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Fabrication of raw and oxidized saw dust reinforced low density polyethylene (LDPE) composites and investigation of their physico-mechanical properties

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Abstract

In this research work, cellulosic waste mango *(Mangifera indica*) saw dust used as the reinforcing material with low density polyethylene (LDPE). A number of samples of saw dust reinforced low density polyethylene (LDPE) composites were prepared by compression moulding technique. In order to improve the mechanical properties of saw dust-LDPE composites, unbleached raw saw dust fibers were modified by oxidation using sodium hypochlorite. FT-IR spectroscopic and scanning electron micrograph (SEM) analyses were done and the results showed the evidence of positive oxidation reaction. The effects of oxidized saw dust on the performance of oxidized saw dust reinforced LDPE composites were studied comparing with the raw saw dust-LDPE composites. The effects of fiber content on the physico-mechanical properties of composites were also studied by preparing the composites with different percentage of fiber loading (from 7.5 wt% to 30 wt%) for each type of composite. Mechanical properties such as tensile strength, tensile modulus, elongation at break, flexural strength, flexural modulus of the resulting composite were measured. Better results were obtained from oxidized saw dust-LDPE composites. Scanning electron micrograph and water absorption tests were carried out for all composites and improved results were found for oxidized saw dust-LDPE composites.

Keywords: Saw dust; LDPE; Composite; Tensile strength; Flexural strength; SEM

Introduction

Lignocellulosic fibers offer many advantages over synthetic fibers as filler reinforcement in thermoplastic matrix. The replacement of synthetic fibers with comparable lignocellulosic fibers provides less weight and decreases the cost without reducing the mechanical properties of the composites (Matuana *et al.*, 2001). Any substance that contains both cellulose and lignin is a lignocellulosic material. Lignocellulosic materials include wood, agricultural crops like jute, kenaf, sisal etc. The advantages of using lignocellulosic materials also include high specific stiffness and mechanical strength, ease availability, lower cost on unit-volume basis, low hardness, which minimizes the wear of processing equipment, renewability, recyclability, non-hazardousness, biodegradability, and so forth (Zadorecki *et al*., 1990, Subyakto *et al.,* 2011 and Ku *et al.*, 2011). Compared with thermosets, composites fabricated from thermoplastic materials typically have a longer shelf life, higher strain to failure, faster to consolidate and retain the ability to be repaired, reshaped and reused as need arises (Wolf *et al*., 1998 and Bismarck *et al.,* 2002). These thermoplastics include polypropylene (PP), polystyrene, vinyls, low-density polyethylene (LDPE) and high-density polyethylene (HDPE). In this research low density polyethylene (LDPE) polymer was used as the matrix material to prepare compression moulded saw dust reinforced LDPE composites.

Mango (*Mangifera indica*) wood is abundantly available in Bangladesh. So mango sawdust, a waste product from the processing of wood, is an important source to use as a lignocellulosic reinforcing fiber in thermoplastics. In this research mango saw dust fiber was used as a reinforcing material to prepare reinforced LDPE composites. Natural fiber reinforced composites are inferior to synthetic fiber reinforced composites in tensile strength and modulus, but they exhibit higher elongation, which provides better tolerance to composite damage. However, a major problem of natural fiber reinforced composites is their susceptibility to fungal and insect attack and to degradation by moisture (Ehrenstein *et al.*, 1992 and Araga *et al.,* 2011). The high moisture absorption of the natural fibers and their low microbial resistance are disadvantages that need to be considered, particularly during shipment and long-term storage as well as during pro-

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cessing of the composites. In addition, their hydrophilic behavior affects the properties of the fibers themselves as well as the properties of their composites (Kazayawoko *et al.,* 1999 and Bismarck *et al.,* 2002). A lot of literature focuses on these limitations and these limitations in performance of the natural fiber reinforced composites can be greatly improved through chemical modification techniques (Kalia *et al.,* 2009, Bodirlau *et al.,* 2009, Dikobe *et al.,* 2010 and Kord, 2011). Cellulose (90%) of cotton is oxidized in two products; one is the reducing oxycellulose in which hydroxyl groups have been converted to carbonyl groups or aldehydes and the other is the acidic oxycellulose in which the hydroxyl groups have been converted to carboxyl groups or acids (Segal *et al.,* 1985). Primary alcohols (hydroxyl group at C_6) are more reactive than secondary alcohol (hydroxyl group at C_3 and C_3) of cellulose anhydroglucose unit but periodic acid, periodate salts and mild oxidizing agent break the anhydroglucose ring between carbon atoms 2 and 3 and convert the two secondary hydroxyl groups to aldehyde groups (Segal *et al.,* 1985). The reaction of sodium periodate with cellulose in raw jute yielded dialdehyde cellulose in oxidized jute (Sultana *et al.,* 2007). In this research, sodium hypochlorite was used as an oxidizing agent to oxidize cellulose of raw saw dust.

Therefore, the aim of this paper was to oxidize cellulose (50%) of unbleached raw saw dust to dialdehyde cellulose and to investigate the effect of this oxidation of saw dust on the physico-mechanical properties of saw dust-LDPE composites.

Materials and methods

Low density polyethylene (LDPE) and Saw dust fiber

Low density polyethylene (LDPE) made by PTT Global Chemical Public Company Limited (Thailand), was used in this study. Melting point of this LDPE was measured and found to be 115 ^{o}C. The saw dust fibers were collected from local saw mills of Narayangonj, Bangladesh. The supplied saw dust fibers were a waste product from mango (*Mangifera indica*) wood.

Treatment of saw dust fibers by oxidation reaction

The raw saw dust fibers were first cleaned manually and sieved with sizes < 0.6 mm in this work. The fibers were then washed with distilled water and dried in open air. Sodium hypochlorite solution of 0.1N concentration was prepared in distilled water. Dried saw dust fibers were immersed in sodium hypochlorite solution. The pH of the solution was adjusted to 7 by adding sulfuric acid. The oxidation reaction was carried out for 48 hours at room temperature (30 ^{o}C). The reaction mixture was stirred by glass rod occasionally during this period. After completion of the reaction the mixture was filtered to isolate the oxidized fibers. The oxidized saw dust fibers were thoroughly washed with tap water and finally with distilled water. The washed oxidized fibers were dried in air and then dried in oven at 105 ^{o}C for 24 hours. This oxidized saw dust fibers were used for composite fabrication.

Composite fabrication

Saw dust-LDPE composites were prepared using raw saw dust and chemically oxidized saw dust following the procedure described below:

The raw and oxidized saw dust fibers were dried in an oven at 105 ^{o}C for 24 hours. Saw dust fibers were mixed thoroughly with low density polyethylene to prepare composites. Blender was used to mix them and the blending time was one minute at 400 rpm for each specimen. LDPE matrix and saw dust fibers were taken in different weight fractions (Table I).

Table I. Relative amounts of reinforcing materials and polymer matrix

Reinforcing material (%)	Polymer Matrix (%)	Composites
None	LDPE: 100.0	100 wt % LDPE
Saw dust: 7.5	LDPE: 92.5	7.5:92.5 (wt. %) Saw dust-LDPE
Saw dust: 15.0	LDPE: 85.0	15:85 (wt. %) Saw dust-LDPE
Saw dust: 22.5	LDPE: 77.50	22.5:77.5 (wt. %) Saw dust-LDPE
Saw dust: 30.0	LDPE: 70.0	30:70 (wt. %) Saw dust-LDPE
Oxidized Saw dust : 7.50	LDPE: 92.50	7.5:92.5 (wt. %) Oxidized Saw dust-LDPE
Oxidized Saw dust: 15.0	LDPE: 85.0	15:85(wt. %) Oxidized Saw dust-LDPE
Oxidized Saw dust: 22.5	LDPE: 77.50	22.5:77.5(wt.%) Oxidized Saw dust-LDPE
Oxidized Saw dust: 30.0	LDPE: 70.0	30:70 (wt. %) Oxidized Saw dust-LDPE

Preparation of composites using compression technique

Compression moulded composites were prepared using Paul-Otto Weber Press Machine (compression moulding machine).The fiber and matrix were mixed and taken into the special moulding device. Sufficient pressure of 200-250 kN was applied at first to get the desired shape and possible homogeneity. The applied pressure was measured by using a pressure guage, set in the device. Heating was done electrically and the temperature was set at 120 ^{o}C for LDPE. The temperature reached to 120 ^{o}C after 30 minutes and the heating time was 10 minutes after reaching the temperature at 120 ^{o}C. After completion of heating the initial pressure was set to zero and an additional pressure of 50 kN was applied to avoid voids and to have a certain thickness. Cooling is essential throughout the curing operation. Cooling was done by tap water through the outer area of the heating plates of the hydraulic press machine. Finally the compression moulded specimen was removed by using a set up device.

Characterization of treated saw dust and prepared composites

The saw dust fibers (treated and untreated) and composite materials were characterized by FT-IR Spectroscopy and scanning Electron Microscopy (SEM) as stated below:

FT-IR Spectroscopy

The infrared spectra of raw saw dust fiber and treated saw dust fiber were recorded on a Shimadzu FTIR-8101 spectrophotometer. IR spectra with all information about absorbance were obtained in the printed form.

Scanning electron microscopy (SEM)

The saw dust fibers (treated and untreated) and the fractured pieces of the bending test specimens of 30 wt-% raw and oxidized saw dust fiber loading LDPE composites were used to examine by a scanning electron microscopy (S-3400 N, Hitachi, Japan). The photographs are presented in the results and discussion section.

Mechanical properties of the composites

In order to investigate the mechanical properties of the prepared composites the following tests were carried out: (a) tensile and (b) three point flexural.

Tensile test

The static tensile test of the composites were carried out in an universal tensile testing machine, H10KS, capacity: 10 kN, Hounsfield, UK at a cross head speed of 2 mm/min. Tensile tests were conducted following ASTM D 3039/D 3039M-00 (2002) method and each test was performed until tensile failure occurred except 100% LDPE composite. Six to ten specimens of each composition were tested and the average values were reported by calculating maximum five values.

Three point flexural test

The static flexural tests of the composites were carried out by same machine that was used for tensile test only by changing the attachment. Flexural tests were conducted following ASTM D 790-00 (2002) method at a cross head speed of 2 mm/min. Span length was 50 mm. Five specimens of each composition were tested and the average values were reported.

Water absorption test of composites

Water absorption tests of the composites were measured according to ASTM D570-99(2002).

The samples were dried in an oven at 105 ^{o}C for 3 h, cooled in a desiccator and immediately weighed. The dried and weighed samples were immersed in boiling water for 2 hours as described in ASTM D570-99 method. Excess water on the surface of the samples was removed by using a soft cloth and then the weights of the samples were taken. The results were presented as average of the tested specimens. The percentage increase in weight during immersion was calculated as follows:

$$\text{Increase in weight, \%} = \frac{(\text{wet wt} - \text{conditioned wt})}{(\text{conditioned wt})} \times 100$$

Results and discussion

FT-IR spectroscopic characterization of oxidized saw dust fibers

The reaction of cellulose in raw saw dust fibers with sodium hypochlorite yielded the oxidized product 2,3-dialdehyde cellulose in oxidized saw dust. The oxidized product 2,3-dialdehyde cellulose has been characterized by infrared spectroscopic analysis. The IR spectrum shows characteristic bands of aldehyde group at the region of 2922 cm^{-1} and 2854.5 cm^{-1} due to C-H stretching and at the region of near

1716.5 cm^{-1} due to carbonyl stretching. Untreated raw saw dust fibers show the absorption band near 1716.5 cm^{-1} due to the carbonyl group of acetyl ester in hemicellulose and carbonyl aldehyde in lignin.

Morphological (SEM) characterization of raw and oxidized saw dust fibers

Morphological information and identification of raw saw dust and oxidized saw dust fibers have been presented in the Figs. 1 and 2 respectively. The role of the chemical treatment of saw dust fibers is mainly to decrease their hydrophilic properties. It is clear from the comparison of the SEM Figs. 1 and 2 that oxidation reaction of saw dust fibers improves the physical structural properties by smoothing cell wall surface of oxidized saw dust fibers than raw saw dust fibers.

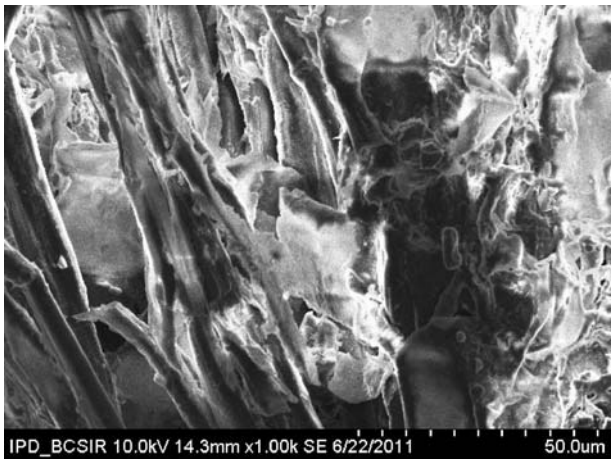

Fig. 1. SEM micrograph of raw saw dust fibers

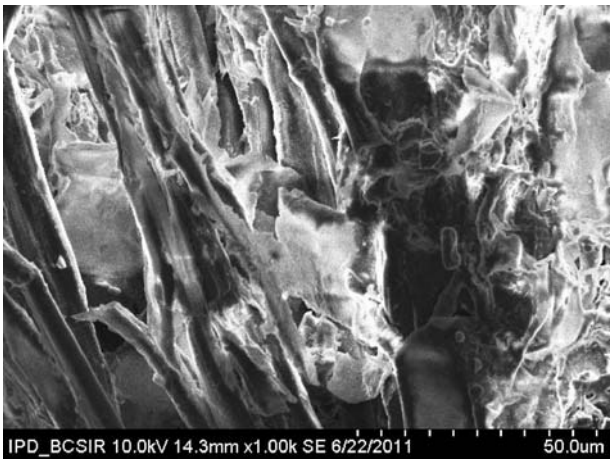

Fig. 2. SEM micrograph of oxidized saw dust fibers

Raw and oxidized saw dust fiber reinforced low density polyethylene (LDPE) composites

As mentioned earlier saw dust reinforced LDPE composites were prepared both by using raw and oxidized saw dust fibers. The results are presented below.

Mechanical properties of raw and oxidized saw dust fiber reinforced low density polyethylene (LDPE) composites

Tensile strength, tensile modulus, elongation at break, flexural strength and flexural modulus of the raw and oxidized saw dust fiber reinforced LDPE composites have determined following the ASTM method described in the experimental sections. The results obtained in this study are presented below.

The tensile strengths of the raw and oxidized saw dust fiber reinforced LDPE composites decrease with increasing saw dust fiber loading by weight fraction from 7.5% (w/w) to 30% (w/w) (Fig. 3). This reduction possibly is due to the lack of stress transfer from the LDPE matrix to saw dust fibers. Approximately similar results were found for kenaf-PP composites (Karnani *et al*.,1997) and empty fruit bunch (EFB) fibers-PP composites (Saad *et. al.*, 2001) in the literature. Tensile strength and tensile modulus of 100 wt-% LDPE are 9.30 MPa and 0.14 GPa respectively. The Tensile modulus increases with increasing fiber loading (Fig. 4) as compared to 100 wt-% LDPE. The higher tensile modulus is found for all oxidized saw dust-LDPE composites with compared to the raw saw dust-LDPE composites. So stiffness of the oxidized saw dust-LDPE composites is higher than raw saw dust-LDPE composites. Elongation at break decreases with increasing fiber loading (Fig. 5). It is also found that elongation at break of oxidized saw dust-LDPE composites is higher than that of untreated raw saw dust-LDPE composites.

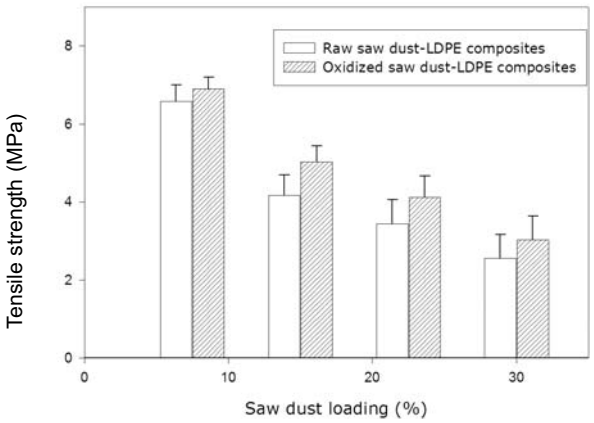

Fig. 3. Tensile strength against saw dust content (wt%) for raw and oxidized saw dust-LDPE composites

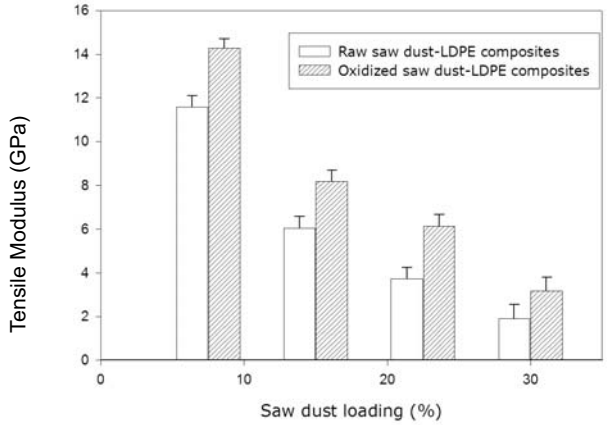

Fig. 4. Tensile modulus against saw dust content (wt%) for raw and oxidized saw dust-LDPE composites

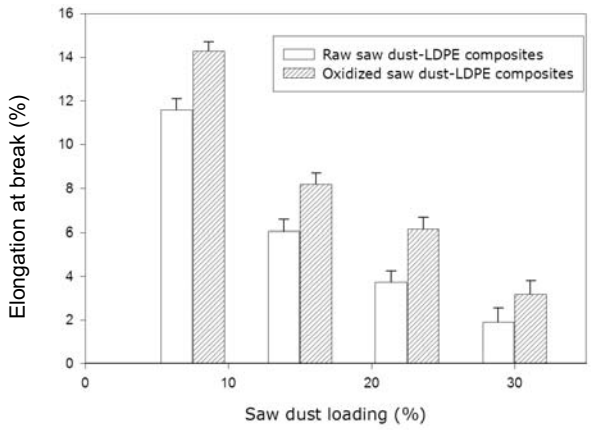

Fig. 5. Elongation at break against saw dust content (wt%) for raw and oxidized saw dust-LDPE composites

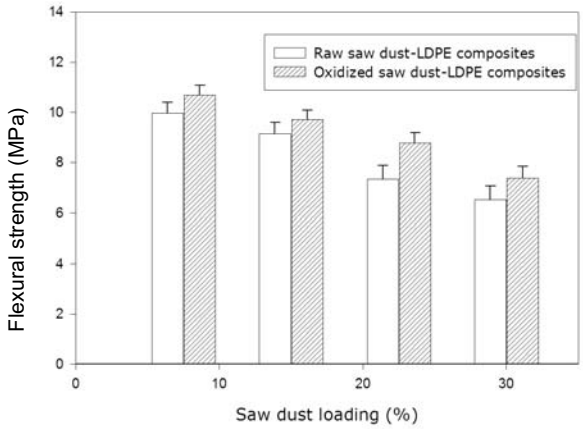

Fig. 6. Flexural strength against saw dust content (wt%) for raw and oxidized saw dust-LDPE composites

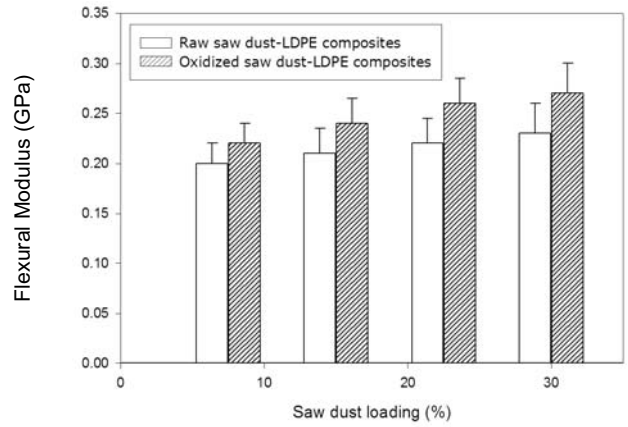

Fig. 7. Flexural modulus against saw dust content (wt%) for raw and oxidized saw dust-LDPE composites

The flexural strength of composites measures the ability of composites to withstand the bending forces applied perpendicular to its longitudinal axis. The experimental results on flexural strength of the raw and oxidized saw dust-LDPE composites have been presented in Fig. 6. Flexural strength and flexural modulus of 100 wt-% LDPE are 8.83 MPa and 0.19 GPa respectively. It is observed from the Figure 6 that the values of flexural strength for both the composites increase with increasing fiber loading up to 15 wt-%, then it starts to decrease at 22.5 wt-% fiber loading as compared to 100 wt-% LDPE matrix. The values of flexural modulus have been presented in Fig. 7. It is observed from the figure that flexural modulus of all composites increases significantly with increasing fiber loading. Higher values of flexural strength and flexural modulus observed in the case of all oxidized saw dust-LDPE composites than that of raw saw dust-LDPE composites.

It is observed from all the mechanical properties according to series of formulation based on the raw saw dust-LDPE and oxidized saw dust-LDPE composites are presented in the Fig. (3-7) that oxidation of saw dust enhanced the mechanical properties of oxidized saw dust-LDPE composites than raw saw dust-LDPE composites. The reasons of these enhancement results may be explained on the basis of hydrophilic nature of saw dust and hydrophobic nature of LDPE matrix. Oxidation of saw dust fibers decreases its hydrophilic nature as compared to raw saw dust fibers. The improved interfacial bonding between oxidized saw dust fibers and LDPE matrix in the oxidized saw dust-LDPE composites increases their mechanical properties as compared to raw saw dust-LDPE composites.

Morphological (SEM) characterization of raw and oxidized saw dust fiber reinforced low density polyethylene composites

Scaning electron micrographs (SEM) were taken using 30 wt-% raw and oxidized saw dust fiber loading LDPE composites. Fracture surfaces of bending test specimens were used to take SEM and are shown in Figs. 8 and 9. SEM of raw saw dust-LDPE composite indicates that there is a weak interfacial interaction and poor dispersion of the hydrophilic raw saw dust fiber in the hydrophobic LDPE matrix. But SEM observation of the oxidized saw dust-LDPE composite (Fig. 9) indicates the better interfacial interaction between oxidized saw dust fiber and LDPE matrix in the oxidized saw dust-LDPE composite as compared to raw saw dust-LDPE composite (Fig. 8). This may be due to oxidation of saw dust fibers because oxidized saw dust fibers contain aldehyde group which are less polar than hydroxyl group of raw saw dust and aldehyde groups are not capable of forming intermolecular hydrogen bonds since aldehyde groups contain hydrogen bonded only to carbon (Clemons *et. al.,* 1999).

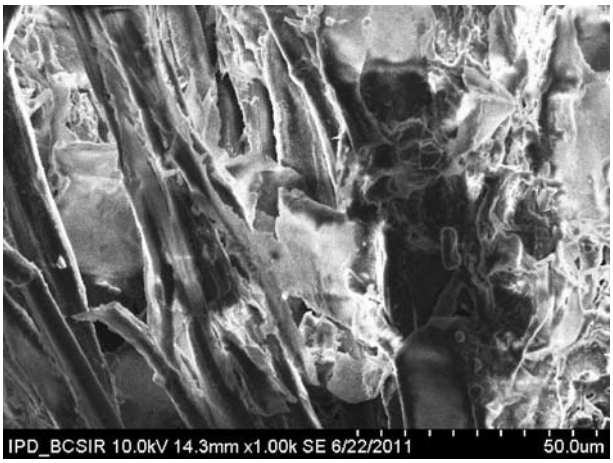

Fig. 8. SEM micrograph of 30 wt-% raw saw dust-LDPE composite

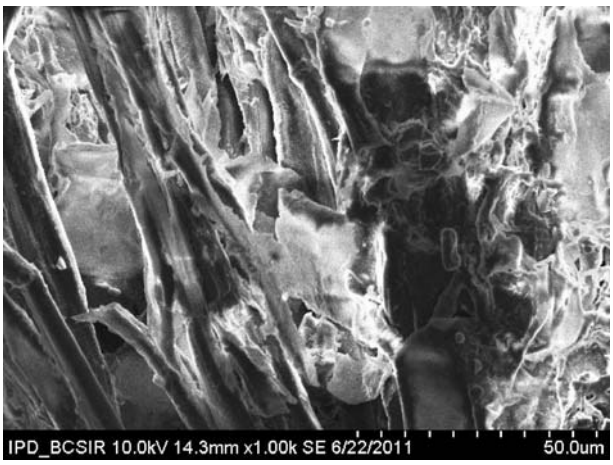

Fig. 9. SEM micrograph of 30 wt-% oxidized saw dust-LDPE composite

Water absorption

Water absorption in lignocellulosic based composites can lead to build up moisture in the fiber cell wall and also in the fiber-matrix interphase region. It is difficult to eliminate entirely the absorption of moisture from the composites without using expensive surface barriers on the composite surface. Proper chemical modification of fibers can reduce the moisture absorption property of the fibers. Results of water absorption obtained in our study are shown in Fig. 10. It is observed from the figure that water absorption of the composites increases with increasing fiber loading but water absorption of all oxidized saw dust-LDPE composites are less than that of raw saw dust-LDPE composites. So it is clear that oxidized saw dust-LDPE composites are less hydrophilic than untreated raw saw dust-LDPE composites. As aldehyde group of dialdehyde cellulose absorbs less water than hydroxyl group of cellulose so water absorption of oxidized saw dust-LDPE composites is less than that of untreated raw saw dust-LDPE composites.

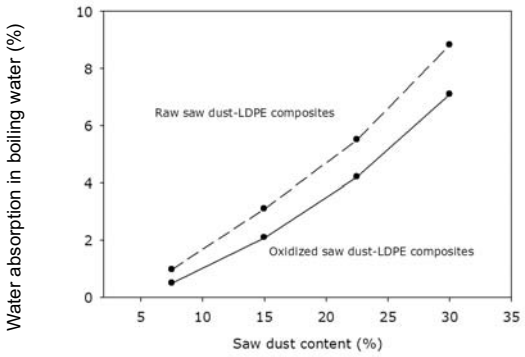

Fig. 10. Water absorption against saw dust (wt%) for raw and oxidized saw dust-LDPE composites

Conclusion

The experimental investigation and theoretical interpretation of raw and oxidized saw dust filled LDPE composites leads to the following conclusions:

Cellulose in raw saw dust fibers can be oxidized at C_2 and C_3 position of anhydroglucose unit to form reducing oxycellulose (dialdehyde) by sodium hypochlorite. FT-IR and SEM analyses have been done to characterize the intermolecular

interaction and physical structural properties of oxidized saw dust fibers.

Tensile modulus, flexural strength and flexural modulus of the raw and oxidized saw dust-LDPE composites are significantly higher than 100 wt-% low density polyethylene (LDPE). Tensile strength and flexural strength of oxidized saw dust-LDPE composites are 4 to 21 % and 6 to 20 % higher than raw saw dust-LDPE composites respectively. Mechanical properties of oxidized saw dust-LDPE composites are higher than those of raw saw dust-LDPE composites. The possible reason for these improvements is due to the absence of intermolecular hydrogen bonding of dialdehyde cellulose in oxidized saw dust fiber which improves the adhesion between fiber and LDPE matrix in the composites interface. As a result oxidized saw dust dispersed more uniformly in the composites than raw saw dust. Morphological studies of these composites show better interfacial bonding between fiber and matrix for oxidized saw dust-LDPE composites and water absorption properties of the composites also show better result for oxidized saw dust-LDPE composites than raw saw dust-LDPE composites.

From these observations, it can be concluded that natural fiber matrix interaction can be improved by chemical modification of fiber by decreasing their hydrophilic nature which enhances the physico-mechanical properties of the natural fiber reinforced composites. And this type of modified natural fiber reinforced composites will show better performance under load bearing conditions.

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