Research Paper

Tensile and Flexural Properties of Compression Molded Composites of Epoxy Reinforced with Treated Sugar Palm Fibre

M.M. Harussani [1,2,3], and S.M. Sapuan [3,*]

[1] Department of Transdisciplinary Science and Engineering, School of Environment and Society, Tokyo Institute of Technology, 2-12-1 S6-10, Ookayama, Meguro-ku, Tokyo 152-8552, Japan; harussani.m.aa@m.titech.ac.jp
[2] Department of Materials Science and Engineering, School of Materials and Chemical Technology, Tokyo Institute of Technology, 2-12-1 S6-10, Ookayama, Meguro-ku, Tokyo 152-8552, Japan; harussani.m.aa@m.titech.ac.jp
[3] Advanced Engineering Materials and Composites Research Centre (AEMC), Department of mechanical and manufacturing Engineering, Universiti Putra Malaysia, 43400 UPM Serdang, Selangor, Malaysia; harussani.m.aa@m.titech.ac.jp; sapuan@upm.edu.my
* Correspondence: sapuan@upm.edu.my

Abstract: The major purpose of this study was to study the effects of acidic and alkali treatments on the chemical, physical and mechanical properties of treated sugar palm fibre reinforced epoxy composites, such as with NaOH solutions (ALSPFN9/epoxy) and acid treatment with HCl solutions (ACSPFN9/epoxy), respectively. The composites were fabricated via compression molding method. From this study, the alkaline treated SPF/epoxy composites (ALSPFN9/epoxy) showed lower water absorption (48.90%) and moisture content (11.40%) when compared to the others. However, alkali and acid treated SPFN9/epoxy composites exhibit reduced tensile and flexural properties compared to the untreated one owing to the high concentration acidic and alkaline solutions utilized during surface treatment, which causes severe fibre breakdown. Thus, further study on surface modification of natural fibre reinforced composites are paramount as it plays an important role for automotive, furniture and packaging applications.

Keywords: Sugar palm fibres; surface treatments; composites; hot compression moulding; composites

1. Introduction

Due to COVID-19 pandemic outbreak, there are several concerns worried by the researchers, environmentalists and technologists, including the increased of environmental concerns as well as reduced the dependence towards petroleum based materials [1]. Furthermore, 381 million tonnes of plastic wastes are produced globally each year, with this figure anticipated to double by 2034. The most regularly employed plastics are polypropylene (PP), high density polyethylene (HDPE), polystyrene (PS), polyethylene terephthalate (PET), and polyvinyl chloride (PVC), with PP and PE being widely utilised polymers in everyday plastic products [2]. Thus, the application of natural resources, such as natural fibres, as fillers/reinforcing agent in furniture, automotive, and packaging industries for possible alternative of fibre reinforced composites is paramount and significant to reduce the uses of plastic domestically. From previous studies, various natural fibres such as bamboo [3], [4], jute [5], [6], flax [7], hemp [8], sisal [9], kenaf [10]–[13], abaca [14]–[16], and sugar palm [17]–[22] were applied in natural fibre reinforced composites.

Natural fibre reinforced composites are by far the most probable candidates to supersede synthetic composites in a variety of engineering applications due to their multiple benefits, including reversibility, biodegradability, less abrasiveness to equipment, ease of fabrication, and relatively inexpensive [23]–[26]. The three key components that transfer the specific features of natural fibres to other inorganic reinforcing fibres are cellulose, hemicellulose, and lignin. The use of natural fibre has a number of advantages, including low density, easy fabrication, availability, renewability, biodegradability, and non-abrasive properties [27]. Thus, a fibre reinforced composite with balanced performance, particularly cost performance and lightweight, can also be manufactured by adding reinforcing biomaterials. Thus, the reinforced composites are environmentally friendly, recyclable, non-abrasive, and they are crucial in reducing our reliance on plastics.

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Unfortunately, natural fibres have some disadvantages which makes them unsuitable for particular applications. Natural fibre reinforced composites have several drawbacks, including sensitive towards moisture and humidity, and incompatible with a variety of polymeric matrix. The fundamental issue with its application is fibre–matrix adhesion, which is caused by the incompatibility of the hydrophilic natural fibres with the hydrophobic polymer matrix. Chemically treating the fibre surfaces may help to solve this problem. Chemical treatment or surface modification can improve the wettability of fibres to a greater extent [24]. There have been several studies throughout the literature where surface treatment has been successful in improving the natural fibre-matrix bonding behaviour to optimize the physical, mechanical, and chemical properties of various natural fibre reinforced composites that have been constructed including sugarcane [28], fan palm [29], kenaf [30], [31], pineapple leaf [32], sugar palm [33], [34], bamboo [35], *Coccinia indica* [36] and *Pennisetum orientale* grass fibres [37].

In this study, attempts had been made in enhancing the surface characteristics of sugar palm fibre (SPF), originated from sugar palm tree (*Arenga pinnata*) planted in local cultivation areas at Kuala Jempol, Negeri Sembilan, Malaysia, by means of numerous chemical treatments using sodium hydroxide (NaOH), and hydrochloric acid (HCl), respectively. It has been profoundly seen to be a paramount source of reinforcing agent for reinforced composites due to their better mechanical properties, and provide synergistic effects as well as its contribution to environmental sustainability [38], [39]. Thus, treated SPF reinforcement in polymer matrix composites remarkably shows a lot of promises in engineering applications based on previous reports by various researchers [23], [40]–[45]. Previously, Bachtiar et al. [46] had treated the SPF with alkali treatment. The treated fibres reinforced epoxy composites, which fabricated via hand layup method, showed higher tensile strength compared to the untreated one. Thus, it proved that alkali treatment was significant in order to improve the interfacial bonding between the hydrophilic fibre and hydrophobic epoxy (matrix). Atiqah et al. [43] had studied the effect of alkali and silane treatment on the tensile and flexural properties of treated SPF reinforced polyurethane hybrids. The silane- and alkali-treated SPF reinforced composites exhibited improved tensile and flexural strength. However, few studies on SPF have focused on the impact of different surface modifications and compression moulding method on the NaOH- and HCl-treated SPF strength.

Therefore, to the best of the author's knowledge, it is critical to broaden the area of study in order to identify the factors that increase SPF-reinforced epoxy composites as well as the fibre properties. This article was mainly to investigate the physical, tensile and flexural properties of the epoxy composites reinforced with treated SPF which originated from Kuala Jempol, Negeri Sembilan, Malaysia (and labelled as SPFN9). Plus, the fabrication method is different from work of Bachtiar et al. [46], compression moulding had been applied to produce composites with improved performance. There are a few or no previous work on SPF surface treatment incorporated with epoxy via compression moulding.

2. Materials and Methods

2.1. Materials

The reinforced composites comprised of sugar palm fibre (SPF) as the reinforcing agent, epoxy as matrix, and methyl ethyl ketone peroxide (MEKP) as hardener. Both chemicals were bought from Chemrex Corporation Sdn. Bhd., Selangor, Malaysia.

Sugar palm fibres (SPF), or also called as black fibre, were collected from trunk of the sugar palm trees that were cultivated from local plantation areas at Kuala Jempol, Negeri Sembilan, Malaysia, see Figure 1. A fully mature sugar palm tree with such a height of 15 m and a lifespan of greater than 6 years was employed in this study. The fibres were cleaned and air dried for 1 day prior to drying process in an oven at 85 ºC for another day. According to their distinct sources, each of these fibres was divided and marked as SPFN9.


(a) (b)

Figure 1. (a) Sugar palm tree and (b) its black fibre or widely called as sugar palm fibre, obtained from Kuala Jempol, Negeri Sembilan, Malaysia

2.2. Chemical pre-treatments

SPF was submerged in a large amount of tap water for a few days, then washed with water and left to dry for a week. Plastic crusher machine was used to breakdown the SPFs into smaller 5–10 mm pieces. The short fibres were then moved to a pulverise machine, which was continued by a siever machine, which collected fibre with an average size of 150–250 m. Preliminary assays on SPF were also conducted to determine the impact of the alkali and acid pre-treatment on the physical and mechanical characteristics of the fibres. Next, the SPFs were then subjected to the following technique of fibre surface modification:

2.2.1. HCl treatment

To allow surface modification of the fibres, a chemical modification was performed. To begin, the hydrochloric acid (HCl) treatment was carried out on 250 g of retted SPF by immersing it in 5 litres of 5% (weight/volume) HCl solution, which amounts to 1:20 (weight/volume) %, for around 2 hours at ambient temperature. By removing excess of the acid solution by rinsing the fibres with 100 ml distilled water, the acid-treated fibres were isolated for further composite fabrication.

$$Fibre - OH + HCl \ \rightarrow Fibre - Cl + H_2O \tag{1}$$

2.2.2. Alkali treatment

he SPFs were pre-treated for 3 hours with sodium hydroxide (NaOH) alkali solutions containing 6% concentration by weight. Surface contaminants and hemicelluloses inside the fibre were removed using alkaline solutions. After that, the SPFs were thoroughly rinsed with 100 ml water to eliminate the remaining NaOH solution, subsequently left to dry at 25 °C for 3 days and placed in an oven for a day at 60 °C to make it dry.

$$Fibre - OH + NaOH \ \rightarrow Fibre - O - Na + H_2O \tag{2}$$

2.3. Preparation of epoxy composites

The compression moulding method was used to create the epoxy-based composite specimens, which included a rectangle mould made of an aluminium sheet (300 x 300 x 3 mm), translucent plastic for the bottom layer, and a spacer frame. To create a matrix, the epoxy and hardener were mixed together in a 4:1 weight % ratio for roughly 10 minutes, which is the ideal technique to ensure good curing and a standard grade specimen. After mixing the epoxy and hardener, the mixture was poured over the SPF into the prepared mould and squeezed with a cold press. The composites were permitted to cure for about a day at room temperature. Finally, the ASTM standard for tensile (ASTM D638-14 [47]) and flexural testing (ASTM D790-10 [48]) was applied to form the produced composites into required dimensions. Acid and alkali-treated SPF reinforced epoxy composites were made using the same techniques.

Table 1. Formulation of untreated and treated SPF reinforced epoxy based composites

Composites	Epoxy (wt.%)	SPF (wt.%)	MEKP (wt.%)	Fibre treatment

USPFN9/epoxy	60	40	2.5	Untreated
ACSPFN9/epoxy	60	40	2.5	HCl treated
ALSPFN9/epoxy	60	40	2.5	NaOH treated

2.4. Chemical characterization

Neutral detergent fibre (NDF) and acid detergent fibre (ADF) were used to investigate the chemical composition of SPF [49]. The main fibre components, such as cellulose, hemicellulose, lignin, and ash, are assessed using this method. Calculate the quantities of cellulose and hemicellulose using the formulas below.

$$Cellulose = ADF - lignin \tag{3}$$

$$Hemicellulose = NDF - ADF \tag{4}$$

2.5. Tensile characterization

Cutting composite boards provided specimens for tensile testing of SPF reinforced epoxy composites. The specimens derived from D638-14 [47] were cut using a jigsaw. For each sample, five specimen replicates were analysed. Tensile tests were performed using an Instron type 5566 universal testing machine (Shakopee, MN, USA). The ASTM D638-99 [50] standard type-I specimen was 3.2 mm thick, 165 mm long, 13 mm wide in the thin section, and 19 mm wide overall, as shown in Figure 2. The displacement was measured with a 50 mm extensometer. At a velocity of 5 mm/min, the specimens were tested. The maximum elongation at break, as well as the tensile modulus and strength, were computed.

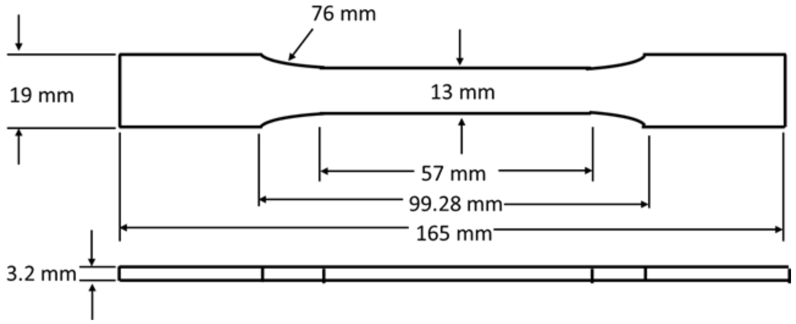

Figure 2. Tensile test sample of treated SPF reinforced epoxy composites

2.6. Flexural characterization

On each of the six rectangular replicates, three-point bending flexural assays were conducted on the SPF reinforced epoxy composites using an Instron model 5567 universal testing machine (Shakopee, MN, USA) following ASTM D790-10 [48] standard with a 5-kN capacity, at a controlled ambient condition of 23 ºC and room humidity of 50%. The samples are 120 mm x 20 mm x 3 mm in size, with such a crosshead speed of 2 mm/mm, see Figure 3.

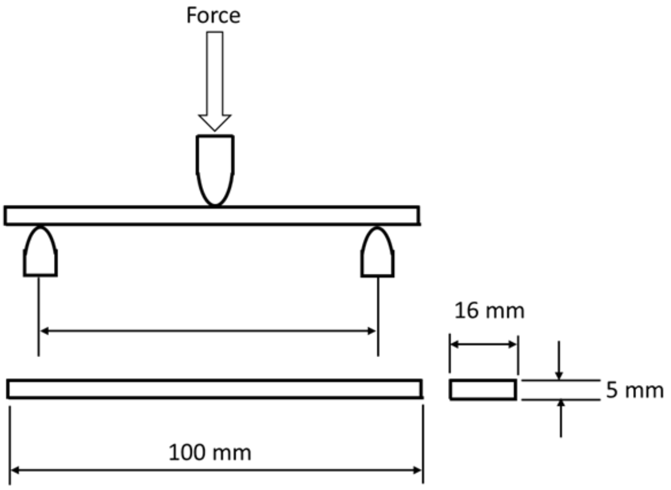

Figure 3. Flexural test setup and sample of treated SPF reinforced epoxy composites

2.7. Statistical analysis

One-way statistical analysis (ANOVA) was used to analyse mechanical and physical characteristics, and Duncan's multiple range tests were used to establish the significance of each mean property value ($p \leq 0.05$).

3. Characterization

3.1. Chemical characterization

The chemical composition of natural fibres has a significant impact on their thermal, physical, and mechanical qualities. Plant fibres are made up of a variety of natural components. Fibres are generally made up of a matrix of crystalline cellulose microfibrils strengthened by hemicellulose and amorphous lignin [23], [51]. The concentrations of these chemicals vary depending on the development settings (sources, climate, soil properties, and nutritional and ageing conditions) as well as the fibre processing/extraction processes [52]–[54]. The chemical composition analysis of raw SPF collected from sugar palm tree at Kula Jempol, Negeri Sembilan plantation area compared to different natural fibres is shown in Table 2. From Table 2, it was seen that flax, hemp, jute and sisal fibres exhibit higher cellulose contents with more than 60 %. Whereas untreated SPF and kenaf fibres comprised of lower cellulose content, 40 % and 44.53 %, respectively. However, USPFN9 comprised of the highest amount of lignin (41.97 %) compared to other fibres such as flax (2 %), hemp (~13 %), jute (0.2 %), kenaf (15 %) and bamboo (~21 %). Plus, USPFN9 exhibits the lowest hemicellulose content than the others, with only 10.01 %.

According to Kumar et al. [55], cellulose accounts for nearly half of all plant materials, along with hemicellulose, lignin, and pectin. The most basic cellulose unit is anhydro-D-glucose, which has three hydroxyls that give it its hydrophilic qualities [56]. Lignin reduces water sorption and increases thermal stability, whereas cellulose has superior mechanical properties. In comparison to other natural fibres, sugar palm fibre that has not been treated has strong mechanical qualities [57]. The hydrophilic characteristic of fibre causes poor fibre dispersion in a matrix, resulting in poor interfaces. The composite's ability to transfer stress between matrix and fibre is reduced due to the incompatibility of hydrophobic matrix material with hydrophilic fibres. Micro cracking is caused by dimensional changes in the fibre. Therefore, surface treatments are significant to modify the surface of fibre to strengthen its interface bonding and improve fibre-matrix adhesion in composite applications as well as reduce moisture sensitivity. To create rough surfaces at the fibre/matrix interface, alkali treatment (mercerization) and acidic treatment (hydrolysis) processes are performed [23], [58]. Chemical treatments reveal short-length crystallites by changing hydrogen bonding with in structure and eliminating partial lignin, waxes, as well as oils [59]. For improved interlocking, treated fibre has a higher concentration of accessible cellulose and a rougher surface. As a result, it aids in the development of better composites with high mechanical properties.

Table 2. Chemical composition of untreated SPF as compared to other fibres

Fibres	Chemical composition (%)			Ref.
	Cellulose	Hemicellulose	Lignin	
Flax	64.1	16.7	2	[60]
Hemp	55–80	12–20	3–13	[61]
Jute	64	12	0.2	[62]
Kenaf	40–50	18–24	15–20	[63]
Sisal	66	12	10	[64]
Bamboo	48–74	12–73	10–21	[65]
USPFN9	44.53	10.01	41.97	This study

3.2. Physical characterization

Water absorption and moisture content of treated and untreated SPF were recorded in Table 3. The characteristic percentage of the water absorption are in accordance with most water absorption studies found in the literatures [66], [67]. As illustrated in Table 3, the raw sugar palm fibre has 0.4 mm in diameters and density of 2.252 g/cm^3.

3.2.1. Water absorption

The untreated SPF (USPFN9) exhibits the highest value of water absorption of 161.96 %, followed by alkali-treated SPF (ALSPFN9) and acid-treated SPF (ACSPFN9) with 48.90 % and

40.44 %, respectively. The water absorption test was calculated based on previous work of Huzaifah et al. [68]. The high amount of water absorption of fibres is mainly due to its hydrophilic nature [56], which then will make it difficult to obtain good adhesion between fibre-matrix bonding. Thus, it affects the mechanical performance of the composites. All lignocellulosic materials consist of cellulose, hemicellulose and lignin, as discussed in previous subsection, which highly contribute to water absorption behaviour. This is due to the fact that these subjects contain many –OH groups that attract water molecules, as discovered by previous work of Sahari et al. [69].

Surface modifications on sugar palm fibre via NaOH and HCl treatments reduce moisture absorption significantly. Higher water absorption may be owing to the poor wettability features of untreated SPF (USPFN9). Whereas, based on Sawpan et al. [70], The contaminants that attach to and hemicelluloses part of sugar palm fibre were eliminated following the alkaline and acidic treatments, and the surface treatment reduced the hydroxyl groups on the sugar palm fibre surface by inducing hydrophobic sodium and chlorine elements. Thus, the treated SPF exhibited lower water uptake value. This observations in line with other researchers which studied with various natural fibres [24], [71]–[73].

3.2.2. Moisture content

Moisture content of natural fibre is a critical point to be considered when applying fibres as a reinforcement in reinforced composites. Table 3 shows that USPFN9 recorded the lowest moisture content percentage, 6.45 %, compared to ALSPFN9 and ACSPFN9, with moisture content of 11.40 % and 13.54 %, respectively. Moisture content of natural fibres affects their fibre dimensional stability, mechanical strength, porosity and swelling behaviour [54]. In production of composites for packaging and structural applications, low moisture content is the most desired characteristics as it will lead to lower degradability due to low amount of water absorbed by the natural fibre itself [74].

Table 3. Physical properties of SPFN9 prior to composite fabrication

Fibres	Diameter (mm)	Density (g/cm^3)	Water absorption (%)	Moisture content (%)
USPFN9	0.4	2.252	161.96	6.45
ACSPFN9	-	-	40.44	13.54
ALSPFN9	-	-	48.90	11.40

4. Results and Discussion

4.1. Tensile properties

The results of the tensile test on the untreated and treated SPFN9/epoxy composites are presented in this section. The tensile strength, Young's modulus and maximum elongation of the treated sugar palm fibre reinforced epoxy composites are represented in Figures 4–6. As shown in the Figure 4, the tensile strength decreases with both HCl and NaOH treatments for the SPFN9/epoxy composites. Plus, the neat epoxy sample show the highest tensile strength, 37.11 MPa. From the observations, the decrease in tensile strength is more pronounced with the alkaline-treated SPFN9/epoxy composite (ALSPFN9/epoxy) which displayed the lowest value of 21.48 MPa, while that of the hydrochloric acid-treated SPFN9/epoxy composite (ACSPFN9/epoxy) recorded 22.98 MPa whereas 27.09 MPa for the untreated SPFN9/epoxy composite (USPFN9/epoxy). According to Zwawi [75], the increase in tensile strength happened by the treated fibre reinforced composites was due to structural changes as a result of the high alkali and acid concentration during the chemical treatments, as observed in few other studies [19], [46]. The fibre and matrix have a weak adherence as a result of this structural alteration. As a result, stress transfer from the matrix towards the fibre is inefficient. High alkali and acidic concentrations have negative effects on fibres, weakening and damaging them. In order to manufacture high-performance composites, the optimal concentration of HCl and NaOH solutions must be achieved to have the most desirable mechanical and physical attributes. Additional explanation for the lower mechanical qualities might be came from the incorrect wetting of the sugar palm fibre with epoxy matrix as a result of manufacturing process errors. Furthermore, the rougher topography generated by the incomplete process of removing waxy and impurity compounds from the fibre.

Similar decreases in tensile modulus were seen comparing the untreated and treated SPFN9 based composites due to chemical treatments. The fibre reinforcement within the epoxy matrix, on the other hand, has a higher tensile modulus than the neat one. Figure 5 illustrates that the untreated SPFN9/epoxy composite (USPFN9/epoxy) has the maximum tensile modulus, 3.09 GPa, relative to ALSPFN9/epoxy and ACSPFN9/epoxy composites, which have 2.49 and 2.33 GPa, respectively, but the addition of treated SPFN9 produces deformation there in epoxy matrix. However, both the acid- and alkali-treated SPFN9/epoxy composites showed an increase in elongation at break percentage, as shown in Figure 6 below. Based on the tensile test findings, it is obvious that alkaline treatment has a greater positive effect on the composite qualities than hydrochloric acid treatment. Though the problem of fibre deterioration caused by excessive alkali concentrations can be mitigated by determining the best concentration and immersion duration for chemical treatment while maintaining the desired characteristics [23], [75]. It was also discovered that adding sugar palm fibre to the composite significantly reduced its overall mechanical performance, possibly due to micro cracking and voids within the composite that were not filled by the epoxy due to the fibre distribution.

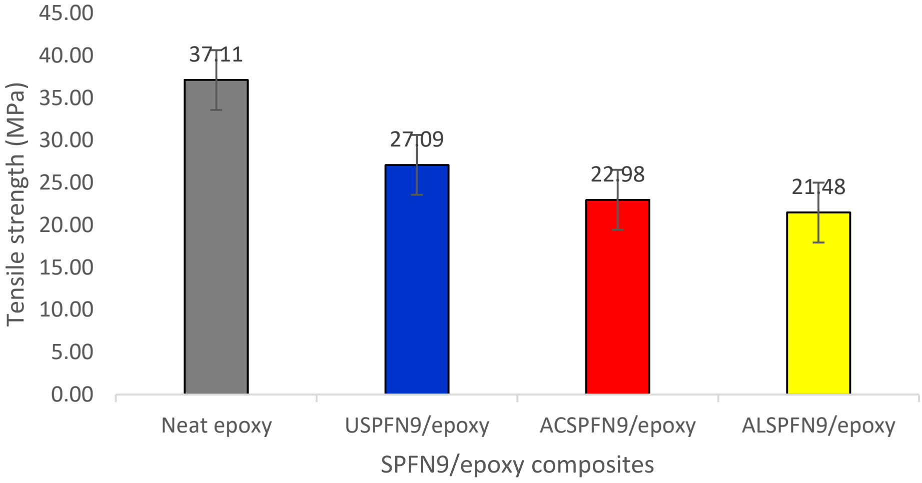

Figure 4. Tensile strength of untreated and treated SPFN9/epoxy composites

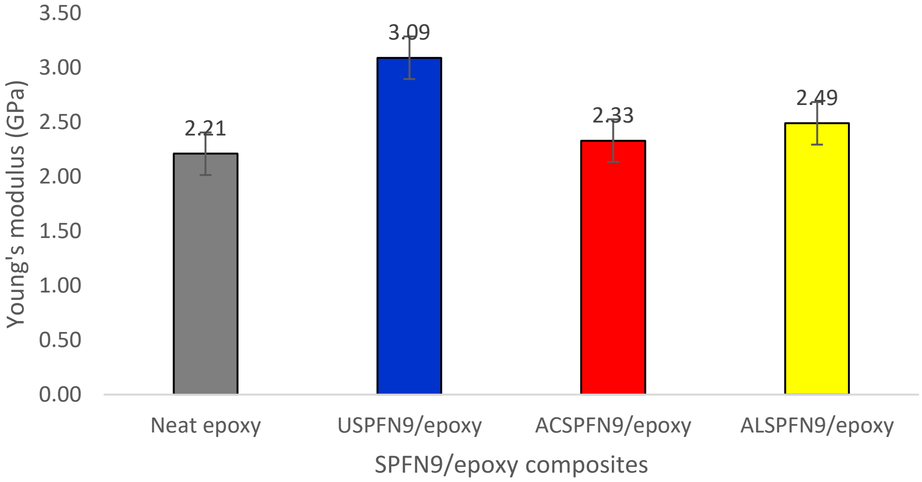

Figure 5. Tensile modulus of untreated and treated SPFN9/epoxy composites

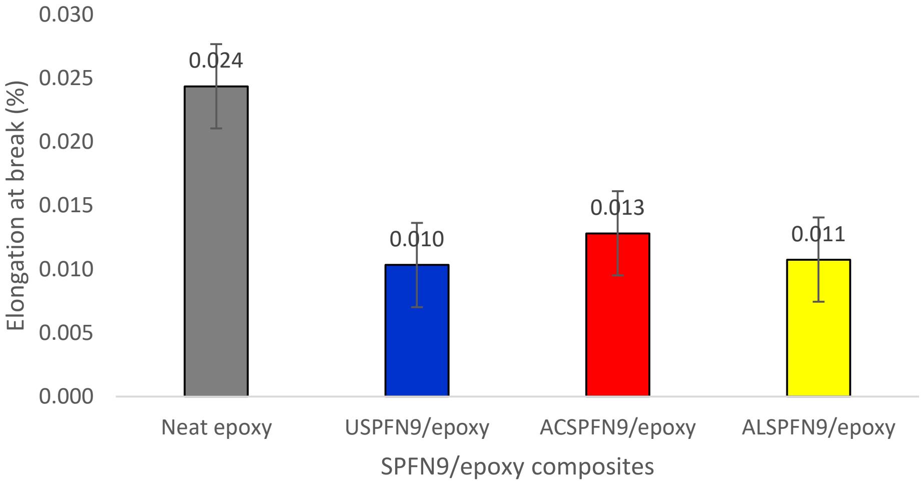

Figure 6. Elongation at break of untreated and treated SPFN9/epoxy composites

4.2. Flexural properties

Figures 7 and 8 illustrate the flexural characteristics of sugar palm fibre reinforced composites consisting of pristine samples, untreated samples, and both the alkali and acid treated specimens. The mean value and standard deviation of the flexural strength and flexural modulus readings were calculated from each of the five specimen specimens. A thorough analysis of flexural strength and modulus ensues for a better comprehension of these data.

The flexural strength of untreated and treated SPFN9/epoxy composites is shown in Figure 7. It can be observed in this diagram that as the type of treatment is changed, the flexural strength changes. According to the findings, acid and alkali treatments lowered the flexural strength of the reinforced composite samples. Furthermore, it has been established that the reinforcement of SPFN9 within the epoxy matrix reduces the flexural strength of the epoxy matrix. The neat epoxy sample has the highest flexural strength of 73.82 MPa, followed by untreated sugar palm fibre reinforced (USPFN9/epoxy) composites, HCl treated composite (ACSPFN9/epoxy), and NaOH treated composite (ALSPFN9/epoxy), all of which have slightly lower values of 54.44 MPa, 48.88 MPa, and 38.18 MPa. The alkali treatment of natural fibre removal of lignin and hemicellulose was reported by Bledzki and Gassan [76]. When hemicelluloses are eliminated from the interfibrillar region, the interfibrillar region becomes less dense and hard, allowing the fibrils to reorganise themselves in the direction of stress loading. Thus, fibrillation is the process of dissolving the hemicellulose in an untreated fibre bundle to break it down into smaller ones. As a result, the effective surface area accessible for interaction with the matrix is increased, improving the interfacial. However, excessive alkali and acid concentrations during surface treatments will result in severe fibre damage. As a result, the lower flexural strength seen in the treated SPFN9/epoxy composites can be explained.

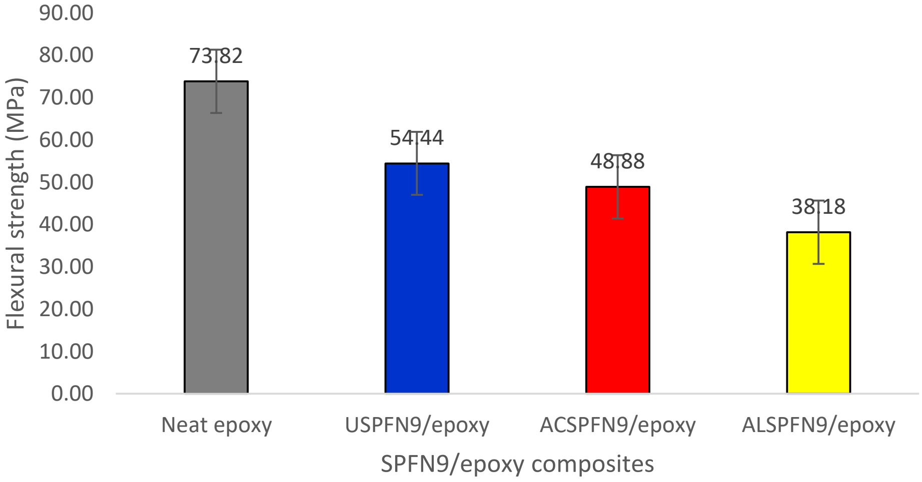

Figure 7. Flexural strength of untreated and treated SPFN9/epoxy composites

The flexural modulus of both the treated and untreated SPFN9/epoxy composites is shown in Figure 8. When different surface treatment types are used, a modest drop in flexural modulus is observed. In general, the stiffness parameter of the composites examined is the flexural modulus. The comparison of flexural modulus of HCl and NaOH treated SPFN9/epoxy composites with the untreated composite can be seen in the results. The flexural modulus of reinforced composites is reduced by alkaline treatment with NaOH (1.74 GPa). While the treatment with HCl solution results in a greater modulus value (1.81 GPa), the flexural modulus of both treated composites was lower than the untreated one (2 GPa). Previous research (please eloborarte) [23], [42], [77], [78] contradicts these findings. Mukhtar et al. [42] found that alkali treatment improves cellulose crystallinity and eliminates hemicellulose and lignin concentration in sugar palm fibres. The enhanced crystallinity of hard cellulose accounts for the greater fibre stiffness. In the case of sugar palm fibre, a similar explanation for the increased flexural modulus is proposed.

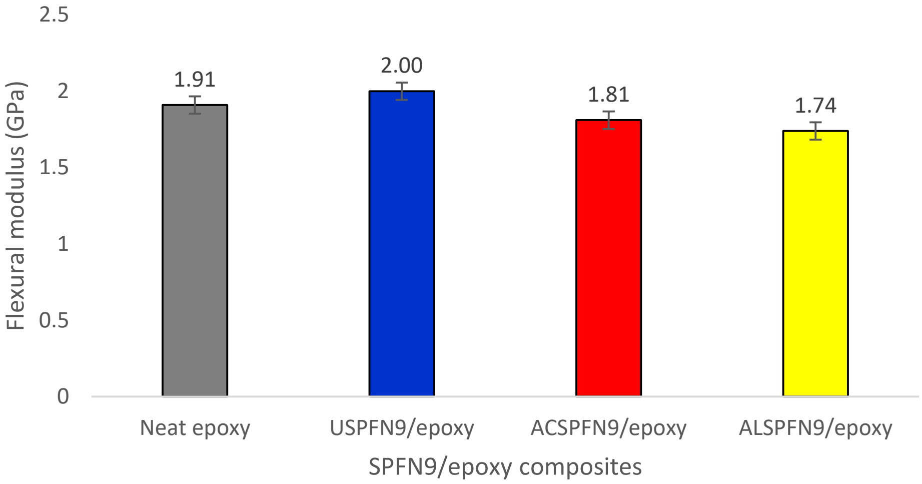

Figure 8. Flexural modulus of untreated and treated SPFN9/epoxy composites

5. Conclusions

To improve the adhesive bonding between SPF and epoxy matrix, alkaline and acidic treatments were used to modify the surface of sugar palm fibre. Physical and mechanical qualities were used to characterise the effects of both treatments on SPF. When it came to physical properties, the alkaline treated SPF/epoxy composites (ALSPFN9/epoxy) exhibited the lowest moisture content and water absorption. Untreated SPFN9/epoxy (USPFN9/epoxy) composites, on the other hand, have better tensile and flexural characteristics due to the high concentration acidic and alkaline solutions used during surface treatment, which induces severe fibre breakdown. In comparison to prior studies by other researchers, these treatments had an insignificantly negative result of tensile and flexural properties as compared to previous works by other researchers. It could be owing to the strong alkali and acidic concentrations used to modify the fibre during the procedure. In order to develop high-performance natural fibre reinforced composites for advanced engineering applications, more research into the appropriate concentration of alkaline and acidic solution is needed.

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