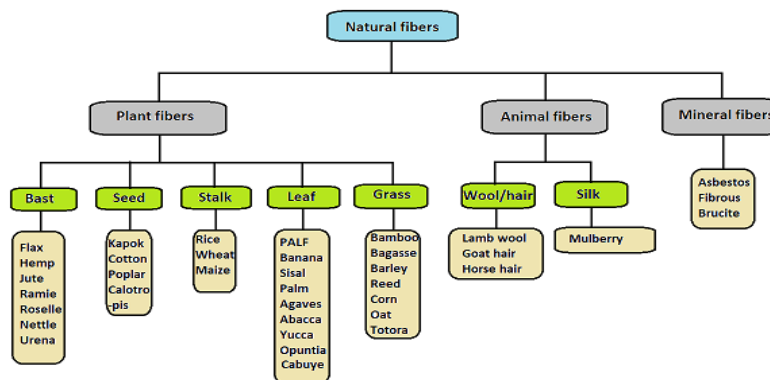


Fig. 2: Classification of Natural fibers [21]

## CHARACTERIZATION OF NATURAL FIBER COMPOSITES

Natural fiber composites are the emerging material technology in polymer science. These composites offer some specific properties as compared to conventional materials. The improvements in specific properties is done using natural fibers hybridization. Hybrid composites are generally referred as a combination of two or more reinforcements with single matrix materials. The potentiality of composites is increased due to hybrid fiber reinforcements with polymer matrix through the superior values of mechanical, thermal, water absorption behavior properties [21]. The addition of two or more natural fiber reinforcements improves the overall properties and is discussed in the following sections. Along with structure and properties, the experimental conditions also impact the properties of natural fiber composites.

## APPLICATIONS

The cost-effective nature of the natural fiber composites is popular especially in construction and in building materials like door and window frames, floors, walls, false ceiling, and partition panels, and other roof tiles materials. These are also used in

the fabrication of storage devices like bio-gas containers, post boxes, grain storage silos, etc. and electrical, automobile components [22]–[25]. Some of the applications are tabulated in Table 2. Mekonnen and Mamo (2020) [26] developed a sustainable green composite made of jute/bamboo/polyester hybrid materials for wind turbine blade application. The hybrid combination of jute fiber (10%), bamboo fiber (20%), and polyester matrix (70%) give a maximum yield strength value of 72.03 MPa and the maximum elongation obtained was 2.98 mm during tensile strength. Also, the combination of bamboo (30%) and polyester (70%) gives maximum flexural strength of 133.9 MPa. By considering all parameters, these hybrids composites are used for small wind blade applications. The sisal fiber reinforced epoxy composites are popular in developing lightweight vehicle body applications in automotive industries [27]–[30].

TABLE 2. Applications of natural fiber composites

Composites	Remarks	Applications	Studies
Sisal/chitosan/glass	It stiffer than human bone and possess a good stress shielding effect.	Bone fracture fixation application	Arumugam et al., [31]
Flax/carbon/epoxy	The composite has higher bending and tensile strength.	Tissue engineering application	Bagheri et al., [32]
Bamboo/polyester	The composite beam stiffness value increases with increasing short bamboo fiber wt. %.	Beam structures	Jena (2018) [33]
Banana fiber/wheat gluten	The physical structures of both proteins and fibers are retained after processing. Composite possess good dielectric properties.	Dielectric applications	Bhuvanewari et al., [34]
Areca sheath fiber/phenolic resin/other ingredients	The lower fiber contents reinforced composites have good wear resistance and coefficient of friction value.	Brake pad applications	Krishnan et al., [35]
Coconut coir/oil palm fiber	Composite exhibits acceptable mechanical and physical properties.	Residential building application	Lertwattanaruk, and Suntiitto [36]

## CONCLUSIONS

This review enclosed the introduction to natural fibers, natural fiber composites, the broad classification of natural fibers, mechanical and thermal characterization of NFRCs, water absorption behavior, and various applications. The different categories of natural fibers were discussed in this review with their examples and also the latest research works were also quoted with their characterization. Usage of natural fiber composite is the best opinion instead of petroleum-based materials because of decrement in CO<sub>2</sub> emission, reduces the oil import and it creates more employment to the rural area peoples. Natural fibers were subjected to chemical treatment while using as a reinforcement to reduce the hydrophilicity. The properties of the composites vary with fiber length and fiber concentration. So, the balancing of fiber percentage with the matrices is very important to get potentiality in the composites, and also the economic considerations are important while using novel materials.

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## ACOUSTIC PROPERTIES OF BIO-BASED POLYMER COMPOSITES – A REVIEW

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### ABSTRACT

In recent days, aircraft and automotive industries focuses on eco-friendly, low cost, lightweight, and good sound absorption properties. Bio based polymer composites are attractive for use of automotive interior applications due to their sound absorption properties. However, enhancing the acoustic characteristics of bio-based polymer composites remains challenging for researchers. This review article discusses the experimental characterization of acoustic properties including sound absorption coefficient, noise reduction coefficient, sound transmission loss of mono fiber and hybrid fiber-reinforced polymer composites. The acoustic characteristics are measured by using various approaches such as reverberation chamber method, impedance tube, in situ, and two microphones free field method. This paper discusses the most extensively used impedance tube method. This method requires small size of samples are which measure the various sound absorption characteristics. Furthermore, the factors influencing the acoustic behavior of bio-based composites are also discussed.

*Keywords:* bio based polymer composites, sound absorption coefficient, noise reduction coefficient, sound transmission loss

### INTRODUCTION

Sound pollution has increased over the past ten decades, affecting different elements like as health, the environment, and the economy [1], [2]. To overcome the noise pollution sound absorption is required nowadays to comfort human health. Sound insulations are used in places like automobile industry, manufacturing units and higher sound pressure equipment to bring effective and beneficial ways in producing sound absorption materials [3]. Acoustic attenuation technique is one of the sound absorption techniques with different porous material have been used to control noise pollution [4]. The three most important acoustic characteristics of materials are noise reduction coefficient, sound transmission loss and sound absorption coefficient. The total amount of a material's sound energy absorbed are calculated by the sound absorption coefficient (SAC). The fraction of occurrence energy that is not reflected or absorbed is known as transmission coefficient [5]. The percentage of sound that the surface absorbs is called noise reduction coefficient [6]. Many techniques used to characterize the sound properties of porous sound absorbers are two-microphone, in situ method, impedance tube and reverberation chamber method [7]. Sound absorption mainly involves two different types, they are impedance tube method and reverberation chamber method. To determine sound absorption for bigger sample reverberation chamber method. And for smaller sample size impedance tube method is preferred [5]. As per the ASTM standard E 1050, with the help of an impedance tube tester the sound absorption coefficient can be measured [8]. The acoustic properties of bio-based polymer composites and hybrid composites, such as sound absorption coefficient, noise reduction coefficient, and transmission loss, were discussed in this review.

### SOUND ABSORPTION BEHAVIOR OF BIO-BASED POLYMER COMPOSITES

The influence of varying volume fractions on the sound absorption coefficient of bio epoxy composites reinforced with coconut, cotton and sugarcane fibers were discussed. The result indicated that sound absorption coefficient (SAC) of coconut fiber reinforced epoxy composites (C/E) had higher than that of sugarcane fiber when reinforced with epoxy composites and cotton fiber reinforced epoxy composites for (10,15,20%) fiber volume fraction which is due to the porosity of fiber [9]. In another work, Prabhakaran et al. [8] stated that the acoustic characteristics of flax/epoxy (F/E) composites were studied and compared with glass/epoxy composites. Their results revealed that flax/epoxy composites showed higher sound absorption coefficient than the glass/epoxy composites at lower and higher frequency level [8].

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Yang weidong et al. [1] investigated that the acoustic properties of jute, ramie and flax fiber reinforced with epoxy composites. It was found that jute fiber reinforced with epoxy composites (J/E) has better sound absorption properties about 1000hz level of frequency. Moreover, jute and flax fiber reinforced with epoxy composites showed greater noise reduction capabilities [1]. The effect of kenaf fiber and rice straw fiber reinforced polypropylene composites on the sound absorption coefficient were investigated by Elammaran Jayamani et al. [10]. The results demonstrate that kenaf fiber reinforced polypropylene

composites (K/PP) were observed to possess greater sound absorption properties than rice straw fiber reinforced with polypropylene composites [10].

Sisal, banana, kusha grass, milkweed and hay fiber reinforced polypropylene composites were studied by Hariprasad et al. [11] investigating the influence of sound absorption properties. Among all the composite configurations, the hay fiber reinforced polypropylene (H/PP) hybrid composites exhibit highest sound absorption properties about 1000hz [11]. Yashwant et al. [12] analyzed on the effect of varying fiber volume fraction on acoustic properties of sisal fiber reinforced polypropylene (S/PP) composites. It can be noticed that maximum sound absorption coefficient and maximum transmission loss of sisal/polypropylene composites were found for 20wt.% and 30wt.% compositions, respectively [12].

Acoustic behavior of luffa cylindrical reinforced epoxy (L/E) composites was analyzed by Garip genc et al [13]. The results showed that sound absorption coefficient and transmission loss level of luffa/epoxy composites will increase with the increase in frequency level. Furthermore, the maximum sound absorption coefficient has been achieved when increasing the thickness of the luffa composites. [13]. K. Saravanan et al. [14] investigated that the acoustic properties of various factors influencing on the compression molded Chicken feather fiber and jute fiber reinforced polypropylene hybrid composites. The 100% of CFF composites showed better sound absorption coefficient and noise reduction coefficient due to their higher porous structure in these composites [14].

## CONCLUSION

In this review, an overview of acoustic behaviors including sound absorption coefficient, noise reduction coefficient and transmission loss of bio-based polymer composites and hybrid composites were presented. Some factors that affect the sound absorption coefficient of bio-based polymer composites were found by some researchers. They are type of fiber, frequency, sample thickness, chemical treatment fiber, orientation and length, fiber content and fiber diameter. Researchers found that to increase the sound absorption coefficient and transmission loss of their composites, should increase the frequency, fiber volume, fiber inclination and sample thickness. The bio based polymer composites were observed good sound absorption quality due to the hollow and porous structure of the natural fibers. It could be extremely promising for usage in practical applications such as aero planes, automobiles, and buildings to increase the use of environmentally acceptable materials.

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**PHYSIO-CHEMICAL AND THERMOMECHANICAL CHARACTERIZATION OF RAW-ALKALI TREATED FIBER REINFORCED BIO-EPOXY COMPOSITE: AN EXPERIMENTAL STUDY**

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**ABSTRACT**

Over the past few decades, there is an increase in environmental concern because of the increased use of synthetic materials. The current work focuses on characterization of the *Digitaria sanguinalis* and its composites. Untreated and alkali-treated fibers were used to manufacture bio-epoxy composites. The composites were subjected to test physico-chemical analysis for their chemical, physical, mechanical properties. From the experimental results, the chemically-treated *Digitaria sanguinalis* fibers and composites performed better in all thermal, physical, and mechanical aspects.

*Keywords:* *Digitaria sanguinalis*, Tensile test, XRD, TGA

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**INTRODUCTION**

Natural fibers have recently sparked a lot of interest due to their superior environmental qualities over synthetic materials [1]. The properties of the natural fibers depend upon various governing factors like part of fiber extraction methods. The cellulose content present in the fiber has a major role in controlling the mechanical strength of the composite [2], [3]. There are some limitations like hydrophobicity and compatibility. To overcome this issue there are various surface modification techniques alkali, silane, etc [4], [5]. These treatments were proven to alter the surface properties of the fibers and enable them to have good interaction and adhesion with the matrix [6]. This research was focus on *Digitaria sanguinalis* plant as a potential resource of raw material.

**MATERIALS AND METHODS**

*Digitaria sanguinalis* was collected, cleaned and fiber were extracted. 5% of alkali treatment were performed and were subjected to physico-chemical analysis. The fabricated of composites were subjected to mechanical analysis. The matrix was prepared by mixing 20 volume % of fiber and 80 volumer % of epoxy resin and left curing for 24 hours. Physical and chemical analysis in current research were conducted according to ASTM standard.

**RESULT AND DISCUSSION**

The results were presented in the figure 1 (a, b, and c). The alkali-treated fiber improved cellulose weight % from 53.27% to 63.12% compared to untreated fiber. The density and diameter of fiber were changed from 0.921 to 1.134 g/cc and from 431.45 to 327.72  $\mu\text{m}$  respectively, after treatment. The X-ray diffractograms of the untreated and alkali treated fiber showed that the crystalline index and crystal size of the alkali-treated fiber increased from 31.27% to 46.05% and 3.15 to 4.16 nm respectively, compared to untreated fiber. The TGA of both treated and untreated fiber showed that thermal stability of alkali treated fiber was shifted from 327 °C to 342 °C compared to untreated fiber. The tensile strength and modulus of alkali treated fiber increased from 38.79 to 54.97 MPa and from 979.19 to 1597.42 MPa respectively, compared to untreated fiber.

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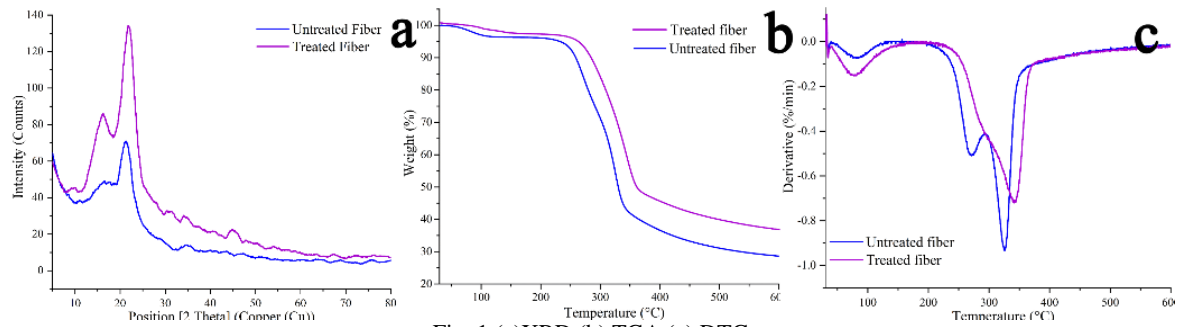


Fig. 1 (a)XRD (b) TGA (c) DTG

## CONCLUSIONS

From the experimental results, *Digitaria sanguinalis fiber* is a potential resource of raw material which can be used as a replacement of synthetic fibers in the polymer matrix, and the alkali-treated fiber reinforced composite performs better when compared to the untreated fiber-reinforced composites.

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## EVALUATION OF THE ACETYLATION OF CELLULOSE EXTRACTED FROM SOYBEAN HULL BY CONVENTIONAL METHOD AND A NEW ECO-FRIENDLY PROCESS

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### ABSTRACT

The extraction of cellulose with peracetic acid from soybean hull (SH) and cellulose acetate (CA) synthesis, employing the conventional acetylation method and an alternative hydrothermal process, solvent and catalyst free, were investigated. The bleached cellulose (BSH) presented an increase in cellulose content from 37.78 to 79.52 %. Triacetates were majorly obtained through conventional acetylation of the BSH, with degree of substitution (DS) ranging from 2.70 to 2.86, in the hydrothermal process, all materials were characterized with monoacetates, with DS around 1.58 to 1.91. The insertion of new functional groups increased the hydrophobicity and altered the morphology of all the samples.

*Keywords:* soybean hulls; cellulose acetate; conventional acetylation; hydrothermal acetylation.

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### INTRODUCTION

The cellulose acetate (CA) is a biopolymer derived of the cellulose modification, it's considered a biodegradable, non-toxic and biocompatible material [1]. However, questions regarding the traditional synthesis of obtaining the CA, have been raised. The first question is the possibility of replacing the pulp cellulosic from wood by lignocellulosic residues, since Brazil is one the world's largest agricultural producers, where a significant amount of these residues is discarded. The second question is the use of sulphuric acid as catalyst of reaction conventional acetylation. Therefore, the objective of this study was synthesize CA from cellulose extracted from soybean hull, employing the conventional acetylation and propose an alternative hydrothermal process, aiming an eco- friendly route, free of solvent and catalyst. The CA obtained was characterized according to the degree of substitution (DS), hydrophobicity and morphology.

### MATERIALS AND METHODS

#### *Materials*

Soybean hulls were kindly provided by SL Alimentos (Mauá da Serra, Paraná, Brazil) and milled to yield particles <0.30mm and dried (45 °C). The chemical reagents used were sodium hydroxide, hydrogen peroxide, acetic acid, sulphuric acid, hexane (Synth, Brazil), acetic anhydride e hydrochloric acid (Química Moderna, Brazil).

#### *Methods*

##### *Extraction of Cellulose*

Approximately of 50 g of SH were dispersed in 500 mL of peracetic acid solution (50% acetic acid, 38% hydrogen peroxide e 12% distilled water) at 80 °C for 24 h. After this treatment, the bleached cellulose of soybean (BSH) was washed with distilled water until pH 5.5-6.5 was achieved, and it was dried at 35 °C until constant weight [2].

##### *Cellulose, hemicellulose and lignin contents*

The cellulose and hemicellulose contents of the raw and bleached SH were determined by the Van Soest method [3] and the lignin content was determined according to a standard method of the Technical Association of Pulp and Paper Industry [4].

##### *Synthesis of cellulose acetate (CA)*

The synthesis of CA through conventional method, occurred by the reaction of BSH with acetic acid as solvent, sulphuric acid as catalyst and acetic anhydride as acetylating agent, according to the methodology described by [5]. Different reaction times were studied (6, 14, 18 and 24 h). The samples produced were named ACS-Cat6h, ACS-Cat14h, ACS-Cat18h e ACS-Cat24h. The hydrothermal method, solvent and catalyst free, was based on [6], with modifications. Approximately 3.0 g of BSH was mixed with 100 mL of acetic anhydride, the solution was submitted to high pressure, in vertical autoclave (Prismatec, modelo CS, São Paulo, Brasil) at 1,5 atm and temperature of 121 °C for different times (1 h; 1 h:30 min e 2 h). Then, 250 mL of distilled water was added to stop the reaction, and the solid portion was filtered, washed with distilled water and ethanol. The material was dried in ventilated oven for 24 h at 29 °C. The samples produced were named ACS-Ht1h, ACS-Ht1:30h e ACS-Ht2h.

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*Degree of substitution (DS)*

The DS of CA was determined by titrimetric analysis, through the mixture of 0.1 g of sample, 0.25 M of NaOH and 5 mL of ethanol. This mixture was left to rest for 24 h, then 10 mL of 0.25 M HCl was added and left to rest for further 30 min. Finally, the solution was filtered and titrated with standardized NaOH. The percentage of acetyl groups (AG) that were substituted in the cellulosic chain and the degree of substitution (DS), were determined by the equations described by [5].

*Hydrophobicity test*

The percentage of hydrophobicity was determined by adding 0.1 g of sample in a solvent system composed by 20 mL water and 20 mL de hexane, and stirred for 3 min. After that, it was allowed to rest for 5 min, the aqueous phase was removed and the material was dried and weighed. The estimates of hydrophobicity were calculated according with the equation described for [6].

*Scanning Electron Microscopy (SEM)*

SEM analysis were performed on the FEI Quanta 200 equipment (OR, USA). The dry samples were assembled for viewing on bronze stumps using double-sided tape. Afterward, the samples were covered with a thin layer of gold (40–50 nm). All samples were examined using a 30 kV voltage accelerator. For comparison, samples of soybean hulls (SH) and cellulose extracted from residue were also analyzed by SEM.

**RESULT AND DISCUSSION***Extraction of Cellulose*

Initially the SH contained 38.78 % of cellulose, after treatment with peracetic acid, the bleached material presented 79.52 % of cellulose, which represents an increase of 105.05 %. In the hemicellulose fraction there was a decrease from 18,14 to 2.15 %, as well as in contents of lignin, from 2.55 to 1.49 %. The use of materials with low lignin content has the advantage of not requiring aggressive treatments in the delignification process, as they have low recalcitrance [7]. The process yield was 29.36 %, that is, each 100 g of raw SH resulted in 29.36 g of bleached material.

*Degree of substitution (DS)*

Of the acetates obtained by conventional methods (Table 2), the samples ACS-Cat14h e ACS-Cat18h presented DS = 2.86 and 2.80, respectively, characterizing them as triacetates. As for ACS-Cat24h, a DS = 2.70 is observed, even with a significant difference when compared to the samples ACS-Cat14h and ACS-Cat18h, it is also considered a triacetate. The ACS-Cat6h sample is considered a diacetate, with DS = 2.09. Through the hydrothermal method, the three samples, ACS-Ht1h, ACS-Ht1:30h and ACS-Ht2h, showed significant difference between them, with DS = 1.91, 1.58 and 1.64, respectively. However, materials with DS < 2 are considered monoacetates [1]. Through the data showed in Table 2, it is noted that, regardless of the acetylation method used, the increase in reaction time was not proportional to the increase in DS. Possibly this is due the cellulose degradation and acetate hydrolysis, resulting in part of the product dissolved in water [8].

TABLE 2: Results of DS to acetylated samples by conventional and hydrothermal method, and percentage of hydrophobicity.

Samples	DS	Hydrophobicity (%)
BSH	–	0 <sup>h</sup>
ACS-Cat6h	2.09 ± 0.14 <sup>c</sup>	89.04 ± 0.98 <sup>d</sup>
ACS-Cat14h	2.86 ± 0.02 <sup>a</sup>	96.90 ± 0.45 <sup>a</sup>
ACS-Cat18h	2.80 ± 0.04 <sup>a</sup>	96.51 ± 0.14 <sup>b</sup>
ACS-Cat24h	2.70 ± 0.01 <sup>b</sup>	93.86 ± 0.91 <sup>c</sup>
ACS-Ht1h	1.91 ± 0.01 <sup>d</sup>	87.94 ± 0.95 <sup>e</sup>
ACS-Ht1:30h	1.58 ± 0.04 <sup>f</sup>	61.73 ± 0.37 <sup>g</sup>
ACS-Ht2h	1.64 ± 0.05 <sup>e</sup>	81.50 ± 0.42 <sup>f</sup>

Means ± standard deviations. Different letters in the same column indicate significant differences between means (Tukey's test  $p \leq 0,05$ ).

Regarding the hydrophobicity, all the samples presented significant increase. This property is a consequence of the presence of the acetyl group in the cellulosic chain, it was observed that the ACS-Cat14h sample, despite having DS without significant difference compared to ACS-Cat18h, exhibited the most considerable increase, with 96.90 % of hydrophobicity. The other samples showed distinct hydrophobicity rates between them, with emphasis on the sample ACS-Ht1:30h, such as DS, exhibited lower hydrophobicity.

*Scanning Electron Microscopy (SEM)*

As observed in SEM micrography (Figure 1), raw SH presented typical aspect of lignocellulosic materials, a more compact and uniform surface with non-fibrous components, hemicellulose and lignin, forming a continuous matrix around the cellulose fibers. After the bleached, observed the disruption of lignocellulosic complex, where the cellulose fiber bundles were exposed and individualized, with shorter rod-shaped fibres, agreeing with the results of other authors who used the same bleaching method [9]. In acetylation by hydrothermal method (ACS-Ht) the material presented greater compaction in CA aggregates, and in the conventional process (ACS-Cat) richer parts of pores can be observed, caused by the difficulty in organizing the acetyl groups in relation to their conformation [10].

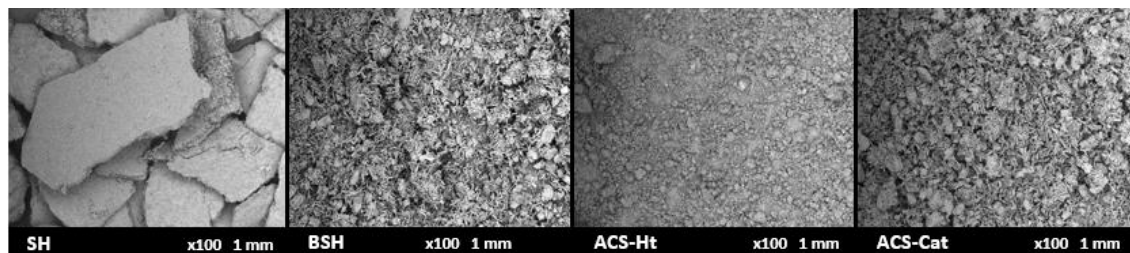


Fig. 1: SEM of soybean hulls (SH), bleached soybean hulls (BSH), samples modified by conventional acetylation (ACS-Cat14h) and hydrothermal (ACS-Ht1h).

## CONCLUSIONS

The treatment with peracetic acid, in addition to being simple and less polluting, proved to be satisfactory in the delignification and extraction of cellulose from SH, being possible to obtain a material richer in cellulose. The two acetylation methods, conventional and hydrothermal, were efficient in the synthesis of cellulose acetate, with different degrees of substitution. It was also concluded that the increase in time was not proportional to the increase in the degree of substitution, and an insertion of acetyl groups confers hydrophobicity to the material, in addition to altering its morphology.

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## DEVELOPMENT OF 3D PRINTING FILAMENT MATERIAL USING RECYCLED POLYPROPYLENE (PP) REINFORCED WITH COCONUT FIBER

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### ABSTRACT

Polypropylene is a recyclable thermoplastic polymer that is used in a wide number of applications. Due to the short shelf life of polypropylene-based packaging, the great majority of these thermoplastics end up in landfills as waste. Polypropylene goods degrade slowly in landfills, requiring between twenty and thirty years to completely degrade. This characteristic contributes significantly to environmental problems. Additionally, conventional filament materials for 3D printing are not recyclable. The fundamental objective of this research is to create a new biodegradable filament for 3D printing. The specific objective is to develop a filament material from a novel blend of recycled polypropylene and coconut fibre.

*Keywords:* 3D printing, coconut fiber, polypropylene (PP).

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### INTRODUCTION

Recycling is one of the most significant acts that can be taken immediately to address the environmental and ecological hazards linked with reduced oil use, carbon dioxide emissions, and trash disposal volumes. While plastic recycling continues to be the most successful means of reducing plastic waste, the material's quality is damaged by polymer cross-contamination, additives, non-polymer contaminants, and deterioration. Although polypropylene is clearly one of the most frequently used plastic packaging materials on the planet, only around 1 percent of it is recycled, implying that the vast majority of it is destined for the garbage. Due to their processing flexibility, high specific properties, high specific stiffness, and low volumetric cost, bio-fibers are gaining appeal as fillers and/or reinforcement in plastics-composites. Additionally, bio-based natural fibre composites are applied to boost electrical resistance, mechanical capabilities, acoustic insulating qualities, thermal properties, and fracture resistance and quality. To assist reduce the amount of polypropylene that ends up in landfills, this study proposes to manufacture a 3D Printing Filament material utilising recycled polypropylene (PP) and coconut fibre for reinforcement.

### MATERIALS FOR 3D PRINTING FILAMENT

3D printing materials come in a wide variety of types and kinds. ABS, polylactic acid (PLA), and polypropylene are the most often used fibres (PP). Tony-Hoffman (2015) [1] concluded that ABS materials are strong, durable, and harmless. Generally, 20% acrylonitrile, 25% butadiene, and 55% styrene are utilised. By adjusting these ratios, one can modify the attributes of ABS; for example, styrene imparts stiffness and brilliance to ABS, whereas butadiene imparts impact resistance and low temperature features to ABS [2], [3]. ABS filament is ideal for printing plastic automobile components, moving parts, musical instruments, household appliances, electronic housings, and a variety of toys, such as LEGO. Apart from 3D printing, it has a slew of additional uses. ABS is a thermoplastic polymer that is used by traditional manufacturers to make plastic wrap, water bottles, and cups, to name a few. According to Ilyas et al. (2018) [4], PLA has a low melting point, it can be utilised at temperatures between 180 and 230 °C, and it is biodegradable and manufactured from plants. According to Ahmed (2010) [5], condensation and polymerization are the two basic processes for the production of polylactic acid, and thermoplastic materials become liquid at their melting point (150-160° for PLA). PLA filament is utilised in medical implants as anchors, screws, plates, pins, and rods, as well as a mesh. PLA is also a biodegradable packaging material that can be moulded, spun, or cast. Cups and purses have been made using this material. While polypropylene (PP) is widely used in the plastic injection moulding industry, it is less frequently used in additive manufacturing due to printing challenges. Because PP is chemically resistant and

hydrophobic, it is an excellent material for packaging cleaning goods, foods, and other liquids. According to Micheal (2019) [6], PP is an ideal material for projects that will come into touch with food due to its chemical stability. Another application is that PP is sufficiently heat resistant to withstand steam sterilisation, making it perfect for medical instruments. Because PP filaments are tough and impact-resistant, they are frequently used in functional parts and prototypes in industrial environments [7]. PP is a widely utilised material because it is resistant to heat and UV radiation, which are two of the most typical concerns faced when 3D printed items are exposed to the outside.

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### COCONUT FIBER AS REINFORCED MATERIALS

Natural fibers are very attractive for composite materials because of their advantages compared to synthetic fibers. These include lower levels of skin irritation and respiratory system during handling, reducing tool wear during the processing, good recyclability, abundant supply, low cost, low density, high strength ratio, non-toxicity and biodegradability. The use of coconut fiber as reinforcement in polymer matrix is important because it is an inexpensive materials when compared with others fibers, decrease the amount of waste accumulated in landfills and yet been studied for reinforcement application in polymers such as Cement Paste, [8] The authors came to the conclusion that the tensile strength and modulus of rupture of cement paste rose up to a specific length and volume fraction, beyond which the composite's strength declined. Asasutjarit et al. (2007) [9] evaluated the physical, mechanical, and thermal properties of a coconut coir-based light weight cement board after 28 days of hydration. The length of the fiber, the coir pre-treatment, and the combination ratio were all studied. Better results were obtained when boiled and washed fibers with a fiber length of 6cm were used. M.Y. Yuhazri and M.M.P. Dan (2007) used coconut fibres in the manufacture of motorcycle helmets. They used epoxy resins derived from thermoset polymers as matrix materials and coconut fibres as reinforcement material. However, the use of coconut fibers in composite materials present a few drawbacks due to some characteristics such as the formation of fiber aggregates during processing, low resistance to moisture, variability of composition and poor compatibility with hydrophobic matrix polymer [10] . In order to improve compatibility of coconut fibers, the alkali (NaOH) treatment was found to improve the tensile and flexural properties while decreasing the impact strength. The ratio is 6% NaOH, 94% distilled water [11].

### RECYCLED POLYPROPYLENE

Polypropylene's resin identification code is 5, which is it can be recycled. Polypropylene recycling is comprised of five steps: collection, sorting, cleaning, reprocessing, and new product manufacture. Based on other researcher's [12] reviewed, Polypropylene reprocessing begins with melting in an extruder at temperatures above 400 degrees Fahrenheit, followed by granulation for use in new manufacture and ultimately degrades thermally, lowering the structural intensity of the plastic as the hydrogen-carbon bonds weaken. To remove impurities, waste plastic is melted to 250 degrees Celsius, followed by vacuum extraction of remaining molecules and solidification at approximately 140 degrees Celsius. This recycled polypropylene can be combined with virgin polypropylene up to 50%. Recycled polypropylene can be found in packaging articles, automotive bumpers, foams, bottle caps, carpets, and home components, as well as straws and sweet wrappers. Bitumen concentrations of 2 to 5% by mass are typically preferred for bitumen asphalt applications. Recycled polypropylene can also be used as 3D printing filament. Based on previous studies, [13] had reviewed Polypropylene recycling can help reduce energy usage and pollutants in the air and water. Industrial waste generated by firms that manufacture plastics is now a substantial cause of pollution. Pollution can be considerably minimised if things are recycled rather than made from scratch on a regular basis. Polypropylene recycling also encourages appropriate plastics management and disposal. However, there are a few drawbacks to recycled polypropylene, such as increased pollution and energy usage [13]. While this may appear to be paradoxical, recycling tonnes of garbage will necessitate waste being transported, sorted, cleaned, and processed in different facilities, all of which will take energy and may produce by products that contaminate the air, water, or soil. Furthermore, it may result in higher processing costs and lower-quality jobs.

### CONCLUSION

In summary, recycled polypropylene has numerous advantages and can be used as a valuable material if it is processed properly. Additionally, pollution and waste can be reduced, including non-recyclable filament. By developing a 3D filament from polypropylene reinforced with coconut fibre, the matrices and fibres are combined to create a biodegradable material that is both environmentally friendly and recyclable. The coconut husk was chosen because it has the best fibre properties and is easy to come by, according to the local population in Malaysia, in particular. Compressive strength, flexural strength, and splitting tensile strength of the filament all increased as a result of the use of coconut fibre as reinforcement material in the filament.

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## SILICA CONTENT OBTAINED FROM SUGAR PALM FIBRE

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## ABSTRACT

Silica is a material derived from natural fibre ash that has a variety of industrial uses, including filler/reinforcement material in composites, glass, electronics, and polymer materials, drug delivery, toothpaste abrasive, insect repellent for agricultural produce, paint additive, drug delivery, and energy storage. X-ray diffraction and energy dispersion spectroscopy were used to examine the ash content of sugar palm fibre following carbonization and calcination. The presence of amorphous silica (SiO<sub>2</sub>), which is more abundant than other minerals, was shown by X-ray diffraction. The energy dispersion spectroscopy revealed that the silica concentration of sugar palm fibre ash was 83.24%, which is greater than some natural fibres.

**Keywords:** Carbonization, sugar palm fibre, silica, Industrial application, XRD, EDS.

## INTRODUCTION

Sugar palm trees, also known as *Arenga Pinnata*, are a popular natural fibre tree that grows primarily in tropical areas. Sugar palm fibre has a wide range of applications, including weaving hats and mats, producing ropes, brooms, road construction, brushes, roof materials, cushion and fish breeding shelters, and polymer composite applications [1]–[3].

Agricultural wastes such as wheat straw, rice husk, rice straw, maize cobs, and bagasse can be used, and they can be extracted using chemical, thermal, or microbiological methods. Control burning of agricultural residues (fibres) produce ash with minor carbon content (10%–20%), substantial silica content, and traces of other inorganic/organic components, depending on the furnace type, burning circumstances, and variety of agricultural wastes [4]–[6]. Fibres are gaining attention as a potential source for silicon-based refractory chemicals which are employed as soil amendment and fertilizer, glassware production, production of silicate based materials, prevention of metal corrosion as green corrosion inhibitor, construction of building materials, protein absorption and separation [7]–[9].

## MATERIALS AND METHODS

*Materials*

Sugar Palm Fibres (SPFs) (Fig. 1a) were collected from a sugar palm tree farm in Jempol, Negeri Sembilan, Malaysia. The black SPFs were cleaned with distilled water to remove dirt and dried for 48 h in the laboratory. The dried SPFs were burned in the open air as depicted in Fig. 1b and the carbonaceous SPF (Fig. 1c) obtained is further calcined in an electric furnace (Fig. 1d) at 650°C for 5h to obtain greyish white sugar palm fibre ash (SPFA) (Fig. 1e) as shown in Fig. 1.

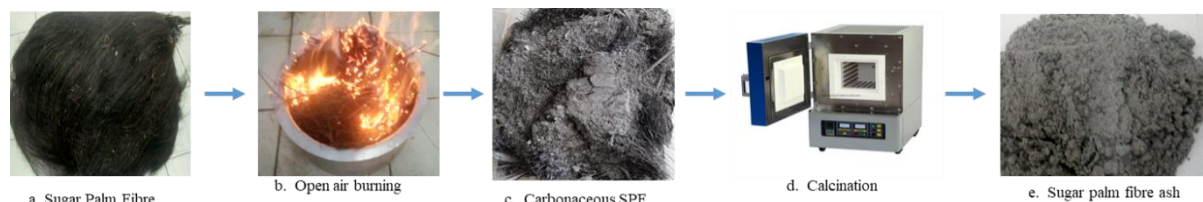


Fig. 1 Carbonization and calcination of SPF

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The diffraction patterns of the SPFA particles were determined using XRD. The test was carried out on a Shimadzu XRD 6100 (30kV, 30mA) with a Cu-Kα1 wave-length of 1.5405 and a rate of 2°/min in the angle range of  $2\theta = (20 - 80^\circ)$ . The XRD characterization was carried out at ambient temperature using CuK radiation. The Energy Dispersion Spectroscopy (EDS) detector connected to the FESEM (Model: Coxem-EM-30AX+) records the elemental components present in the SPFA sample.

## RESULT AND DISCUSSION

The XRD analysis was used to examine at the SPFA's diffraction pattern. There are three prominent silica peaks seen in the XRD diffraction patterns of SPFA displayed in Fig. 2 at the corresponding angles of 30.4o, 38.2o, and 44.7o. The XRD diffraction pattern revealed quartz (Q) to be the predominant constituent, with a fewer trace of small peaks of Wollastonite (CaSiO<sub>3</sub>) (W) and Feldspar (F), both of which have triclinic crystal systems. The existence of silica (SiO<sub>2</sub>) is confirmed by the broader diffraction peaks in the XRD patterns that correspond to quartz (Q), which is amorphous in nature. The peaks for quartz (Q) predominate in the XRD diffraction patterns (Fig. 2), indicating that silica is the most common ingredient in the SPFA.

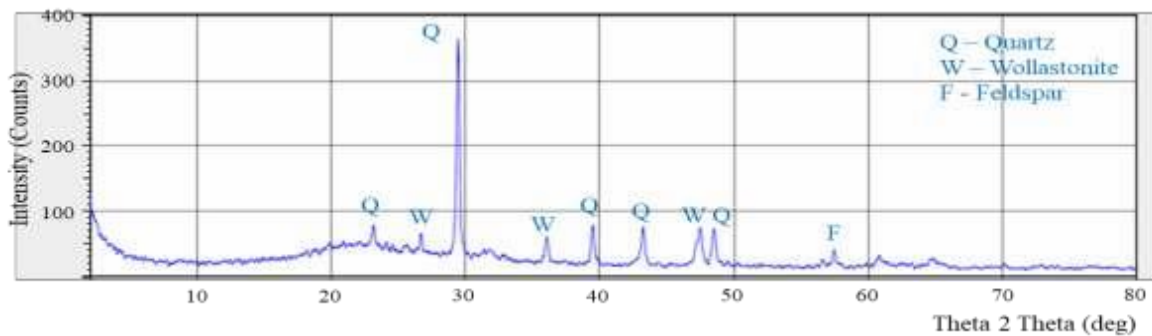


Fig. 2 XRD pattern of calcine SPFA

SPFA's EDS reveals the presence of minerals other than C, O, and Si (Fig. 3). The highest elemental value in the EDS spectrum was Si (silica), confirming its existence in SPFA coupled with residues of impurities. The presence of oxygen in the EDS test suggested a close contact with Si, equivalent to silica (SiO<sub>2</sub>). Table 1 shows that silica (SiO<sub>2</sub>, 83.24%) is the most abundant ingredient in SPFA, which is similar to other siliceous pozzolans such rice husk ash. This verified that the most prevalent element in the SPFA is silica (SiO<sub>2</sub>), with varying levels of Ca, C, Mg, Al, and other inorganic impurities.

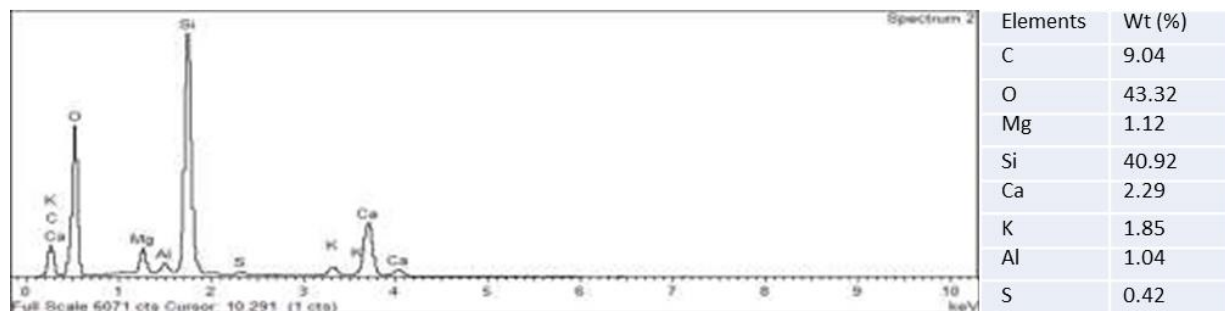


Fig. 3 EDS of SPFS

TABLE 1. Chemical makeup of SPFA

Elements	SiO <sub>2</sub>	C	CaO	MgO	K <sub>2</sub> O	Al <sub>2</sub> O <sub>3</sub>	FeS <sub>2</sub>
Content(%)	83.24	9.04	2.29	1.12	1.85	1.04	0.42

## CONCLUSION

The presence of silica (SiO<sub>2</sub>) as a main ingredient, as well as other trace compounds such wollastonite and feldspar, was revealed by XRD patterns of the sugar palm fibre. The EDS confirmed that silica is the main component of SPFA, amounting to 83.24 %.

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## BIO-NANOCOMPOSITE FILMS REINFORCED WITH CELLULOSE NANOCRYSTALS FOR IMPROVED BARRIER PROPERTIES

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### ABSTRACT

The pearl millet starch-based biodegradable films incorporated with cellulose nanocrystals were investigated as a substitute for petro-based films. The nanocomposite films were formulated using the solution casting process by blending starch and glycerol with different cellulose nanocrystals compositions (1-7%). The pearl millet starch reinforced with cellulose nanocrystals films possessed improved oxygen and moisture-barrier properties after reinforcement. Therefore, cellulose nanocrystals reinforced starch film offers a promising candidate for developing biodegradable films with improved barrier properties.

*Keywords:* Pearl millet starch; cellulose nanocrystals; nanocomposite films; barrier properties

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### INTRODUCTION

The packaging industry is the third-largest industry globally, and more than two-thirds of packaging material is exclusively used in the food market. This number is constantly growing due to the growing population, different food habits, and preparation methods. Due to the highest volume of plastic consumption, packaging industries have become the primary source of plastic waste accumulation at an alarming rate. Therefore, the demands for biodegradable and renewable materials for packaging applications have increased tremendously. Natural polymers like cellulose, chitosan, starch, collagen, and proteins could be used as prospective alternatives to conventional packaging material because of their availability, renewability, eco-friendliness, and degree of functionality. Among biopolymers, starch stands out as the future green biopolymer to substitute non-biodegradable plastic. The pearl millet starch-based active nanocomposite films reinforced with cellulosic nanocrystals (CNCs) from Kudzu have been proven an alternative option for existing biodegradable plastic packaging. CNCs are prepared by subjecting plant fibers to the processes such as depolymerization followed by bleaching, acid hydrolysis, and mechanical dispersion.

### EXPERIMENTAL PROCEDURES

The method adopted by Niliswan et al. (2018) [1] was used to determine the barrier properties of films.

### RESULTS AND DISCUSSION

Films and coatings for foodstuffs are often necessary to prolong shelf life and maintain important texture, taste, and mouthfeel properties. The uptake of moisture is often a crucial factor in the rate of degradation of a given product; therefore, films/coatings acting as moisture barriers are of the utmost interest in the food industry. Barrier properties of films may be accessed by water vapor permeability (WVP) and oxygen permeability. WVP measures the migration of water vapors in a material. As such, hydrophobicity is one of the fundamental characteristics that should be considered when selecting a material for food packaging applications as it affects end-product performance [2]. Films with low WVP reduce or prevent water vapor transfer between the surroundings and the food, and therefore are suitable for food packaging applications. Also, decreased WVP of nanocomposites indicates better moisture protection [3].

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The highest WVP and oxygen permeability was observed for PMS film. The highest WVP of starch films could be due to starch hydrophilicity or water sensitivity, a factor that was difficult to manage. Reinforcing CNCs to pearl millet starch reduced the WVP and oxygen permeability of the control films (Fig. 1). The decrease in WVP could be attributed to the tortuosity produced by the rigid crystalline structure and dispersing nature of CNCs in the pearl millet starch, resulting in hindrance in the path of molecules by the film matrix (Fig. 2) [4].

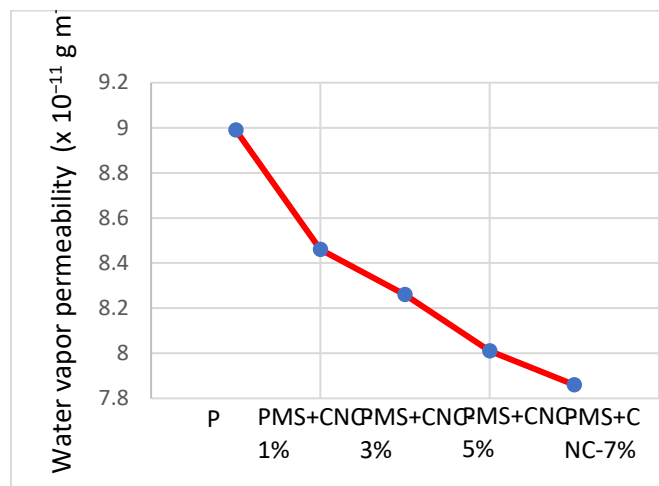


Fig. 1. Water vapor permeability of pearl millet starch films reinforced with cellulose nanocrystals at 0, 1, 3, 5 and 7%

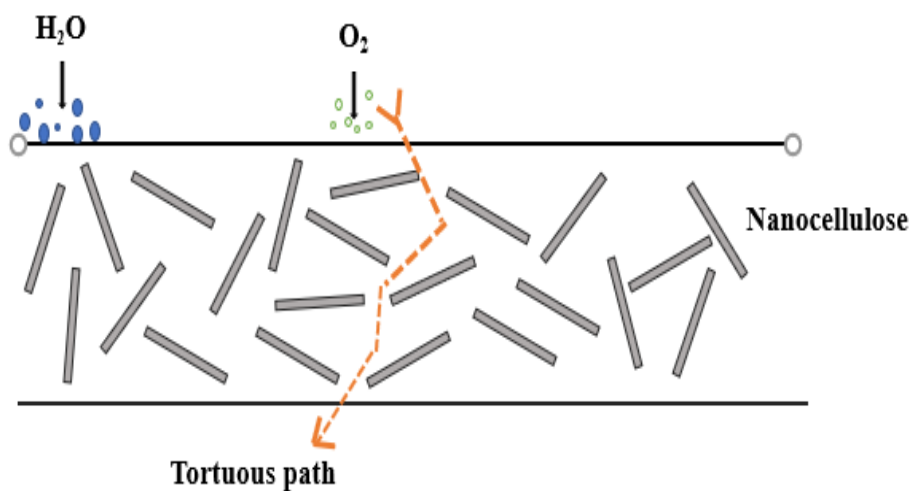


Fig. 2. Tortuous path by cellulose nanocrystals

## CONCLUSIONS

The performance enhancement of pearl millet starch films reinforced with CNC is most likely due to the high compatibility of intermolecular H-bonding interaction between starch and CNCs due to their chemical similarities and the improved dispersion and adhesion of CNCs within the pearl millet starch matrix. For flexible starch films, the optimum level of CNC as a reinforcer was 5% to improve the oxygen and water vapor permeability. Overall, the present study shows the real potential of the CNCs nanocomposites for food packaging applications.

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## EXPERIMENTAL ANALYSIS OF KERF TAPER ANGLE IN CUTTING PROCESS OF SUGAR PALM FIBER REINFORCED UNSATURATED POLYESTER COMPOSITES WITH LASER BEAM AND ABRASIVE WATER JET CUTTING TECHNOLOGIES

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### ABSTRACT

In this research, the effect of processing input parameters on the kerf taper angle response of three various material thicknesses of sugar palm fiber reinforced unsaturated polyester composite was investigated as output parameter from abrasive waterjet and laser beam cutting techniques. The main purpose of the study is to obtain data that includes the optimum input parameters in cutting the composite utilizing these two unconventional techniques to avoid some defects that arise when using traditional cutting methods for cutting the composites, and then make a comparison to determine which is the most appropriate technique regarding the kerf taper angle response that is desired to be reduced. In laser beam cutting process, traverse speed, laser power, and assist gas pressure were selected as the variable input parameters to optimize the kerf taper angle. While the water pressure, traverse speed, and stand-off-distance were the input variable parameters in the case of waterjet cutting process, with fixing all of the other input parameters in both cutting techniques. The levels of the input parameters that provide the optimal response of the kerf taper angle were determined using Taguchi's approach, and the significance of input parameters was determined by computing the max-min variance of the average of the signal to-noise ratio (S/N) for each parameter. The contribution of each input processing parameter to the effects on kerf taper angle was determined using analysis of variation (ANOVA). Compared with the results that were extrapolated in the previous studies, both processes achieved acceptable results in terms of the response of the kerf taper angle, noting that the average values produced from the laser cutting process are much lower than those resulting from the waterjet cutting process, which gives an advantage to the laser cutting technique.

*Keywords:* Laser cutting, abrasive waterjet, natural fiber, composite, kerf taper angle.

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### INTRODUCTION

Natural fibers have been primarily viewed as waste and remnants until recently, as they were not efficiently exploited. However, its use is spreading due to the advantages of natural fibers that made them to be an acceptable alternative to synthetic fibers in many applications, especially considering the property of natural decomposition of natural fibers, which makes them environmentally friendly materials [1], [2], in addition to that they are extracted from renewable resources that require no energy consumption to produce them unlike the synthetic fibers production processes. They also have certain other advantages, such as low density, low cost, enhanced recovery, and flexibility [3]–[5]. Owing to mentioned benefits, natural fibers have attracted a lot of attention in advanced polymeric composites field for a variety of engineering applications as a reinforcement material for a broad spectrum of matrices [6], [7]. Although composites are formed close to near-net shape, final processes such as drilling, cutting, trimming, and profiling are still required [8]. Due to the cutting forces associated with conventional cutting methods and the heterogeneous nature of composites, in addition to specimen fixing that requires a relatively large clamping force, several serious defects appear with the application of traditional cutting techniques in the composites cutting processes, such as, material damage, poor surface quality, delamination, fiber fraying, and dimensional instability [9]–[12]. In

order to avert these flaws, non-traditional techniques were considered [8]. Laser beam machining (LBM) is the most prominent unconventional technology utilized in cutting composites due to their high efficiency and productivity [7], [8]. In this context, the current study investigates and analyzes the influence of significant input parameters on the kerf taper angle response in cutting three different material thicknesses (2, 4, and 6 mm) of sugar palm fiber reinforced unsaturated polyester (SPF-UPE) composite cut with Laser beam technique. As one of the most interesting composite materials reinforced with sugar palm fibers is the sugar palm fibers reinforced unsaturated polyester (SPF-UPE), on which several studies have been conducted regarding the evaluation of its physical and chemical properties [13], [14]. In this study, the focus was on testing the

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SPF-UPE under unconventional cutting conditions, as the principle target is to collect data that involves the optimum input parameters in cutting the composite using laser technology to prevent or limit the defects that emerge with using conventional machining processes for cutting the SPF-UPE. The data provided in this research would contribute to the exploitation of natural fiber composites, especially the material under study in various applications, such as automobiles, aerospace, construction industries, marine applications, packaging, sporting products, and electronic industries applications.

## MATERIALS AND METHODS

### Materials

#### Fabrication of composites

Sugar palm fiber reinforced unsaturated polyester (SPF-UPE) composite was used for the research. Sugar palm fibers (SPFs) were cleaned with pure water, dried by hot air, and then treated with 0.25 M/L NaOH with one-hour immersion duration, as this treatment demonstrated good improvement in the mechanical and physical properties of SPF [13]. The fibers have been cut manually with lengths from 5 to 10 mm (average aspect ratio 25). The matrix used is unsaturated polyester (UPE) with fiber loading by 30%, as this fiber content showed good mechanical and physical properties [15]. Three molds with three different depths were utilized to make three different types of specimens with thicknesses of 2, 4, and 6 mm and lengths of 210 mm and widths of 120 mm. The hand lay-up technique was used to perform the composite specimens. The molds were subsequently disassembled and the specimens were removed after 24 hours of being covered with a 40 kg weight.

### Methods

#### Experimental setup

Laser beam cutting experiments were carried out using CO<sub>2</sub> laser cutting machine (AMADA FO 3015 M2 NT) with a CNC worktable with a maximum power of 4000 W in 1500 Hz pulsed mode. The laser beam was focused on the top surface of the material using a 7.5" focal length lens, the nozzle diameter was 2 mm, the nozzle stand-off distance was 1.5 mm, and air was utilized as the assist gas. Traverse speed, assist gas pressure, and laser power were taken as the input parameters as they demonstrated a significant influence on the kerf taper angle [7]. Other parameters, such as nozzle diameter, focal length, and nozzle stand-off distance, were kept constant. The various input parameter values were used to investigate three different material thicknesses of 2, 4, and 6 mm.

## RESULTS AND DISCUSSIONS

The ranges of input parameters that produced the defects shown in Figure 1 have been excluded, and the parameters in Tables 1-3 that gives good observed quality of cutting zone, have been considered for optimizing kerf taper angle property of sugar palm fiber reinforced unsaturated polyester cut with laser beam cutting technique.

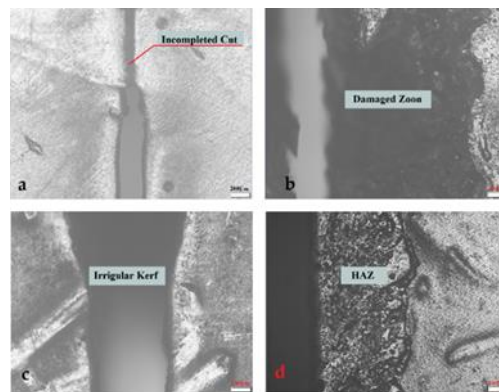


Fig. 1. (a) Incomplete cut. (b) Damage at the cutting zone. (c) Irregular kerf. (d) High extension of the heat-affected zone (HAZ).

The contributions of the input parameters to the effect on the kerf taper angle of each material thickness are represented in Figure 2. In small material thicknesses (2 mm), gas pressure takes the largest contribution, as the higher value of assist gas pressure produce the better response of kerf taper angle as this is consistent with what Girish Dutt Gautam et al. [16] found, while traverse speed comes second in terms of effect on kerf taper angle and no significant effect of laser power was observed. This is in contrast to the larger material thicknesses (4 and 6 mm) where the laser power has the largest importance in affecting the kerf taper angle followed by the traverse speed with little effect of assist gas pressure. The best response of taper angle in the case of 4 and 6 mm is obtained with high and medium laser power and low traverse speed which consistent with Ali Solati et al. [17] found as this study did not demonstrate an importance of assist gas pressure. The high laser power and low traverse speeds allow a large possibility and time to complete the thermal decomposition of the material in the cutting area and thus completely removing it by assist compressed gas, which may justify obtaining the best kerf properties under these conditions.

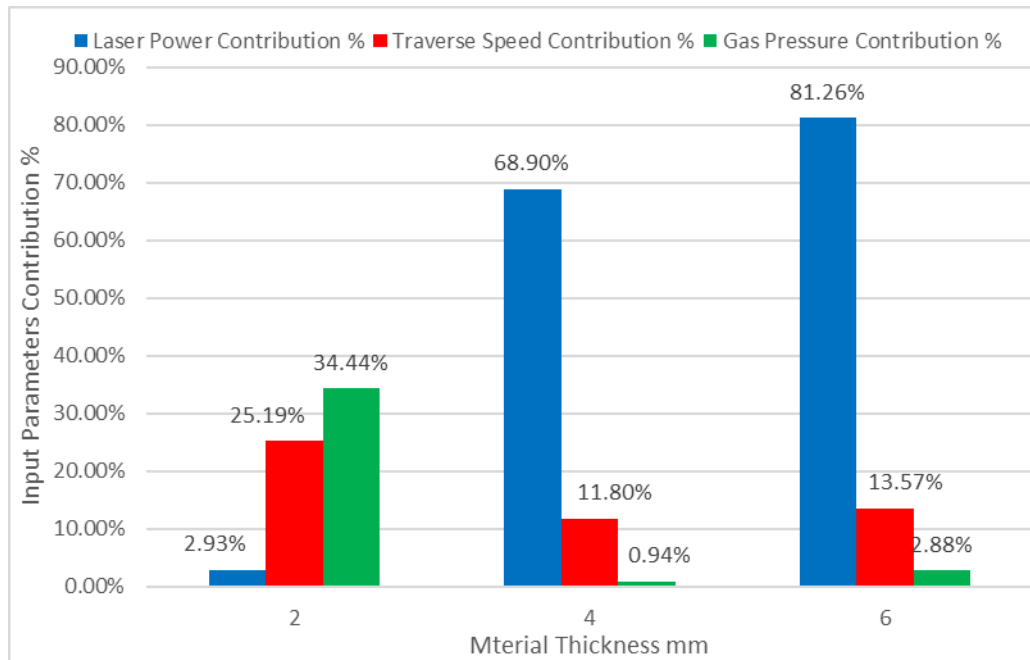


Fig. 2. Input parameter contributions to kerf taper angle of the various material thicknesses of SPF-UPE composite cut with laser beam cutting technology.

## CONCLUSIONS

The laser beam cutting process of sugar palm fiber reinforced unsaturated polyester composite is completed satisfactorily, with the following findings:

- In laser beam cutting process, assist gas pressure has the largest influence on the kerf taper angle response, followed by traverse speed and laser power, respectively, for 2 mm material thicknesses, meanwhile, laser power took the greatest influence on the kerf taper angle, followed by traverse speed with small contribution of assist gas pressure in the cases of 4 mm and 6 mm specimen thicknesses.
- Optimum input parameters that produced the best response of kerf taper angle in laser cutting process based on DOE Taguchi and ANOVA analysis methods were, 4 bar assist gas pressure, 200 mm/min traverse speed, and 400 W laser power for 2mm material thickness. In the case of 4 mm material thickness the optimum input parameters were, 1300 W laser power, 5600 mm/min traverse speed, and 2 bar assist pressure, meanwhile the optimum input parameters for 6 mm specimen thickness were, 2600 W laser power, 7600 mm/min traverse speed, and 4 bar assist pressure.

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## NATURAL FIBRE REINFORCED RUBBER-BASED MEMBRANE COMPOSITE

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Membrane technology for wastewater treatment has shown constant and progressive improvement. This technology has become a popular alternative to several commonly used techniques due to its benefits. Membrane selectivity and permeability are depending on the membrane's surface characteristics, particularly the pores. Many studies have been undertaken to improve the performance of rubber-based membranes in many applications. The reinforcement of filler in the ENR/PVC matrix has been shown to be beneficial for gas and water permeation applications. The addition of natural fibre as reinforcing filler and pore former in the composite membrane compromises improves performance and mechanical properties of the membrane. Besides, it could help to resolve the agricultural and environmental problems by using abundant agricultural waste.

*Keywords:* reinforcing filler, pore-forming agent, rubber-based membrane, water treatment application.

**INTRODUCTION**

The membrane separation processes are the excellent medium of separation without the addition of adsorbent. This technology offers several advantages such as ease to operate, less environmental impacts, minimal energy consumption and more efficient than conventional processes [1]. Separation by using membranes have several applications for industrial effluents and in food and pharmaceutical industries [2], [3]. However, the performance of the membrane is the most important part in the membrane separation process. Therefore, the suitable materials and method for water treatment application is highly needed. In the membrane processes, the most common ones used are polymeric membranes.

Rubber thin film exhibits elastic, flexible, ductile, and tough behaviors that are usually used for clothing, food packaging, and separation process. Compared to other non-rubber polymers, most of the rubber thin film are uncured due to poor crosslinking, poor selectivity, and are difficult to prepare, limiting its applications. The blending of thermoplastics with elastomer was found to improve the physical and mechanical properties of the thin film. Thin film made from thermoplastic elastomer exhibit excellent chemical, mechanical and thermal stabilities. ENR, a polymeric rubber-based material, is polar, high in strength, high oil resistance, and high tensile and tear strength. Due to its stiffness, flexibility, durability, good abrasion resistance, and low cost, polyvinyl chloride (PVC) is a polymer with extraordinary qualities that is utilized to make microporous membranes [4]. Epoxidized Natural Rubber (ENR) is a versatile elastomer that polar, high in strength, high oil resistance, and high tensile and tear strength. The blending of PVC with ENR is beneficial where the PVC is expected to impart high tensile strength, good chemical resistance whereas ENR have good tear strength and acts as a permanent plasticizer to PVC [5]. This thermoplastic elastomer was reported to improve the physical and mechanical properties of the material and to form miscible blend at any proportion [6].

Several researchers have looked into the usage of rubber-based membranes for various industrial separation processes [7], [8]. Vane et al. [9] studied the separation properties of the ethanol-water mixture through silicone rubber mixed matrix membrane filled with zeolite by zeolite and silicone component for pervaporation application, while Achalpurkar et al. [10] investigated the gas permeation capabilities of amine substituted silicon rubber (ASR) membranes. Jon et al. [11] reported the ENR/PVC/SiO<sub>2</sub> membrane exhibited CO<sub>2</sub> gas permeability. Samad et al. [4] studied the potential for rubber-based membrane in water permeation application with the addition of natural fiber as reinforcing filler and pore former. Recently, many researchers investigated the rubber-based membrane in Palm Oil Mill Treatment (POME) treatment [12]–[14].

It shows that the rubber-based membranes are suitable for many applications separations process depends on the structure and the presence of pores. Membranes made of thin-film composites with pores can be used to separate water and gases [15]. The porous structure is important to induce flux, permeability, and selectivity in membrane to be applicable for water treatment. Various methods are used to prepare porous membranes such phase inversion method and the addition of reinforcing filler [4].

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**NATURAL FIBRE REINFORCED RUBBER BASED MEMBRANE COMPOSITE**

The addition of reinforcing filler to thin-film composites has improved mechanical properties [16]. By including reinforcing fillers in the polymer matrix, thin-film selectivity and strength can be increased [17]. Ray et al. [18] reported that filler loading natural rubber (NR) membranes showed better toluene selectivities than unfilled membranes. The addition of fillers may increase the surface area and mechanical strength of the membrane. Table 1 shows the addition of filler in ENR/PVC matrix improves the properties of

composites. Increasing use of natural fibers reinforced composites attracted much attention in the past few years [19], [20]. Agricultural fillers (such as kenaf, pineapple, rubberwood, and palm oil empty fruit bunch) have been used to improve the material properties of polymer composites because of their low cost, low density, high specific strength, and modulus, environmental friendliness, and renewable nature [21]–[23]. Normally, fiber type fillers improved tensile strength because the fibers are able to support stresses transferred from the polymer [24].

TABLE 1: Filler to ENR/PVC matrix composites

Filler	Fabrication Techniques	Properties	Applications	Ref.
<b>Oil palm empty fruit bunch (OPEFB)</b>	Electron beam-Irradiation	Tensile strength, Young's modulus and gel content increases with a concurrent reduction the elongation at break (Eb) of the composites.	Composite material	Ratnam et al. [25]
<b>Oil palm empty fruit bunch (OPEFB)</b>	Melt blending	Young's modulus, hardness, and flexural modulus of the PVC/ ENR blend increase with the increase in OPEFB loading	Composite material	Raju et al. [26]
<b>Rubber-wood</b>	Melt blending	Flexural modulus, Young's modulus and hardness increased with the RW loading. The impact strength, Ts and Eb decrease with the increase in RW loading	Composite material	Ratnam et al. [24]
<b>Titium dioxide (TiO<sub>2</sub>)</b>	Melt blending, radiation	Good distribution of TiO <sub>2</sub> in the PVC/ENR blends matrix	Composite material	Ramlee at al. [27]
<b>Pineapple leaves fibre cellulose</b>	Solution blending, casting technique, phase inversion method	Number of pores increased with addition of cellulose. Decoloration of palm oil mill effluent after treated by ENR/PVC/Cell-20% and ENR/PVC/Cell-g-PMMA-10% membranes.	Composite material	Shamsuddin et al. [13]
<b>Rice husk powder</b>	Solution blending, casting technique, phase inversion method	Relative humidity (RH) reduces tensile strength and increases the tensile modulus. The number of pores increased with the increasing wt % of RH.	Water permeation	Ab et al. [4]
<b>Silica</b>	Solution blending, casting technique, phase inversion method	Thermal and mechanical stability of the membranes improved with the incorporation of silica. CO <sub>2</sub> and N <sub>2</sub> gas permeation of silica-filled membranes increased with increasing silica content	Gas permeation	Jon et al. [11]
<b>Silica</b>	Solution blending, casting technique, phase inversion method	Silica as pore former. mechanical properties of the membrane improved by the addition of silica. COD and BOD showed a reduction of 44% and 38.3%, respectively, after POME	POME treatment	Ismail et al. [14]
<b>Magnesium Oxide, MgO</b>	Solution blending, casting technique, phase inversion method	Pores were developed as fillers were introduced to the membrane. Permeability values of CO <sub>2</sub> and N <sub>2</sub> increased with the addition of MgO.	Gas permeation	Mohd Nor et al. [28]
<b>Microcrystalline Cellulose, MCC</b>	Solution blending technique	Chemical oxygen demand (COD), biochemical oxygen demand (BOD) and total suspended solid (TSS) were reduced to 99.9%, 70.3%, and 16.9%, respectively.	POME treatment	Arman Alim et al. [12]

## CONCLUSIONS

Rubber-based membranes exhibit behaviors which are elastic, flexible, ductile and tough that usually used in pervaporative separation process. The blending of thermoplastics (PVC) with rubber (ENR) was found to improve the physical and mechanical properties of membranes which is did not require additives and other support materials because the elastic nature thermoplastic elastomer is unique to generate membrane that good in selectivity, flux and resilience to stress. The addition of filler improved mechanical properties and performance of ENR/PVC matrix. The ENR/PVC membrane with the addition of filler showed development of pores on the membrane surface increased the water content, flux and permeability for the membrane to be applicable for gas and water separation process. From the table, we can summarize that ENR/PVC matrix compromises an ideal polymeric membrane in the presence of filler as reinforcing filler and pore former to membrane conducting to gas and water permeation application.

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**MODELLING THE LOW VELOCITY IMPACT OF 3D WOVEN FLAX  
REINFORCED COMPOSITE MATERIALS**

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**ABSTRACT**

Composites materials are used in products from daily life items to advanced applications. Latest advancements and research work is being performed to access strength of natural fiber-based composites and replace synthetic fibers with them as they are environment friendly, renewable resource, abundantly available, and cheaper in cost. Structures from 2D laminated composites are already being produced and tested from natural fibers while 3D woven reinforced composites need to be developed, tested, and optimized for their properties. It is also one of the major limitations in the advancement of 3D woven natural fiber composites. This study focuses on the impact properties of 3D woven flax structures. Four different hybrid 3D woven structures were produced, and composites were manufactured using green epoxy using vacuum bagging. Developed composite samples were investigated for drop weight impact strength and compression after impact. Composite samples were cut and impacted with a speed of 2.70 m/s and impact energy of 10J. The impact from drop weight did not cause complete damage of specimen. Absorbed energy was obtained and partially damaged zone was analyzed. Geometry and shape of damage zone helped to analyze the damage initiation, propagation, and failure of composite plate. The compression after impact (CAI) test was performed to determine the residual compressive strength of the composite material after drop weight impact test. Results were simulated and validated using Ansys Explicit dynamics.

*Keywords:* composite, natural fibre, low velocity impact

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**MECHANICAL AND THERMAL PROPERTY EVALUATION OF HYBRID FLAX  
/CARBON/KEVLAR BASED EPOXY COMPOSITES FOR AUTOMOTIVE  
APPLICATIONS**

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**ABSTRACT**

Natural fibre-reinforced polymeric composites are gaining significant attention in engineering applications. Hybridizing the natural fibres with synthetic fibers are being carried out has affected in an improved composite material system in terms of mechanical and thermal properties. The present work is to assess the thermo-mechanical performance of Flax/ Carbon/Kevlar fibre hybrid composites reinforced with epoxy. The composite laminates were fabricated using hand layup followed by compression moulding. All the laminates were prepared in 250 mm ×250 mm different stacking sequences. The specimen preparation and testing were carried out as per ASTM standards and tested for mechanical and thermal properties. The results showed the improvement of mechanical properties with hybridization of flax with synthetic fibers.

*Keywords:* Flax, Carbon, Kevlar epoxy; Mechanical Properties

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**EFFECT OF WATER ABSORPTION OIL PALM FIBER (OPF) REINFORCED  
POLYLACTIC ACID BIOCOMPOSITE: A REVIEW**

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**ABSTRACT**

In context of sustainability, environmental friendly and quality performance, biocomposites gaining much interest among researchers and industries over the last few years. Biocomposites is composed of biopolymer or biodegradable polymer as matrix and natural fiber as reinforcement. Generally, Polylactic Acid (PLA) are biopolymer that has a special interest as matrix in composite due to the environmental friendly material that has been recognized to replace non-degradable polymer materials due to their biodegradability, availability, renewability, and compostibility. However, shortcoming of PLA like inherent brittleness, high cost and low crystallization rate are considered as major limitation in broad application. Blending PLA with natural fiber like oil palm fiber (OPF) can solve the problem due to low density, lightweight, low cost, biodegradability and renewability. Few studies also reported that blending with starch can enhance the biodegradability, lower the cost of finished product and improved in water absorption (degradation). Among the environmental condition, water absorption is one of the most important ground to degradation which resist them to be used in outdoor applications. Several studies have been carried out on water absorption of OPF/PLA biocomposites and further consequences of water uptake on mechanical performance of biocomposites.

*Keywords:* Bio based polymer composites, sound absorption coefficient, noise reduction coefficient, sound transmission loss

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**STUDY ON THE EFFECTS OF TREATED/UNTREATED NATURAL FIBRE  
REINFORCED COMPOSITE – A REVIEW**

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**ABSTRACT**

Natural fiber reinforced composite materials attracts the wide commercial markets in the automotive, aerospace and marine industries. The cost efficient nature of the material and also the light weight properties, stability of the materials are some of the salient features and reason for the attraction. Hence researchers are in the quest of new materials as a reinforcement in a monolithic resin. The materials used for the reinforcement are mostly natural and artificial element such as natural fiber and carbon/glass/ceramic fiber. The natural fibers have the vast attention because of its availability and stability. Researchers also studied the effects of treated and untreated natural fiber composite materials. These studies reported that the treated natural fiber reinforced composites possesses the high stability in all the aspects of strength, thermal and dynamic mechanical properties. The treatment of these fibers were happened with the alkaline solutions to remove the hemicellulose content and improve the roughness over the surface of the natural fiber to improve the bonding between the fiber and matrix. For the treated fiber, the less amount of the fiber pullouts and fiber cracks were seen when compared to the untreated one. Hence, this review is intended to discuss the various aspects of the treated/untreated natural fiber reinforced composites and its advantages over the other polymer composites materials.

*Keywords:* Natural fiber, treated/untreated fiber, alkaline solution..

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**DEVELOPMENT OF NATURAL FABRIC REINFORCED FIRE-RETARDANT COMPOSITE**

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**ABSTRACT**

A composite is a hybrid material which is made up of a matrix and a reinforcement. A matrix is a material which is utilized as a binder while reinforcement provides fortification to the binder material. In recent times, great advances in material science have led to a significant interest of material scientists towards hybrid materials. The commonly utilized significantly strong hybrid materials are composites. There are many types of composites based on the type and structure of material. The highly used composites are the fiber reinforced composites. These composites have high strength to density ratio, low cost to strength ratio, significant weather resistivity, high solvent resistivity, good chemical resistivity, and poor moisture absorbance. All these qualities make fiber reinforced composites an ideal replacement of conventional material-based furniture. The most commonly used conventional material is wood. Thermosets are the mostly as used matrix in composites. While epoxy has better strength as compared to all the thermosets. Therefore, it is mostly used in furniture-based application. In current times, there are rising environmental concerns growing due to pollution which have driven the replacement of synthetic fibers to natural and biodegradable fibers. Fabric has more strength as compared to fibers as yarns are weaved to form a well oriented and organized two- dimensional continuous fiber structure which provides better strength as compared to simple fibers. However, there is a common ground for wood and natural fiber composites which is that they can steadily burn to ashes when subjected to combustion. Although, fire-retardants can be incorporated into the matrix of composites. Although, the fire retardants may be toxic to the environment as they may cause suffocation to living beings. Therefore, ecofriendly fire retardants are a good replacement for toxic fire retardants. Overall, it can be concluded that ecofriendly fire-retardant based natural fabric-based epoxy composites are a promising candidate for fire retardant furniture.

*Keywords:* Composite, natural fabric, ecofriendly fire retardant

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**INVESTIGATION OF MECHANICAL PERFORMANCE OF JUTE WOVEN  
REINFORCED THERMOPLASTIC COMPOSITE MADE USING YARN  
COMMINGLING TECHNIQUE**

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**ABSTRACT**

Composite materials have two components: reinforcement and resin. Depending upon the nature of resin, the composite materials are characterized as thermoplastic and thermoset composites. While the reinforcement can be synthetic or natural. There is huge pressure to make composites which are made with natural fiber and can be recycled. The thermoplastic composites are preferred due to low cost, low density and recyclability. The jute is natural bast fiber and found in abundance, it is used extensively in composites due to better specific mechanical performance. In the current study the mechanical characterization of jute woven reinforced thermoplastic composites were done made using thermoforming techniques while Polypropylene was used as thermoplastic resin. During the reinforcement manufacturing the jute yarn was singed to remove the issue of protruding fiber and it has improved the strength of jute yarn along with its easy processing on the machine. There is specific issue of resin distribution in the thermoplastic matrix due to its high viscosity, to address that issue the yarn commingling technique was used to improve overall impregnation of reinforcement. The jute and PolyPropylene yarn were commingled before weaving of reinforcement. The tensile, flexural, short beam shear and Charpy impact test were done for developed commingled and non-commingled composites. The mechanical results showed significant improvement in the mechanical performance of commingled composites. The microscopic cross section of tested samples also showed improved impregnation of commingled composites.

*Keywords:* Jute fiber, bio-composite, thermoforming technique, thermoplastic matrix

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**INVESTIGATION OF 2-WAY SHAPE MEMORY EFFECT OF BIO-BASED COMPOSITE STRUCTURE WITH DUAL-PROGRAMMING**

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**ABSTRACT**

The past decade has observed outstanding improvements in stimuli-responsive polymers (SMPs) along with its possible applications in advanced materials, automotive, textile composites, biomedical equipment, aerospace industry, electrical devices and engineering sectors. Bio based shape memory polymer composites have boosted and broadened the applications of shape memory polymers. Likewise, in order to reinforce bio-based composites, environmental-friendly natural fiber is being known as a promising alternate to synthetic fiber. Also, over the last decade, biomaterials have gained tremendous popularity both in scientific and technology-based researches. Flexible bio-based reinforcement could make it possible to improve the actuation property of the shape memory polymeric composites like shape memory, shape fixity and shape recovery effects in order to reduce the volume of large structures. This feature work presents up- to-date research on single and dual shape memory effect from bio-based polymer composite structures. In this research, the single layer bio-based shape memory polymer composite is developed and characterized, that have two-way effect and provide the more activation displacement at low temperature. For this purpose, composite samples are fabricated by using different lengths of jute yarn. After that the composite plates are characterized at different temperatures in order to obtain the single and dual shape memory effects from it. Furthermore, the effect of different lengths of jute yarn on single and dual shape recoveries are also evaluated by using external stimulus (thermal) and room temperature. The resultant single layer structure of natural fiber reinforced composite successfully shows the more activation displacement in dual shape memory programming cycle at low temperature. Also, it is found that the shape memory and recovery property decrease as the length of the jute reinforcement increases because the long length of the jute fiber impart the rigidity in the structures.

*Keywords:* Composite structure, shape memory, jute fibre

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**LVI PROPERTIES OF HYBRID NAURAL FIBER METAL LAMINATES**

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**ABSTRACT**

Fibre metal laminates (FMLs) have better low-velocity impact (LVI) performance as compared to its constituent materials due to the combined effect of composite and metal. Conventionally, these are developed by sandwiching man-made Uni-Directional(UD) or cross-ply fibre layer with aluminium, while the epoxy is used as a resin. The UD or cross-ply reinforcement offer poor LVI performance as compared to 3D-woven reinforcement, while both synthetic material and epoxy raise environmental concerns due to non-recyclability and non-biodegradability. Natural fibres can replace synthetic fibre subject to address low mechanical performance. Therefore, in this article an effort is made to investigate the LVI performance of novel FML made with hybrid reinforcement produced by sandwiching 3D-woven jute core with plain-woven skin, while both thermoset and thermoplastic matrix employed, respectively. Emphasis was placed on making the Jute hybrid reinforced FMLs with improved impact properties. Several configurations were manufactured and tested leading to a parametric study integrating both the type of weaving (2D and 3D) and the nature of the polymeric matrix (thermoplastic, thermosetting). This study showed the contribution of jute fibers as well as thermoplastic matrices for the impact performance of these new sandwich materials. The new hybrid reinforced FMLs made with thermoplastic matrix and jute reinforcement showed far superior results than conventional thermoset based FMLs. The solution proposed in the current research is relatively low cost, environmentally friendly and thus will expand its application area.

*Keywords:* hybrid reinforcement, low-velocity impact performance, X-ray computed tomography, fibre metal laminates, thermoplastic matrix.

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**NATURAL FIBER HYBRID COMPOSITE BALLISTIC HELMET DEVELOPMENT  
PROCESS WITH CONCURRENT ENGINEERING BETWEEN INDUSTRIAL  
DESIGN AND ENGINEERING: A REVIEW**

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**ABSTRACT**

In particular, ballistic helmets serve to provide protection to the head from bullet attacks as well as explosive sparks. This study aims to discuss the redevelopment of the ballistic helmet design known as Personnel Armor System for Ground Top (PASGT) based on the existing design. The first part of the study is to discuss the problems on the existing ballistic helmet design such as weight, comfort, material and also cost. To overcome the problem, a study on ballistic helmet manufacturing materials should be conducted to identify potential materials. Next, this study will discuss/list natural fiber hybrid composite (NFRHC) materials that have the potential to minimize the use of synthetic fiber. This study also identified the advantages and disadvantages of the mechanical and physical properties of natural fibers such as kenaf, bamboo and sugar palm (*Arenga Pinnata*). Natural fibers have good properties to produce lightweight ballistic helmets, good thermal insulation and are a source of renewable materials. The development of ballistic helmet design requires Concurrent Engineering (CE) method which is the integration between material and design for manufacturing. The implementation of CE is very important in the development of a composite product because most of the composite products must be determined/set/selected during the early stages of the design process. Finally, the review of this study will identify existing problems on existing ballistic helmet designs and will suggest the use of natural fiber materials in the redevelopment of these designs.

*Keywords:* natural Fiber composite, sustainability design, product development, concurrent engineering.

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**GRAPEHENE FACE (GF) SUPER MASK**

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**ABSTRACT**

The emergence of a pandemic affecting the respiratory system can result in a significant demand for face masks. These include the use of cloth masks by large sections of the public, as can be seen during the current global spread of COVID-19. However, there is limited knowledge available on the performance of various commonly available fabrics used in cloth masks. Importantly, there is a need to evaluate filtration efficiencies as a function of aerosol particulate sizes in the 10 nm to 10 µm range, which is particularly relevant for respiratory virus transmission. We have carried out these studies for several common fabrics including, graphene, cotton, silk, chiffon, flannel, various synthetics, and their combinations. Although the filtration efficiencies for various fabrics when a single layer was used ranged from 5 to 80% and 5 to 95% for particle sizes of <300 nm and >300 nm, respectively, the efficiencies improved when multiple layers were used and when using a specific combination of different fabrics. Filtration efficiencies of the hybrids (such as graphene-cotton-silk,) were >90% (for particles >300 nm). We speculate that the enhanced performance of the hybrids is likely due to the combined effect of aerosol layer, mechanical layer and electrostatic layer-based filtration. Graphene is a super- material that is nano sizes and produces a continuity graphene fabric layer. Nevertheless, the graphene layer may increase the temperature to 80-degree Celsius on the surface of the face mask when we leave it under sunlight for 20-30 minutes. Cotton, the most widely used material for cloth masks performs better at higher weave densities (i.e., thread count) and can make a significant difference in filtration efficiencies. Our research also implies that gaps (as caused by an improper fit of the mask) can result in over a 60% decrease in the filtration efficiency, implying the need for future cloth mask design studies to take into account issues of “fit” and leakage, while allowing the exhaled air to vent efficiently. Overall, we find that combinations of various commonly available fabrics used in cloth masks can potentially provide significant protection against the transmission of aerosol particles.

*Keywords:* graphene, fabric, face mask, covid 19

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**EVALUATION ON HYBRID CORN HUSK FIBER/HIBISCUS TILIACEUS FIBER  
POWDER REINFORCED COCONUT SHELL/POLYESTER COMPOSITE:  
EFFECT OF VOLUME FRACTION ON MECHANICAL AND MORPHOLOGY  
PROPERTIES**

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**ABSTRACT**

Composites sourced from natural fibers and renewable fillers are continuously being developed to get the best performance compositions. This study aimed to evaluate the mechanical and morphological properties of hybrid cornhusk fiber/Hibiscus T. fiber powders (HTP) reinforced composite coconut shell (CSP) were fabricated by hot press technique. The effect of volume fraction of hybrid CHF and HTP has been evaluated through tensile strength, flexural strength, and impact strength tests. Morphological analysis of composite fractures has been analyzed by SEM. The results showed that the tensile and bending strength of 5% CSP/polyester composite and 10% CSP/polyester tended to decrease with increasing volume fraction of HTP; or tensile strength increases by about 7.42% - 16.1% and 12.3% - 15.9% respectively, and bending strength increases by about 4.2% - 53.3% and 3.8% - 56.3% respectively with increasing CHF. The best tensile strength and flexural strength were obtained from a 5% CSP composite with a volume fraction of 25% CHF and 10% HTP. Meanwhile, the highest impact strength was obtained from the 10% CSP composite. A large amount of CHF in the composite acts as a reinforcement resulting in high strength, whereas the HTP filler acts as a defect causing reduced mechanical properties. SEM analysis presents the interface bonds between CSP-CHF-HTP- polyester, voids, and CHF pullout with increasing CHF and HTP in the composite.

*Keywords:* Composite, cornhusk fiber, hibiscus tiliaceus powder, coconut shell powder, mechanical properties, SEM

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**ANALYSING THE IMPACT PROPERTIES OF HOLLOW GLASS  
MICROSPHERES FILLED NATURAL FIBRE REINFORCED POLYPROPYLENE  
COMPOSITES**

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**ABSTRACT**

Fibre-reinforced composites are vigorously used for engineering and structural applications owing to their superior performance characteristics. The detrimental ecological impacts of synthetic fibres led the focus of research towards natural fibres to provide sustainable, biodegradable and economical solutions. However, poor performance characteristics confined their applications which can be enhanced by the addition of fillers such as hollow glass microspheres (HGMs). HGMs were incorporated in woven commingled hemp and flax fibre fabrics through the dip and dry method. Composite laminates with HGMs (1.5 % and 3 % by vol.%) and without HGMs were developed using the compression moulding technique during this research work. The fabricated laminates were characterised in terms of drop weight impact and compression after impact (CAI) test. The flax fibre reinforced composites depicted superior drop weight impact properties (absorbed less energy, low deformation) compared to hemp fibre reinforced composites laminates. Furthermore, the addition of 1.5 % HGMs improved the impact properties, while a decrement was witnessed by 3 % HGMs addition due to the agglomeration of particles.

*Keywords:* Natural fibre, thermoplastic composites, fillers, hollow glass microspheres, impact properties, compression moulding.

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**MECHANICAL PROPERTIES OF ORTHOGONAL WOVEN STRUCTURE  
CARBON FIBER REINFORCED COMPOSITE**

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**Abstract**

In this research, orthogonal structure carbon fiber reinforced composite was developed and its properties were compared with those of laminated structure composite. To solve the problem of yarn damage caused by friction during the weaving process, a custom-built weaving device was build up. Compression and vacuum assisted resin infusion molding (VARIM) techniques were selected and compared. It was found that fiber geometries could be well retained using the later one. The orthogonal and laminated composites prepared by the VARIM technique with the fiber volume fraction were 32.76 % and 38.64 %, respectively. Flexure and impact tests were performed to evaluate the properties of laminated and orthogonal composites. The results show that orthogonal composite exhibits superior properties in the warp and weft direction. In addition, due to its straight yarn path and insertion of carbon fiber in the through-thickness direction, orthogonal fabric reinforced composite exhibits an improved bending strength over composites reinforced by laminated plain woven fabric, although the fiber volume fraction of orthogonal composite is much lower than that of laminated composite. The findings are indicating that orthogonal structure composite provides profound guiding significance for product development and application of carbon fiber composite.

*Keywords:* Carbon fiber; orthogonal structure; laminated structure; VARIM technique.

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**MECHANICAL PERFORMANCE OF JUTE-HEMP/EPOXY HYBRID COMPOSITES FOR FURNITURE APPLICATIONS**

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**ABSTRACT**

Due to recent global warming and threats of mass pollution concerns provoke researchers to investigate some alternatives of synthetic materials. Also, new environmental laws urged consumers towards eco-friendly products by emphasizing manufacturers on the production of recyclable and bio-degradable composite materials. Natural fiber reinforced polymer composite (NFRPC) consists of polymer matrix embedded with natural fibers. Type of fibers and source decide the different properties of NFRPC. Keeping in view the new environmental threats, different natural fibers e.g., jute, flax, hemp, sisal, and banana gaining importance day by day on industrial level in the field of composite materials and replacing synthetic fibers such as glass. In this work, effect of hybridization by increasing or decreasing the layers of jute(J)/hemp(H) reinforcements on mechanical properties of their corresponding epoxy composites was elaborated. Plain woven jute and hemp woven reinforcements were developed with similar areal density i.e., 350 g/m<sup>2</sup>. Jute and hemp reinforced composites were fabricated by compression molding technique using epoxy resin. Five cross ply laminates were fabricated with different stacking sequence of jute and hemp reinforcements i.e., H<sub>4</sub>, J<sub>1</sub>-H<sub>3</sub>, J<sub>2</sub>-H<sub>2</sub>, J<sub>3</sub>-H<sub>1</sub> and J<sub>4</sub>. Number of layers and fiber volume fraction in all samples were 4 and 37%±0.7 respectively. Tensile, flexural, Charpy and drop weight impacts were performed on these composite samples to compare their mechanical properties. Hybridization of layers showed a significant change in the mechanical properties of composites with respect to their controlled samples. Hybrid composite (J<sub>2</sub>-H<sub>2</sub>) having equal number of layers of both reinforcements showed the highest value of tensile strength, tensile modulus, flexural strength, flexural modulus, and Charpy impact strength. Drop weight impact test results revealed that J<sub>2</sub>-H<sub>2</sub> hybrid composite showed the highest values of maximum force for deformation and work done, while less surface damage was observed in hybrid composites as compared to controlled one.

*Keywords:* Jute, Hemp, reinforcement hybridization, epoxy, composites, stacking sequence, mechanical performance

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**THE EFFECT OF DENIM WASTE ON THE MECHANICAL PROPERTIES OF THE FIBER REINFORCE CONCRETE AND MORTAR**

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**ABSTRACT**

Use of different natural fibers waste in the manufacturing of concrete and mortar along with other materials will increases the mechanical properties of the concrete and mortar as well as the waste will be used in a better way instead of dumping or burning. As by burning of waste with other fuels it will add to the environmental pollution. So, the topic recycling or reuse of waste in concrete and mortar will help to reduce the waste as well as environmental pollution. In this current research the denim waste was collected in different sizes and recycle it into fibrous form to be used in fiber reinforced concrete (FRC) and in fiber reinforced mortar (FRM). The fibers are added in the concrete and mortar in three different ratios 1, 2 and 3 % by the weight of cement. Three replicates were manufactured with each ratio to check the mechanical properties and compare with the samples without fibers. After manufacturing of samples, they are left to be cured for 28 days. After the completion of curing the mechanical properties of these cured samples were checked like Flexural strength, Charpy impact strength of the FRC and FRM were checked along with the controlled samples (without fibers). After analysis of the results obtained from the Charpy impact strength and flexural strength it was concluded that by adding the fibers in mortar and concrete their flexural strength and Charpy impact strength was increased significantly with the increasing percentage the fiber directly up to 3%. Also, it was noted that by using the fibers in the FRC and FRM the cracks formation and propagation was reduced. The breaking pattern of the samples also vary from the pattern of the samples without fibers. So, the use of fiber in FRC and FRM is useful not only the utilization of waste but also help to reduce the environmental pollution.

*Keywords:* water barrier, biodegradable films, nanocellulose, food application.

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