

Research Paper

Effect of Silane treatment on Rise straw/High density polyethylene Biocomposites

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Abstract: Polymer composite has been subjected to increasing interest, study, and utilization for some decades. The depletion of petroleum resources coupled with increasing environmental regulations are acting synergistically to provide the impetus for new materials and products that are compatible with the environment and independent of fossil fuels. Waste minimization is one of the problems that need to overcome. Rice straw is abundantly available and renewable in nature. As open-field burning of rice straw is being phased out in Malaysia, rice growers and government agencies are looking for new rice straw uses. In this study, a formulation was designed to produce silane crosslinked rice straw/High Density Polyethylene (RSPE) compound suitable for injection molding process. The formulation consists of High Density Polyethylene (HDPE) as the base polymer, rice straw as the filler, processing aids and a mixture of crosslink chemicals. Crosslink chemicals consist of vinyltrimethoxysilane (VTMO) as crosslinking agent, dicumyl peroxide (DCP) as the initiator and dibutyltin dilaurate (DBTL) as the condensation catalyst. The rice straw was oven dried at 70°C for 24 hours and grinded. A counter rotating twin shaft high speed mixer was utilized to mix the rice straw, HDPE and the processing aids. Test specimens were prepared via injection molding process followed by oven cured at 90°C. VTMO was used as coupling agents to adhere fillers to a polymer matrix, stabilizing the composite material. Mechanical properties of Crosslink RSPE biocomposite showed improvement in tensile strength, flexural strength and impact strength as a result of strong interfacial bonding between the filler and polymer matrix compared to uncrosslink.

Keywords: Bio-composites; Rice Straw; Natural fillers; Polymer; Renewable resource; Injection Molding

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1. Introduction

Biopolymers or synthetic polymers reinforced with natural fiber frequently termed 'biocomposites' can be viable alternatives to glass fiber reinforced composites. Natural fiber are chosen as reinforcement since they can reduce the chance of tool wear when processing, dermal and respiratory respiration [1]. The combination of natural fibers like kenaf, industrial hemp, flax, jute sisal, wood, and various straws with polymer matrices from both non-renewable (petroleum-based) and renewable resources to produce composites materials that are competitive with synthetic composites such as glass-polypropylene, glass-epoxies is gaining attention over the last decades. Although the strength of such fibers is lower than that of the traditional advanced composites, in certain

extent, the strength of plant-based fiber reinforced composites is sufficient for domestic or household plastic products.

Recently, natural fibers have been investigated as filler materials capable of serving as localized tensile reinforcement and volume fillers within several types of polymer matrices. The widespread use of biocomposites in a variety of contemporary applications is due to their various potential benefits, which include their high strength-to-weight ratio, non-toxicity, self-lubricating properties, lightweight, corrosion resistance, ease of fabrication, design versatility, better coefficient of friction, and wear resistance [2]. Presented study report by [3] state that the tensile strength of vakka fiber composite at highest volume fraction of fiber is much higher than those of sisal and banana composites and comparable to that of bamboo composites. Among organic filler, wood and cellulose fibers offer a number of benefits as reinforcement for synthetic polymers because of their high specific strength and stiffness, relatively low density, biodegradability and low cost on a unit volume basis [4]. From previous research, rice husk ash obtained from burning rice husk in an opened area, was reported to be apply as filler for rubber such as natural rubber [5] and for thermoplastic such as high-density polyethylene blend with wood fiber [6], [7], [8]. It was found that tensile modulus was improved with increasing filler loadings and the specimen became brittle at higher crosshead speeds as report by [9]. These composites would resolve environmental problems and offer the possibility of producing products having a range of different physical properties and functions.

Natural fibers are gaining progressive account as renewable, environmentally acceptable, and biodegradable starting material for industrial applications, technical textiles, pulp, and paper, as well for civil engineering and building activities ([10]–[12]). The best-known bast fiber plants are flax, hemp, linseed, jute, sisal, kenaf, yucca, abaca, and ramie which some can be seen in Figure 1. The advantages of plant fibers are low cost, low density, acceptable specific strength, good thermal insulation properties, reduced tool wear, reduced dermal and respiratory irritation, renewable resources and recycling possible without affecting the environment ([13]–[15]). These fiber plants have been grown, processed, and used in different climatic zones of the world.



Figure 1. Various type and sources for biofibers.

Straw is produced throughout the world in enormous quantities as a by-product of cereal cultivation. Half of the straw is simply burned on the fields just to get rid of it or else it is buried in greater quantities than what is needed to replenish organic matter in the soil; both of these practices contribute to environmental problems. Grain straw from rice, wheat, maize, oats, barley, and rye, left after harvesting forms a large source of fibrous biomass for the Western world. From the previous work showed that cereal straw can be a wood substitute as a fiber source for building panels and wheat straw that manufactured furniture panels which is advantageous to the environment and the economy [10]. Straw offers some technological advantages over wood because it allows better packing of the fiber strands and the degree of bonding between them is much better. Wheat straw fiber reinforced polypropylene composites exhibited significantly enhanced properties compared to virgin polypropylene as reported by Panthapulakkal (2006) [16].

Rice straw which is low-cost lignocellulosic biomass has become attractive as a renewable resource because it is available in large quantities and routinely cultivated as shown in Figure 2, 1575MT of rice were produced in 2009. Rice straw is the residue and the excesses production of rice that was not utilized. Burning rice straw in the field is a long-standing agricultural practice [17]. In traditional rice cropping systems, rice straw was either removed from the field at harvest time, and stored as stock feed or burnt in the field [18]. From the data available, the quantity of rice straw produced that may be available as a feedstock ranges from 1.0 to 1.4 million tonnes per year [19]. The options for the disposition of rice straw are limited by the great bulk of material, slow degradation in the soil, harboring of rice stem disease, and high mineral content. Nowadays, in California the rice growers have developed an alternative uses for the straw that is left over after harvest. With straw burning now so limited, many growers are tilling the straw into the soil and adding water in an effort to decompose the straw but this is a costly activity and may fosters rice diseases.

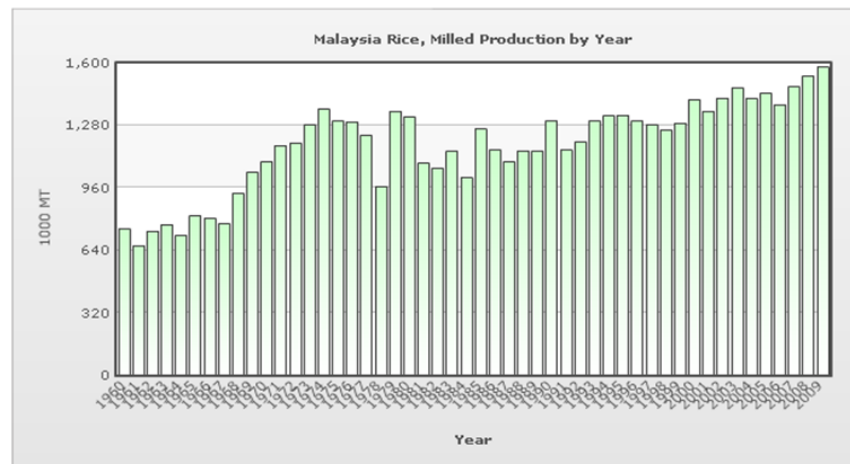


Figure 2. Malaysia Rice, Milled Production by year 1960-2009.

At harvest, the moisture content of straw is usually more than 60% on a wet basis, however, in dry weather straw can quickly dry down to its equilibrium moisture content of around 10-12%. Rice straw is unique relative to other cereal straws in being low in lignin and high in silica [20], [21]. In round numbers, on a dry basis rice straw contains 43 to 49% cellulose, 25 to 28% hemicelluloses, 15.8% lignin, and 12 to 15% silica [22]. Resins, gums, proteins, and mineral compounds are also presents. Various fiber components have different chemical constituents, especially cellulose and residual ash contents as shown in Table 1, which may contribute differently to the properties of rice straw fiber-reinforced thermoplastic composites [23].

Table 1. The ranges of the chemical constituent in cell wall

Constituent (%)	Wood		Others			Rice Straw			
	Soft wood	Hard wood	Flax	Jute	Sisal	Husk	Whole Straw	Leaf	Stem
Cellulose	40-45	45-50	64	64	66	35-45	41-57	37-41	24-46
Hemicellulose	25-30	21-36	17	12	12	19-25	33	22-25	24-28
Lignin	26-34	22-30	2	12	10	20	8-19	7-8	4-6
Residual ash	-	-	7	2	2	14-17	8-38	26-33	8-16

Compare with wood, straw tissue contains more silica and extractive and has less cell wall substances. Individual straw fibers have a much lower tensile strength and lower compressive strength as compared to other plant fibers. Like other fibers, rice straw can be easily crushed to chips or particles, which are similar to wood particle or fiber, and may be used for wood based raw materials [24]. Rice straw as an agricultures residue has been used in pulp and paper production for a long time and remains one of the major raw materials in many countries [25]. As future worldwide fiber shortages are predicted, agricultures fibers are believed to be potential substitution for wood fibers in certain applications. Use of the rice straw as a filler or reinforcement in the production of plastic composites alleviate the shortage of wood resources and can have the potential to start a

natural fiber industry in countries where there are little or no wood resources left. Rice straw has been used to reinforce clay in the making of bricks. It can also be used as a component in constructing drains and pipelines because it is made up of strong fibers that do not easily decay.

It has been reported that in last two decades, use of straw has been gaining much research attention as a potential alternative lignocellulosic raw material replacing wood for making composites particularly for construction application [26]. Han Seung Yang et al., (2003) [27] has developed rice straw wood based composite for sound absorbing wooden for construction materials. They revealed that the rice straw bending modulus of rupture (MOR) of the rice straw-wood particle composite board with a specific gravity of 0.8, have slightly better bending MOR than wood particle board (as control board). Han Seung Yang et al., (2004b) [28] also have developed rice straw-waste tire particle composite boards and report that the composite had better flexural properties than wood particleboard, insulation board, fiberboard, plywood and various other construction materials. This means that they can be used for specific purposes, for example as flexural insulating materials for curved walls. Composites with longer and wider rice straw particles showed better bending MOR. The sound absorption coefficients of rice straw-wood particle composite boards were higher frequency range than commercial wood based materials, such as particleboards, fiberboard and plywood which can contribute as a suitable sound absorbing insulation material in wooden construction [24], [28].

In spite of all the advantages mentioned about natural filler, there are also drawbacks in using natural filler as reinforcement in thermoplastics. One difficulty that has prevented the use of natural filler is the lack of good adhesion between the hydrophilic filler and the hydrophobic polymer [29]. The adhesion between the reinforcement fillers and the composite and the matrix in composite plays an important role in the final mechanical properties since the stress transfer between matrix and fillers determines reinforcement efficiency [30]. This is the main reason for using silane technology in crosslinking polyethylene-rice straw composites. Silane was used as coupling agents to adhere fillers to a polymer matrix, stabilizing the composite material. In general, the use of this coupling agent significantly improves the mechanical properties of the composites [31]. Thus, objective of this study was to investigate the effect of crosslinked rice straw/HDPE biocomposited on mechanical properties.

2. Materials and Methods

2.1. Materials

High density polyethylene (HDPE) thermoplastic was used as the base resin in the biocomposite compound. The high-density polyethylene used in this study was

purchased from Polyethylene Malaysia Sdn. Bhd. with the brand name, Etilinas (HD6070UA). The physical properties of this injection grade resin were listed in Table 2.

Table 2. Physical properties of HDPE grade HD6070UA

Property	Typical Value	Unit
Melt flow rate (190°C/2.16 kg load)	7.6	g/10 min
Density	960	kg/m ³
Melting point	132	°C
Vicat softening temperature	127	°C

Vinyltrimethoxysilane (VTMO) was used as a crosslinking agent in the formulation. The ability of VTMO to act as crosslinking agent was evaluated via gel content test. The organofunctional silane used in this research was Dynasylan® VTMO from Degusa and was used as received. Table 3 shows the specification for Dynasylan® VTMO.

Table 3. Specification for Dynasylan® VTMO

Data	Value
Appearance	Colorless liquid
Purity (GC-TCD)	≥ 98%
Hydrochloride	≤ 1.0 mg/kg
Boiling Temperature	123°C
Density (20°C)	0.97 g/cm ³

Rice straw was used as filler in the plastic compound. Rice straw is available in large quantities and routinely cultivated in Malaysia. In this study, the grinded rice straw particle was obtained from Persatuan Penanam Padi Tanjung Karang, Selangor. Rice straw tissue contains about 12-15% silica and has less cell wall substances [22]. The moisture content of bale straw is 20-25%. Rice straw was grounded and sieved into a particle size of 250 – 300 µm, 125 – 250 µm and 75 – 125 µm. After that, the ground rice straw was dried in a bench-top oven at 105 °C for 24 h. The rice straw and HDPE as well as the dispersing agent lubricant (Ultra-Plast TP01) and binding agent (Ultra-Plast TP10) were mixed in a high-speed mixer and then melt compounded using a co-rotating and intermeshing twin screw extruder at temperature of 180 – 190 °C. Vinyltrimethoxysilane (VTMO) was used as a crosslinking agent in the formulation. The pelletised compound was then injection molded into ASTM standard specimens for characterisation.

2.2. Compounding of Crosslinkable RSPE Formulation

Crosslinkable RSPE formulations were prepared by first placing the RSPE pellets in a dry sealable cylindrical mixer filling the mixer to about three quarters of its capacity. Pellets were then injected with the prepared silane chemical liquid mixture by using a syringe and blended on a laboratory scale Fielder mixer for 2 minutes. At the end of this time, silane chemical mixture was seen uniformly coated onto the RSPE pellets. The coated pellets were allowed to rest for 2-3 hours, the time necessary for complete absorption of the silane chemical liquid mixture. These pellets were then injection molded on injection molding machine model JSW N100 BII into ASTM standard specimens for mechanical testing. Machine settings that have been used were: 150 ° C first zones, 160°C zone two, 170°C zone three and 180°C for zone four. Cooling time was 60 s, injection hold pressure time 3 s, injection time 1.8 s and injection pressure 80 kgf/cm² [32]. RSPE biocomposite after molding were not fully cured. The curing process was accomplished by curing the sample in oven for 24 hours and the temperature used was 90°C.

2.3 Gel Content Test

Degree of crosslinking of the silane crosslinked RSPE sample was measured by determining their gel content after crosslinking reactions. The degree of grafting was regulated both by the silane and the peroxide concentration [33]. The determination of the gel content test has been performed by extracting the soluble component with decahyronaphthalene with refluxing at 200°C for 6 hours according to *ASTM D 2765-1995 Method B*

2.4 Impact Test

The impact test was carried out on Izod impact tester model LS 22005 according to ASTM D 256. Definition of Izod impact is the kinetic energy needed to initiate fracture and continue the fracture until the specimen is broken. Izod impact as shown in Figure 3 was a single point test that measures a material resistance to impact from a swinging pendulum. The RSPE biocomposite specimens were molded by injection molding. Notching at the middle of the specimen has been carried out using the notching tool. The notch depth fixed at 2.5 + 0.02 mm. The test conditions used were as in Table 4.

Table 4. Test Condition for Impact Test Testing

Velocity	=3.0m/s
Angle	=90°
Hammer energy	=3.87J

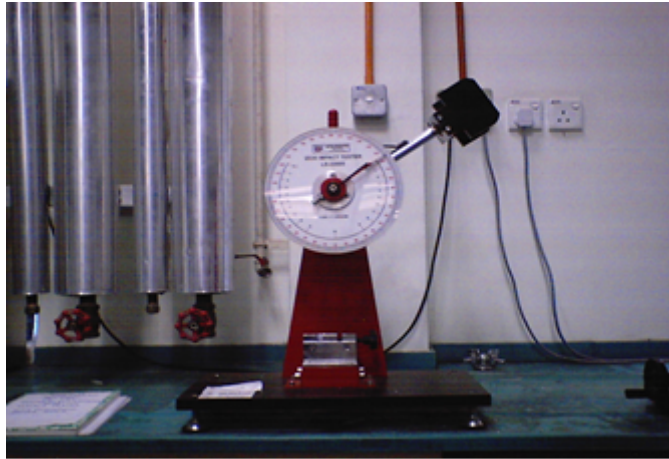


Figure 3. Izod Impact Tester

2.5 Flexural Test

Flexural test measures the strength of the RSPE biocomposite when a force was applied perpendicular to the longitudinal axis sample. Lloyd machine model EZ 20 was used for this testing according to ASTM D 790 three-point bending system. 3 mm/min cross head-speed was used, and the test was performed at a temperature of 25°C and humidity measurement of 58%-61%. A jig that allows span 51mm was used to maintain the consistency.

2.6 Tensile Test

The main factors that determined the tensile properties of composites beside the processing period were the filler dispersion, particle size, degree of filler adhesion and degree of degradation of polymer. Lloyd machine model EZ 20 as shown in Figure 3.3 was used for the tensile test according to ASTM D 638. Shape of dumb-bell specimens 3 mm thick was cut from the mould sheets. 50 mm/min of crosshead speed was used, and the test was performed at 25°C, and humidity measurement was 58%-61%.

3. Results and Discussion

3.1 Effect of VTMO Silane Crosslinker

3.1.1 Gel Content Test

The degree of crosslinking in the composites was determined by gel content measurements. Crosslinked polyethylene is insoluble in boiling decalhydronaphthalin while the uncrosslinked part is soluble [34]. Figure 4 shows the effect of vinylmethoxysilane (VTMO) concentration on the gel content of crosslinked rice straw/HDPE biocomposite. With increasing VTMO concentration, the gel content of the composite increase slowly from 17.14% to about 20.1% at low crosslinker concentrations (1 to 2 phr), and the gel percentage increased rapidly at high crosslinker concentrations (2.5 to 3 phr) indicates of crosslinks

formation. From previous study with silane grafted HDPE, it was shown that filler could be incorporated in the crosslink network since the silanol groups could react with hydroxyl groups in filler as well as with other hydroxyl groups grafted on the polyethylene backbone [35]. As shown from Figure 4, no gel was formed in the composites where no silane solution was added. A dosage of low crosslinker concentration does not significantly improve the crosslinking results.

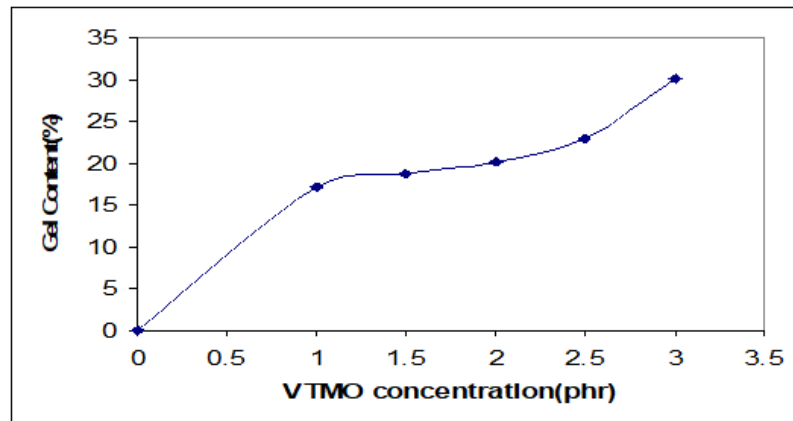


Figure 4. The Effect of the VTMO concentration on gel content of RSPE biocomposite

3.1.2 Impact Strength

Impact strength of the composites was one of the criteria used besides rheological properties. Previous research shows that the impact strength of pure HDPE decreases with the incorporation of filler [36]. The results for the impact strength test at various VTMO concentrations are shown in Figure 5. From the results increment of VTMO content increases the impact strength because of the toughness imparted by the crosslinked RSPE biocomposite. As can be seen from the figure, the silane crosslinkable at 2.5 to 3phr VTMO concentration composites with 30% rice straw content have at least 35% to 47% higher impact strength than uncrosslinked RSPE biocomposite. Crosslinking of the matrix is one of the ways of improving the toughness of the polymer matrix. With the incorporation of rice straw in the composites, the impact strength decreases, but the crosslinked composites still have higher impact strength than do the uncrosslinked ones. There is also indication of improved adhesion between the rice straw and the polyethylene matrix in the cross-linked composites. The improved toughness of the matrix and enhanced adhesion between rice straw and polyethylene upon crosslinking have also been reported by Magnus *et.al* (2006) [7].

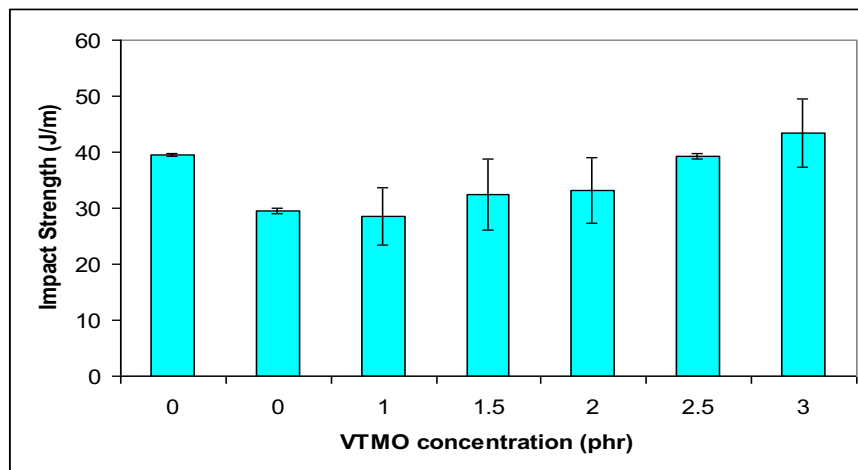


Figure 5. Effect of VTMO concentration on impact strength of RSPE biocomposite

3.2 Flexural Strength

The flexural properties of the biocomposites were determined in accordance with ASTM D790. The uncrosslinked composites showed inferior flexural strength compared to the composites in which silane was added. In previous study by Magnus and Kristina (2006) [29] on wood flour/polyethylene silane crosslinked composites gave similar result. It is observed that the flexural strength increases from 20.7 to 31.05 MPa for crosslink RSPE biocomposite with 3 phr VTMO concentration as shown in Figure 6. At 2 phr to 3 phr VTMO, demonstrates significant flexural strength increment of RSPE biocomposite compared to uncrosslink RSPE. However, lower flexural strength is seen at 2.5 phr of VTMO of the crosslinked samples could be due to unreacted silane. It is expected that some of the silane is just blended into the system without reacting. Part of the silane also evaporated or volatilized out of the system during processing. The excessive amount of unreacted silane could act as a plasticizer and lower the strength of the composites. This result is in accordance with gel content data as shown in Figure 4, which exhibits low gel content at 2.5 phr of VTMO.

Improved adhesion between the rice straw and the polyethylene matrix is also most likely the reason for the significant improvement in flexural strength of the crosslinked composites. The improved adhesion comes from the covalent bonding between filler and polyethylene through either condensation or free radical reaction. Moreover, hydrogen bonding between silanol grafted on polyethylene and hydroxyl groups on filler, as well as van-der-Waals forces between condensed silane on filler and polyethylene matrix, can improve the adhesion between the phases [7]. The improved adhesion between rice straw and polyethylene also provides explanation for the superior flexural strain before break of the crosslinked biocomposites. Crosslinked biocomposites did not break during the experiment, but the uncrosslinked, on the other hand, all was broken. Without interfacial adhesion, the gap between the rice straw and polyethylene phases provides an area of weakness which easily propagates a crack through the material.

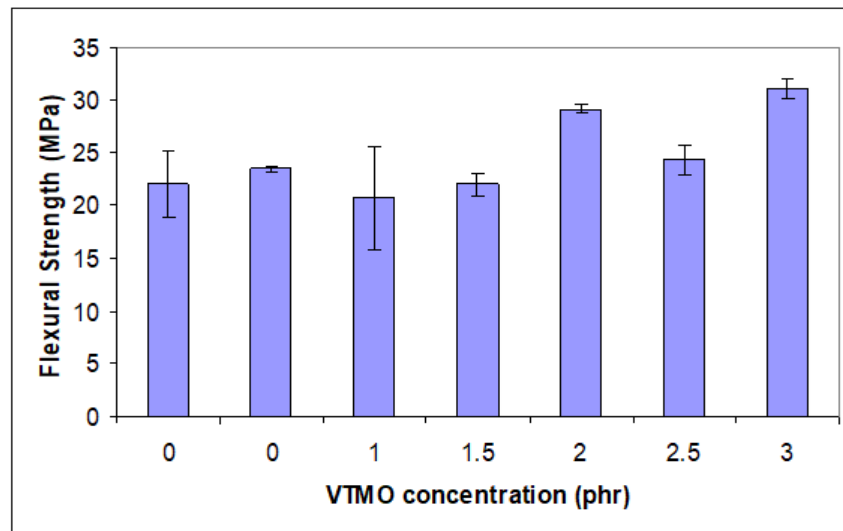


Figure 6. Effect of VTMO concentration on flexural strength of RSPE biocomposite

3.3 Tensile Strength

The tensile strength of crosslinked rice straw/HDPE biocomposite with various formulation of VTMO (Vinylmethoxysilane) is shown in Figure 7. The tensile strength of the composite increased gradually as the amount of VTMO is increased. Figure 7, demonstrates that the trend was more obvious at the higher composition of VTMO which is 3 phr. Crosslinking the RSPE biocomposite has improved the tensile strength above the uncrosslinked RSPE biocomposite. Tensile strength of crosslinked rice straw/HDPE biocomposite is correlated with gel content as reported by Toh (2005) [37] which is shown in Figure 4. As the graph indicate, at 17% of gel content (low value), the tensile strength is comparable to uncrosslinked virgin HDPE but higher than uncrosslinked RSPE biocomposites.

Manufacturing parameter is one of the reasons which can influences the drop in tensile strength whereby the composites melt experience higher internal stress during molding into product samples. During reactive injection mold process, melt flow is decreased due to grafting process and viscosity of the melt becomes low. This is another possibility causing the lowering of the tensile properties. At higher gel values (30%), the tensile strength seems to be higher compared to uncrosslinked virgin HDPE. This shows that VTMO has significantly increased the tensile strength of RSPE biocomposite due to the improvement in interfacial adhesion between the rice straw filler and polyethylene matrix.

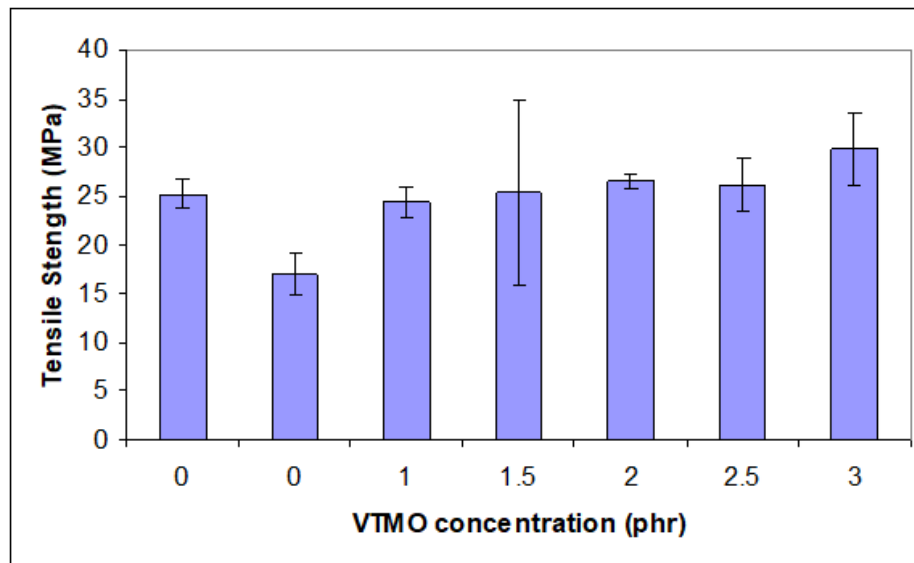


Figure 7. Effect of VTMO concentration on tensile strength of RSPE biocomposite.

4. Conclusions

Mechanical properties of RSPE biocomposite showed improvement in tensile strength, flexural strength and impact strength as a result of strong interfacial bonding between the filler and polymer matrix. Crosslinked RSPE biocomposites exhibit good mechanical properties compared to uncrosslink. It is showed that the tensile strength of cross-linked rice straw/HDPE biocomposite is correlated with gel content. At high percentage of gel content, tensile strength also increased. The improvement in impact strength and flexural strength could not only be due to enhanced adhesion between the phases, but could also be related to strengthening of the matrix upon crosslinking.

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