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Characterization and Properties of Durian Skin Fiber Filled Polylactic Acid/Polypropylene Compatibilized with Glut Palmitate Salt

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ABSTRACT

PLA matrix provides good mechanical, physical and degradability properties, but somehow PLA alone is not good enough to produce a product as PLA has poor toughness resulting a brittle material. Natural fiber such as Durian skin fiber (DSF) can be incorporated into PLA/PP as a biopolymer to achieve the desired properties and better degradation which resulting a good bio-composite. Therefore, the study was carried out to produce bio-composite of DSF filled with PLA/PP and glut palmitate (GP) salt as compatibilizer. The bio-composites was successfully developed using different filler (DSF) loadings (0 php, 15 php, 30 php, 45 php and 60 php) with melt mixing blending. The FTIR analysis showed the new peak existed at 1595 cm^{-1} revealed a good interaction in the composite system which come from the bending of NH_2 vibration from GP. The mechanical properties (tensile properties) and physical properties (water absorption ability and density) of prepared bio-composite were determined. Increasing of DSF filler loading to 60 php DSF in PLA/PP bio-composite with the addition of GP increased in tensile strength (5.06 ± 0.09 MPa) and elongation at break (3.74 ± 0.18 %) due to enhanced interfacial bonding and strong adhesion between the polymer matrix and the filler due to the presence of compatibilizer. However, increased filler loading to 60 php DSF, showed a decreasing trend in the tensile modulus which represents high flexibility of the bio-composites. Furthermore, PLA/PP/DSF bio-composites absorption of water increases with the increasing filler loading until it reaches at

saturation point. The study concludes that the DSF reinforced PLA/PP with the addition of GP was suitable to be used in the production of bioplastic by many industries which can promote a sustainable environment for society.

Keywords: Hybrid Bio-Composite; Compatibilizer; Durian Skin Fiber; Polylactic Acid; Glut Palmitate

INTRODUCTION

Biopolymers are the ideal option to replace petroleum-based polymers and minimize environmental issues associated with waste because of their renewability, biodegradability, and commercial viability. Polylactic acid (PLA) is one of the most used biopolymers which is made from renewable sources such as corn, potato, cassava, and rice by bacterial fermentation [1]. PLA has become one of the interesting biopolymers due to its properties such as high strength, high modulus, compostable and regarded as a safe material for food packaging application [2]. PLA is a biodegradable plastic making it become an answer to "white pollution" and energy problems [3]. PLA has low thermal stability and low elongation property and adding reinforcement agent or filler to form bio-composites can improve the performance of PLA. The low flexibility properties of PLA were improved by blending PLA with polypropylene (PP) in the production of composite [4]. Anuar *et al.* [4], suggested that the incorporation of durian skin fiber (DSF) as micro and nano size filler in PLA and PP composites will enhance the capabilities of the bio composites. DSF reinforced with PLA/PP will produce good interfacial adhesion between the natural fillers and matrix with the help of compatibilizer. Hence, DSF can be potentially turned into benefit biomass through incorporation as a natural filler or reinforcement into polymer matrix. According to Wan Nazri *et. al* [5] reported that the tensile strength of low linear density polyethylene reinforced with Durian skin (LLDPE/DSF) declined as the DSF content increased. The incorporation of durian skin fiber (DSF) as micro and nano size filler in PLA and polypropylene (PP) composites will enhance the capabilities of the bio-composites [6]. The addition of fillers to polymer increases the strength and rigidity of the pure polymer [7]. PLA reinforced with banana fibers and treated with alkaline solution has better tensile properties and impact properties as compared to untreated composite rod [8]. A study by Yusoff *et. al* [9] reported PLA

incorporated with tapioca starch enhanced tensile strength with the greatest tensile strength achieved was higher than plain PLA at 30 wt loadings and then gradually decreasing with the addition of tapioca starch.

Nevertheless, of the type of natural fiber chosen as reinforcement in PLA matrix, the properties of the resultant composite are mainly influenced by the interaction between the hydrophilic natural fiber and hydrophobic polymer matrix. Compatibility of the combined constituents is the key in ensuring the performance of the developed bio-composite. To enhance the physical properties of plastic, additives are used in the production of bioplastics. Glut Palmitate salt (GP) is newly discovered as compatibilizer to enhance the adhesion in bio-composites production. GP is a non-toxic substance with amphiphilic properties where the palmitic segment is hydrophobic and glutamine segment is hydrophilic [10]. Hence, the lengthy chain of palmitate interacts with the non-polar matrix, while polar fillers can easily connect with the glutamine segment due to having the same characteristics [11]. Thus, making GP as a good compatibilizer in production of bio-composite consisting polymer matrix and natural fiber (DSF) as natural filler.

Although polymer composite is easily found in open literature, research on durian skin fiber reinforced with polylactic acid/polypropylene specifically treated with glut palmitate salt has never been explored. This study represents a new idea of utilizing durian skin waste as reinforcing fiber for polylactic acid/polypropylene compatibilized by glut palmitate salt. Therefore, the objective of this study is to determine the characteristics and properties of durian skin fiber as natural filler treated with GP in hybrid bio-composite of PLA/PP.

EXPERIMENTAL DETAILS

Materials

Polylactic acid (PLA) and Polypropylene (PP) were obtained from Nature Works and Vistec Sdn. Bhd., Puchong, Selangor, respectively. Durian skins were collected from Durian Central at Macalister Road, Pulau Pinang. Glut Palmitate (GP) salt was synthesized in Makmal Lateks Universiti Teknologi MARA (UiTM), Cawangan Perlis.

Synthesis of Glut Palmitate (GP) Salt

Glut palmitate (GP) salt was used as a compatibilizer to DSF/PLA/PP hybrid composite. The GP was synthesized by titrating a clear homogenous solution of 2 mol palmitic acid and 1 mol L- Glutamine solution at 60 °C and the solution was stirred vigorously using magnetic stirrer for 30 minutes until precipitates were formed. The precipitates were vacuum-filtered and then dried in oven at 80 °C for two hours.

Bio-Composite Preparation

The composition of durian skin fibers (DSF) was varied from 0 (control), 15, 30, 45 and 60 php. In each formulation of bio-composite, 3 php of glut palmitate (GP) was used. The amount of polylactic acid (PLA) and polypropylene (PP) were fixed at 75 php and 25 php, respectively due to the brittle properties of PLA. The formulation of the hybrid bio-composite as shown in Table 1.

Table 1: The formulation of PLA/PP/DSF hybrid bio-composite treated and untreated with GP

Sample	Composition			
	DSF (php)	PLA (php)	PP (php)	GP (php)
1	0	75	25	0
2	15	75	25	3
3	30	75	25	3
4	45	75	25	3
5	60	75	25	3
6	60	75	25	0

Each formulation of the sample was added together and mixed in melt mixer based on their melting point starting with PP, PLA, DSF and GP. PP has a melting point of 200 °C [12] while PLA has a melting point of 155 °C [13] and range below 200 °C [14]. The hybrid bio composite took six to 25 minutes to be fully homogeneous. Then, each mixture of the sample was poured into a mould that had been pre-heated previously for four minutes at 180 °C. Compression machine was used to mould the hybrid bio-composites to turn the mixtures into rectangular sheet in 2 mm thickness. Then, each sample slabs were fully compressed for seven minutes at 180 °C. Lastly, each sample slab was allowed to cool for 20 minutes before cut into different size of specimens that were used in characterization, mechanical

and physical tests.

FTIR Analysis

The spectroscopic equipment that used is Perkin Elmer FT-IR Spectrometer Frontier in order to determine the functional groups of PLA/PP (control), PLA/PP/DSF (60 php DSF) with GP (treated) and PLA/PP/DSF (60 php DSF) without GP (untreated). The spectrum was run within range of frequency 4000-550 cm^{-1}. All FTIR spectra that obtain from each specimen was recorded [10].

Tensile Test

A specimen of the sample 1 to 6 was cut in size 150 mm length and 15 mm width to be tested by using Universal Instron Tester to determine the tensile properties; tensile strength, tensile modulus, and elongation at break of the samples. The tensile testing was performed according to the standard ASTM D 638, crosshead speed was 20 mm/min [6]. The gauge length used was 100 mm. Six sample slabs were tested in triplicate. Results from tensile test were recorded.

Density Test

A specific size of 2 cm x 2 cm of the specimens from each sample slabs 1 to 6 were cut from the rectangular compressed sample. Each specimen was tested by using Electronic Densitimeter MD-300S to determine the density of the plastic. The standard used was according to ASTM D 792 [5].

Swelling Test

A specific size with 2 cm x 2 cm of the specimens from each sample slabs 1 to 5 were cut from the rectangular compressed sample according to standard ASTM D 570 for the testing of thickness swelling. Each specimen was soaked in distilled water and leave them for 7 days. After 7 days, all specimens were observed and calculated the thickness swelling (TS) according Eq. (1) [15];

$$\text{Thickness swelling} = \frac{TI - TO}{TO} \times 100\% \quad (1)$$

where,

TO: The thickness of specimen before immersion

TI : The thickness of specimen after 7 days of immersion in distilled water

Statistical Analysis

Statistical analysis is carried out by using Microsoft Excel 2013. All values obtain in the study presented in mean ± SD.

RESULTS AND DISCUSSION

For sample 1 (PLA/PP) which is the control sample, it produces a clear yellowish sample sheets while the other sample with DSF loading produces a brownish sample sheet. Figure 1 shows the sample slabs of DSF reinforced PLA/PP bio-composites and Table 2 shows the composition of DSF in PLA/PP polymer matrix treated and untreated with GP.

Table 2: Composition of DSF in PLA/PP polymer matrix treated and untreated with GP

Sample	Composition of DSF (php)	GP
1 (control)	0	Untreated
2	15	Treated
3	30	Treated
4	45	Treated
5	60	Treated
6	60	Untreated

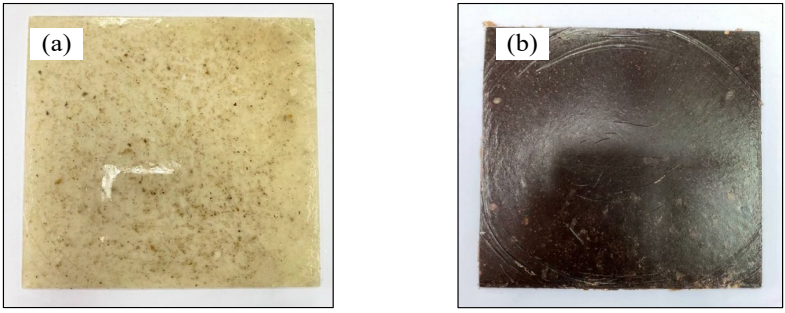

Figure 1: Sample slabs of (a) PLA/PP polymer matrix and (b) DSF filled PLA/PP

Characterization of DSF Filled PLA/PP Biofilm by FTIR Spectroscopy

The FTIR analysis was performed to examine the presence and interaction of functional groups in the composites. The infrared spectra of the samples PLA/PP, PLA/PP/DSF, and PLA/PP/DSF + GP are shown in Figure 2. The PLA/PP spectrum displayed the typical peak of PLA at 1746 cm^{-1} attributed to C=O stretching, 1183 cm^{-1} identified for C-O- bond stretching in CH-O-, three peaks at 1124 cm^{-1}, 1081 cm^{-1}, and 1044 cm^{-1} attributed to -C-O stretching vibration in -O-C=O, as well as the amorphous and crystalline phases of PLA associated with the peaks at 869 cm^{-1} and 750 cm^{-1}, respectively. Popa *et al.* [16] also had reported similar distinctive peaks. The typical CH_3 peaks for both PLA and PP structures were found at the peaks 2995, 2875, 1448, 1360, and 956 cm^{-1}, which, respectively, reflect asymmetrical stretching CH_3, symmetrical stretching CH_3, asymmetrical bending CH_3, and rocking CH_3. The peak observable features for CH_2 in the PP structure were 2948, 2831, 1452, and 702 cm^{-1}, respectively. These characteristics correspond to asymmetrical stretching CH_2, symmetrical stretching CH_2, symmetrical scissoring CH_2, and CH_2 rocking [17]. PLA/PP with the inclusion of a DSF sample exhibits shaped peaks that are similar in appearance but less intense. DSF is primarily made up of cellulose, hemicellulose, and lignin, all of which include functional groups that comprise CH_2 and C-O and share the same IR absorbance characteristics as PLA and PP [18, 19]. Moreover, the Carbonyl region, C=O, which is linked to the stretching vibration of the ester bond in DSF, was obscured. Due to the system's reduced PLA/PP concentration after the addition of 60 phr of DSF, as well as the poor interaction between the DSF and PLA/PP, the intensity reduced when DSF was introduced [19]. In the spectrum PLA/PP/DSF + GP, the GP was added to the PL/PP/DSF composites. Based on the spectra, a peak that is similar to the PLA/PP/DSF peak was seen, but with a stronger frequency and newly developing peaks at about 1595 cm^{-1}. The more intense frequency revealed a good interaction took place in the composites system, while the new peak suggested the bending of NH_2 vibrations originating from GP. This validated the presence of the GP [20].

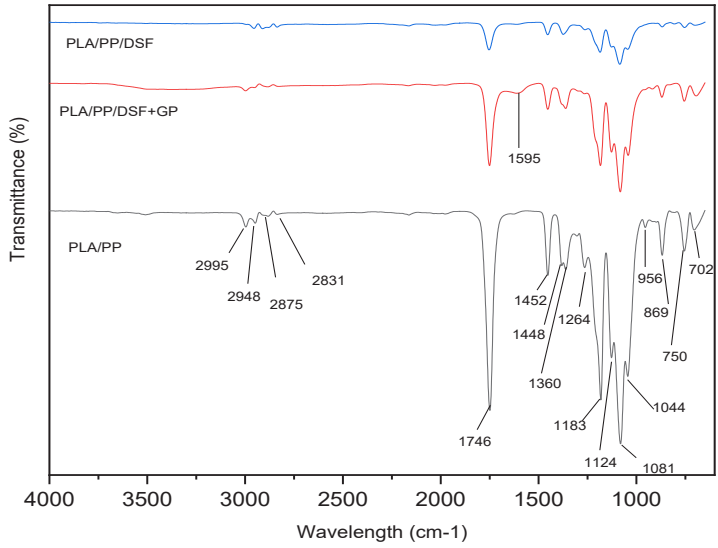

Figure 2: Infrared Spectrum of PLA/PP, PLA/PP/DSF and PLA/PP/DSF+ GP composites

Tensile Test

The PLA/PP/DSF bio-composite samples were tested using tensile test following the ASTM D 638 test standard. From this test, the tensile strength, tensile modulus, and elongation at break results of the bio-composite sample were obtained. Figure 3 shows the tensile strength for each sample of PLA/PP/DSF bio-composites at different DSF filler loading.

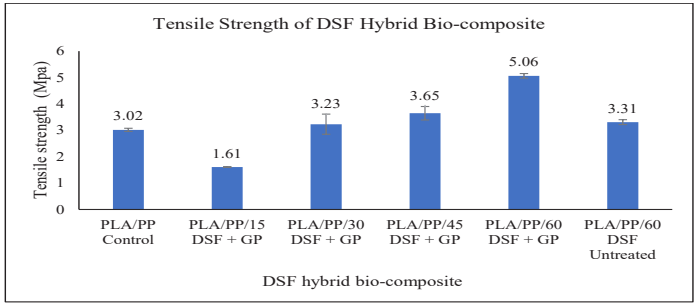

Figure 3: Tensile strength of PLA/PP with different DSF loading treated and untreated with GP

Figure 3 shows the presence of DSF treated with GP (15, 30, 45 and 60 php) altered the tensile properties of PLA/PP. The increase of DSF filler loading lead to gradual increase of tensile strength of bio-composites. The control sample (PLA/PP without DSF) compute a tensile strength of 3.02 ± 0.06 MPa. There is gradual increment in composite tensile strength as the amount of DSF was increased from 1.61 ± 0.01 MPa to 5.06 ± 0.09 MPa. As the amount of DSF increased, reactive site of hydroxyl group (OH) present in the composite is also increased. This OH create the hydrogen bonding with NH_2 from GP. This finding agreed with Lee *et al.* [21], whom claimed that increased in the tensile strength of PLA/DHF (durian husk fiber) with existing compound of fatty acid amide used as lubricant for composite in their study. GP increased interfacial compatibility, which facilitated stress transmission between matrix and filler [10]. This finding is supported by FTIR spectrum as shown in Figure 2. A peak existed at 1595 cm^{-1} indicated GP promoted the interaction of the constituents in the composite sample compared to untreated composite.

The tensile modulus indicates the stiffness of the composite. Reduced tensile modulus observed by increment DSF as shown in Figure 4 indicated that DSF can overcome the brittleness properties of matrix material. This result is in line with elongation at break, indicated better flexibility of the composite sample than that of the control sample as shown in Figure 5. It seems that DSF have the capability to promote ductility behaviour onto the composite sample. Zaini *et al.* [11] reported that the increased in elongation at break is due to the greater ductility of the composites caused by the uniform dispersion of filler and matrix inside the composites with the addition of GP. Sahi *et al.* [22] also found the addition of plasticizer increases the elongation at break. This is due to the plasticizing of GP which confers to the composite's properties of flexibility. Bio-composite sample of 60 php DSF without GP shown 36 % and 46 % decreased of tensile modulus and elongation at break than treated sample at the similar loading of DSF, respectively. This means the untreated bio-composite is more brittle as there is no compatibilizer acting on the composite making the bonding of the filler and polymer matrix is brittle and poor interfacial compatibility of the constituent's materials in the composites.

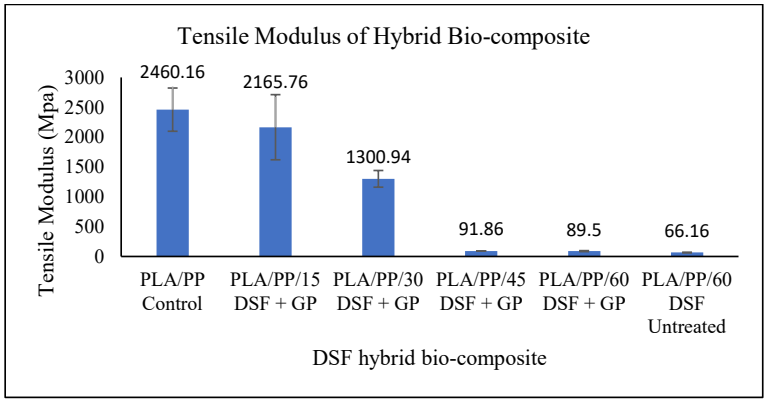

Figure 4: Tensile modulus of PLA/PP with different DSF loading treated and untreated with GP

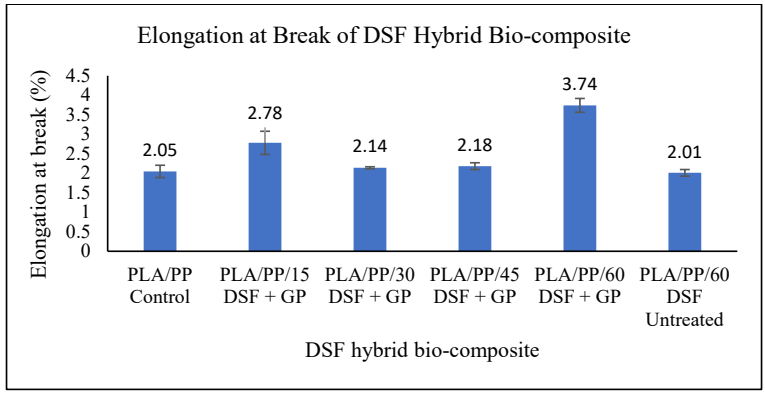

Figure 5: Elongation at break of PLA/PP with different DSF loading treated and untreated with GP

Density Test

Density test was used to indicate the presence of fibers in the composite. Table 3 shows the result of density test for PLA/PP/DSF bio-composite at different filler loading. There were no remarkable increased in density of bio-composite.

Table 3: Density of PLA/PP+DSF bio-composites

DSF filler loading (php)	Density (g/cm^3)
0 (control)	1.18 ± 0.01
15	1.18 ± 0.01
30	1.20 ± 0.01
45	1.24 ± 0.01
60	1.24 ± 0.01

Notes : Data are presented in mean ± SD, n=3

The findings showed that bio-composite sample with 0 (control) and 15 php DSF loading has the lowest density with 1.18 ± 0.01 g/cm^3 each. As filler loading increased to 30 and 45 php, the density of the composites increased from 1.18 ± 0.01 g/cm3 to 1.20 ± 0.01 g/cm^3 and 1.24 ± 0.01 g/cm^3 respectively. This showed an increasing in density of the bio-composites as DSF filler loading was increased. However, after further increasing the DSF filler loading to 60 php DSF, the data remains constant. Higher density related to the higher content of PLA/PP polymer matrix and DSF filler in the bio-composites. The density of the composites is relevant according to JIS A 5905: 2003, stated that the density of bio-composite materials should be between 0.35 g/cm^3 to 1.3 g/cm^3. More filler resulting more content in the composite making the composite become more compact and thus, resulting in high density level.

Swelling Test

Swelling test was used to study the water absorption of the composite. This water absorption was carried out to find out the amount of water can be absorbed by the composites under specified conditions. Figure 6 shows the result of swelling test for PLA/PP+DSF bio-composite at different filler loading.

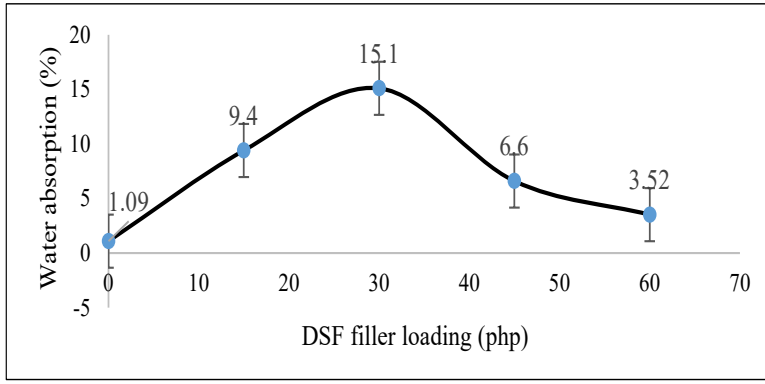

Figure 6: The effect of filler loading on water absorption of PLA/PP/DSF bio-composites

The control bio-composite sample (0 php DSF) has the lowest percentage of water absorption (1.09 ± 0.22 %) compared with others. The findings revealed the percentage of water absorption increased from 1.09 ± 0.22 % to 9.40 ± 1.50 % and 15.10 ± 6.06 % when the PLA/PP reinforced with 15 php and 30 php of DSF treated with GP respectively. However, as the filler continue increasing the percentage of water absorption is decreased. The percentage reduced from 15.10 ± 6.06 % (30 php DSF) to 6.60 ± 0.82 % (45 php DSF) and 3.52 ± 1.17 % (60 php DSF). The composites had significantly increase in water absorption upon immersion in the water until it reaches at a certain period, the composites started to reduce the absorption of water from the surrounding until it reaches saturation point [23]. Saturation point refers to the composite absorb no more water and the water content in the composite remain constant [24]. This is because the DSF have absorbed all the moisture from the water making it turgid. Sahari *et al.* [25], reported that the amount of water absorbed in the composite increases over time until it stabilizes.

CONCLUSION

GP was able to be used as a compatibilizer in enhancing the properties of DSF filled PLA/PP biocomposites. In FTIR, a new peak at about 1595 cm^{-1} was seen in the spectrum PLA/PP/DSF + GP, revealed a good interaction in the composite system which come from the bending of NH_2 vibration

from GP. Incorporating DSF into PLA/PP polymer matrix with addition of GP leading to the increase of tensile strength because of strong adhesion between filler and matrix interfaces, which resulted from the presence of compatibilizer that plays a role in effectively transfer the stress in the composite. GP impart flexibility to the composites, reflected from decreased tensile modulus test results, thus reduces the stiffness of the composites. This finding is supported by improve in elongation at break of the composites as compared to pristine PLA. The presence of DSF in the composite was indicated by increase in the density test results. Furthermore, the swelling test indicates the percentage of water absorption of bio-composite DSF filled PLA/PP risen with the increased filler loading until it reached at saturation point.

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