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INFLUENCE PRE-TREATMENT ON THE PROPERTIES OF LIGNOCELLULOSE BASED BIOCOMPOSITE

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ABSTRACT

Natural fibers are gaining progressive account as a renewable, environmentally acceptable, and biodegradable reinforcing fibre which combine acceptable mechanical properties with a low density. The utilization of biomass (lignocelluloses) for the processing of novel composites has attracted growing interest because of its eco-friendly and renewable nature. The major challenge for natural fibre composites is their inherent poor adhesion between fibre and the matrix. In this study the lignocelluloses were treated with alkaline peroxide to remove lignin. Treatment time, temperature and concentration were optimized on the basis of removal of noncellulosic compound. The non-cellulose and celluloses fibres were blended in different ratios up to 50 wt% with polypropylene (PP) matrix using Twin-screw extruder. Samples were prepared by using injection molding machine and tested using tensile, flexural and impact tester. It was found that the mechanical properties were improved by the alkaline peroxide treatment of fibre. Also, elimination of lignin contents of fibre strongly influenced on the properties of polypropylene composite which was evaluated by using Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectrophotometry (FTIR).

Keywords: Lignocellulose, Alkaline peroxide, Oil palm empty fruit bunch fibre, Composites.

INTRODUCTION

Agricultural residues such as empty fruit bunch, straw and sugar-cane bagasse represent an abundant, inexpensive, and readily available source of renewable lignocelluloses materials. Tailoring new composites within a perspective of eco-design or sustainable development is a philosophy that is applied to more and more materials. Ecological concerns have resulted in a resumed interest in renewable resources-based products. It is the reason why material components such as natural fibers, biodegradable polymers can be considered as environmentally safe – alternative (Digabel and Averous, 2006; Rozman et al., 2003). Indeed enormous interest in the development of new composite materials filled with natural fibers has been shown by important industries such as the automotive, construction or packaging industry. Composites have encompassed almost all material domains. All synthetic polymers (thermoplastics, thermosets, and elastomers) can be used as matrices. As fillers, besides inorganic powders, extensive use has been made of inorganic man-made fibers such as glass, silicium carbide,or organic fibers like carbon and aramid, in the form of individual fibers (chopped or continuous), mats, or textiles (Habibi et al., 2008; Bengsston et al., 2007). Compared to inorganic fibers, natural fibers present some well-known advantages such as lower density and lower price. They are less abrasive to the processing equipment, harmless, biodegradable, renewable, and their mechanical properties can be comparable to those of inorganic fibers (Spoljaric et al., 2009; Ratnam et al., 2008; Yang et al., 2006; Georgopoulos et al., 2005; Rozman et al., 2003).

While, based on Zain et al. (1994), almost more than 30 million metric ton of entire oil palm production is considered by product or agriculture material and about 8 million metric ton is empty fruit bunch materials. It is also reported that estimated production of heat, electricity and fuels could be increased by 90 million tonnes of oil-equivalent corresponding to 14 million hectares of land by 2010 from 40 million tonnes in 1995 by the agro based renewable materials. A viable solution is to combine the petroleum and bio resources (plant and agriculture stock) to produce a useful product having the requisite cost-performance properties for real world applications (Khalid et al., 2008; Ratnam et al., 2007; Rozman et al., 2001).

Many studies carried out based on composites containing natural lignocellulosic fibers from agro industrial or agricultural residues. It depends on origin, natural fibers such as Empty fruit bunch (EFB) in fruit fibers group is a byproduct from the oil palm industry. It consists of 65% cellulose and 19% lignin (Ratnam et al., 2008; Rozman et al., 2001). Cellulose is the main component of natural fibers, and Cellulose is organised into fibrils, which are surrounded by a matrix of lignin and hemicelluloses, which contains three hydroxyl (OH) groups. These hydroxyl groups form hydrogen bonds inside the macromolecule itself (interamolecular) and between other cellulose macromolecule (intermolecular). Therefore, all natural fibers are hydrophilic in nature. However, the main disadvantage of natural fibers/plastic composites is the poor compatibility between the mainly hydrophobic polymer-matrix and the hydrophilic fibers. This leads to the formation of a weak interface, which results in poor mechanical properties; where the stress transfer at the interface between two different phases is determined by the degree of adhesion. A strong adhesion at the interfaces is needed for an effective transfer of stress and load distribution throughout the interface. Thus, enhanced interfacial adhesion for composites containing natural fibres can be achieved either by fibre and matrix modification with chemical/physical treatments or by use of interfacial additives such as delignition, bleaching and chlorination (Habibi et al., 2008; Ratnam et al., 2008; Pracella et al., 2006). Only a few researches were carried out to study potential of treated EFB fibers filled in polypropylene composite (Khalid et al., 2008).

Due to the utilization of chlorine and chlorine derivatives in conventional bleaching processes, large amounts of chlorinated organic compounds are generated, which presents serious environmental problems. Therefore, the use of chlorine-free bleaching processes has become increasingly important. Hydrogen peroxide is the most widely used bleaching agent in the lignin-retaining bleaching of mechanical, thermomechanical, chemi-mechanical, and semi-chemical pulps. It has been generally accepted that the bleaching action of hydrogen peroxide is attributable to the hydroperoxide anion (HOO^-), which is dominantly present under alkaline conditions and responsible for the elimination of chromophoric groups from lignin.

The spectroscopic studies showed that the brightening effect of hydrogen peroxide is attributed to its ability to react with various colored carbonyl structures present in lignin including quinones and cinnamaldehyde structures. Studies with model compounds indicated that the addition of hydroperoxide ions (HOO-) to quinines and non-conjugated carbonyl groups and double bonds leads to the formation of carboxyl fragments (Sun et al., 2002; Mitra et al., 1998). On the other hand, hydrogen peroxide is susceptible to decomposition, particularly in the presence of some heavy metal ions, into more reacting species such as hydroxyl radicals (HO-) and oxygen, which may lead to lignin degradation. A few studies have been performed on delignication of lignocelluloses materials with alkaline hydrogen peroxide (Ratnam et al., 2008; Magnus et al., 2007; Georgepoulos et al., 2005; Sun et al., 2002; Ray et al., 2001). This study was to investigate the influence of pre-treatment of empty fruit bunch using alkaline peroxide as alternative of chlorination method which environmental friendly as well as to study the influence on mechanical properties of polypropylene composite materials.

MATERIALS AND METHODS

Materials

Empty fruit bunch fibers (EFBF) was collected from Felda Palm Industries Berhad and homopolymer polypropylene (PP) (Melt flow Index 12g/10min, density 0.91g/cm^3) was supplied by MTBE (M) Sdn Bhd. No coupling agent was used in this study. The noncelluloses and celluloses fibers were derived from EFBF at lab scale by alkaline peroxide treatment method. All chemical such as sodium hydroxide (NaOH) and Hydrogen peroxide (H_2O_2) and acetic acid used were of analytical or reagent grade. The proximate chemical composition of EFBF is given in table 1 as below:

Table 1: Proximate chemical composition of EFBF

Composition	Weight (wt %)
Lignin	25
α-cellulose	17.2
Hemicellulose	18.0
Holocellulose	50.5
Ashes	3.5

Pre-treatment of Empty fruit bunch (EFB) –Alkaline Peroxide

In this study, there are two stage of treatment in order to prepare lignocelluloses fibers. Firstly, physical treatment where collection residue waste of empty fruit bunch which already through hammer mill process. Dried it in the oven for overnight, then was cut using crusher machine, sieved it to get uniform sizes and remove ashes. After that, EFB fibers was soaked with hot distilled water at temperature around 65ºC- 80ºC for at least 12 hour to eliminate impurities and large particles. Secondly, EFB fibers was treated with alkaline peroxide by placing 20g in 500ml Erlenmeyer flask of distilled water containing sodium hydroxide (NaOH).These mercerization (delignificated fibers) process were soaked in 15%(w/v) NaOH maintained in water bath at temperature 75ºC, speed 40rpm about 3hr to remove lignins content. Next, fibers was washed with distilled for a few time, treated with distilled water containing 2% H_2O_2 in water bath at temperature 45°C, speed 40 rpm for 8 hour. Continued reaction will remove more lignin content and active the OH group of the cellulose. It was indicated 10-30% (w/v) sodium hydroxide solution produced the best effects on natural fibers properties (Sun et al., 2004). After that, EFB fibers were taken out and washed with distilled water. Then, was treated with 10% (v/v) acetic acid (at room temperature) and treat it for 30min to neutralize the excess NaOH present in treated fiber residue. The washings were repeated until cellulose residue is free from acid. Then, the treated fiber was dried in an oven at 70°C for overnight.

Preparation of composite

The compounding of polypropylene, untreated and treated EFB Fibers was carried out in Twin screw extruder machine (MODEL LTE26-40) at 180°C at a roller speed around 50 rpm (pressure is 48 bar) and L:D is 40:1. The compositions of composite are given in table 2 and moulded by using injection molding machine at temperature 190°C, sheets size is approximately 3-4mm thickness. After molding, test specimens were conditioned at 23 ± 2°C, 50 ± 5% RH for at least 40 h according to ASTM D618-99 standard.

Table 2 Composition of PP/ lignocellulose composite

PP-Untreated Lignocellulose (wt %)	**PP-Treated Lignocellulose (wt %)**
100/0	100/0
90/10	90/10
80/20	80/20
70/30	70/30
60/40	60/40
50/50	50/50

MECHANICAL TESTING

Mechanical tests had been carried out on the samples. All tests were performed at room temperature

Composite tensile test

Tensile testing was carried out according ASTM D636-03: Standard test Method for Tensile Properties of Plastics. All tension testing specimens were cut in dog-bone shape using Universal Testing Machine, Shimadzu (MODEL AG-1) fitted with load cell of 5kN, operated at a crosshead speed of 10 mm/min and with a gauge length is 115 mm. Test was performed until tensile failure occurred. Six specimens were tested and at least five replicate specimens were presented as an average of tested specimens.

Three points bend test

Three point bend testing was carried out according ASTMD790-97: Standard. All flexural samples were cut into rectangular specimens. The Flexural modulus was determined using Universal Testing Machine, Shimadzu (MODEL AG-1). The support was 51 mm while the crosshead speed utilized was 1.26 mm/min. Six specimens were tested and at least five replicate specimens were presented as an average of tested specimens

Izod impact test

The izod impacts tests were conducted according to ASTM D256: Standard. All impact samples were cut into rectangular specimens and notched. The impact test was determined using Impact pendulum tester, with a 1J hammer (MODEL TOYOSEIKI). Six specimens were tested and at least five replicate specimens were presented as an average of tested specimens

Interfacial morphology analysis (SEM)

Fracture surface of fibers before and after pre-treatment preparation were observed with a scanning electron microscope (SEM) using JOEL (MODEL OXFORD)

Fourier Transform Infrared Spectrophotometry (FTIR)

Interfacial and esterification morphology of fibers before and after pre-treatment preparation also were analyzed by means of Fourier Transform Infrared Spectrophotometry (FTIR) using Therma (MODEL THERMO)

RESULTS AND DISCUSSION

Morphology of EFB fibres before and after alkaline pre-treatment (SEM)

Based on SEM morphology studies in Figure 1, it was showed surface roughness of lignocelluloses EFB before pre-treatment which indicated as residual of lignin. By applied pre-treatment of alkaline peroxide, residual lignin was eliminated. Elimination of lignin was breakdown the fibre bundles led increasing the effective surface area and surface charge exposed further hydroxyl and carboxyl groups. This may be because alkaline reacts with cementing materials of the fibre, splitting the fibers into finer filaments. In addition, combination alkaline peroxide enhanced the porosity and pore size on the fibre size where improved physical interlocking leading better interfacial bonding between fibre and matrix. Hence, alkaline peroxide enhanced the surface charge, porosity and pore size on the fibre surface.

EFB fibres before and after alkaline pre-treatment (FTIR Spectroscopy)

Figure 2 shows the FTIR spectra of the lignocelluloses EFB fibers before and after alkaline peroxide pre-treatment. The region from 4000 to 1900 cm^{-1} did not give any useful information since it is largely responsible for the O–H and aliphatic C–H stretching frequencies (Rozman et al., 2003; Sun et al., 2003). The major peaks which show up in the spectra are the broad band's at 3465.42 cm^{-1} and 3476.86cm^{-1}, stretching vibration peaks at

3382.74cm^{-1}, 3316.57cm^{-1} rather than at 3428.92cm^{-1}, 3322.16cm^{-1} as attributed to hydroxyl groups in phenolic and aliphatic structures, and the bands centred between 2909.2 cm^{-1} and 2932.31 cm^{-1} predominantly arising from C–H stretching in aromatic methoxyl groups. The remaining region, 1900-500 cm^{-1}, was far more informative and the results below will be confined to this region. Comparison was obviously showed by the virtual absence of absorbance peak at 1740.24 cm^{-1} which is due to ester carbonyl vibration in acetyl, feruloyl, *p*-coumaroyl, etc. groups in lignin and hemicelluloses(Sun et al., 2002). The stretching peaks at about 1654.65 cm^{-1} and 1610.65 cm^{-1} are originated from conjugated carboxyl–carbonyl stretching and are of particular interest indicating that the ester bonds between hydroxycinnamic acids and lignin were cleaved during the alkaline peroxide treatments or the content of these groups is below the detection limit for the spectroscopical methods employed.

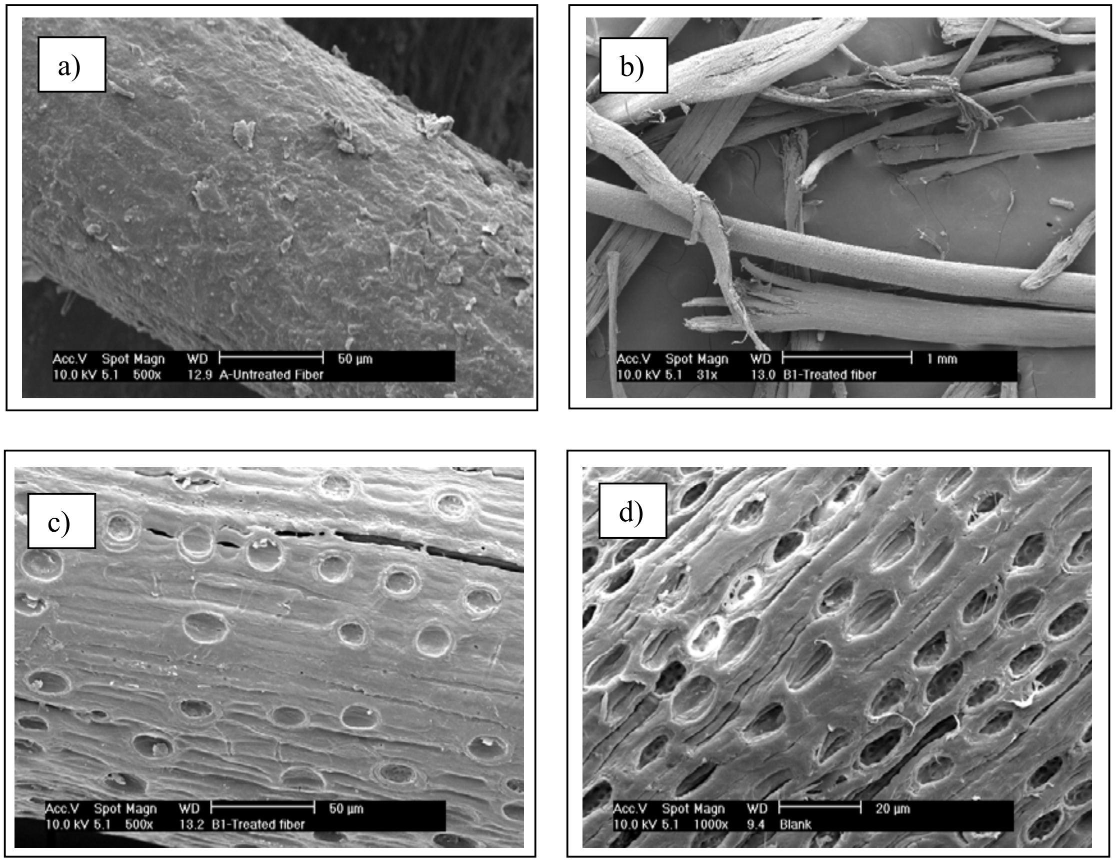

Figure 1: a) EFB fiber before pretreatment, b) EFB fiber after pretreatment, c) Pre-treatment with NaOH + H_2O_2(2hr), d) Pre-treatment with NaOH + H_2O_2 (8hr).

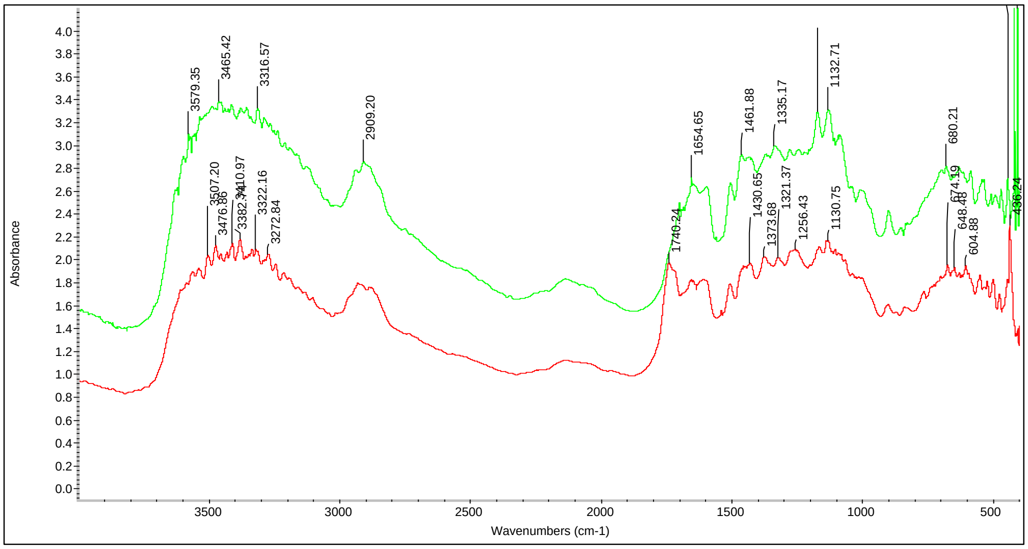

Figure 2: The FTIR spectra of Empty fruit bunch (EFB) fibre, before (spectrum 1) and after (spectrum 2) Alkaline peroxide pre-treatment

Generally, the alkaline peroxide treatment does not seem to have any dramatic consequences on the structure of lignins exception for an increase in carboxylic acid content and a decrease in phenolic hydroxyl content which shown by a relative increase in carbonyl group stretching at around 1256-1132.71 cm^{-1}(Sun et al., 2003). In these reactions, unsaturated and saturated carboxylic acid structures are formed, and the reaction is dependent on the stability of the peroxide solution; an increased decomposition resulting in the formation of hydroxyl radicals and superoxide radical ions leads to a great oxidation of phenols. Aromatic skeleton vibrations in the lignin preparations are assigned at 1600cm^{-1}, 1498cm^{-1}, 1430 cm^{-1}, 1321.37 cm^{-1}, 1256.43 cm^{-1} and 1130.75 cm^{-1}. Further bands are located at 1461.88 cm^{-1} (CH in CH2 and CH3), 1321.37 cm^{-1}, 1335.17 cm^{-1} (guaiacyl ring breathing with C=O stretching), 1256.43 cm^{-1} (guaiacyl ring breathing with C–O and C=O stretching), 1132.71 cm^{-1} (aromatic CH inplane deformation). On the other hand, the introduction of carboxy groups into the lignin polymer is important, since this functional group is hydrophilic and thus facilitates dissolution of the lignin in water. It is, therefore, very likely that the hydroxyl radicals and superoxide radical ions formed promote the oxidation and degradation of the lignin.

Tensile properties

Filler plays an important role in determining the mechanical properties of cellulose filled-thermoplastic composites. The most crucial factor that affects the mechanical properties of fiber-reinforced materials is the fiber matrix interfacial adhesion. The quality of interfacial bonding is determined by several factors, such as the nature of fiber and polymer components, the fiber aspect ratio, the processing method and the treatment of the polymer of the fiber (Ratnam et al., 2008; Sun et al., 2005). In the case of PP-lignocelluloses (EFBF) composite without pre-treatment, adhesion between the two materials was expected to be rather poor than the PP-lignocelluloses (EFBF) composite with application of chemical pre-treatment upon EFBF. EFBF has polar nature whereas polypropylene is characterized by non-polar groups. Due to the changes in physical structure it was expected for treated lignocelluloses (EFBF) to show some changes in the overall properties. The tensile properties of composites are presented in Figure. 3.

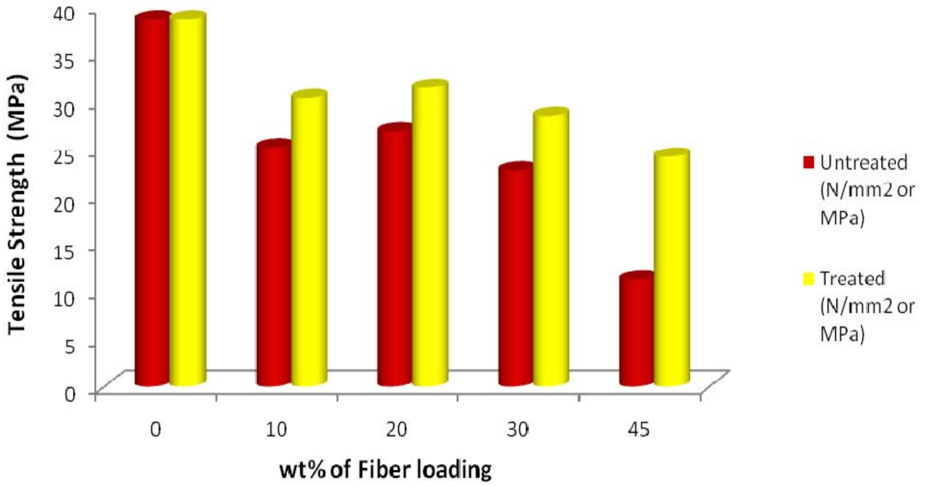

Figure 3: Tensile strength of lignocelluloses based biocomposite

As expected with increase in the filler loading in polymer matrix, PP-untreated EFBF composite showed a common phenomenon of decrease tensile strength as shown by conventional lingocellulosic thermoplastic composite (Ratnam et al., 2008; Pracella et al., 2006; Sun et al., 2001). Filler incorporation in the polymer matrix caused an interruption in stress transferring along the applied force. Above all, lack of significant interfacial interaction between fillers and polymer matrix intensified the problem. Irregularly shape fillers as in the case EFBF filler, were generally not be able to support stresses transferred from the polymer and thus weakened the composite material. PP-untreated and treated EFBF composites appeared to have slightly increased as the filler loading was increased till about 20wt%, while decrease after 30 wt% above. However, the tensile strength of PP-treated composite increased significantly rather PP-untreated EFBF composite as per expectation. The PP-treated EFBF composite till around 45 wt% loading was still higher by almost 50% enhancement rather than untreated EFBF. This indicates that pre-treatment EFBF enhanced cellulose chain mainly which molecular structure of cellulose is responsible for its supermolecular and this, in turns determine many of its physical and chemical properties (Ratnam et al., 2008).

Alkali peroxide treatment reduces the fiber diameter and increases the aspect ratio. Some studies on the alkali treatment of jute-fibers reported (Ray et al., 2001) on the removal of lignin and hemicellulose, which affects the tensile characteristics of the fibers. When the hemicellulose is removed, the interfibrillar region is likely to be less dense and less rigid and thereby makes the fibrils more capable of rearranging themselves along the direction of tensile deformation. When natural fibers are stretched, such rearrangements amongst the fibrils would result in the better load sharing by them and hence result in higher stress development in the fiber. In contrast, softening of the inter-fibrillar matrix adversely affects the stress transfer between the fibril and thus, the overall stress development in the fiber under tensile deformation. As lignin is removed, the middle lamella joining the ultimate cell is expected to be more plastic as well as homogeneous due to the gradual elimination of microvoids, while the ultimate cells themselves are affected slightly.

Furthermore, some researchers reported on the change in the crystallinity through alkaline treatment on sugarcane (Luz et al., 2008), coir and flax (Bledzki and Gassan , 1999). The increase in the percentage crystallinity index of alkali peroxide treated fibers occurs because of the removal on the cementing materials, which leads to better packing of cellulose chain (Luz et al., 2008). In addition, treatment with NaOH leads to decrease in the spiral angle, i.e. closer to fiber axis, and increase in the molecular orientation. A fair amount of randomness is introduced in the orientation of the crystallites due to non-cellulosic matter (Sun et al., 2004). Additionally to increase in the mechanical properties through alkalization peroxide (Luz et al., 2008; Digabel et al., 2006), an increase in the composite quality is expected due to the improved fiber-matrix bonding.

Flexural modulus

Modulus (stiffness) is another basic property of composites; the primary intention of filler incorporation is usually to increase the stiffness of the resultant material. The elastic modulus of bulk natural fibers such as wood is about 10 GPa. Figure 4 shows the flexural modulus of the before and after alkaline peroxide pre-treatment composite.

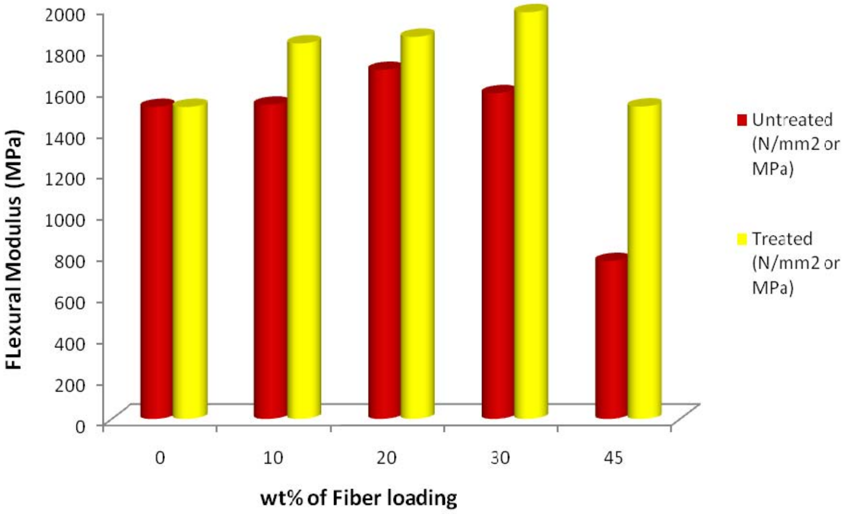

Figure 4: Flexure modulus of lignocelluloses based biocomposite

As expected, pre-treatment of EFBF provided higher flexural modulus rather than without pre-treatment of EFBF. With increasing filler content, slightly increased till about 30wt%, started decreased after 45%. However, PP-treated EFBF composite showed enhancement almost 50 % compared untreated EFBF. There are many factors affecting the modulus of the composites. Those factors are filler content, modulus and aspect ratio. The elastic moduli of fibers, for instance are expected to increase with increasing degree of molecular orientation. Well-oriented cellulosic fibers such as flex have much higher Young's modulus than fibers with medium orientation, such as cotton.

Impact behaviour

The effect of impact strength of PP-untreated EFBF and treated EFBF composite for notched samples is shown in Figure 5. PP-treated EFBF composites appeared to have slightly lower impact strength at lower filler loading however the impact strength of both composites increase with the filler loading. PP-treated EFBF composites show higher degree of impact strength as compared to PP-untreated EFBF composite. The possible reasons for this kind of behaviour may be due to the modification of fiber which tends to have improved energy absorption due to better interfacial bonding (Luz et al., 2008; Ratnam et al., 2008).

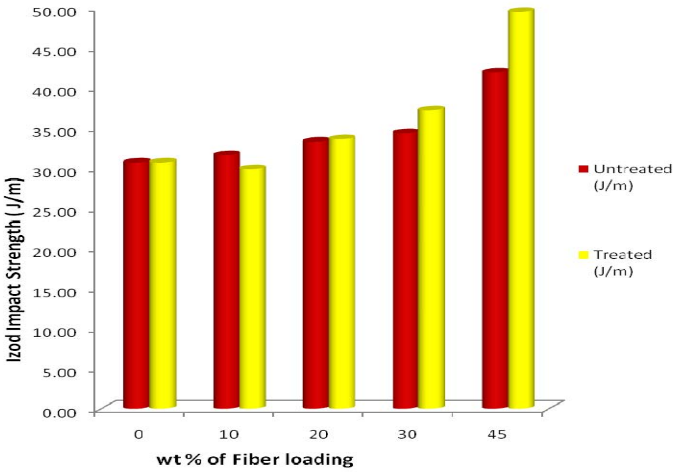

Figure 5: Impact on strength of lignocelluloses based biocomposite

CONCLUSION

This study suggest influence of pre-treatment of EFB fiber provided better interfacial interaction with polypropylene matrix in term of mechanical properties which potentially attractive thermoplastic filler compared to untreated EFB fiber. The Tensile and flexural modulus indicated treated EFB fiber is much higher than untreated EFB fiber composite by using alkaline peroxide treatment which more environmental friendly. However, mechanical properties of these composite may be further improved by using suitable coupling or bonding agents .Successive studies will be conducted using various type of coupling agent namely Maleic anhydride grafted polypropylene (MAPP) to improve the bonding between the cellulose and polymer matrix.

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