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Potential of Using Polyester Reinforced Coconut Fiber Composites Derived from Recycling Polyethylene Terephthalate (PET) Waste

Nurul Munirah Abdullah and Ishak Ahmad*

School of Chemical Science and Food Technology, Faculty of Science and Technology, The National University of Malaysia,
43600 UKM, Bangi Selangor, Malaysia

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Abstract: Unsaturated polyester resin synthesized from glycolyzed product of polyethylene terephthalate (PET) waste was used as a matrix to form coconut fiber/polyester composites. PET wastes were recycled through glycolysis and polyesterification reaction to produce a formulation for unsaturated polyester resin (UPR). FTIR spectra of glycolyzed product and prepared resin revealed that cross-links between unsaturated polyester chain and styrene monomer occurred at the saturated sites which resulted in the forming of cross linking network. To improve the adhesion between coconut fiber and polyester resin, various concentrations of alkali, silane and silane on alkalized fiber were applied and the optimum concentration of treatments was determined. The influence of water uptake on the sorption characteristics of composites was studied via immersion in distilled water at room temperature. Surface treatment of coconut fiber caused a significant increase in the tensile properties with the optimum treatment is 0.5 % silane on the 5 % alkalized coconut fiber/polyester composites. It was also observed that the treated fiber composites showed lower water absorption properties in comparison to those of untreated fiber composites. This observation was well supported by the SEM investigations of the fracture surfaces. From the study, it was concluded that polyester reinforced coconut fiber composites derived from recycling polyethylene terephthalate (PET) waste may have the potential application in the fields of construction and automotive interior substrates.

Keywords: Polyethylene terephthalate, Unsaturated polyester resin, Natural fiber, Coconut fiber, Mechanical properties

Introduction

The increases in the amount of plastics and agricultural wastes have contributed to various environmental problems. This has attracted the attention of researches to find new ideas to solve the problem. Polyethylene terephthalate (PET) is one of the versatile engineering plastics that are used to manufacture films and bottle for packaging. PET does not create any direct hazard to the environment, but due to the increasing in the plastics waste and its high resistance to the atmosphere and biological agents, it could be considered as pollutant materials. Therefore, recycling of PET will not only serve as a partial solution to the solid waste problem but also contribute to the conservation of raw petrochemical products and energy. In other words, product made from recycled plastics can results in about half energy saving [1]. The use of UPR from recycled PET as a binder is important for the construction industry and also helps in the long-term utilization of PET waste in an effective and economical manner [2].

Source from agricultural waste such as natural fibers have recently become an excellent reinforcement in composite materials. Furthermore, these fibers can also be used as replacement to man-made fiber such as carbon, fiber glass, Kevlar, nylon and asbestos to make a good ecologic and economic composite material. Because of their biodegradable nature, natural fibers have been increasingly adopted to replace synthetic polymers in the industrial applications [3]. Natural fibers are very attractive for composite materials because of their low cost, eco-friendly, available in high quantities, renewable, biodegradable, have excellent mechanical properties and low density [4]. However, the use of natural fibers as reinforcement agent, particularly in hydrophobic polymeric matrices has some drawbacks such as poor adhesion in the fiber/matrix interfacial region. This is because the natural fibers are highly polar due to the high polysaccharide content (hemicelluloses and cellulose), making the lignocellulosic fibers to also be highly hydrophilic [5]. Moreover, the large moisture sorption of natural fibers adversely affects adhesion with hydrophobic matrices that may cause material degradation and loss of strength [6].

Although a small number of studies have been carried out to utilize the polyester combined with coconut fiber to form coconut fiber/polyester composites, no report on using coconut fiber as reinforcement in polyester synthesized from recycled PET. Hence, the aim of this research is to use coconut fiber (CF) as fibrous reinforcement in unsaturated polyester resin (UPR) synthesized from glycolyzed product of waste PET. CF is obtained from the extraction external layer of the exocarp and from the endocarp of the fruit [7]. Coconut fiber has certain benefits in comparison to other natural fibers that have high failure strain and high weather resistance due to higher amount of lignin. CF also absorbs less amount of water because of the less cellulose content. However, as other natural fibers, coconut fiber is also found to be a poor reinforcement due to the large and variable diameter, high microfibril angle and high lignin and hemi-cellulose content but these disadvantages can be solved through chemical

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*Corresponding author: gading@ukm.my

treatment on the surface of fiber [8,9].

The objective of this study is to analyze the three most used treatments on natural fibers: alkali, silane and silane on alkalized CF treatment as a function of the variation concentration of the treatment. Despite the fact that a lot previous work were done studying the same treatments, most of the work did not take into account the variation of concentration of the treatment. Therefore, this investigation seek to find the optimum concentration of fiber surface treatment of CF and to study the effect of the treatment on the mechanical, water sorption and morphology UPR/CF composites based on recycled PET.

Experimental

Material

Drinking bottles of PET were collected and then grounded into fine pieces. Glycolysis of PET was done together with ethylene glycol and zinc acetate. Both chemicals were supplied by MERCH and Riedel-de Haën. Maleic anhydride, hydroquinone and styrene monomer from MERCK were used for the synthesis of unsaturated polyester resin. Curing process of UPR was done by using Methyl ethyl ketone peroxide (MEKPO) as accelerator and Cobalt octoate as an initiator respectively supplied by Revertex Sdn. Bhd. and Aldrich Co. The coconut fibers (CF) that act as a reinforcing agent were obtained from Fibromat (M) Sdn. Bhd. To modifiy the surface of CF by chemical treatment, Sodium hydroxide (SYSTERM) and silane (Supplied by Dow Corning) were used.

Glycolysis of Waste PET

Fine pieces of PET were put into a four-necked flat bottom flask with the capacity of 1 *l*. Ethylene glycol was added into the flask at the ratio of PET:EG=1:4 (w/w) together with 0.5 % zinc acetate based on weight of PET as a catalyst. Then, magnetic stirrer was put into the flask to ensure the mixture occur uniformly. This was carried out under reflux in nitrogen atmosphere with temperature at 190 °C for 8 h by using heated silicon oil.

Preparation of Unsaturated Polyester Resin

Unsaturated polyester resin (UPR) was prepared by using polyesterification reaction. Glycolysed product from glycolysis reaction was reacted with maleic anhydride at a fixed ratio of 1:1 for the hydroxyl to carboxyl groups. The polyesterification was carried out in a 1 *l* four-necked round bottom flask connected to a reflux condenser under nitrogen gas at 200 °C. The experiment was done by heating the reactant in an oil bath. The acid value was monitored during the reaction and determined by the titration of 0.1 N NaOH into the weighed resin in 100 m*l* acetone according to ASTM D 1639-90 method. The reaction was stopped when the acid value reached 29-34 mg of KOH/g. The liquid resin was then cooled to 120 °C and 0.45 % hydroquinone based on weight PET was added to prevent the precuring of the resin. Finally, the liquid resin was dissolved in styrene monomer to achieve a 40 % w/w styrene in the resin that equals to commercial UPR [10].

Coconut Fiber Treatments

Modification on the CF has been done to get a uniformed thickness of fiber mat. CF in the tangled form was vacuumed and compressed with a thickness around ~1-2 mm. CF in the form of unwoven mat was cut according to the size wanted. Before specific treatments were carried out, the fiber was first rinsed with distilled water to remove the dirt on the fiber surface. Washed CF was left to dry at room temperature and finally dried in oven for 2 hours at 60 °C.

CF has been treated with various concentrations of alkali between 2.5 % to 10 % (w/w). The fiber was immersed in alkaline solution for 2 hours then neutralized with acetic acid and washed with distilled water repeatedly until all sodium hydroxide was eliminated. Finally, the fiber was washed with distilled water and dried at room temperature for 48 h [11].

Various concentrations of silane solution were used in fiber treatment, ranging between 0.25 % to 1 % w/w. For each concentration, silane was dilute with distilled water and stirred using a glass rod for 15 minutes to form an aqueous solution. The silane solution was then added with acetic acid until pH of solution becomes 4 in order to optimize the performance of reinforcing material. CF was immersed in the solution for an hour. Finally, the fiber was washed with distilled water and dried at room temperature for 2 days.

CF that has been pretreated with alkali solution was immersed in dilute silane solution using the same method as for silane treatment of CF. Percentage of different concentrations used for each sample is shown in Table 1.

Preparation of the Composites

In order to prepare CF mat/polyester composites, hand lay-up method was applied. A stainless steel mould with the dimensions of (235×235×235) mm^3 and 3 mm of thickness was used. The curing of the polyester resin was done by the incorporation of 0.75 wt.% cobalt octoate and 1.5 wt.% Methyl Ethyl Ketone Peroxide (MEKPO) as a initiator and

Table 1. Percent concentration of the chemical treatment of each sample

Sample	Concentrations (%)		
	Alkali	Silane	Silane on Alkalized CF
A	2.50	0.25	2.50+0.25
B	5.00	0.50	5.00+0.50
C	7.50	0.75	7.50+0.75
D	10.00	1.00	10.00+1.00

accelerator respectively. The mixture was then poured on the mat form of CF. The volume fraction of CF in the composite, V_f, was approximately 0.30 with the thickness of specimens was 3 mm. The composite was cured at room temperature for 24 h before removing it from the mold. Finally, the composite was post cured in the oven at 60 °C for 6 h.

Analysis and Testing

Fourier transform infrared (FTIR) spectroscopy (Perkin Elmer, BX Model) was used to obtain spectra of functional groups for the glycolyzed product and prepared resin with the spectral range 4000-400 cm^{-1}. The IR spectra were presented in absorbance and were assembled in a common scale.

Tensile test on composites were determined according to ASTM D-638-91 standard. The measurements were done using Instron universal tensile machine (Model 5567) at a speed of 5 mm/min at room temperature.

The impact test was carried out using Universal Fractoscope (CEAST) 6546/000 impact tester according to ASTM D256-88 at room temperature. It was used to test the strength of composite samples when the load was suddenly applied. At least 7 samples were tested for each mechanical measurement.

Rectangular specimens with dimensions of (25.4×76.2) mm^2 were cut from each sample. 7 replicate specimens were tested and the results were presented as an average of the tested specimens. The samples were dried in an oven at 60 °C for 24 h, cooled in desiccators and immediately weighed to the nearest 0.001 g. In order to measure the water absorption of the composites, all samples were immersed in water for about 24 h at room temperature as described in ASTM D 570-99 procedure. Excess water on the surface of the samples was removed before weighing. Weight change was determined by means of a microbalance calculated at 10 mg [12].

The scanning electron micrograph of fracture surfaces following the tensile test of composite materials was taken using scanning electron microscope (SEM), Philips XL30. The samples were first dried in oven to remove air moisture and then coated with a thin layer of gold by using Sputter Coater machine. Examination of microscopy has been done at 500X magnification with the highest voltage of 20 V.

Results and Discussion

Characterization of Glycolyzed Product and Prepared Resin

The FTIR spectra for glycolyzed product (BHET) and unsaturated polyester resin (UPR) from recycled PET are shown in Figure 1 and the corresponding peak assignments are include in Table 2. From the table, it can be conclude that almost all the spectra for both samples were same whereas hydrogen bond OH, C-H stretching, carbonyl group (C=O) and aromatic ring were appeared. However, the absorption peak at wavelength of 776 cm^{-1}, 977 cm^{-1} and 1644 cm^{-1} can

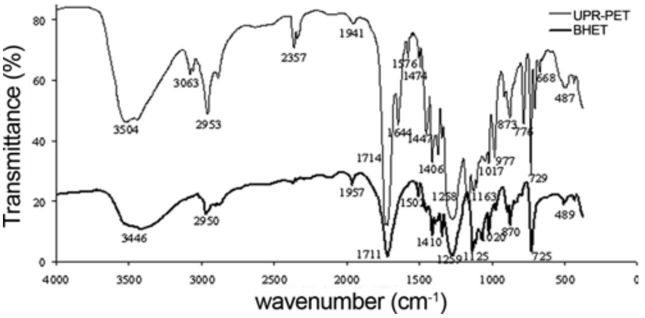

Figure 1. FTIR spectra of (a) glycolyzed product and (b) prepared unsaturated polyester resin (UPR).

Table 2. Characterization of FTIR spectra for glycolyzed product (BHET) and unsaturated polyester resin (UPR) from recycled PET

	Wavelength number (cm^{-1})	
BHET	3446	Hydrogen bond
	2950	C-H stretching
	1711	Carbonyl group (C=O)
	1125	C-O-C asymetric stretching
	489-870	Aromatic ring
UPR	3504	Hydrogen bond (OH)
	3063	C=C stretching (aromatic ring)
	2953	C-H stretching
	1714	Carbonyl group (C=O)
	1644,776	C=C stretching (vinyl)
	1163	C-O-C asymetric stretching
	977	C=C trans
	487-873	Aromatic ring

Table 3. Comparison of wavelength number for unsaturated resin from recycled PET (UPR-PET) with commercialized unsaturated polyester resin (UPR-C)

Wavenumber (cm^{-1})		Functional group
UPR-PET	UPR-C	
3504	3530	ν(OH)
3063	3080	ν(C=C) φ
2953	2983	ν(CH, CH_2, CH_3)
1714	1726	ν(C=O)
1644	1644	ν(C=C) vinyl
1474	1493	ν(C=C) φ
1258	1286	ν(C-O-C)
1163	1123	ν(φ-O-C)
1017	1070	δ(φ-H) in planar
977	982	δ(C-H) out planar
776	778	ν(C=C) vinyl
729	703	δ(C=C) φ at planar ring

(ν) streching, (δ) bend, and (φ) aromatic ring.

just be seen at FTIR spectra for UPR only and did not appeared at BHET spectra. This is due to the present of unsaturated double bond (C=C) in UPR as results of polyesterification reaction between glycolyzed product and maleic anhydride and also refer to the vinil group at styrene monomer.

FTIR analysis for commercial UPR (UPR-C) was also has been done to compare with UPR from recycled PET. Comparison of absorption peaks for both samples that which related are summarized in Table 3. Refer to the table, it can be seen that overall absorption peaks for UPR-PET are similar with UPR-C whereas unsaturated acid group in UPR-PET appear through wavelength number at 1644 cm^{-1} and 776 cm^{-1}. Both absorption peaks was obtain from molecular chain of unsaturated polyester and also styrene monomer.

Tensile Test

Mechanical properties of composites were strongly influenced by the adhesion between the matrix, the fibers and interphase properties. Due to the presence of hydroxyl groups in natural fibers, this makes the fiber to be of high moisture absorption and lead to the poor wettability and weak interfacial bonding between fibers and hydrophobic matrices. Therefore, in order to produce a composite with better mechanical strength, a modification on the fiber surface by using chemical treatments is needed to make the fibers more hydrophobic [13]. Figure 2 shows the analysis results of tensile strength for untreated CF and treated CF composites. It can be seen that the tensile strength increased with the treatment. This increase in tensile strength indicates that modifying the surface of CF improved the interfacial adhesion between CF and polyester matrix. On the other hand, untreated fiber composite has the lowest tensile strength is due to the weak compatibility between fiber and matrix. Figure 2 also shows that optimum concentration is obtained for sample B for all treatments. In sample B, the tensile strength increased 34 % for alkali treatment in comparison to the untreated CF composites. This is an indication that alkali treatment improves the fiber-matrix adhesion because of the removal of natural and artificial impurities. Moreover, during the alkali treatment, NaOH reacts with hydroxyl groups of the cementing material hemicelluloses and it leads to the destruction of the cellular structure and therefore the fibers splits into filaments. This phenomenon is called fibrillation, which breaks the untreated fiber bundle down into smaller fibrils by the dissolution of the hemicelluloses. This fibrillation then increases the effective surface area available for contact with the matrix. In addition, alkali treatment reduces fiber diameter and thereby increases the aspect ratio, improving mechanical characteristics of the resulting composites [14].

Silane treatment on the surface of CF also increases the tensile strength of the composite with the improvement in the tensile strength is 28 %. Silane coupling agents may reduce the number of cellulose hydroxyl groups in the fiber-matrix interface. Silane can be hydrolyzed into silanol when it reacts with moisture in the fiber. This silanol groups reacts with the hydroxyl groups on the surface of fiber, forming a stable covalent bonds on the cell wall. Meanwhile, the remaining silanol groups are adequate of hydrogen bonding or condensing with adjacent silanol groups (Si-O-Si). Therefore, the hydrocarbon chain of polymerized silane can adhere to the matrix mainly because of the van der Waals type of attractive forces. As a result, silane-coupling agent forms a bridge at the interface and improves the adhesion between fiber and matrix [15].

However, chemical modification of fiber surface with only alkali treatment was not enough to increase the strength of composite materials. Further treatment with a silane coupling agent solution to provide improvement adhesion between fiber and matrix by chemical interaction was necessary. The improvement of tensile strength for silane treated on alkalized CF composites is higher than that of untreated CF composites with a 41 % increase. The increase in tensile strength of the silane treatment on alkalized CF composites is due to the improvement of adhesion between the CF and thermoset matrix. Moreover, due to the intra and interfibrillar swelling of alkali treatment during first step of pretreatment and the present of covalent bonds between the fiber and matrix after the silane treatment, composites treated with silane on alkalized CF have the highest improvement in the mechanical performance [16].

The tensile strain of CF/UPR composite as a function of

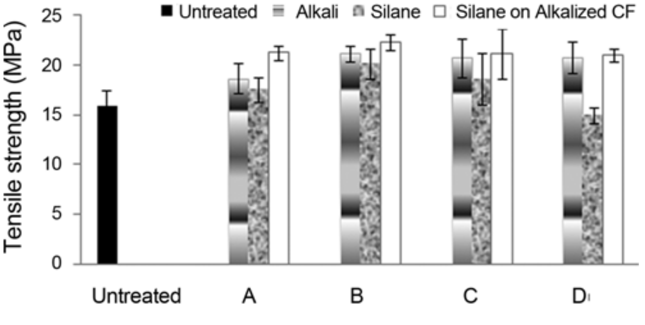

Figure 2. Tensile strength of various treated CF/UPR composites.

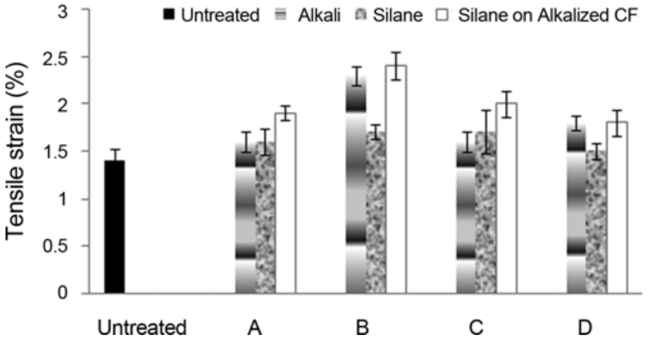

Figure 3. Tensile strain of various treated CF/UPR composites.

variation fiber surface treatment is shown in Figure 3. It can be seen that untreated CF composite has the lowest tensile strain in comparison to all treated CF composites. Based on Figure 3, silane treated on alkalized CF composite for sample B gave the highest value of tensile strain with the optimum concentration. It can be noted that composites reinforced by silane on alkalized CF were characterized by a little improvement in their resistance (higher tensile strength). During the reaction of silane treatment on alkalized CF, silanol forming stable covalent bond grafted between the microfibrils and leading to a high increase in the tensile strain. It is suggested that by providing additional sites of mechanical interlocking, silane treatment on alkalized CF leads to the improvement of the CF/UPR composites [17].

Impact Test

Figure 4 shows the impact strength of CF/UPR composites with variation of fiber surface treatment. Relative to the untreated CF composite, the value of treated CF composites are higher. These results suggest that the treatment had improved adhesion between the CF and UPR matrix and can act to prevent the propagation of cracks generated during the impact test [18]. The fiber/matrix adhesion mainly determines the strength of composites. Improved adhesion after treatment can lead to perfect bonding and thus the failure of composites can occur at relatively high impact strength. Composite having better interfacial bonding need more energy absorbing capacity when the crack propagation occur along the fiber/matrix interface and this is lead to a significant increase of impact strength [19]. The highest value of impact strength is for silane treated on alkalized CF composites with sample B is the optimal treatment and these are similar to the tensile analysis results. Further treatment with silane coupling agent on alkalized CF has induced chemical interaction between the fiber and the matrix. Hydroxyl group on the cellulose fibers could interact better with the silane due to the availability of possible reaction sites in large numbers. Hence, chemical treatment with silane on alkalized CF has increased the mechanical properties of composite from the aspects of tensile and impact strength.

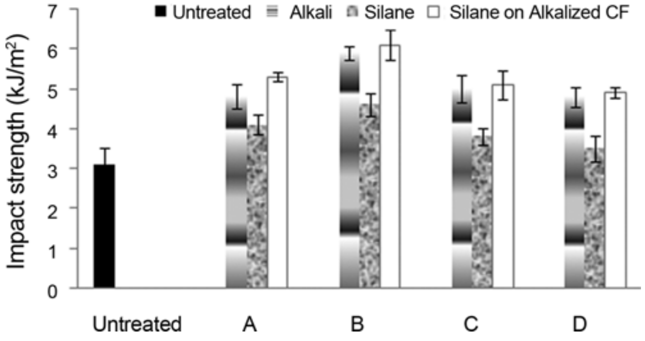

Figure 4. Impact strength of various treated CF/UPR composites.

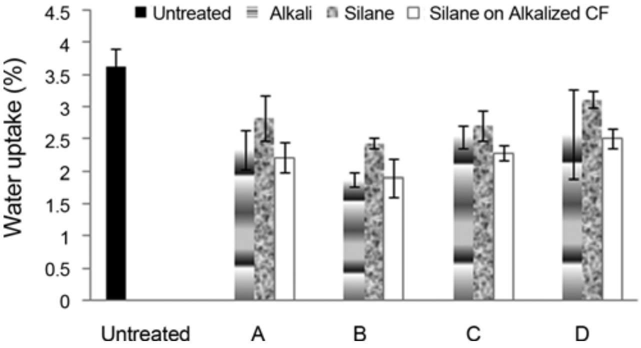

Figure 5. Water uptake of various treated CF/UPR composites.

Water Absorption

The water absorption behaviors of untreated and surface modified CF polyester composites are shown in Figure 5. The extent of water absorption decreases considerably on surface modification of CF. Treated CF shows considerable reduction in water absorption, which again confirms a better adhesion between treated CF with polyester resin. In case of untreated CF/UPR composites the poor wettability and adhesion between untreated CF towards polyester resin are due to hydrophilic nature of CF. This hydrophilicity is responsible for the higher percentage water uptake in untreated CF composites. Meanwhile, in case of surface modified CF composites, the fibers get masked with the polyester resin in the laminate with a stronger adhesion, resulting in greater hydrophobicity and lesser water absorption [20]. As it can observe in Figure 5, silane treated alkalized CF composite shows lesser water absorption in all sample. The order of decreasing value of water absorption composites are as follows: untreated < silane < alkali < silane on alkalized CF. After treatment, the first layer of fibers sorption have been modified by creation of a water resistant resin. It can be observed that the silane treatment on alkalized CF, which is the most efficient in terms of moisture resistance, is characterized by the lowest polarity parameter. Therefore, silane treatment on alkalized fiber had enhance the fiber-matrix adhesion and lead to a most hydrophobic surface [21].

From the Figure 5 for sample B, the water uptake of alkali treated CF composite (1.87 %) showed a close values to silane treated on alkalized CF composite (1.90 %). Other than the removal of impurities such as waxes, pectins, and mineral salts as well as treatment with NaOH, pretreatment with alkali promotes the activation of hydroxyl groups of cellulose unit by breaking the hydrogen bond [22]. Thus, after going through the silane treatment, a stable covalent bond has been accepted between fiber and matrix made the surface topology of fiber varies and the fiber becomes more hydrophobic in nature leading to good fiber/matrix interaction.

Scanning Electron Microscopy

Scanning electron microscopic analysis examined the

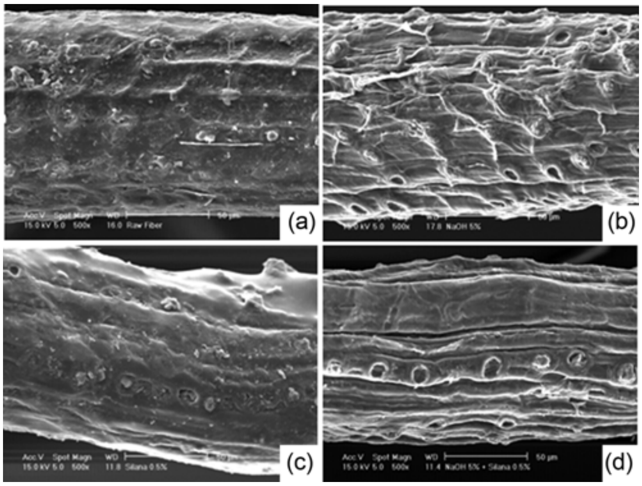

Figure 6. SEM micrographs of fiber surface for the (a) untreated CF, (b) alkali treated CF, (c) silane treated CF, and (d) silane treated on alkalized CF.

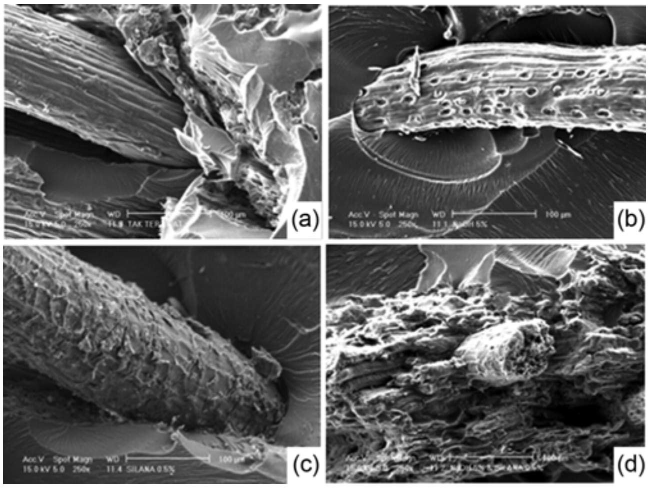

Figure 7. SEM micrographs of tensile fracture surface for the (a) untreated CF composite, (b) alkali treated CF composite, (c) silane treated CF composite, and (d) silane treated on alkalized CF composite.

surface morphology of treated and untreated CF. The removal of surface impurities on plant fibers is advantageous for fiber-matrix adhesion as it facilities both mechanical interlocking and the bonding reaction [23]. Figure 6 shows the SEM images of CF surfaces before and after modification with alkali, silane and silane on alkalized CF. After the alkali treatment (Figure 6(b)), the surface of the fiber become rougher and micropores were more visible compare to untreated fiber. Alkali treated CF surface showed rough surface topography due to the removal of hemicelluloses, lignin and amorphous waxy layer. Figure 6(d) shows the SEM micrograph of silane treatment on alkalized CF surface structure. The surface of the fiber becomes rougher in comparison to untreated fiber and micropores were not so apparent in comparison to alkali treatment and silane treatment. Cellulose is generally unreactive to many chemicals and the OH groups of the microfibrils have very low accessibility. Based on this fact, an optional step is to activate the alkoxysilane by hydrolyzing the alkoxy groups, thereby forming a more reactive silanol groups. As a result, the silanol may react with the hydroxyl groups of fibers and facilitate the enhancement of interfacial adhesion of treated fibers and polymer matrices [24].

Morphological studies of untreated and surface modified CF/UPR composites were also carried out to investigate the fiber-matrix interaction by scanning electron microscopy. Figure 7(a)-(d) displays the SEM micrographs of the untreated, alkali treated fiber, silane treated fiber and silane treated on alkalized CF composites fracture surface respectively. In other words, it appears that the fibers are extracted from the matrix. These clearly indicate that the interfacial adhesion between the fiber and the matrix is poor for untreated composites.

However, treated fiber composites (Figure 7(b) to 7(d)) have a different fracture surface in which the matrix debonding cannot be properly seen. It also can be seen that polyester resin adhered to the fiber surfaces of treated fiber composite. This may support good adhesion between treated fiber and polyester matrix. In the case of silane treated on alkalized CF composite, a large amount of polyester resin was adhered more to the fiber surfaces clearly and this indicates silane treatment on alkalized CF lead to better adhesion between the fiber and the polyester resin. From SEM observations, it can be concluded that modification of alkalized CF with silane coupling agent improved the adhesion between the fiber and the matrix. Therefore, the silane treated on alkalized CF composites has the highest tensile and impact strength in comparison to untreated and other treated fiber composites.

Conclusion

Chemical recycling of waste PET bottles through glycolysis was successfully performed to produce unsaturated polyester resin (UPR) which was suitable to be a matrix for the preparation of composite polyester reinforced with coconut fiber. The FTIR of prepared resin showed that the present of unsaturated double bond (C=C) in polyester molecular chain which is absent in the chemical formula of the glycolyzed product and there was also no considerable differential with spectrum of commercialized resin. Coconut fiber has been treated with different chemicals; such as alkali, silane and silane on alkalized coconut fiber at different concentrations. It may be pointed out that silane treatment on alkalized coconut fiber results in an increase in tensile strength as well as tensile strain. Moreover, from impact results of silane treatment on alkalized coconut fiber, leads to the impact strength increase in coconut fiber/unsaturated polyester resin based on recycled PET composites, which can be related to the effect of improved interfacial adhesion between coconut fiber and polyester matrix. The water absorption studies showed that treatment decreases the water uptake of the composites, which supports a better fiber/matrix interaction. The SEM of the treated fibers revealed that the fiber became porous and fibrillated by alkali, silane and silane on alkalized CF. The microscopic investigation of the fracture surfaces of composites also revealed enhanced bonding between the matrix and the fiber. The experiments also reveal a new opportunity for the reuse of PET waste products as components of coconut fiber reinforced composites.

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