Effects of Ultrasound Treatment and Impact Modifier on Mechanical Properties of Oil Palm Fiber Reinforced Poly (Lactic) Acid Composites

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Abstract— In this study, composites were prepared from ultrasound treated oil palm empty fruit bunch fiber (EFB) and poly (lactic) acid (PLA) through extrusion and injection moulding. For better compatibility between fiber and matrix, the fiber was delignified through ultrasound treatment and the treated fibers were utilized for composites fabrication. Additionally, different content of impact modifier was added to study possible improvement in impact and energy absorption properties of the composites. Surface morphology and structural changes due to treatment of fibers was observed by using scanning electron microscope (SEM) and Fourier transforms infrared spectroscopy (FTIR). For composite characterization, mechanical testing such as tensile, flexural and impact were carried out. Improvements of up to 23.5%, 33.6%, 16.8% and 19.6% respectively for TS, TM, FS and FM was obtained for treated fiber composites as a result of effective delignification leading to good mechanical interlocking between the de-lignified fiber and PLA matrix. Furthermore, impact strength of poly (lactic) acid was enhanced by incorporating biostrong impact modifier, which resulted to about 38% increase in impact strength of the resulting composite.

Keywords-composites; ultrasound; mechanical interlocking; delignification; impact modifier

1. INTRODUCTION

The evolution of polymer composites came as a result of the search for new low cost materials with high performance, in a bid to meet up the expanding demand from various consumer groups. The preferential desire for polymers as matrix for composites was associated with its potentiality as a great raw material for many structural applications. Synthetic fiber based polymer composites took the early stages of this development because of its high dimensional properties compared to the natural fibers. Not quite long before it started to receive strict competition from natural fibers due to cost implication of the required energy in production and recycling of synthetic fiber based polymer composites, as well as environmental concerns because they were perceived to pose great threat to the environmentNatural fibers quickly came to the forefront based on its low cost, less abrasiveness, low density, worker friendliness and possession of good load bearing capacity. All these coupled with the fact that natural fibers are recyclable and biodegradable among other desirable properties, gave natural fibers its wide range of application in sectors such as construction, manufacturing, packaging, automotive and even in medicine. Natural fibers are derived either from plant, animal or mineral sources, but the commonly used of these three is the plant fiber because of its renewability, wide range availability and shorter period of biodegradability compared to the other groups. Within the plant fiber group, the bast and leaf fibers are the most often used in polymer composites [1, 2]. Few among natural fibers which have been utilized for polymer composites includes but not limited to flax, bamboo, sisal, hemp, ramie, jute, oil palm and wood fibers.

Incorporation of natural fibers in polymer composites however produced composites with poor mechanical strength due to weak interface between the fiber and the polymer matrix. In order to improve the mechanical and tensile properties of natural fiber reinforced polymer composite, there is need for a modification of the fiber surface so as to remove the non-cellulosic components like lignin and hemicellulose which are of less significance to the overall mechanical properties of natural fibers [3-5]. Several conventional surface treatment methods have been applied to manipulate natural fiber surfaces to make then align with the hydrophobic nature of polymer materials. This could be done either by introducing new groups unto the fiber surface or by merely activating the hydroxyl of the cellulose, causing it to rearrange at the molecular level for an improved fiber-matrix surface relationship [3, 4]. Some of the conventional methods includes but are not limited to alkali treatment, [5] isocyanate treatment, [6] acrylation, [7] benzoylation, [8] latex coating, [9] permanganate treatment, [10] acetylation, [11] silane [12] and peroxide treatment [13]. It was noted however that most of these methods have some adverse effects on the environment.

Recently, it has been observed that ultrasound treatment affords numerous advantages for fiber purification. Few among these are: better purification with minimum alkali concentration, minimum exposure time in the reaction chamber, lower treatment temperature and better mechanical strength due to the lower degree of damage. Ultrasound treatment of natural fibers in alkali medium of low concentration can greatly improve the mechanical performance of the resulting natural fiber reinforced composites due to enhancements in interfacial relationships between the fiber and polymer matrix[14, 15].

On the other hand, poly (lactic) acid is a biodegradable thermoplastic which has enjoyed preferred interest over the years as a result of the aesthetic product it produces, good adaptability to varying techniques of processing as well as mechanical strength which is desirable in several applications [16], and comparable to other petroleum base polymers like polyethylene terephthalate (PET), polyethylene (PE), polypropylene (PP) and polystyrene (PS) [17]. The major setback to the use of PLA is its inherent brittleness. This can however be overcome by reinforcing it with short filler fibers or some impact modifiers to improve its toughness [18]. In this study, ultrasonic cavitation were used to dislodge lignin and other unwanted constituents from the oil palm empty fruit bunch fiber, thereby making the cellulose to re arrange at the molecular level. To study the effect of ultrasound, composites were also fabricated using oil palm empty fruit bunch fiber (EFB) and poly (lactic) acid (PLA). For further enhancement in properties, the brittleness of PLA matrix was improved via the incorporation of an impact modifier into the EFB/PLA composite formulations.

2. MATERIALS AND METHODS

A. Materials

The thermoplastic poly (lactic) acid resin used in this study is a Natureworks IngeoTM Biopolymer 3051D grades, supplied by Unic Technology Ltd, China with density of 1.24g/cm³, and melting temperature between 160-170°C. Analytical grade sodium hydroxide and acetic acid were procured from Merck, Germany. Impact modifier (Biomax[®] Strong 120) was collected from Dupont, Switzerland. DupontTM Biomax[®] strong (biostrong) is an ethylene-epoxy based copolymer grafted on to modify PLA. Tetraoxosulphate (VI) acid (96%) was supplied by sigma Aldrich USA. Raw oil palm EFB fibers were collected from the LKPP Corporation Sdn. Bhd., Kuantan, Malaysia.

B. Fiber pre-treatment

The first stage is physical treatment where empty fruit bunch were air dried for 3 days, they were thereafter cut using hammer mill and crusher machine in order to obtain uniform size of empty fruit bunch fiber. In order to remove the impurities which were contained in the raw oil palm fiber, it was washed in water flow such that these materials which are capable of constituting a nuisance can be easily removed. The targeted impurities include sand particles, stones, mud, etc. Then the raw fibers were dried in air for 24 h, after which it was chopped into shorter fiber to get a uniform size of between 2-5 mm. Fiber treatment was carried out with the help of ultrasound both in water and alkali medium as described in previous research work [19].

C. Scanning electron microscopy (SEM)

The morphological changes to the surface of EFB fiber before and after ultrasound treatment were studied using a scanning electron microscope. Test samples were initially air dried to make them moisture free. These samples were placed unto specimen holders having a metal base, with the help of a double sided sticky carbon tape. Prior to SEM observation, samples were coated with gold using a vacuum sputter-coater in order to make test sample conductive.

D. Fourier transforms infrared microscopy (FTIR)

Fourier transforms infrared spectroscopy (FTIR) was done in order to get infrared spectrum for raw and treated EFB fibers. This analysis was carried out with a Fourier transform infrared spectrophotometer (model- THERMO) having an OMNIC software, using the standard KBr method. Appearance and disappearance of peaks with respect to composites were also analysed.

E. Composite fabrication

For composite fabrication, extrusion of EFB fiber and PLA was done using an extruder (model - THERMO SCIENTIFIC PRISM EUROLAB-16) with temperature profile shown in Table 1. Composites were prepared for raw fibers and treated fibers. EFB/PLA composite with 10, 20, 30 and 40 wt% raw fiber were prepared and the optimum fiber content was noted based on the tensile strength result obtained for these formulations. Further composites with treated EFB fibers were prepared based on the optimum fiber content as obtained from raw EFB/PLA composites. Prior to composite fabrication, both fiber and PLA were dried in oven at 60°C for 8h to avoid bubble effects during extrusion. Extruded composites were pelletized to 3 mm length using a fixed length

pelletizer. Tensile test specimen were prepared using injection moulding machine (model- DR BOY 22M) with temperature parameter as included Table 1. The specimen prepared for testing are: Pure PLA (PLA), optimized-content raw EFB/PLA composite (ORFPC), water-medium ultrasound treated EFB/PLA composite (WUFPC) and optimized ultrasound alkaline-medium treated EFB/PLA composite (OUFPC). To improve the impact strength of PLA, an impact modifier (biostrong) was incorporated into the OUFPC formulation, with optimum fiber content (30 wt%) at both 2 and 5 wt% of impact modifier to PLA content. Test specimens prepared for these two formulations were termed 2% IMFPC and 5% IMFPC respectively.

F. Tensile testing of composite

Tensile testing was done according to ASTM D638-08 using a shimadzu universal tensile testing machine (model - AG-1) having a load cell of 5KN, running at a crosshead speed of 10 mm min⁻¹. Average of five tested samples with 65 mm length was recorded for each composite type to obtain tensile strength (TS) and tensile modulus (TM) values.

G. Flexural testing

Flexural testing was conducted according to ASTM D790-97 using a shimadzu universal tensile testing machine (model - AG-1) having a load cell of 5KN, at a speed of 10 mm min⁻¹ having a base support set at 50 mm distance apart for samples with dimension 125 mm x 3.3 mm x 12 mm. Flexural strength (FS) and Flexural modulus (FM) values were obtained as the average of five tested sample for each composite category.

H. Charpy Impact testing

Charpy impact testing was conducted after the EN ISO 179 on rectangular test samples with dimensions 80 mm x 3.5 mm x 10 mm using a Ray-Ran universal pendulum impact tester with an impact velocity of 3.04 m/s and hammer weight of 0.168 kg. A Ray-Ran motorised notching cutter was used to notch samples to a depth of 0.25 mm prior to impact testing. Average of five test results were recorded to obtain the impact strength (IS) for each composite category.

3. RESULTS AND DISCUSSION

A. Surface Morphology of Fiber

The SEM images for raw and treated EFB fiber are put together in Fig. 1. Ultrasound treatment of EFB fiber can be seen to have offered conspicuous changes to the fiber surface. The surface of the untreated fiber was seen to be composed of binding structures which makes the surface even and seemingly non-penetrable (Fig. 1a). However, the treated EFB, (Fig. 1b) shows the opened pore spaces on the fiber surface and also reveals uneven and rougher surface topography as well as fibrillation when compared to the untreated fiber. These changes observed on the surface of the treated fiber is a as a result of fiber deagglomeration brought about by ultrasonic cavitation which produce both mechanical and thermal energy, causing the binding structures like lignin and hemicellulose to be disengaged, thereby leading to fibrillation[20]. Ultrasonic cavitation having dislodged the non-cellulosic substances from the fiber surface causes them to naturally dissolve into the liquid medium and as a consequence opens up the pore spaces on the fiber surface.

EM Conditions Temperature Profile: Zone Temp.		IM Conditions		
		Temperature Profile: Section	Temp.	
Feeding zone: Mixing zone: Metering zone: Die:	110 °C 175-185 °C 190 °C 185 °C	Feeding section: Compression section: Metering section: Nozzle: Mould:	160 °C 175-185 °C 190 °C 180-185 °C 25-30 °C	
Screw speed: Torque:	100-110 rpm 55-60 (%)	Screw speed: Screw position: Injection time: Cooling time:	155 rpm 28.5 mm 0.45 sec 25 sec	

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Figure 1: SEM image of (a) untreated and (b) treated EFB fiber

Table 2: Summary	of observable	peaks for both	untreated and	ultrasound	treated EFB fibers.
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Functional group representation		
-OH bond structure of cellulose		
C-H stretching vibration of cellulose		
C=O stretching		
=CH vibration from aromatic skeletal		
-C=C bending in lignin		
C-H bending in lignin		
-CH ₃ asymmetric and C-H symmetric deformation of		
lignin		
C-O stretching in lignin		
-C-O-C- β-glycosidic linkage in cellulose		

B. Fourier transforms infrared spectroscopy (FTIR) of fiber

Summary of the conspicuous peaks obtained for both untreated and ultrasound treated EFB fibers are put together in Table 2. The broad peaks obtained for treated and untreated EFB fibers are 3400-3200 cm⁻¹ which represents –OH stretching in cellulose, 2932-2917 cm⁻¹ represents –C-H vibrational stretching of methyl and methylene components in cellulose and hemicellulose, 1740-1723 cm⁻¹ which is as a result of C=O stretching of ester and carboxylic components in hemicellulose, 1514 cm⁻¹ is due to –C=C bending in aromatic lignin, 1506 cm⁻¹ reveals the C-H bending in lignin, 1376-1338 cm⁻¹ is due to C-O stretching in lignin and 1243-1183 cm⁻¹ which signifies the –C-O-C- asymmetric stretching of β-glycosidic bond in cellulose chain [21, 22].

In general, the spectrum of both untreated and ultrasound treated EFB fibers are in many ways similar as illustrated in Fig 2. The main difference is the reduction in intensity of peaks after treatment. Also the disappearance of band around 1730 cm⁻¹, and weakening of 1645 cm⁻¹, 1423 cm⁻¹ and 1376-1338 cm⁻¹ after ultrasound treatment which could be as a result of the removal of non-cellulosic components like lignin, waxes, pectin and hemicelluloses from the EFB fiber subject to ultrasound treatment[23].



C. Mechanical properties of composites

The variation of tensile strength (TS), tensile modulus (TM), and flexural strength (FS), flexural modulus (FM), with fiber content of untreated EFB/PLA composite is shown in Fig. 3 a and b. The highest mechanical properties were obtained from 30 wt% EFB fiber reinforced PLA composite. This was regarded as the optimum fiber content and other composite samples were prepared based on this (30 wt%) fiber content. Samples which were prepared for analysis includes pure poly (lactic) acid (PLA), optimum content (30 wt%) raw fiber reinforced PLA composite (ORFPC), water medium ultrasound treatment fiber reinforced PLA composite (WUFPC), optimized ultrasound (2% NaOH, 90°C and 100 mins) treated fiber reinforced PLA composite (OUFPC), and impact modified EFB/PLA composites at 2 wt% (2% IMFPC) and 5 wt% (5% IMFPC) respectively. Fig. 4 shows the TS values of 55.90, 59.24, 65.08, 73.14, 59.54 and 58.68 MPa; TM values of 1195, 1571, 1825, 2099, 1541 and 1435 MPa respectively for PLA, ORFPC, WUFPC, OUFPC, 2% IMFPC and 5% IMFPC. Moreover, FS values of 83.20, 99.18, 110, 115.88, 105.06 and 89.32 MPa; FM values of 936, 1469, 1477, 1758, 1721 and 1588 MPa are plotted for PLA, ORFPC, WUFPC, OUFPC, 2% IMFPC respectively Fig. 4. The highest performance from TS, TM, FS and FM was obtained from OUFPC, with an increase in TS, TM, FS and FM of 23.5%, 33.6%, 16.8% and 19.6% from ORFPC to OUFPC respectively.

The observed enhancement in properties from pure PLA to ORFPC might not necessarily be associated with surface morphology of the EFB fiber. It is rather as a consequence of the highly desirable TS and TM of individual EFB fibers [24]. However, the improved performance as obtained from the OUFPC samples could be attributed to effective fiber delignification leading to better interfacial adhesion between the fiber and PLA matrix which invariably brought about an effective stress transfer from the PLA to the EFB fiber, as also reported elsewhere [25]. It should be noted however that above the 30 wt% optimum fiber content, the mechanical properties start to decrease due excess fiber within the composite which led to fiber-agglomeration caused by fiber-fiber interactions when they come too close to each other within the composite [23, 26].



Figure 4: Effect of different treatment conditions on TS, TM, FS and FM of EFB/PLA composites

In another vein, at the incorporation of biostrong impact modifier, there were observable decreases in TS, TM, FS and FM values obtained for OUFPC composites. These decreases became larger as the impact modifier content increased from 2 wt% to 5 wt%. In any case, the values obtained from impact modified composites at both 2 and 5 wt% still show some degree of improvements in mechanical properties above pure PLA which could be due to the initial inherent brittleness of PLA. The increase in TS, TM, FS and FM between pure PLA and impact modified composites are 6.5%, 30%, 26.5% and 83.9% respectively for 2 wt% impact modifier content and 5%, 20.1%, 6.3% and 69.7% respectively for 5 wt% impact modifier content of those properties could be associated with formation of film in between two successive layers of PLA which tends to increase yield strength [27]. At 5 wt% impact modifier content, there might be an excessive formation of rubbery films, leading to developed secondary flexibility phase. Thus, an increase of local stress concentration occurred which leads to the development of triaxial stress in the impact modified particle films leading to agglomeration, and thereby reducing the tensile properties [27].

Impact strength of composites is showed in Fig. 5. It was observed that due to addition of fiber, impact strength reduced (Fig. 5a) for the composites with 10 wt% and 20 wt% of fiber, compared to control PLA. This initial decrease could be due to development of local stress concentration within the matrix and subsequently reduced yield strength. Therefore, the composites with 10 wt% or 20wt% became brittle. Furthermore, the fiber content was not sufficient enough to absorb energy necessary for toughening of the PLA [28]. The IS was found to increase due to further addition of fiber (30 wt%) and also found a slight increment compared to control PLA. This is owing to increased effective stress transfer ability within the matrix, which was supported by sufficient amount of fiber, necessary to absorb maximum energy. This therefore reduced the shock effect on the PLA matrix, and increased energy absorption [29]. Above 30 wt% fiber loading the IS however started to decrease again. This could be as a result of weak interfacial interaction due to less wettability of the excess fiber by the matrix, leading to the formation of micro cracks at impact points due to reduction of surface area available for redirecting cracks [29].

For composites based on treated EFB fibers Fig. 5b, the IS values are 14.1, 14.38, 14.72, 16.23. 19.48 and 16.71 for PLA, ORFPC, WUFPC, OUFPC, 2% IMFPC and 5% IMFPC respectively. The highest values were obtained for the impact modified composites with the largest belonging to 2% IMFPC. The increase in IS from PLA to 2% IMFPC and 5% IMFPC is 38.2% and 18.5% respectively. This notable increase could be as a result of transfer films formation on the surface of the composite because of the rubbery nature of biostrong impact modifier [27], as well as increased homogeneity within the matrix as a result of the rubbery nature of the impact modifier [30, 31].

4. CONCLUSION

Ultrasound treatment of oil palm empty fruit bunch fibers produced fibers with greatly reduced non-cellulosic components. SEM image of treated fibers confirmed the effectiveness of ultrasound treatment, by showing cleansed surfaces as well as opened pore spaces available for interlocking with the matrix. Composites fabricated with treated EFB and PLA showed great enhancement in properties with up to 23.5%, 33.6%, 16.8% and 19.6% respectively for TS, TM, FS and FM, compared to untreated fiber composites. Incorporation of biostrong impact modifier with as low as 2 wt% impact modifier content, was able to improve the impact properties of PLA with about 38 % increase in impact strength of the resulting composite.

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Figure 5: Variation of Impact strength with (a) EFB fiber content and (b) EFB/PLA composites at different treatment conditions

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