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Impact Modified Oil Palm Empty Fruit Bunch Fiber/Poly(Lactic) Acid Composite

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***Abstract*—**In this study, composites were fabricated from oil palm empty fruit bunch fiber and poly(lactic) acid by extrusion followed by injection moulding. Surface of the fiber was pre-treated by ultrasound in an alkali medium and treatment efficiency was investigated by scanning electron microscopy (SEM) analysis and Fourier transforms infrared spectrometer (FTIR). Effect of fiber treatment on composite was characterized by tensile strength (TS), tensile modulus (TM) and impact strength (IS). Furthermore, biostrong impact modifier was incorporated into the treated fiber composite to improve its impact properties. Mechanical testing showed an improvement of up to 23.5% and 33.6% respectively for TS and TM of treated fiber composite above untreated fiber composite. On the other hand incorporation of impact modifier led to enhancement of about 20% above the initial IS of the treated fiber composite.

***Keywords*—**Fiber treatment, impact modifier, natural fibers, ultrasound.

I. Introduction

THE increasing trend of awareness about environment sustainability, and especially the regulation and legislations towards a greener environment are compelling researchers' especially material engineers to search out more eco-friendly materials to be incorporated into their products. This is because applications that made use of synthetic and man-made fibers like glass fibers had often led to environmental pollution, and especially respiratory tract related diseases for workers. Also production and recycling of materials based on synthetic fibers normally demand a great deal of energy [1], [2].

Natural fibers are fast becoming the desired alternative in polymer composite materials due to its many advantaged like biodegradability, renewability, recyclability, reduced abrasiveness to production equipment, reduced exposure of workers to health hazards, great load bearing potential, low cost and low weight. All of these contribute to the widespread suitability of natural fibers in applications such as aircraft, packaging, construction, medicine, automotive parts, furniture and also in footwear. Several investigations had been carried out on the potentiality of natural fibers as reinforcing material for composites [3]-[7]. From most of these researches, it was found that natural fibers do possess desirable stiffness and strength. However, natural fiber reinforced polymer composites produced low tensile properties due to poor adhesion between the surfaces of the hydrophilic fibers and the hydrophobic polymer matrices. To overcome this challenge, surface of the fibers need to be modified to enhance its adhesion with the polymer matrix [7], [8]. Some of the natural fibers which had been surface modified and effectively used as reinforcement in polymer composites include oil palm empty fruit bunch fiber, coir, flax, ramie, jute, bamboo and kenaf fibers.

On the other hand, some of the eco-friendly polymers available for renewable and biodegradable composite materials include poly(lactic) acid (PLA), lignin and starch based plastics, poly hydroxyl butyrates as well as cellulose esters [1]. PLA is of particular interest due to its suitability and adaptability to be processed with several production techniques like extrusion, blow moulding, injection moulding and thermoforming. PLA is versatile thermoplastic polyester often obtained from annually renewable materials from agricultural origin, which are fermented into lactic acid. PLA being a fully biodegradable matrix also possesses high modulus and strength as well as highly stiff and brittle. It is therefore very important to incorporate impact modifiers and plasticizers into PLA composites to improve the impact and toughening properties of the composite [1].

In this study, composites were fabricated from oil palm empty fruit bunch (OPEFB) fiber and poly(lactic) acid (PLA) matrix. Surface of the fibers were modified through pre-treatment with ultrasound in NaOH solution. Furthermore, PLA was toughened by biostrong impact modifier to improve its impact properties. Surface morphology of the fiber with respect to treatment was investigated by scanning electron microscopy (SEM) whereas the functional group analysis was investigated by Fourier transforms infrared spectroscopy (FTIR). On the other hand, mechanical properties of the composite was studied by tensile and impact testing.

II. Materials and Methods

A. Materials

The reinforcing filler used for this study is oil palm empty fruit bunch fiber which was collected from LKPP Corporation Sdn. Bhd., Kuantan, Malaysia. It was obtained as raw wastes. On the other hand, the polymer matrix used is poly(lactic) acid which was supplied by Unic Technology Ltd, China. The supplied grade is a 3051D Natureworks Ingeo^{TM} thermoplastic

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poly(lactic) acid Biopolymer. It has a melting temperature of 160-170^{0}C, density of 1.24 g/cm^{3}and melt flow index of about 30-40g / 10min. (190^{0}C/2.16kg). Acetic acid and Sodium hydroxide of analytical grades were purchased from Merck, Germany. The impact modifier used is an ethylene-epoxy based Dupoint^{TM} Biomax® strong 120 copolymer grade which was collected from Dupoint, Switzerland.

B. Methods

1. Fiber Treatment

Treatment of oil palm empty fruit bunch (OPEFB) fibers was done by initially washing the raw fiber in ordinary water to remove the accompanying substances which came along with the fiber from the oil palm factory. After washing, the fibers were dried in air for about 3 days after which it was chopped to shorter length with a plastic crusher machine. Crushed fibers were sieved by a mechanical sieve shaker to length of between 2-5 mm, as well as to eliminate ash. For chemical fiber surface modification, OPEFB fibers were weighed and put into large beakers containing 2% (w/v) NaOH solution, maintaining 1:20 (w/v) ratio for fiber to solution and the beaker as well as its content was then placed in an ultrasound bath (CREST-ultrasonics). Fiber treatment in ultrasound bath was continued for about 100 min at 90^{0}C and ultrasound power of 9 Watts. Optimization of ultrasound bath working condition as well as NaOH concentration was initially carried out as described in literature [8]. After ultrasound treatment, treated fibers were washed in distilled water continuously to remove excess alkali. Few drops of very dilute acetic acid were added and washing was continued until wash water no longer indicated a sign of alkalinity i.e., pH of 7 was reached. At this stage, the fibers were dried at room temperature for 24 hr and later dried in hot air oven set at 70^{0}C for 8 hr.

2. Composite Production

For preparation of composites from untreated and treated oil palm empty fruit bunch fiber and PLA, compounding was carried out using a THERMO SCIENTIFIC PRISM EUROLAB-16 twin screw extruder. This was followed by sample preparation by DR BOY 22M injection moulding machine. Initially, untreated OPEFB fibers were compounded with PLA at varying fiber content (0-40 wt%) to obtain the optimum fiber content. Tensile and impact testing showed that 30 wt% fiber content was the optimum content. For comparison purpose, other composite categories were prepared based on treated OPEFB fiber, maintaining the 30 wt% fiber content, and impact modifier was incorporated where necessary. Optimization of impact modifier content was initially carried out and 2 wt% being the optimum content, it was incorporated into the treated fiber composite. Samples which were prepared for testing are pure PLA, raw fiber composite (RFC), treated fiber composite (TFC) and impact modified treated fiber composite (IMTFC). Summary of the different composite categories prepared and their code names are put together in Table I.

TABLE I
MIXING FORMULATIONS FOR COMPOUNDING OF PLA AND OIL PALM EMPTY FRUIT BUNCH FIBER WITH TWIN SCREW EXTRUDER

CODE	PLA (wt%)	OPEFB (wt%)	Biostrong (wt%)
0	100	0	-
10	90	10	-
20	80	20	-
30	70	30	-
40	60	40	-
Pure PLA	100	0	-
RFC	70	30	-
TFC	70	30	-
IMTFC	68	30	2

3. Scanning Electron Microscope (SEM) Analysis of Fiber

SEM analysis was used to observe the surface morphology of OPEFB fibers before and after treatment, using, a ZEISS, EVO 50 scanning electron microscope. To make test sample conductive, they were initially sputter coated with gold before SEM observation.

4. Fourier Transforms Infrared Spectroscopy (FTIR) Analysis of Fiber

FTIR was done to collect infrared spectra of untreated and treated OPEFB fibers, and also to analyze the effect of fiber treatment on the functional groups present in the fiber. Spectra for each fiber type was collected with the help of an OMNIC software connected to the FTIR spectrophotometer (THERMO), using the standard KBr technique.

5. Mechanical Testing of Composites

Tensile testing of composites was carried out using an AG-1 shimadzu universal tensile testing machine, fitted with a 5 KN load cell. Test samples were prepared in accordance with ASTM D638-08 - Standard. During testing, the tensile testing was carried out on 65 mm gauge length samples at a 10 mm min^{-1} crosshead speed. Six samples were tested for each composite category and replicate of five samples were presented as average of tested samples to obtain tensile strength (TS) and tensile modulus (TM).

Impact testing of composites was done in accordance with EN ISO Plastics – Determination of Charpy impact strength. The dimension of test samples were 55 x 3.3 x 10 mm and testing was carried out using a Ray-Ran universal pendulum impact tester on sample which were initially notched to a depth of 1.5 mm by a motorized notch cutter. Impact velocity and weight of hammer are 3.04 m/s and 0.168 kg respectively. Five replicate samples out of six tested samples were presented as average of tested samples to obtain the impact strength (IS).

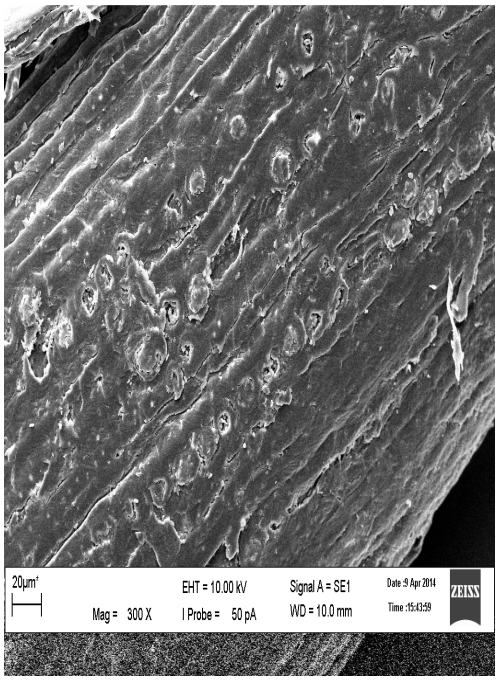
III. RESULTS AND DISCUSSION

A. Surface Morphology Analysis of Fiber (SEM)

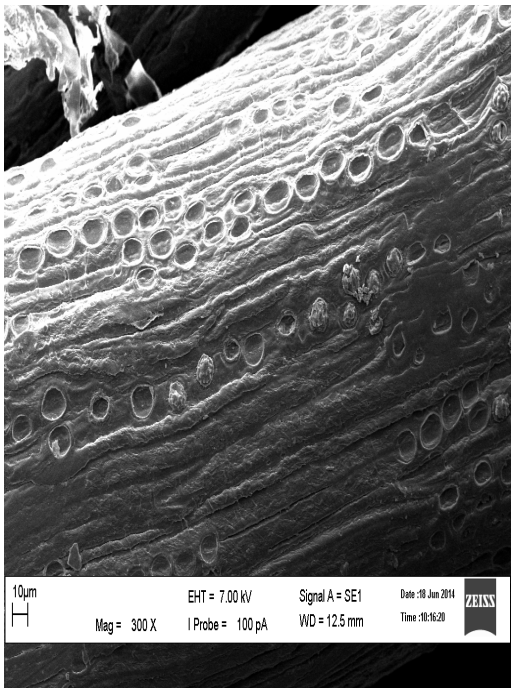
Figs. 1 (a) and (b) represent the SEM micrographs of untreated and treated OPEFB fibers. It can be seen from the image of the untreated fiber in Fig. 1 (a) that the pore spaces on the fibers surface are blocked by certain sizing and cementing substances. These substances could include lignin,

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hemicellulose, waxes, silica and other dirt substances. Treatment of fibers had been reported to dislodge as much of this substances as possible and at the same time activate the hydroxyl groups of the fiber cellulose [9]-[11].



(a)



(b)

Fig. 1 SEM micrographs of (a) raw and (b) treated OPEFB fiber

From Fig. 1 (b), surface morphology of the treated fiber reveals several opened pore spaces. This increased porosity observed for the treated fiber indicates an effective removal of sizing materials from the fiber through the applied treatment method. This also indicates changes to the structural network in the fiber perhaps through disruption of the hydrogen bonding and rearrangement of cellulose components through formation of new hydrogen bonds *via* intramolecular hydrogen bonding [12]. Rearrangement of cellulose, exposure of cellulosic hydroxyl groups as well as opening of pores on the fiber surface have all been reported to be great perquisites for efficient physic-chemical interlocking between natural fibers and the polymer matrix counterparts [8], [13]. Therefore it is expected that incorporation of the treated fiber in Fig. 1 (b) should produce composites with better mechanical properties compared to untreated fiber composite.

B. Functional Group Analysis of Fiber (FTIR)

The FTIR spectra for raw and treated OPEFB fibers are illustrated in Fig. 2. The broad band at 3500-3200 cm^{-1} is a characteristic stretching from vibrations of the bonded hydroxyl groups of carbohydrate (hemicellulose and cellulose) and lignin of OPEFB fiber [14]. It can be seen that the intensity of this band increased for treated OPEFB fibers as well as shifted towards higher wavelength. This indicates the presence of more intramolecular hydrogen bonding in the cellulose structure of the OPEFB fibers perhaps due to the exposure of more hydroxyl groups on the fiber surface after treatment with ultrasound in alkali medium [8], [12]. The IR band around 2900 cm^{-1} represents stretching vibrations from the methyl and methylene groups of the OPEFB fiber components. This band was found to split after treatment of fiber indicating structural changes to the fiber though removal of certain proportions of lignin and hemicellulose from the fiber by ultrasound alkali treatment. The peak at 1749 cm^{-1} signifies the carbonyl group stretching of the acetyl and carboxylic acid groups of lignin and hemicellulose components of OPEFB fiber. The conspicuous disappearance of this peak from the spectrum for treated OPEFB fiber confirms the effectiveness of the applied treatment at removing large proportion of lignin and hemicellulose from the fiber. Gradual reduction in intensity of the peak at this wavelength with increasing fiber treatment intensity had also been reported for the case of empty fruit bunch in literature [12]. Other notable peaks are the peaks at 1644 cm^{-1} and 1514 cm^{-1} which represents aromatic skeletal =CH vibrations and –C=C bending in fiber lignin respectively. Asymmetric $–CH_3$ and symmetric C–H deformation of fiber lignin are represented by the peak at 1422 cm^{-1} whereas the peak at 1318 cm^{-1} represents C=H stretching of methylene, methoxy and methyl groups of lignin. Peaks at 1241 cm^{-1} and 1034 cm^{-1} represents –C-O-C- stretching of the β-glycosidic bond of cellulose chain and C–H in-plane deformation of lignin respectively [8]. In general, the noticeable differences to the fiber spectrum after treatment either through shifting of peak to lower or higher wavelength, disappearance of certain peaks, or perhaps the reduction or increase in intensity of absorbance peak at certain points, all suggest structural changes to the OPEFB fiber. These can be associated with the effectiveness of the treatment method which might have modified the fiber

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structural build up. Structural changes to fibers after treatment were also reported in other literature [7], [13], [15]. Summary of the noticeable peaks and their band representation is included in Table II.

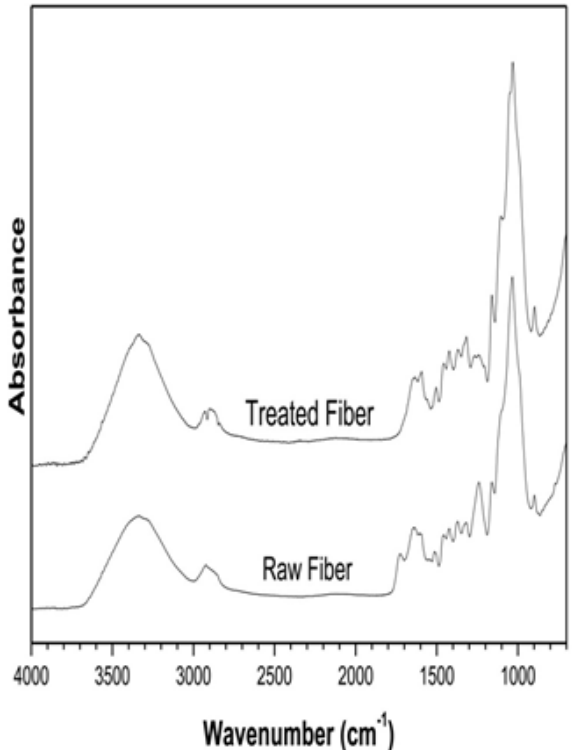


Fig. 2 FTIR spectra of raw and treated OPEFB fiber

TABLE II
SUMMARY OF FTIR SPECTRA FOR UNTREATED AND TREATED OOPEFB FIBERS

Position of bands (cm^{-1})		Functional group representation
Raw	Treated	
3335	3385	-OH bond structure of cellulose
2924	2922	C-H stretching vibration of cellulose
1749	-	C=O stretching
1644	1595	=CH vibration from aromatic skeletal
1514	1509	-C=C bending in lignin
1422	1424	$-CH_3$ asymmetric and C-H symmetric deformation
1318	1317	C=H stretching in methyl, methylene and methoxy groups
1241	1240	-C-O-C- β-glycosidic linkage in cellulose
1034	1031	Aromatic C-H in plane deformation
898	986	Si-O asymmetric stretching

C. Tensile and Impact Properties of Composites

Fig. 3 illustrates the effect of fiber content on tensile strength (TS) and tensile modulus (TM) of OPEFB/PLA composite. For both TS and TM, the trend shows an increase with increasing fiber content up to 30 wt% fiber content after which both of TS and TM decreased. The recorded increase in TS and TM at 30 wt% fiber content above pure PLA is 5% and 31% respectively, which is a reasonable improvement. These observed increases could be associated with the desirable strength of the individual OPEFB fibers or perhaps due to even distribution of fibers within the matrix at 30 wt% [7], [8]. Below 30 wt% there might be localization effect of fiber on the matrix leading ineffectiveness in stress transfer between fiber and matrix. This could be the reason for the less TS and TM values at 10% and 20% compared to 30% fiber content [8]. On the other hand, above 30%, the reduced TS and TM obtained could be due to uneven distribution of fiber within the composite. It had been pointed out that when composite materials are overcrowded with excess fiber, the fibers come too close to each other leading to fiber agglomeration. Agglomeration within the composite would then lead to excessive break in stress transfer between matrix and fiber and the consequence of this would be a reduction in tensile properties [8], [16], [17].

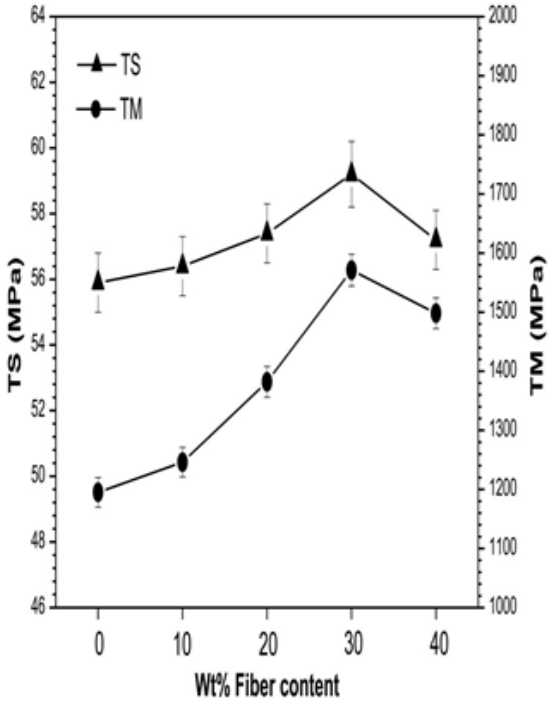


Fig. 3 Variation of TS and TM with wt% fiber content of OPEFB fiber and PLA composite

The variation of impact strength with fiber content of untreated OPEFB fiber and PLA composite is shown in Fig. 4. The trend can be seen to be an initial drop of impact strength of pure PLA after the incorporation of OPEFB fiber. However, an in increase of impact strength was noticed as fiber content increased as also reported elsewhere [1], [3]. The maximum IS value of 14.4 Jm^{-1} which was obtained for OPEFB/PLA composite, was reached when the fiber content is 30 wt%. This figure is about 2% improvements above pure PLA (14.1 Jm^{-1}). In a similar research, the maximum IS was also attained for cordenka reinforced poly lactic acid when the fiber content is 30 wt% [3]. This suggest that at 30 wt% fiber content, the fibers are sufficient to absorb maximum energy and reduce the effect of shock on the PLA matrix, and thereby toughening the composite through increased energy absorption [18]. Above 30 wt% fiber content, the IS was found to reduce to 13.5 Jm^{-1} at 40 wt% fiber content. The initial decrease of IS for 10% and

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20% could be associated with the low population of fiber within the composite, leading to the formation of concentrated sites for local stress as well as reduced yield [18]. It could as well be that the fiber content was too low to absorb enough energy to toughen the PLA matrix [19]. On the other hand above 30 wt% fiber content, the decrease of IS could be as a result of over population of fiber within the matrix which might have led to fiber agglomeration. Excessive loading of the matrix with fiber would lead to poor surface interaction due to less wettability of the fibers by the matrix and invariable reducing the surface area available for redirecting cracks. Moreover, there is more possibility for formation of micro cracks at impact points when the fiber content exceeds an optimum value as reported in literature [18], [20].

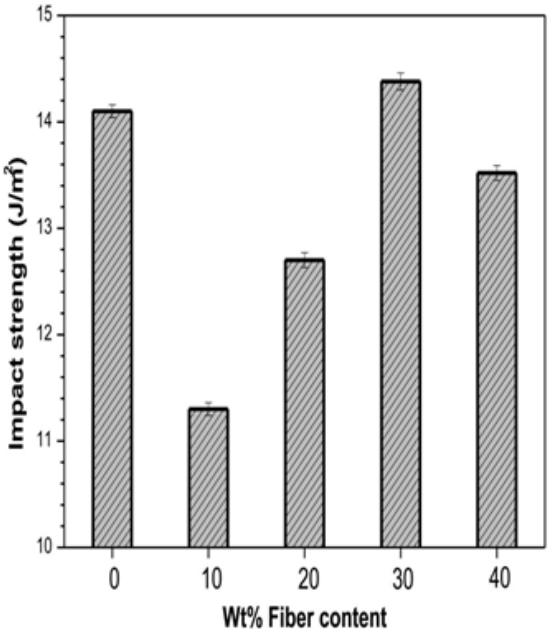


Fig. 4 Variation of IS with wt% fiber content of OPEFB fiber and PLA composite

Figs. 5 and 6 represent the comparison of TS, TM and IS for pure PLA, raw OPEFB fiber composite (RFC), treated OPEFB fiber composite (TFC) and impact modified treated OPEFB fiber composite (IMTFC). Clearly from Fig. 5, it can be seen that the maximum values for TS and TM were obtained from treated fiber composite (TFC). For TFC, the TS and TM values are 73.1 MPa and 2099 MPa respectively. This is an improvement of about 23.5%, and 33.6% respectively over RFC which has TS and TM values of 59.2 MPa and 1571 MPa respectively. These figures are reasonable enough for enhancing the selection and properties materials. The observed increases in these properties could be as a result of improved surface interaction between the treated OPEFB fibers and PLA matrix [8]. Treatment of OPEFB fibers led to opening of pore spaces on the fiber surface as seen in the SEM image in Fig. 1 (b), perhaps due to effective removal of non-cellulosic components from the fiber surface. Removal of non-cellulosic components like lignin and hemicellulose through fiber treatment would lead to structural changes to the hydrogen bond within the fiber, making the cellulose components to rearrange at molecular level through intramolecular hydrogen bonding as illustrated in literature [12]. This would further lead to the exposure of more hydroxyl groups on the fiber surface that can interlock with the polar groups of PLA through hydrogen bonding, bringing about an improved mechanical interlocking and invariably enhanced mechanical properties [21]. However, incorporation of biostrong impact modifier into the formulation of the treated fiber composite, led to reduction of both TS and TM. The rubbery nature of biostrong impact modifier might have led to the formation of rubbery films, bringing about the creation of secondary phase of flexibility within the composite. Furthermore, this could lead to generation of triaxial stress in the impact modified composite (IMTFC), as well as a reduction in the effectiveness of stress transfer between the OPEFB fibers and PLA matrix leading to the observed decrease in TS and TM [18].

On the other hand from Fig. 6, it can be seen that the maximum IS value was obtained from impact modified treated fiber composite (IMTFC), followed by treated fiber composite. This suggests that incorporation of biostrong impact modifier into OPEFB/PLA composite led to increase in the toughness properties of the composite. The increase of IS from 16.23 Jm^{-1} (TFC) to 19.48 Jm^{-1} (IMTFC) indicates that incorporating biostrong led increase in the yield strength of PLA perhaps through the creation of stress films between successive layers of PLA as reported elsewhere [1], [18]. It could as well be as a result of increased homogeneity within the composite which might have led to increased toughness properties as reported elsewhere [22].

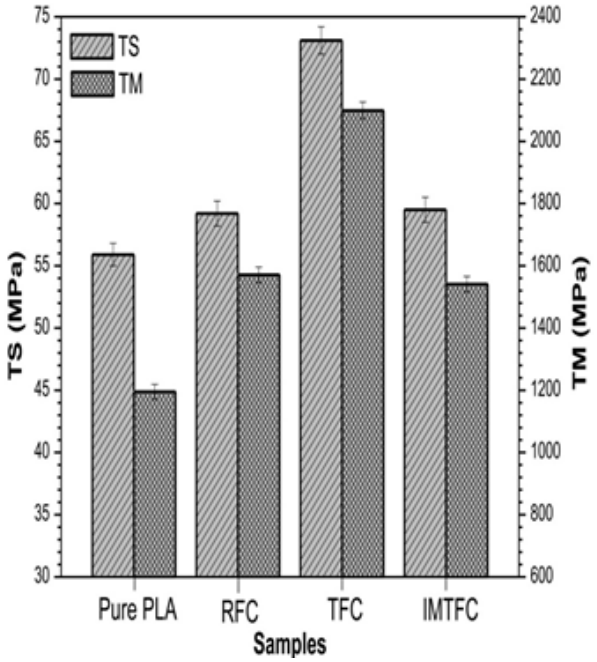


Fig. 5 Effect of fiber treatment and impact modifier on TS and TM of pure PLA, raw fiber composite (RFC), treated fiber composite (TFC) and impact modified treated fiber composite (IMTFC)

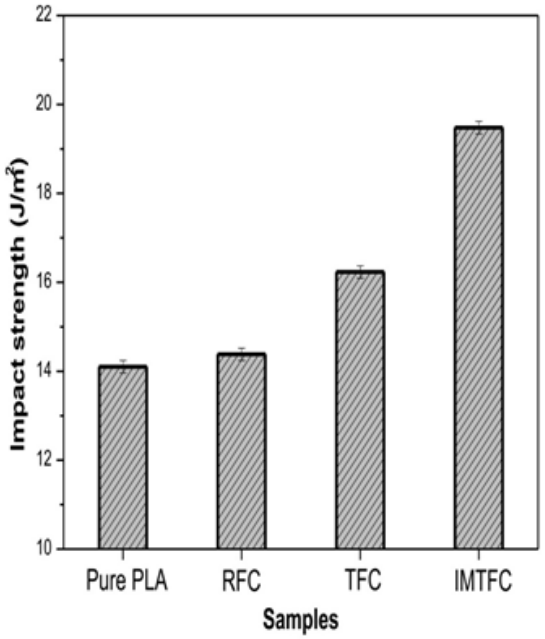


Fig. 6 Effect of fiber treatment and impact modifier on IS of pure PLA, raw fiber composite (RFC), treated fiber composite (TFC) and impact modified treated fiber composite (IMTFC)

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