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Influence of Chemical Treatments of Sawdust Fibers on the Physical Properties of Their Reinforced Polypropylene Composites

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ABSTRACT

In order to achieve superior composite properties, sawdust fibers (SDF) were chemically treated by NaOH, $NaClO_2$, $KMnO_4$, acetic anhydride and n-substituted methacrylamide. Composites of SDF and PP were fabricated by simple hot press molding method using various proportion (0, 10, 20 and 30 wt%) of raw and chemically treated SDF with respect of matrix weight in composites. The composites were characterized by means of tensile, Rockwell hardness, water absorption and thermal measurements. The tensile moduli of all types of composites were increased with the increase of fiber proportion, whereas the tensile strength, elongation at break and Rockwell hardness revealed inverse trend. Composites with 10 wt% fiber loading were exhibited higher tensile strength, Rockwell hardness and elongation at break than that of 20 and 30 wt% fiber loaded composites. Alkali treated, acetylated and nMA treated SDF-PP composites were exhibited significant improvement in tensile strength, water absorption and Rockwell hardness properties. Thermal stability of the composites have been assessed as well. The main degradation temperature of the SDF was shifted to higher after composite fabrication. Fiber treatments also showed positive impact on the thermal properties of composites.

1. Introduction

Over the last decade, use of agro-residual/forest biomass fiber reinforced thermoplastic composites become popular as a replacement of traditional fiber composites [1, 2]. Sawdust fiber is a good representative of abundant natural fiber containing high percentage of cellulose [3]. In Bangladesh, SDF obtain from sawmills are usually thrown away after timbers are sawed in sawmills sometimes it is as mulch, fuel for brickfield, household cooking etc. It is often used in mostly for packaging materials, insulator, decorative materials etc. Till date, a few numbers of studies have been done on the fabrication of composite materials from sawdust [4, 5]. Thermoplastices polyolefins (polyethylene and polypropylene) and polyesters are the suitable matrix materials for cellulose fiber. Among them polypropylene (PP) is most extensively used plastics due to high mechanical strength, thermal stability, recycle ability and low price [6, 7].

The desired properties of SDF reinforced polypropylene composites is hardly found because of the weak mixing capability of hydrophilic natural fiber and hydrophobic synthetic polymer. The presences of large number of hydroxyl groups of cellulose make poor resistance to moisture absorption. To overcome this drawback, various modifications like physical, radiation, chemical etc. have been investigated. The physical modification such as Corona treatment, plasma treatment etc. didn't get reputation due to complex and expensive instrumental setup. UV radiation, gamma radiation, ultrasonic shock wave are the most effective ways to modify natural fiber. But it is very difficult to get repeated results from radiation methods. Chemical methods are comparatively free from the above mentioned drawbacks. For these reasons, most of the modification was done by chemical methods. Alkali treatment, acetylation, acrylation, vinyl treatments, silane treatment etc. are the major methods applied to interface modification of natural fiber [8-10]. Among the chemical modifications, alkali treatment is most popular. It may increase the amorphous part of cellulose and decrease number of intra- and intermolecular hydrogen bonding in the cellulose structure. Alkali pretreatment is often performed before the chemical modification via substitution reactions [9, 10]. Bleaching is generally used in textile application of natural fiber. It is very efficient method to remove lignin and other impurities from natural fiber. Khan et al., used sodium chlorite bleaching agent for the surface treatment of jute, banana, okra, pineapple leaf fibers on their several studies [11, 12]. $KMnO_4$ act as a strong oxidizing agent in presence of concentrated sulfuric acid. The primary hydroxyl groups of cellulose may transform into carbonyl compounds followed by carboxylic acids. In addition, this treatment increases the nucleating ability and thermal properties of natural fiber PP composites [13]. Acetylation is most powerful method to increase the surface hydrophobicity of the natural fibers. The hydroxyl groups (-OH) of the fiber reacts with acetyl groups ($-CH_3CO$), thereby decrease the number of hydrophilic hydroxyl groups as well as surface covering may formed onto natural fibers [14, 15]. Graft copolymerization through vinyl monomers often employed to improve surface properties of natural fibers. Methacrylamide (MA) monomer attached with –OH group of cellulose and propagate the polymerization reaction on fiber surface. As a result, increase hydrophobic nature of fiber which is more compatible with thermoplastic resin to get high mechanical properties in composites [16-19].

In the present investigation, composites of SDF and PP were prepared by simple hot press molding method. It is one of the cost effective method of composite preparation. A number of chemical modifications of SDF have been done to find the suitability of SDF as reinforcement in PP matrix. The change of physical properties of the composites with fiber loading and chemical treatments have been performed.

2. Experimental Methods

2.1 Materials

Commercial grade polypropylene (PP) supplied by Yokkaichi, Japan was used as a matrix without further purification. The technical specifications of PP are shown in Table 1. SDF was used as reinforcing agents obtained from local saw mill of Kushtia district, Bangladesh. The adhering impurities of SDF were removed by the scouring treatment fiber with a solution of 3.5 g/dm^3 Na_2CO_3 and 6.5 g/dm^3 Jet powder (detergent) at 343 K in a beaker. Jet powder was purchased from Kallol Group of

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Companies Ltd., Bangladesh. The other chemicals and reagents used in this investigation were analytical reagent (AR) grades.

Table 1 Properties of virgin polypropylene

Properties	Value
Melting temperature	441-445 K
Density	0.905 g/cc
Tensile strength	31-33 MPa
Young's modulus	1.00-1.15 GPa
Elongation at break	10-12%

2.2 Chemical Treatments

The bleaching treatment of SDF was carried out with 7 g/dm³ $NaClO_2$ aqueous solution at 363-368 K for 90 min. The pH of the solution was adjusted 4 by dilute acetic acid. Then 10v/v% acetate buffer was added to keep pH constant. The SDF was then taken into the solution with the liquor ratio 1:50. After complete the reaction, the fiber was taken off from the solution and washed thoroughly with sufficient water. The fiber was submerged in 2 g/dm³ $Na_2S_2O_5$ solution for 15 min to stop the oxidation and finally washed repeatedly with distilled water [4].

SDF was soaked in 100 g/dm³ NaOH solution for 10 hrs with occasional stirring. The alkali treated SDF was washed repeatedly with large amount of water, dilute CH_3COOH and distilled water. The fibers were dried at room temperature and then at 323 K in air oven for 24 hrs [4].

Small amount of SDF was dipped in 0.05 g/dm³ $KMnO_4$ solution in acetone for 10 min. 1 to 2 drops of concentrated H_2SO_4 was added during the treatment and stirred continuously. Finally, the fiber was washed with acetone and distilled water. The dried SDF was stored for further use [4].

10 g SDF was soaked in 100 mL glacial acetic acid for 1 hr at ordinary temperature. The fiber was taken out from the solution and then immersed in 1 g/dm³ $(CH_3CO)_2O$ solution having few drops of concentrated H_2SO_4 for 10 min. The fiber was filtered by Buchner funnel, and washed repeatedly with distilled water. The acetylated SDF was dried in air oven at 323 K for 24 hrs [4].

n-substituted methacrylamide (nMA) monomer was dissolved in distilled water. The reaction vessel containing 1 w/w% nMA monomer, 0.01 w/w% $K_2S_2O_8$ and 0.01 w/w% $FeSO_4$ was placed in a hotplate equipped with magnetic stirrer. The weight percentage of monomer and reagents were calculated respect to the weight of fiber and fiber to liquor ratio was fixed to 1:50. The reaction was carried out at 363 K for 60 min with continuous stirring. During the reaction, the loss of liquid from reaction vessel was compensate by hot distilled water. After completion of reaction the vessel was allowed to stand for further 30 min till it cools down. Then the fiber was refluxed by CH_3OH to remove the adhering homopolymer, washed with hot distilled water and dried at room temperature followed by air oven at 323 K for 24 hrs.

2.3 Composites Preparation

The SDF was placed in an electric oven for moisture removal at 378 K for 24 hrs. Composites were prepared by compounding PP matrix with the untreated and treated SDF in a compress molding machine. Predetermine amount of PP pellets and dried SDF were taken in a stainless steel mold and placed in molding machine. A mold release spray was used for easily taking composite from mold. During composite fabrication 50 KN pressure was fixed and mold was heated at 443 K for 30 min. Mold was then suddenly cooled by tap water. Finally composite sample was taken out from mold and sized for tensile measurement. The specimens for tensile (dimensions: 110×15×3) mm³ tests were prepared using a cutting machine [21].

2.4 Tensile Measurements

The tensile tests were performed according to ASTM D882 (2012) standard method using Hounsfield Universal Testing Machine (UK). The method was operated by QMAT software. The distances between two jaws were fixed 15 mm and crosshead speed was set 5 mm/min [10]. At least 5 specimens were taken for each type of sample.

2.5 Rockwell Hardness Test

Rockwell hardness number is a value derived from the net increase in depth impression as the load on an indenter increased from a fixed minor load to a major load and then returned to a minor load. Rockwell Hardness test of composite sample was conducted according to ASTM D785-93 standard method. The specimens were conditioned at controlled atmosphere of 298±2 K and 50 ±5% RH for specified duration. At least 5 measurements were taken for each type of sample and the results were averaged.

2.6 Water Absorption

The percentage of water absorption of composite samples was measured according to ASTM-D-570. The specimens (dry weight W_1) were subjected in water at 50 °C for 24 hrs, and after conditioned weight (W_2) was taken carefully. At least 5 measurements were taken for each type of sample and the results were averaged. Percentage of water absorption was calculated as follow:

$$\text{Water absorption } \% = \frac{(W_2 - W_1)}{W_1} \times 100$$

2.7 Thermogravimetric Analysis

The Thermal Gravimetric Analyzer (DTA6300 supplied by TA instruments) was used to measure the thermal stability of the fiber and composite samples. About 2 mg of each sample was taken on sample holder heating was started by the rate of 10 °C/min from 303 to 773 K. All of the tests were carried out at nitrogen atmosphere.

3. Results and Discussion

3.1 Tensile Properties of the Composites

The tensile properties of composite using 0, 10, 20 and 30 wt% untreated fiber are demonstrated in Table 2. Among the various fiber loading, tensile strength (TS) is found higher in 10 wt% fiber loading. The comparatively lower amounts of fiber are well distributed in PP matrix and therefore SDF and PP are tightly bonded at their interface. Above 10 wt% fiber loading, the tensile strength decreases, probably due to the relatively large amount of SDF make agglomeration inside the matrix network which causes non-uniform stress transfer [22]. In addition, incompatible effect between hydrophilic fiber and hydrophobic matrix is more pronounced which may promote micro-crack formation at the interface. Tensile modulus (TM) was calculated from tangent of initial point of tensile stress-strain curves (Fig. 1). From the Table 2, it has been seen that TM increases and elongation percentage decreases with the increase of fiber loading. Since the stiffness of the natural fiber is much higher than PP matrix, that may influence the overall performance of composites. Thus with the increase of fiber content the stiffness as well as TM increases. Similar trend of TM is found for betel nut fiber-PP composites in previous study [21]. Conversely, the percent of elongation of composites decreases with the addition of SDF. The result indicates the reduction of ductile nature of composite samples. This is also reported in previous investigation [23]. This result can be explained the crystalline nature of fiber may decrease the chain mobility of PP and deformability of stiff interface between SDF and PP. Consequently, causes the reduction of ductility of composites.

Table 2 Effect of fiber loading on tensile properties of composites

PP : SDF	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation (%)
100:0	76.2	4.11	11.0
95:5	83.0	5.42	6.60
90:10	88.7	6.67	4.70
80:20	62.5	7.61	3.40

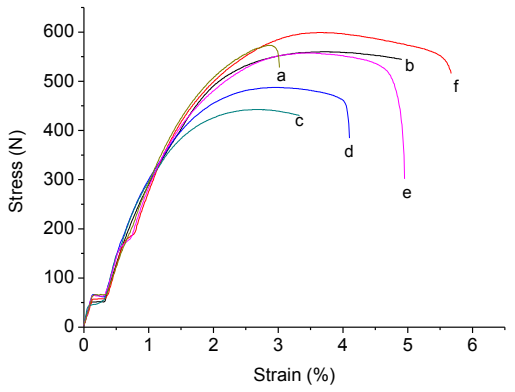

Fig. 1 Tensile stress-strain curves of 10wt% SDF loaded composites (a) untreated, (b) alkali treated, (c) bleached, (d) $KMnO_4$ treated (e) acetylated and (f) nMA treated fiber

The change of tensile properties of 10 wt% fiber loaded PP composites with the effect of chemical treatment of SDF is listed in Table 3. The bleached SDF reinforced PP composite showed a little lower TS compare to untreated SDF composite. In bleaching operation, fiber loss the cementing material and fiber became lighter. Again due to the increase of

relative quantity of cellulose, fibers turn into more hydrophilic. This causes lowering of interface bonding between fiber and matrix as well as lower tensile strength. Although alkali treatment of fiber removes most of the hemicelluloses and other incrusting materials of cellulose, it shows positive effect to improve the tensile properties of a composite. Because it creates the fiber rough surface by leached out pectin, fats, waxy matter etc. from outer surface of the fibers. The polymer matrix is then mechanically interlocked of those rough surfaces which attributed to the improvement of tensile strength. Permanent treatment of natural fiber is employed to clean the surface impurities and change assembly, dimensions and morphology. The highly reactive permanganate ions creates the active sites in cellulose which improve the fiber matrix adhesion. However, no significant change is observe on the tensile strength $KMnO_4$ treated SDF-PP composite compare to untreated fiber SDF-PP composite. Probably, the treatment with very low concentration of $KMnO_4$ has no effect on the tensile properties of composite. It is apparent from the Table 3 that the tensile strength of acetylated SDF-PP composite is higher than those of the untreated SDF-PP composites. This may be attributed that the hydroxyl groups (-OH) of cellulose react with anhydride via substitution process that means the hydroxyl groups (-OH) are replaced by $-CH_3$ groups. Therefore fiber turn into hydrophobic which is more compatible with hydrophobic PP. Table 3 also represents the effect of nMA treatment of SDF on TS of the composites. The effect of nMA treatment on TS of composite is evaluated and made comparison with untreated SDF-PP composite. The increment of TS for 10 wt% nMA treated fiber loaded composites is found 11.2%. The enhanced TS of nMA treated fiber composite is due to better wetting and adhesion ability of SDF with the matrix PP. The order of TS of treated SDF composites is as: nMA treated > alkali treated > acetylated > $KMnO_4$ treated > untreated > bleached SDF-PP composites. TM of SDF reinforced PP composite showed similar trend like TS while no effect is found on elongation at break of composites on treatments of fiber.

Table 3 Effect of fiber treatments on the properties of composites

SDF/PP Composites	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation (%)	Water Absorption (%)
Untreated	88.7	6.67	4.7	4.2
Alkali-treated	94.5	7.91	4.5	4.0
Bleached	86.3	6.63	4.8	4.1
$KMnO_4$ treated	89.1	6.72	4.7	3.9
Acetylated	91.8	7.76	5.1	2.3
nMA treated	98.6	8.08	5.2	2.1

3.2 Water Absorption Properties of the Composites

The water absorption properties of the untreated and treated SDF reinforced PP composites are shown in Table 3. The value of water absorption is decreased significantly for acetylated and nMA treated fiber composites. nMA treated fiber showed greater reduction of water absorption (2.1%) compare to others. In natural fiber reinforced composite, the amount of water absorption depends on the nature of fiber and the micro-channel due to void contain inside the composite. Actually, water is absorbed from the cutting edge of the composites and penetrate through the interface of fiber. Therefore adhesion of fiber and matrix should be compact to stop the water absorption. The surface modified SDF composites especially acetylation and nMA treated SD-PP are masked by PP in composites resulting in greater hydrophobicity and lesser water absorption. The percentage of water absorption of alkali treated, $KMnO_4$ treated and bleached fiber-PP composites are slightly less than the untreated fiber-PP composites. No swelling effect is shown after submersion of the specimens of both untreated and treated SDF-PP composites, which may also due to encapsulation of the SDF by the PP matrix.

3.3 Hardness Properties of the Composites

The Rockwell hardness C-scale of untreated and treated sawdust reinforced PP composites with different proportion of fiber content (0, 10, 20 and 30 wt%) are compared in Fig. 2. It has been found that the value of Rockwell hardness of composites is decreased with the increase of SDF content. Among the chemical treated fiber composites, alkali and nMA treated fibers reinforced composites showed slightly higher Rockwell hardness values in 10 wt% fiber loading. In most cases, better wetting of fiber in polymer matrix gives higher hardness of composites [24]. The 10 wt% fiber loading alkali and nMA treated fibers are distributed compactly in composites. Higher percentage of fiber does not well disperse into the matrix.

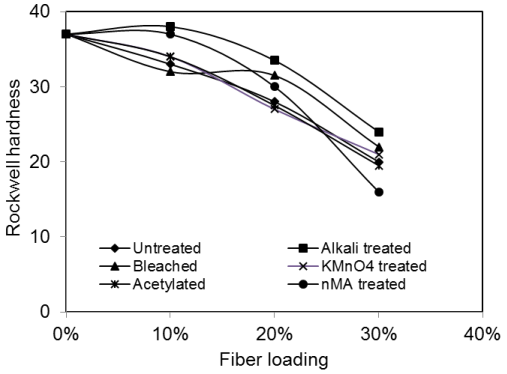

Fig. 2 Rockwell hardness C-scale of 10 wt% SDF loaded composites

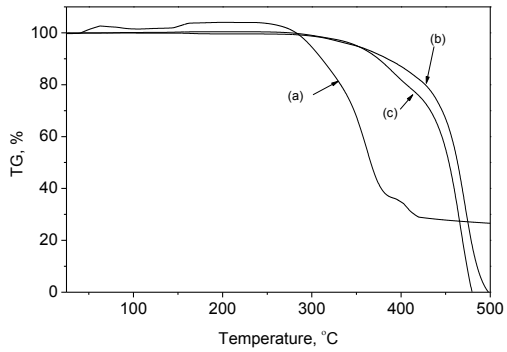

Fig. 3 TG curves of (a) untreated fiber, (b) PP and (c) 10 wt% untreated SDF-PP composites

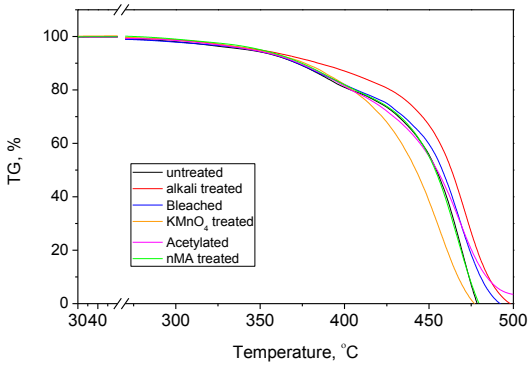

Fig. 4 TG curves of 10wt% fiber loaded composites

3.4 Thermal Properties of the Composites

Fig. 3 showed the percentage of weight loss of untreated SDF, PP and untreated SDF-PP composites with temperature. The TG curves of untreated SDF showed weight loss before main decomposition. The degradation of SDF can be divided into three steps (30-150 °C, 150-280 °C and 280-400 °C). The moisture presence in SDF is removed at 30-150 °C, hemicelluloses and low molecular weight fractions are decomposed at 150-280 °C, cellulose together with lignin are decomposed at 280-400 °C as well. The TG curves of PP and composite looks solid before 320 °C, thereafter decomposition starts and continues upto 480 °C. The decomposition temperature of virgin PP is slightly higher than composite. At very high temperature the linkage between fiber and PP may be broken down. Therefore, composite has lesser thermal stability than PP. However, no weight loss is found before 320 °C for the composite. The fiber in composite does not show the decomposition nature like individual fiber.

The thermal stability of 10 wt% (untreated, bleached, alkali treated, $KMnO_4$ treated, acetylated and nMA treated) fiber loaded composites are shown in Fig. 4. All TG curves of the fiber loaded composites shoed similar trend with two steps degradation. The first step degradation found at 280-400 °C due to SDF together with PP of composites. The second step degradation is found for PP at 400-490 °C. Among the composites, alkali treated fiber reinforced composite revealed higher thermal resistance probably due to the better adhesion with PP matrix. The residual char content is found almost zero at 500 °C for PP and composites attribute the absence of inorganic matter in composite though SDF contain a significant amount of residue.

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4. Conclusion

The properties of SDF-PP composites with fiber loading (0, 10, 20 and 30 wt%) were examined by tensile, water absorption, Rockwell hardness and thermogravimetric analyses. 10 wt% fiber loaded composite showed higher tensile strength. The chemical modification of the fiber surface increases the mechanical properties of composites. Among the treated SDF reinforced PP composites, nMA grafted fiber composite showed better tensile strength, tensile modulus and elongation at break. nMA grafted fiber composites showed 11.2% increase in tensile strength compare to untreated fiber composite. Bleached, alkali treated, $KMnO_4$ treated and acetylated SDF composites also show better tensile strength and modulus than untreated fiber composites. Rockwell hardness value is found decreases with the increases of fiber loading in composite. The thermal stability of the composites is also improved.

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