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Coir Fiber Reinforced Polypropylene Composites: Physical and Mechanical Properties

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Abstract

Coir fiber reinforced polypropylene composites were manufactured using a single extruder and an injection molding machine. Raw coir was chemically treated with benzene diazonium salt to increase its compatibility with the polypropylene matrix. Both raw and treated coir fiber was utilized and five levels of fiber loading (15, 20, 25, 30 and 35 wt%) were used during composite manufacturing. Microstructural analysis (Fourier transform infrared spectroscopy and scanning electron microscopy) and mechanical (tensile, flexural, impact, hardness and water absorption) tests were conducted. Chemically treated coir fiber reinforced specimens yielded better mechanical properties compared to the raw ones. For the fiber loaded samples, 30% fiber reinforced composites had the optimum set of mechanical properties. Authors propose that the bonding between the polypropylene matrix and chemically treated coir fiber must be increased in order to have improved mechanical properties at higher fiber content.

Keywords

Coir fiber, polypropylene, composite, physical properties, mechanical properties

1. Introduction

There has been an increasing interest recently in the use of biodegradable polymers, due to the serious environmental pollution arising from consumed plastics with time. Biodegradable polymers may be obtained from renewable resources, can be synthesized from petrobased chemicals or microbially synthesized in the laboratory [1]. Polypropylene (PP) is one of the most extensively used plastics both in developed and developing countries as it provides advantages in regard to econ-

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omy (price), ecological (recycling behaviour) and technical requirements (higher thermal stability).

Today, synthetic polymers are combined with various reinforcing fillers in order to improve the mechanical properties and obtain the characteristics demanded in actual application [2–4]. Research is going on in order to replace synthetic fibers with lignocellulosic fibers as reinforcing fillers [5–9]. The lignocellulosic fibers (corn stalk, rice husk, rice straw, coir, palm, jute, abaca, saw dust, wheat straw and grass) are lightweight, decrease wear in the machine used for their production, easily available, renewable and inexpensive. Furthermore, they are biodegradable and do not leave residues or result in by-products that are toxic [1, 10]. The cost of producing lignocellulosic polymeric composites is quite low. Hence these composites have attracted much attention and are becoming increasingly important for the production of a large variety of cheap light-weight environmentally friendly composites [11].

In current work, coir fiber was used as the reinforcing material since it is produced in large scale in the Indian sub-continent, especially Bangladesh and has a minimal effect on the environment because of its biodegradable properties [12–15]. So far, only little work has been conducted on chemical treatment of coir fiber to improve the mechanical properties of the coir fiber reinforced polymer composites [12]. In the current work, raw coir fiber was chemically treated with benzene diazonium salt to increase the compatibility of the coir fiber with the polypropylene matrix. Thus the aim of this study is to manufacture composites from raw and treated fiber and bio-degradable PP and subsequently characterize those using microstructural analysis and mechanical testing. The effect of the fiber loading on the mechanical properties and morphology of the coir fiber reinforced PP are also reported.

2. Materials and Methods

2.1. Materials

The thermoplastic polymer polypropylene (PP), used as matrix material, was supplied by the Polyolefine Company, Private Limited Singapore in the form of homopolymer pellets. It had specific gravity of 0.90–0.91, melting temperatures of 165–171°C and crystallinity of 82% [16]. The coir, used as reinforcing fiber, was collected from a rural area of Bangladesh. It is composed of 43.44% cellulose, 45.84% lignin, 0.25% hemicellulose, 3% pectin, 5.6% ash and 7.47% other materials [17]. The diameter of the coir fiber used was 400 μm. Chemicals used in this study to treat coir were HCl (Merck, Germany), $NaNO_2$ (Merck, Germany), $C_6H_5NH_2$ (Merck, Germany) and NaOH (Merck, India).

2.2. Treatment of Coir Fiber

Benzene diazonium salt was synthesized by the standard diazotization method. Coir fiber was dried at 105°C for 24 h in order to reduce its moisture content to 1–2% and then kept in a sealed container. 1000 ml 5% NaOH solution was taken in a 2000 ml beaker. 500 g coir was submerged into the solution for 10 min at about 5°C in an ice bath. A freshly prepared cooled solution of benzene diazonium salt was then poured slowly into the above mixture with constant stirring and kept for about 10 min. Coir was then taken out, washed with soap solution followed by water and finally dried in open air.

2.3. Fabrication of Composites and Test Specimens

Coir fiber was cleaned and chopped into an approximate length of 3 mm. Dried coir was randomly mixed with PP granules in order to prepare the composites. Both raw and treated coir was taken with the PP matrix at 15, 20, 25, 30 and 35 wt% separately. Composites were prepared by passing the mixtures through a single screw extruder machine at a constant temperature of 135°C ± 5°C. The extruded composites were cut into 15–20 cm long small pieces. All the pieces were then crushed into smaller granules using a grinding machine (Model FFC-23, Machinery Company Limited India). The granules were dried in a vacuum oven at 65°C for 1 h and fed into an injection molding for making specimens.

The dried granulates were molded according to DIN EN ISO 572-2 Type 1A and DIN EN ISO 179, Type 1 using the injection molding machine at a molding temperature of 165°C in order to prepare tensile and flexural test specimens. Molds with single cavity were used during the process.

2.4. Micro-structural Analysis

2.4.1. Fourier Transform Infrared Spectroscopy (FT-IR)

The infrared spectra of the raw and treated coir were recorded on a Nicolet 380 Spectrophotometer at a scan range of 4000–400 and resolution of $<0.5\ \text{cm}^{-1}$. The obtained spectra are discussed in the Results and Discussion section.

2.4.2. Scanning Electron Microscopy (SEM)

The interfacial bonding between the coir fiber and PP matrix in manufactured composites and tensile and flexural fracture surfaces of the same composites were examined using a Scanning Electron Microscope (Philips XL 30). The micrographs, taken at a magnification of 300, are presented in the Results and Discussion section.

2.5. Mechanical Testing

Tensile, flexural, charpy impact, hardness and water absorption tests were conducted. For each test and type of composite, ten specimens were tested and the average values are reported.

2.5.1. Tensile Test

Tensile tests were conducted according to ASTM D 638-01 [18] using a Universal Testing Machine (Model: MSC-5/500, Agawn Seiki Company Limited, Japan). The tests were performed at a crosshead speed of 10 mm/min. The dimensions of the specimens used were 148 mm × 10 mm × 4.1 mm.

2.5.2. Flexural Test

Static flexural tests were carried out according to ASTM D 790-00 [19] using the same Testing Machine mentioned above at same crosshead speed. The dimension of the specimen used was 79 mm × 10 mm × 4.1 mm. The flexural strength and modulus were calculated using the following equations:

$$\text{Flexural strength} \quad \sigma_{\mathrm{f}} = \frac{3PL}{2bd^2}, \tag{1}$$

$$\text{Flexural modulus} \quad E = \frac{L^3 m}{4bd^3}, \tag{2}$$

where P is the maximum applied load, L is the length of support span, m is the slope of the tangent, b and d are the width and thickness of the specimen, respectively.

2.5.3. Charpy Impact Test

Dynamic charpy impact tests were conducted according to ASTM D 6110-97 [20] using a Universal Impact Testing Machine. Notched composite specimens were used during the experiment. The dimensions of the specimen used were 79 mm × 10 mm × 4.1 mm.

2.5.4. Water Absorption Test

In order to measure the water absorption characteristics of the composites, rectangular specimens were prepared having dimensions of 39 mm × 10 mm × 4.1 mm. The specimens were dried in an oven at 105°C, cooled in a desiccator using silica gel and immediately weighed. A Denver Instron balance was used for weight measurement. The dried and weighed specimens were immersed in cold and hot distilled water according to ASTM D 570-99 [21] for 24 and 2 h, respectively. After immersion, the excess water on the surface of the specimens was removed using a soft cloth. The final weight of the specimens was then taken. The increase in the weight of the specimens was calculated using the following equation:

$$\text{Water absorption (\%)} = \frac{\text{Final weight} - \text{Original weight}}{\text{Original weight}} \times 100. \tag{3}$$

2.5.5. Hardness Test

The hardness of the composites was measured using a Rockwell Hardness Testing Machine according to ASTM D 785-98 [22]. The Rockwell hardness values were 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 returned to a minor load. The indenter had a diameter of 6.35 mm, while the values of the major applied load was 490 N. Results are shown in the following section.

3. Results and Discussion

3.1. Structural Analysis

In order to improve mechanical properties of the composites, coir was chemically treated using benzene diazonium salt. The hydroxyl group in the raw coir is responsible for high water absorption and weak interfacial bond between the coir fiber and PP matrix. There are actually three hydroxyl groups present in a cellulose anhydroglucose unit. One is a primary hydroxyl group at C_6 and the other two are secondary hydroxyl groups at C_2 and C_3. Although the primary hydroxyl group is more reactive than the secondary groups, the diazonium salt breaks the OH group of carbon 6 and carbon 2 during the reaction. This converts the two hydroxyl groups into diazo groups and results in an azo product, 2, 6-diazo cellulose, as illustrated in Fig. 1 [23]. The FT-IR spectroscopic analysis of the raw (Fig. 2) and treated (Fig. 3) coir confirms this phenomenon. The IR spectrum of treated coir

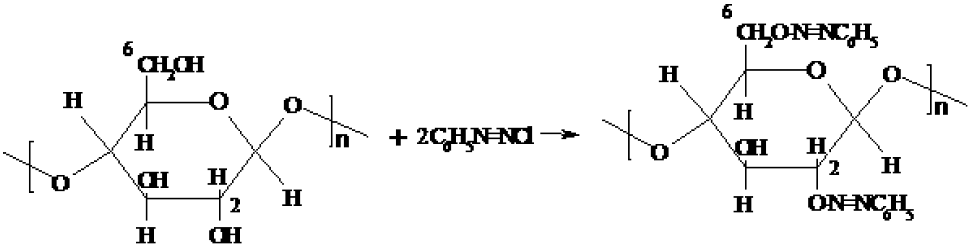

Figure 1. Treatment of cellulose in coir with diazonium salt.

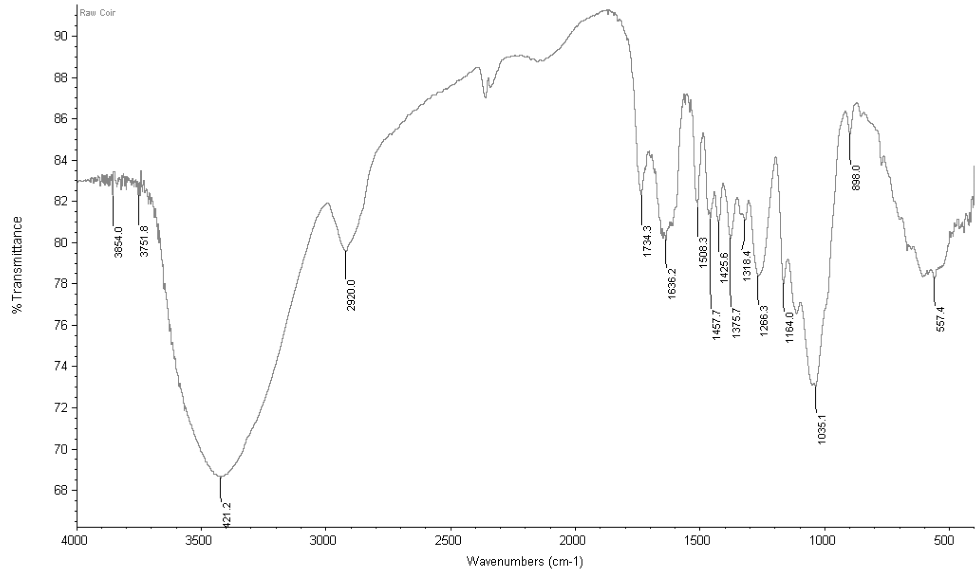

Figure 2. FT-IR spectrum of raw coir.

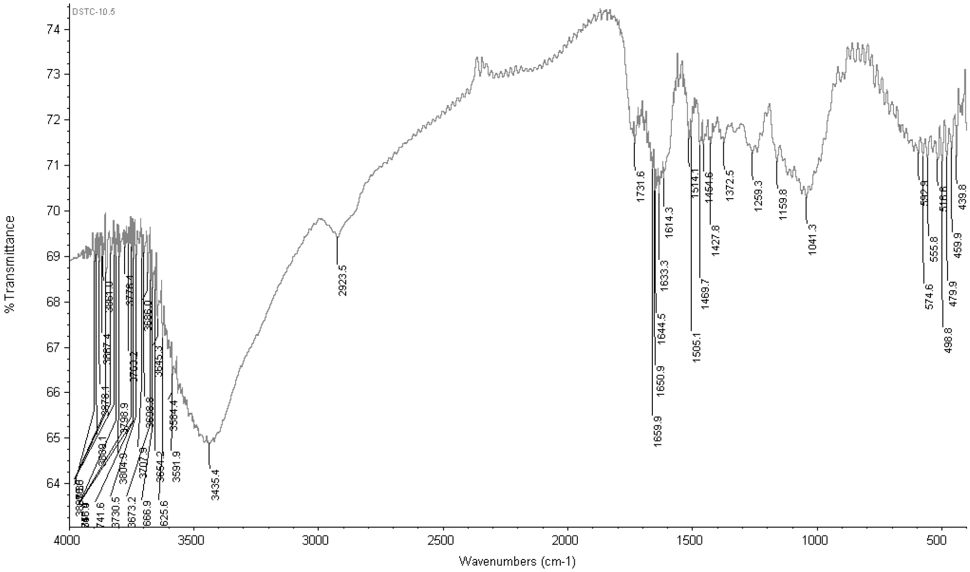

Figure 3. FT-IR spectrum of treated coir.

clearly shows the presence of the characteristic band of the NO group in the region of 1600–1700 cm^{-1}, an absorption band of N=N stretching near 1614 cm^{-1} and a C–O stretching band at the region of 1300–1000 cm^{-1}. Again the IR spectrum of the raw coir shows the absorption band at the region near 1732 cm^{-1}. This absorption band may be due to the carboxyl group of acetylester in cellulose and the carboxyl aldehyde in lignin [24].

3.2. Tensile Properties

The tensile strength of the raw and treated coir fiber reinforced PP composites at different fiber loading is shown in Fig. 4. The tensile fracture behaviour of the composites was ductile. For the raw coir fiber reinforced composites, the tensile strength decreased with fiber loading [2, 3, 5, 7, 25, 26], which is in accordance with the findings other researchers. As the fiber load increased, the weak interfacial area between the fiber and matrix increased, which consequently decreased the tensile strength.

In order to increase the compatibility of the coir fiber with PP matrix, raw coir fiber was chemically treated with benzene diazonium salt. The reaction took place deep down in the specimen, as evidenced by improvement of the mechanical properties described later. The dimensions of the specimen remained the same after reaction, which proves that there was no detrimental effect of the reaction on the deformation behaviour of the composites. The chemical treatment increased the tensile strength of the 15% fiber loaded treated composites compared to the PP matrix itself (0% coir fiber). These results suggest that stress is expected to transfer from the matrix to the fiber, indicating a better interfacial bonding with a consequent

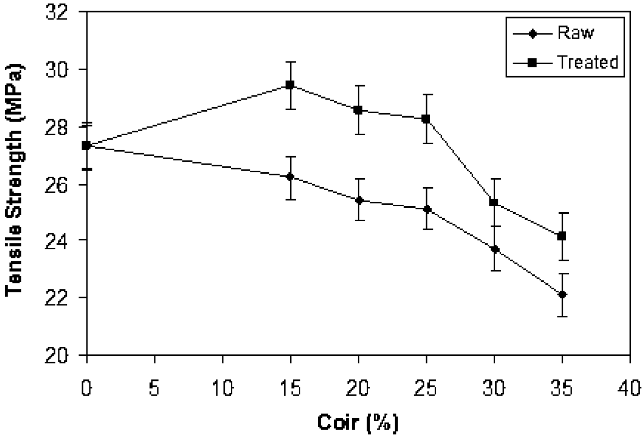

Figure 4. Variation of tensile strength at different fiber loading.

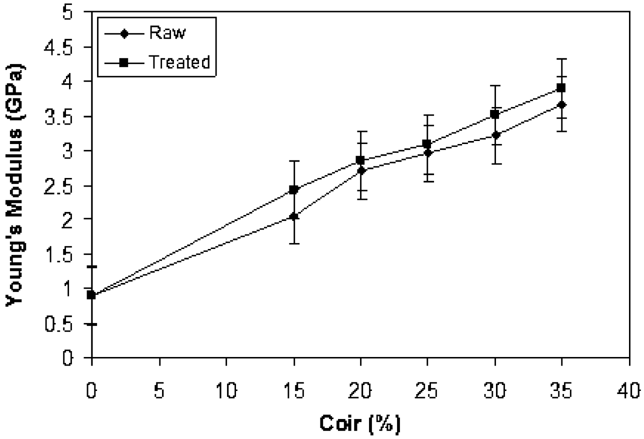

Figure 5. Variation of the Young's modulus at different fiber loading.

improvement in the mechanical properties. The tensile strength of the chemically treated composites decreased with fiber loading due to the same reason mentioned above. It is found that the tensile strength increased approximately 6.8% to 12.5% over the raw coir fiber composites. The range of the tensile strength obtained in the current work is (22.1–29.43) MPa, which is higher than the range obtained in previous research (5 to 25 MPa) using the same fiber material [13, 14]. This could be due to better interfacial bonding between the coir fiber and PP matrix obtained in current research, which subsequently improved the tensile strength.

Figure 5 shows the variation of the Young's modulus at different fiber loading. The Young's modulus increased with fiber loading in accordance with the results of other researchers [5, 7, 8, 26, 27]. During tensile loading, partially separated micro-spaces are created, which obstructs stress propagation between the fiber and matrix [28]. As the fiber load increases, the degree of obstruction increases, which consequently increases the stiffness. The Young's modulus of the treated composites was higher than those obtained for the raw ones. It is found that the Young's modulus increased from 4.4% to 16.5% over the raw coir fiber composites. The range of the Young's modulus found in current work is (2.05–3.89) GPa, which is higher than those obtained in previous research (0.56–1.3 GPa) using the same fiber material [13, 14].

3.3. Flexural Properties

Flexural strength and modulus of the raw and treated coir fiber composites at different fiber loading are shown in Figs 6 and 7 respectively. The flexural fracture behaviour of the composites was ductile. The flexural strength increased with fiber loading [8, 27]; however, there was a decrement from 30% to 35% fiber loaded composites (Fig. 6). It is found that the flexural strength increased approximately

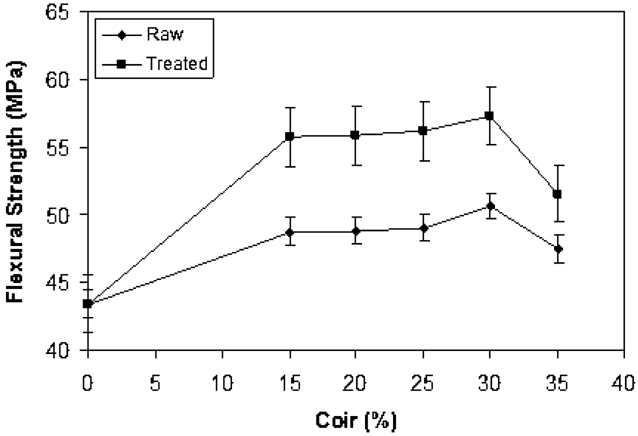

Figure 6. Variation of flexural strength at different fiber loading.

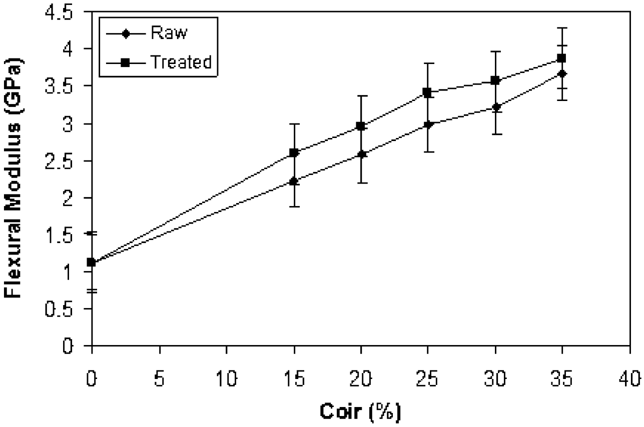

Figure 7. Variation of flexural modulus at different fiber loading.

8.6% to 14.6% over the raw coir fiber composites. The range of the flexural strength obtained in current research is (47.45–57.32) MPa. According to Fig. 7, flexural modulus increased with fiber loading as found by other researchers [8, 25, 27]. Since coir fiber is a high modulus material, higher fiber concentration demands higher stress for the same deformation. Increased fiber–matrix adhesion provides increased stress transfer between them. The flexural modulus increased approximately 3.9% to 16.1% over the raw coir fiber composites. Again the range of the flexural modulus obtained in current research is (2.23–3.87) GPa.

3.4. Impact Strength Results

Variation of the charpy impact strength with fiber loading for both raw and chemically treated fiber composites is shown in Fig. 8. Impact strength increased with fiber loading [27, 29, 30]. However, there was a decrease in the impact strength from 30% to 35% fiber loaded composites. The impact strength of the fiber reinforced polymeric composites depends on the nature of the fiber, polymer and fiber–matrix interfacial bonding [31]. It has been reported that high fiber content increases the probability of fiber agglomeration which results in regions of stress concentration requiring less energy for crack propagation [32]. As presented in Fig. 8, impact strength of all composites increase with fiber loading. This result suggests that the fiber was capable of absorbing energy because of strong interfacial bonding between the fiber and matrix. Another factor that influences the impact failure of a composite is fiber pull-out. With increase in fiber loading, a larger force is required to pull out the fibers. This consequently increased the impact strength. The impact strength of the treated composites was 3.9% to 10.1% higher than that of raw composites, while the manufactured composites yielded impact strength values in a range from 38.75 J/m to 56.12 J/m.

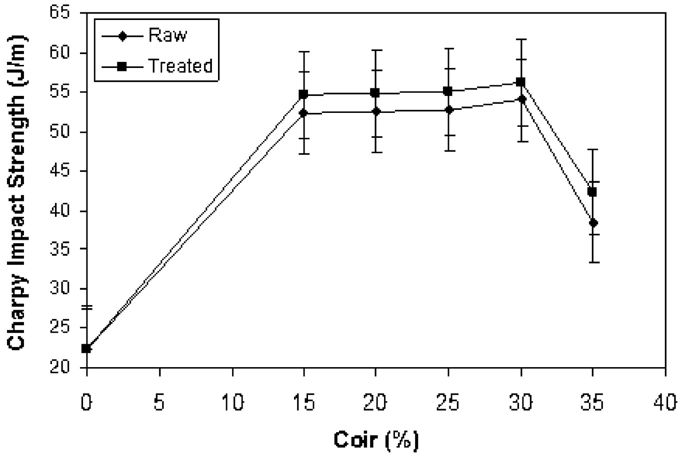

Figure 8. Variation of impact strength at different fiber loading.

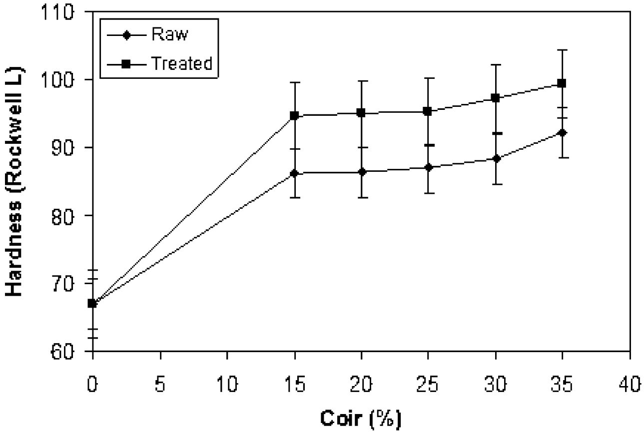

Figure 9. Variation of hardness at different fiber loading.

3.5. *Hardness Results*

Figure 9 shows the hardness of various manufactured composites at different fiber loading. Average hardness increased with chemical treatment of coir fiber and fiber loading [33]. This is due to the increase of stiffness of the respective composites. It is found that hardness values increased from 7.7% to 10.1% over the raw coir fiber composites. The range of hardness obtained in current work is 86.29–99.32 R_L.

3.6. *Water Absorption Characteristics*

Water absorption characteristics of manufactured composites against fiber loading are shown in Figs 10 and 11. Figure 10 shows the results for cold water immersion, while hot water immersion results are shown in Fig. 11. Water absorption (%) increased with an increase in fiber loading [2, 23]. As mentioned earlier, the hydroxyl groups in coir fiber are responsible for high water absorption. With increase in fiber loading, the number of hydroxyl groups in the composites increased, which in turn increased the water absorption. Chemically treated coir fiber reinforced composites had lower water content compared to the raw ones, as the treatment decreased the number of hydroxyl groups in the resultant composites. Water absorption (%) decreased approximately from 25% to 28.6% over the raw composites.

3.7. *SEM Morphology*

SEM micrographs of the raw and treated 20% coir fiber composites are shown in Figs 12 and 13, respectively. The SEM image of raw coir fiber PP composites (Fig. 12) shows agglomeration of untreated coir in the PP matrix. This feature suggests weak interfacial bonding between the fiber and matrix. On the other hand, chemically treated coir fiber PP composites (Fig. 13) show better dispersion of the fiber into the matrix. This resulted in a better interfacial bonding between the fiber and matrix.

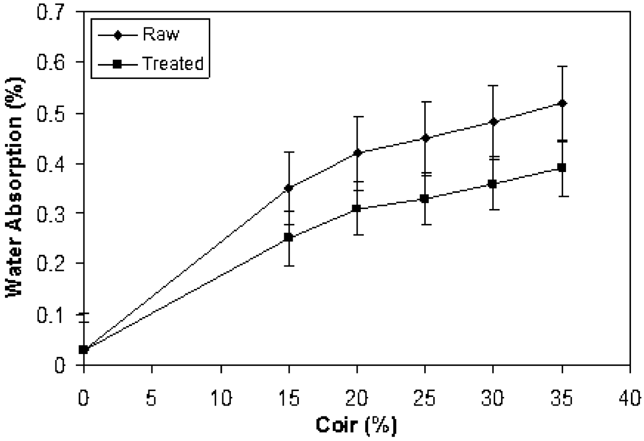

Figure 10. Variation of cold water absorption at different fiber loading.

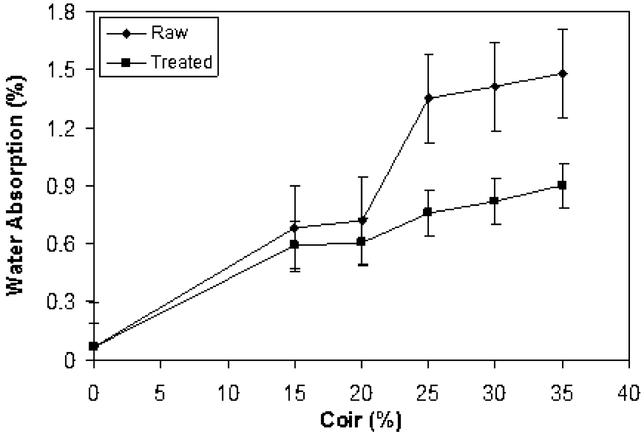

Figure 11. Variation of hot water absorption at different fiber loading.

SEM micrographs of the tensile and flexural fracture surfaces are shown in Figs 14 and 15, respectively. Figures 14 and 15 show uneven rough surfaces, which prove that both the tensile and flexural fracture of the coir fiber reinforced polypropylene composites were ductile.

Tensile fracture surfaces of the raw and treated coir fiber composites are shown in Figs 14 and 16, respectively. Both figures show uneven rough surface, which are proofs of ductile fracture behaviour of the coir composites. Compared to the fracture surface of the raw composite, the fracture surface of the treated composite found is smoother. This indicates that the fracture behaviour of the treated fiber composite was less ductile compared to that of the raw fiber composite.

According to the results discussed above, diazonium salt chemical treatment enhanced the mechanical properties of the coir fiber reinforced PP composites by improving the interfacial bonding between the coir fiber and PP matrix. Based on

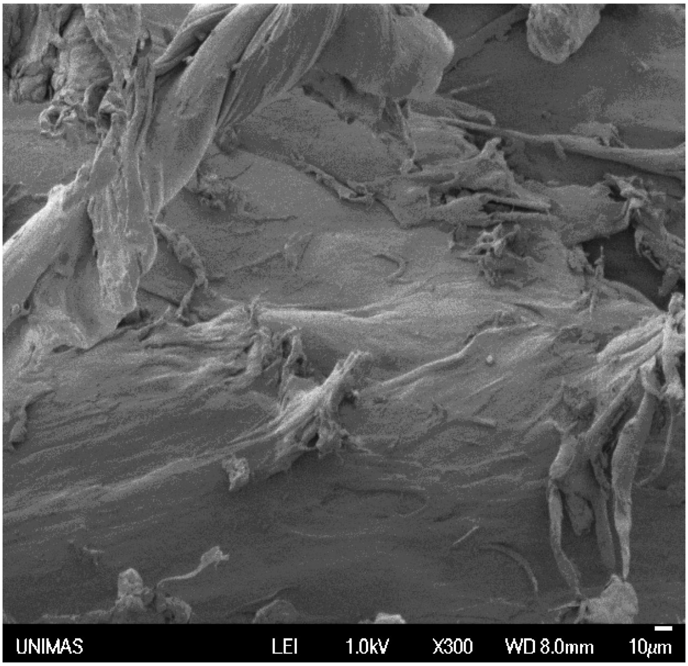

Figure 12. SEM micrograph of 20% raw coir fiber reinforced PP composite.

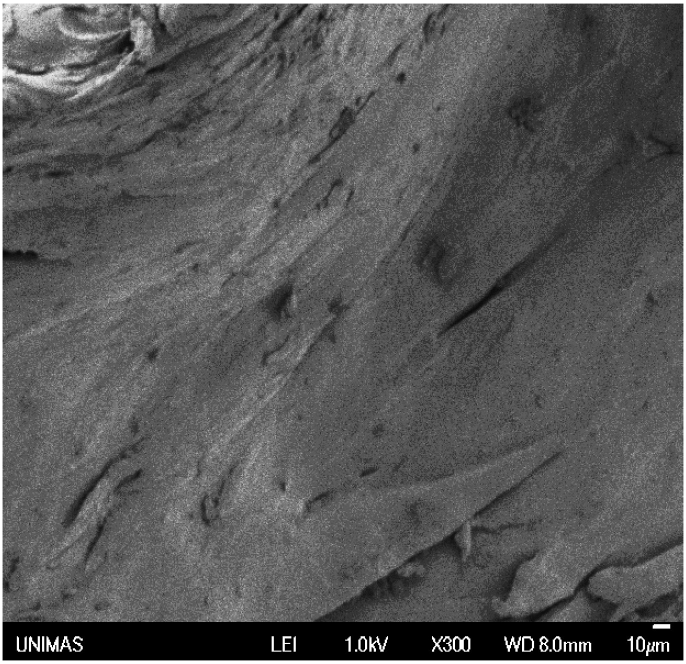

Figure 13. SEM micrograph of 20% treated coir fiber reinforced PP composite.

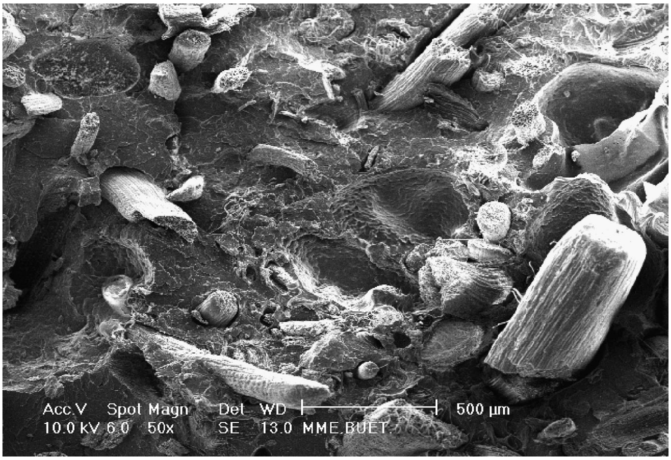

Figure 14. Tensile fracture macrograph of 20% raw coir fiber reinforced PP composite.

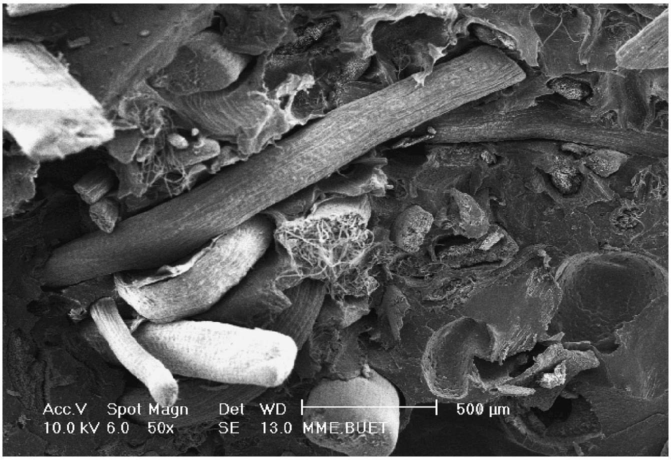

Figure 15. Flexural fracture macrograph of 20% raw coir fiber reinforced PP composite.

the fiber loading, 30% fiber reinforced composites had the optimum set of mechanical properties. When fiber content was increased to 35%, the flexural and charpy impact strength decreased. The authors propose that the bonding between the PP matrix and chemically treated fiber must be increased in order to have improved mechanical properties at higher fiber content. This may be achieved by chemical modification of the PP matrix, thereby changing its hydrophobic nature to hydrophilic nature.

4. Conclusion

In the current work, coir fiber reinforced PP composites were manufactured using a single extruder and injection molding method. Raw coir was chemically treated

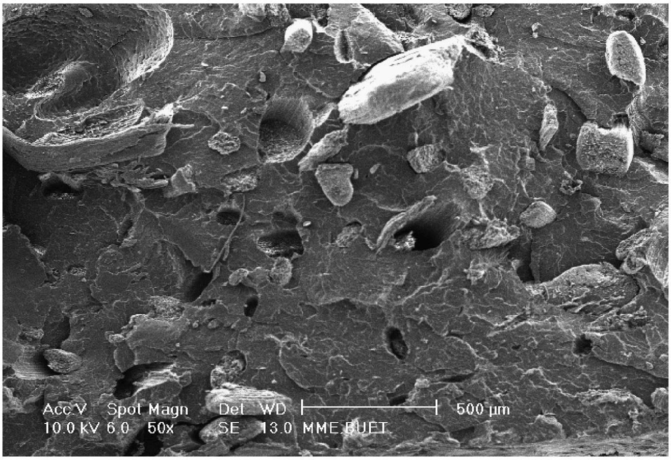

Figure 16. Tensile fracture macrograph of 20% treated coir fiber reinforced PP composite.

with benzene diazonium salt to increase the compatibility of the coir fiber with the PP matrix. A significant effect of the chemical treatment is observed on the physico-mechanical properties and micro-structural analysis. The following conclusions can be drawn from the experimental results of this study:

(i) The tensile strength of the composites decreased with an increase in the coir fiber loading. However, there was an increase in the tensile strength of the 15% treated coir fiber reinforced composite compared to the PP alone.

(ii) The Young's modulus, flexural strength, flexural modulus, charpy impact strength and hardness of the composites increased with an increase in the fiber loading. However, the 35% fiber loaded composites had lower flexural and impact strength compared to the 30% ones.

(iii) The tensile strength, Young's modulus, flexural strength, flexural modulus, impact strength and hardness of the diazonium salt treated coir fiber reinforced composites were higher than those of the raw ones.

(iv) The authors propose that the 30% coir fiber reinforced PP composites had the optimum set of mechanical properties in comparison with other manufactured composites. To have better mechanical properties at higher fiber content, the bonding between the coir fiber and PP matrix must be improved. Chemical modification of the PP matrix can achieve this by changing its nature from hydrophobic to hydrophilic.

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