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Original Research

Surface Modified Benzoylated Okra (*Abelmoschus esculentus*) Bast Fiber Reinforced Polypropylene Composites

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Abstract: Nonwoven unidirectional Okra fiber (OF)-reinforced polypropylene (PP) based composites were manufactured by a hot press machine and their interfacial, physicomechanical features, thermal degradation, and weather test were examined. This paper reports the effect of OF content, alkaline treatment of fiber, as well as inclusion of benzoylation as coupling agent on the features of PP/OF composites, was studied. The outcomes indicated that the OFs performed as reinforcing fillers, developing the mechanical features (e.g., tensile strength, tensile modulus, impact strength, and hardness) of the composites. PP/OF composites with benzoylation after alkaline pretreatment exhibited better mechanical features than alkali treatment and untreated composites. The interfacial feature was examined by scanning electron microscopy (SEM) and it was found that the interfacial interaction between PP and OF was increased due to the treatment of the fibers, which validates the obtained mechanical features of the composite. Moisture absorption experiments have shown that benzoylation treatment of OF composites shows lower water absorption than both alkali-treated and untreated composites. Thermal degradation and weather tests of the composites were also investigated.

Keywords: Okra bast fiber; Polypropylene; Composite; Alkali-treatment; Mechanical properties.

Introduction

Recently polymer composites have been widely used in a wide variety of fields due to their great and unique combination of physical and mechanical properties and they are widely used in civil construction, chemical equipment and machinery construction, electrical and electronic equipment, automobile and marine industries, aircraft manufacturing and many more [1-4]. Many studies have been done on the synthetic matrix-based synthetic fiber (e.g., glass, carbon, nylon, and Kevlar) reinforced composites [5, 6]. Synthetic fiber reinforced thermoplastic/thermoset composites are influential on natural fiber reinforced composites due to their advanced strength, durability, corrosion, and moisture resistance features. Scientists prefer thermoplastic composites over

thermosets because of lower production costs and lower processing. The use of synthetic fibers is a great ecological problem because these fibers are not biodegradable. Composites made with ligocellulosic components as reinforcing fiber and thermoplastic/thermoset polymers as the matrix is explored day by day due to increased environmental awareness.

Natural fibers (NFs) have some advantages over conventional plastic or synthetic components in terms of low price, low-density, appropriate definite strength, renewability, recyclability, and biodegradability. Of all the natural fibers, we chose okra fiber because at present this fiber has no economic value because the plant burns. Okra bamia (botanical name Abelmoschus esculentus) is a monocotyledon herbaceous plant under the family of Malvaceae, which mainly exists in Bangladesh and some other tropical countries of the world. From okra bark, fibers can be extracted as nutrients added to gums and pectins for some use in the food sector. Nevertheless, the main use of okra fiber in components is limited to employing mucilage as a moisture absorber [7]. Okra is a source of mucilage polysaccharides, which can be used with chemical grafting suitable for the synthesis of biodegradable polymers [8]. The composition of OF is hemicellulose (15-20%), α-cellulose (60-70%), pectin (3-5%), lignin (5-10%), and some water-soluble substances related to jute, flax, pineapple leaf fibrous, etc. [9]. Cellulose is the most important chemical constituent of NFs, particularly in composite making [10]. These fibers will be considered for use in composites. Recent studies of their thermal and mechanical behavior have indicated okra fibers as potential candidates for the production use of biodegradable composites [11]. Although there are multiple advantages, cellulose fibers reduce adverse reactions to high temperatures and the omnipresence of moisture absorption. Hydroxyl groups represent cellulose to form various hydrogen bonds and make cellulosic fibers hydrophilic. Prepared composites, including nonpolar thermoplastic matrix and hydrophilic natural fibers, reduce the mechanical features due to the weakness between the plastic component and the fiber [12]. The mechanical features of the composites can be improved by modifying the natural reinforced fibers by chemical treatment [13]. Chemicals can effectively interlock with cellulose and activate the hydroxyl group that can induce the necessary properties of the polymer. Alkali treatment is a process that enhances mechanical properties. Alkali treatment involves the removal of lignin, hemicellulose, and pectin from hydrophilic substances that alter the status of hydrophobic substances. Due to the huge damage to hemicellulose, the fibers can lose their cement capacity and as a result, they become separated from each other and turned into fine [14].

In this study, PP was chosen as a thermoplastic resin because it has several outstanding features such as transparency, superior surface strength, high impact strength, high heat distortion temperature, and dimensional stability. PP is also very suitable for filling, reinforcing, and blending. PP with fibrous natural fibers is one of the potential routes to create natural synthetic polymer composites. In this present study, unidirectional PP/OF composites with various fiber contents were manufactured and their physicomechanical properties were evaluated. Both alkaline pretreatment and subsequent benzoylation treatment were used to increase the adhesion between these two dissimilar surfaces. The interfacial feature was evaluated by analysis of SEM. Water absorption capacity and weather tests were also observed for optimized composites to investigate the suitability of composites for wide-ranging applications.

Materials and Methods

The PP (trade name: Cosmoplene) used in this study was supplied by the Polyolefin Company, Private Ltd., Singapore. Sodium hydroxide (NaOH), benzoyl chloride, acetic acid, and ethanol were

collected from Sigma-Aldrich Chemical Co. Inc. (St. Louis, USA). Okra plant was collected from Manikganj District (Bangladesh). Five months later and about 2 m tall, trees were collected. After collection, the plant was placed underwater for bacterial decay. The fiber was separated from the stacks by water retting for about 15 days. Then the fibers were washed several times using distilled water. They were dried in the open air and reserved in clean containers.

To remove natural impurities (lignin, pectin, and wax), the fibers were mercerized at 60 °C for 30 min using a 10% NaOH solution. Mercerized fibers were then thoroughly cleaned several times in water to detect any traces of NaOH. However, the final washing was done with 2% acetic acid to handle the last trace of NaOH. Finally, the washed and dried fibers were reserved in an air dryer for 24 h. The treated fibers were suspended in a 10% NaOH solution and stirred vigorously with 50 ml benzoyl chloride. The mixture was allowed to stand for 15 min, then filtered, washed with water, and dried at room temperature. The separated fibers were soaked in ethanol for 1 h to remove unwanted benzyl chloride and finally washed with water and dried in an oven at 60 °C for about 24 h. A set of formulations was arranged and the formulated compositions are presented in Table 1.

Table 1: Formulations of untreated and treated PP/OF composites.*

Ingredients (wt%)	Sample designation		
	PP/OF	**PP/AOF**	**PP/ABOF**
PP	100	100	100
OF	0, 20, 30, 40, 50	–	–
Alkalized OF	–	0, 20, 30, 40, 50	–
Alkali pretreated benzoylated OF	–	–	0, 20, 30, 40, 50

*PP: Polypropylene, OF: Okra fiber.

Preparation of OF-Reinforced PP Composite

The PP sheet was made from PP pellets using a hot press machine at 170 °C with a pressure of 8 MPa for 5 min. In the manual rotation method, the OFs were set lengthwise on the PP sheet. PP sheet layers (PL) stacked OF layers (OFL) were formed in steps similar to PL-OFL-PL..., the outer layers were made by two layers of PP sheet. Composite samples were made using a hot press at 180 °C for 10 min at a pressure of 10 MPa and then cooled to another press with the help of two steel plates. The thickness of the resulting composites was kept at 2 mm. Figure 1 presents the processing method of PP/OF composite and the temperature-time-pressure profile used for composite production.

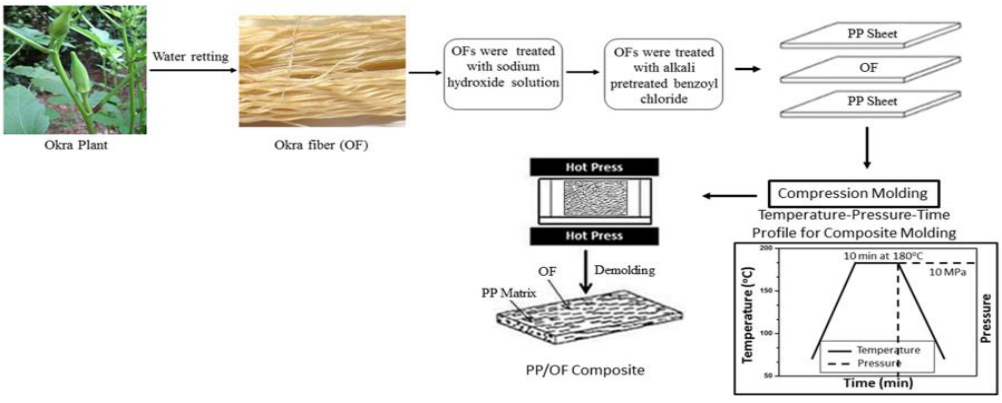

Figure 1: The schematic illustration of the manufactured PP/OF composite and its processing profile.

Characterization

Tensile features of the composite sample were measured using Shimadzu UTM (Model AG-1, Japan) with electrical weight cells of 6 kN following the ASTM-D 638-03 standard. The tensile test was achieved at a crosshead speed of 10 mm/min and a gauge length of 20 mm. According to ASTM-D 256 standards, the Izod impact strength was performed using an impact machine (model, Toyo Seiki Co., Japan). The dimensions of the sample were 63.5 × 12.7 × 3 mm^3. The hardness of the composite samples was tested by an HPE Durometer (model type 60578, Germany) according to DIN 53505 standard. The mechanical features of the unreinforced PP sheet were also tested according to the above-mentioned. All experiments were achieved at 23°C ± 2°C and relative humidity of 50 ± 5 %. Reported values averaged over five measurements. The SEM micrographs of untreated and treated composite samples were analyzed by a Zeiss, Evo 50 scanning electron microscope. The fracture edges of the specimens were embedded in an aluminum spit and covered with a thin layer of gold to disperse the electric charge throughout the test. Water absorption of untreated and treated composite samples was determined by dipping the sample in a glass beaker covering water at 23 °C. After a constant time break, the sample was taken out of the water, drained, and weighed. To determine the thermal aging, a thermo-stated oven was selected and the test was continued for 30 days. The model of the device was Denver, AA-160. After a certain time (5 days), the samples were taken out from the oven and the tensile features were kept at 25 °C for 24 h. The composite sampling was conducted by the Accelerated Weathering Tester (Model Q-U-V, Q-Panel Company, USA). Untreated, alkaline treatment and alkali pretreated benzoyl chloride treated composite specimens were tested. The treatment differed between 60 °C ± 2 °C (sunshine) and 40 °C ± 2 °C (condensation) over 4 h of sunshine and 2 h of condensation for a period of 600 h. The specimens were dried in an oven for 0.5 h and measured in their tensile properties due to weather testing.

Results and Discussion

Mechanical Features of the Composites

The mechanical features of composites strongly affected the fiber-matrix interfacial bond strength, the nature of the fiber and the matrix, and the role of the fiber load. Dispersion and interfacial bond between hydrophobic matrix and hydrophilic filler to determine composite mechanical features.

Tensile Features; Effect of OF Loading

Figures 2 (a) and 3 (a) display the tensile strength and tensile modulus of untreated and treated OF-reinforced PP composites as a function of OF loading. The tensile strength and modulus of virgin PP were found to be 24.4 MPa and 533.5 MPa, respectively. To untreat OF reinforced composites, the tensile strength of the composites increased from 28.3 MPa to 35.3 MPa as the fiber contents increased from 20 wt% to 40 wt%. According to other researchers 15, 16, tensile strength increased with fiber loading. This conduct is mainly accredited to the transfer of even stress from the matrix to the fiber. However, the inclusion of more fiber (50 wt%) did not increase the tensile strength. Exceeding 40%, a declining trend in tensile strength has been observed, possibly due to the effect of an agglomeration on poorly dispersed fibers in the polymer matrix at higher fiber loading, resulting in a non-uniform transition to applied stress and weaker fibers-matrix adhesion. In contrast, the inclusion of OFs in variable fiber loading from 20 wt% to 40 wt% resulted in a steady increase in composite tensile modulus (Figure 3 (a)). As the OFs increased from 20 wt% to 40 wt%, the tensile modulus increased to 29%, 55%, and 94% with 688.4 MPa, 825.5 MPa, and 1033.7 MPa,

respectively, compared to the virgin PP. The increase in tensile modulus may be related to the increase in stiffness of the composites due to OF loading. Loaded composites of 50 wt% fiber show tensile modulus reduction. Deviation of higher fiber loading may occur due to fiber packing and insufficiently rich polymer regions. Moreover, fiber entanglements and agglomeration result in composite, which reduces the stress transfer between the matrix and the fiber. If the matrix was scarcely available, the fibers were no longer surrounded by the matrix at high fiber loads, and voids were produced in the composite. It is known that most of the features of composites were affected by the presence of voids [17]. As can be seen from Figures 2 (a) and 3 (a), the tensile strength and modulus of the composite prepared with 50 wt% OF was less than in the sample with 40 wt% OF. That is, 40 wt% OF was selected to study the effect of fiber modification on composite features.

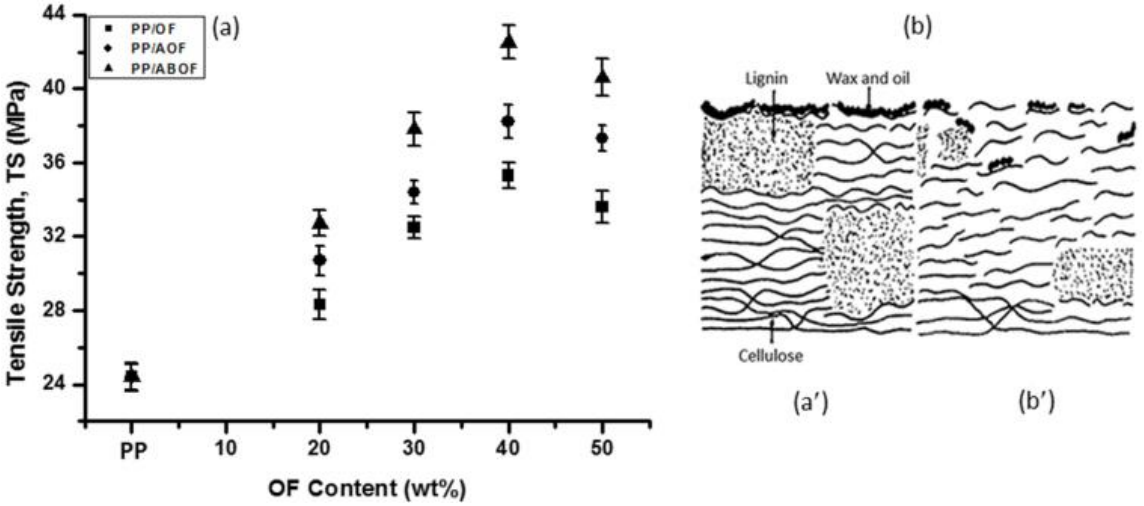

Figure 2: (a) Tensile strength of PP/OF, PP/AOF, and PP/ABOF composites at different fiber loading; **(b)** Characteristic structure of **(a')** untreated and **(b')** alkaline cellulosic fibers.

Effect of Alkali Treatment

The effect of alkaline treatment on the tensile strength of PP/AOF composite is shown in Figure 2 (a). As the load level increased from 20 to 40 wt%, the tensile strength of the PP/AOF composite increased from 26 %, 41 %, and 57 % to 30.7 MPa, 34.4 MPa, and 38.2 MPa, respectively, as compared with virgin PP, then decreased with a further increase in reinforcement (50 wt%). Alkali treatment removes a certain number of natural and artificial impurities such as lignin, hemicellulose, wax, and oil, covering the outer surface of the fiber cell wall (Figure 2b (a')), thus resulting in the better mechanical interlocking of the surface friendliness and increasing the surface roughness of cellulose exposed on the fiber surface. It indicates that alkalization depolymerizes native cellulose I molecular structure produces short-length crystallites (Figure 2b (b')). This allows a possible increase in the number of reaction sites and better wetting of the fiber, thus increasing the tensile strength. In addition, alkalization reduces the diameter of the fiber, increasing the aspect ratio which leads to the peak development of the rough surface which enhances the good fiber-matrix interface bonding and mechanical features [18]. The possible reaction of fiber and NaOH is shown in Figure 3b (a'). One-way fiber orientation requires less fiber overlapping of the fibers and thus pulls out the fiber, reducing fiber agglomerations. Air penetration was less likely due to one-way fiber orientation. The ingress of air leads to the formation of tip forms which leads to weak stress transfer between the fiber and the matrix. After 50 wt% alkalized OF load, the tensile strength of composite reduces. This may be due to the depletion of cellulose and the introduction of internal cracks in the fibers.

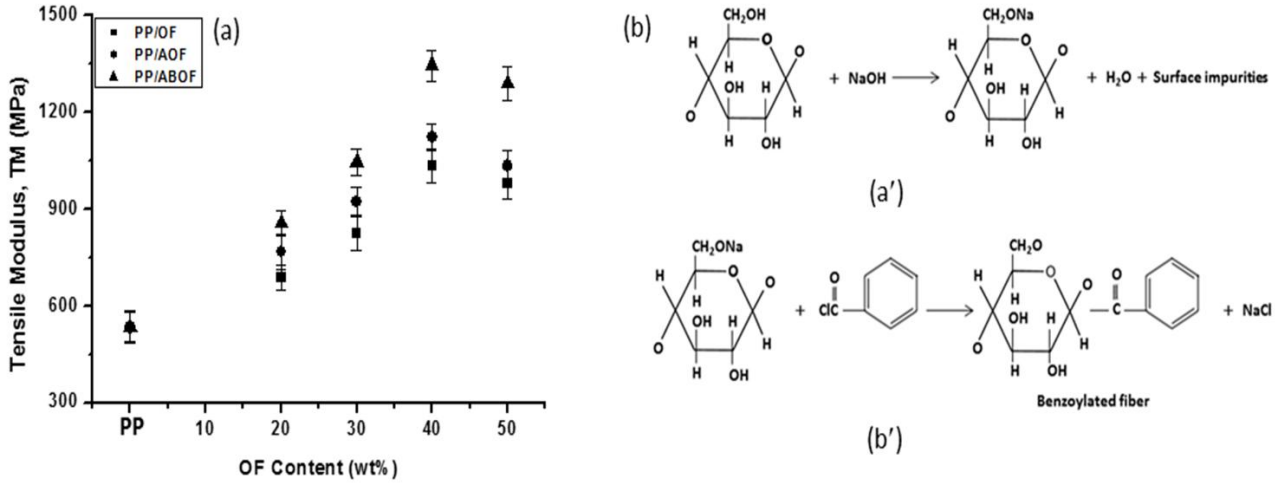

Figure 3: (a) Tensile modulus of PP/OF, PP/AOF, and PP/ABOF composites at different fiber loading; **(b)** The possible reaction mechanism of OF with **(a')** NaOH and **(b')** benzoylation.

Figure 3 (a) displays the correlation between tensile modulus and OF load with and without alkali treatment. Loading levels increased from 20 wt% to 40 wt%, the tensile modulus improved from 44 % to 111 % compared to virgin PP, and then the tensile modulus tends to opt towards lower value with further increase in fiber content. The currently available tensile modulus range used was 767.8 MPa to 1123.8 MPa using the same fiber content. The progress in tensile modulus may be related to the increase in stiffness of the composites due to OF loading.

Effect of Benzoylation Treatment

Figures 2 (a) and 3 (a) display the effect of benzoylation of alkali-pretreated OF on tensile strength and modulus of PP/ABOF composites. As the load level increased from 20 to 40 wt%, the tensile strength and modulus of the PP/ABOF composite increased from 34 %, 55 %, 74 % and 61 %, 96 %, 152 %, respectively, as compared with virgin PP, then decreased with a further increase in reinforcement (50 wt%). The increase in tensile strength and modulus may be due to the removal of lignin and extractable, a slight increase in cellulose content, and the conversion of a small portion of hemicellulose to benzoylated hemicellulose. Benzoylation also reduces polarity by covering the hydroxyl groups located on the cell wall of the OF. The hydrogen atoms of the hydroxyl group have been replaced by the benzoyl group as illustrated in Figure 3b (b'). By improving the mechanical features of benzoylated composites, the introduction of the benzoyl group is attributed to the reduction of the hydrophilicity of the fibers, which gives the fibers a more hydrophobic character and therefore provides the surfaces with better interaction of the hydrophobic matrix [19]. SEM studies of benzoylated fibers (Figure 5, c) show that benzoylation makes the surface of the fiber very rough and increases fibrillation and provide better mechanical interlocking with the PP. The development in tensile features is due to the good bond between the benzoylated fibers and the PP matrix, which can be supported from the SEM photomicrograph of the tensile fracture surface of the composite. The tensile features of the alkaline pretreated benzoylated fiber composites were found more than untreated fiber composites and more than alkaline treated composites.

Impact Toughness and Hardness Features

Impact strength is the capacity of a substance to absorb energy under a shock load or to prevent a fracture under a load applied at high speeds. Figure 4 (a) displays the variation in impact strength

with different OF loading for untreated and chemically treated (alkali or both alkali and benzoylation) composites. From the diagram, it was observed that the impact strength showed the same trend as the tensile features. The optimal property was observed at 40 wt% fiber loading. This outcome proposes that the fiber was able to absorb energy due to the strong interfacial bond between the fiber and the matrix. Above 40 wt% of OF, the impact strength tends to reduce. When the amount of fiber exceeded 40 wt%, the wetting of the fibers by PP resin was insufficient and many voids were present, resulting in a fiber-PP bond. For the treatment of PP/OF composites, the impact strength value has been observed to indicate a gradual increase with an increase in fiber content up to 40 wt%, which plays a role in the positive impact energy absorption of the fiber. Chemically treated PP/OF composites showed much higher impact strength values than untreated ones. Alkalization outcomes in improved interfacial debonding of fibers, including PP, probably due to additional sites created for mechanical interlocking [20]. This treatment increased the impact strength by 21 % at 40 wt% fiber loading compared to untreated ones. On the contrary, the addition of benzoylated OF to PP indicates a 23 % improvement in impact strength compared to alkaline treated OF composite. According to Mishra et al. [21], this improvement in the impact strength was due to the minimal interfacial debonding. The influence of fiber content on the hardness of PP/OF, PP/AOF, and PP/ABOF composites is illustrated in Figure 4 (b). In general, fibers that increase the modulus of composites increase their hardness in thermal applications. It was found that the hardness of the two types of modified composites increased by 3% and 5 %, respectively, compared to the PP/OF composite, at 40 wt% OF content. The inclusion of fillers in the PP matrix reduces polymer chain mobility in rigid composites. Better hardness is seen in a composite of two types of treatment than in untreated composites. This can be attributed to the good dispersion of fibers in the matrix so that the voids are reduced and there is a strong interfacial bond between the matrix and the filler.

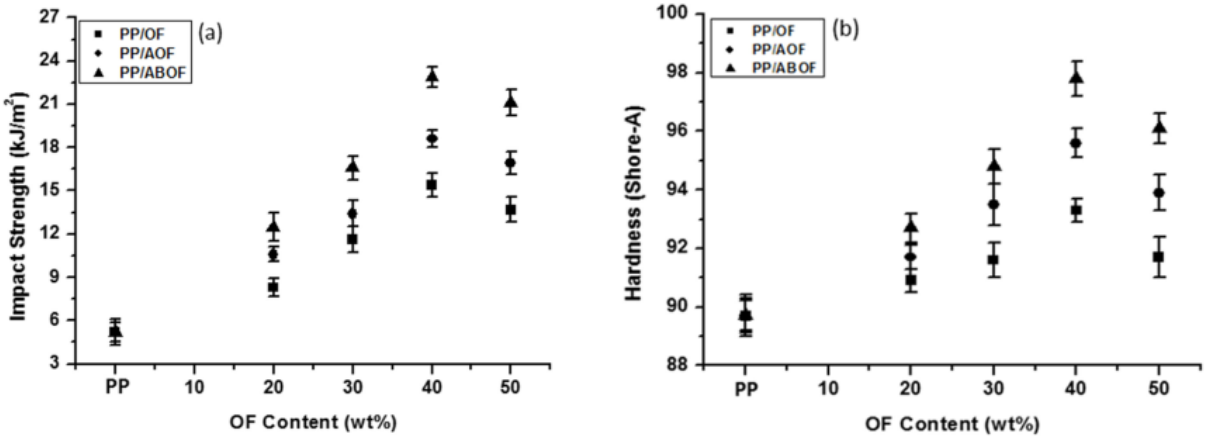

Figure 4: (a) Impact strength of PP/OF, PP/AOF and PP/ABOF; and **(b)** Hardness of PP/OF, PP/AOF, and PP/ABOF composites at the different fiber content.

Morphological Study

Fiber-matrix adhesion, which was inferred from the resulting mechanical features as predicted in the previous section, was used to mark the fracture surfaces of the tensile specimen of the SEM. Figure 5 displays SEM images of fracture surfaces of (a) untreated, (b) PP/AOF, and (c) PP/ABOF composites. Photomicrographs (Figure 5(a)) of reinforced composites with OF without treatment shows weak adhesion between the OF surface and the matrix. Many large voids were enclosed in

the OF and the fibers lacked contact with the matrix due to the incompatibility of these elements. It has been observed that the interfacial structure of this composite cannot effectively stress transfer. This observation was consistent with the low tensile strength values described in Figure 2 (a). Treated specimens showed better bonding than the untreated one. Alkaline treatment increased its dimensional stability and surface roughness of OF and thus improved bonding with PP (Figure 5, b). We further noticed that the OF was partially wetted by the matrix. In the case of benzoylated reinforced composites (Figure 5, c), the OFs were somewhat elongated during the fracture process and most of the PP matrix was attached to the surface of the OFs and adhered. Fiber seems to be less prolonged for PP/AOF composites than PP/OF composites. This has confirmed a good interaction between OF and PP. SEM photomicrographs show evidence of increased mechanical features, and moisture absorption features of PP/OF composites by alkaline treatment or both alkali and benzoylation treatment.

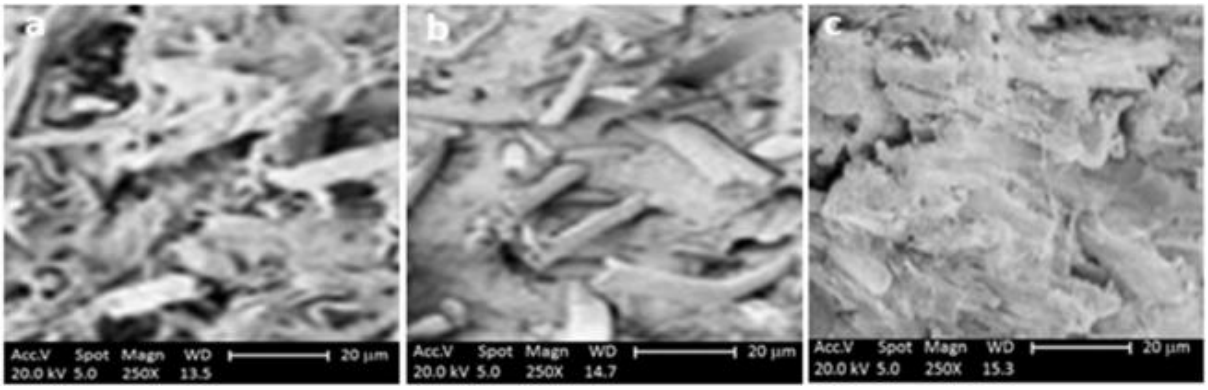

Figure 5: SEM images of the fracture surfaces of **(a)** PP/OF (untreated), **(b)** PP/AOF, and **(c)** PP/ABOF composite at 40 wt% fiber content.

Water Absorption (WA) Behavior

The outcomes of WA values of PP/OF, PP/AOF, and PP/ABOF composites as opposed to OF content are presented in Figure 6 as a function of water soaking time (24 h). Soaking time and the amount of OF contents are substantial issues, which affect the WA of the composite. The WA of PP was 0.05 % and was negligible compared to the WA of 50 wt% OF-reinforced PP composite (1.69 %). From the test results described in Figure 6, the amount of WA has obtained after 24 hours immersion in water in the range of 0.73 % for 20 wt% fiber loading to 1.69 % for 50 wt% fiber loading due to the higher availability of cellulose –OH groups that can absorb water. Natural fiber-reinforced composites typically demonstrate momentously moisture absorption due to the presence of micro-voids in the filler-matrix interface which occurs due to incomplete wetting of the fibers through the matrix [22]. At a loading level of 50 wt%, the WA of PP/AOF composite (approximately 57 %) decreases compared to the untreated composite. This may be due to the removal of lignin and hemicellulose components of the fiber by alkali treatment. In contrast, benzoylated composites (PP/ABOF) in 50 % fiber content showed about 63 % WA less than in untreated composites. It was further found that the WA of PP/ABOF composites was less than that of PP/AOF composite in 50 % fiber content. The WA reduction of PP/ABOF composites is due to the reaction of the benzoyl group which reduces the -OH group on the surface of the fiber and hence the fibers become more hydrophobic. Modification in the fiber cell wall, -OH groups reduce WA and are responsible for increasing matrix-fiber contact. In this regard, details of such processes are exhibited in Figure 6 for showing the variations in 24 h.

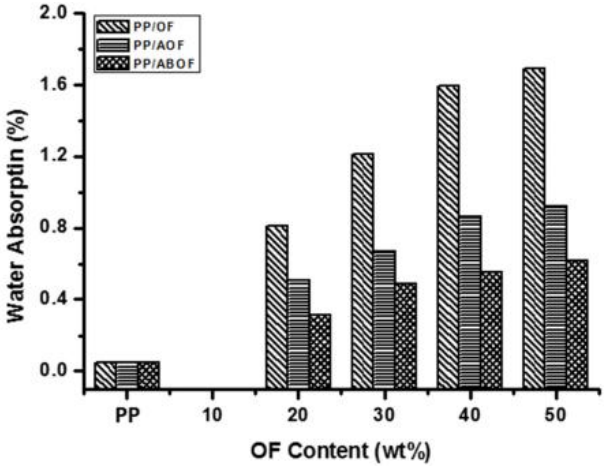

Figure 6: WA values of PP/OF, PP/AOF, and PP/ABOF composites against different fiber loading as a function of water soaking time (24 h).

Weather Effects

Untreated and treated specimens were exposed to simulated sunshine and severe weather tests over 500 h of condensation in the periodic cycle. The tensile strength (TS) and tensile modulus (TM) of the specimens were determined periodically. Figures 7 (a) and 4 (b) represent a reduction in TS and TM of the specimens due to weather. In most observations, the loss of the TS of the untreated specimen was about 27 %, while the treatment composite of PP/AOF and PP/ABOF was about 13.5 % and 9 %, respectively. Similarly, the TM loss of untreated specimens was about 24 %, whereas PP/AOF and PP/ABOF specimens were about 11 % and 8 %, respectively. Weather tests showed that untreated specimens lost tensile features (TS and TM), but treated specimens retained tensile features for decades despite being exposed to intense weather for 500 h. The PP/ABOF specimen exhibits greater weather acceptance and is more durable than other specimens.

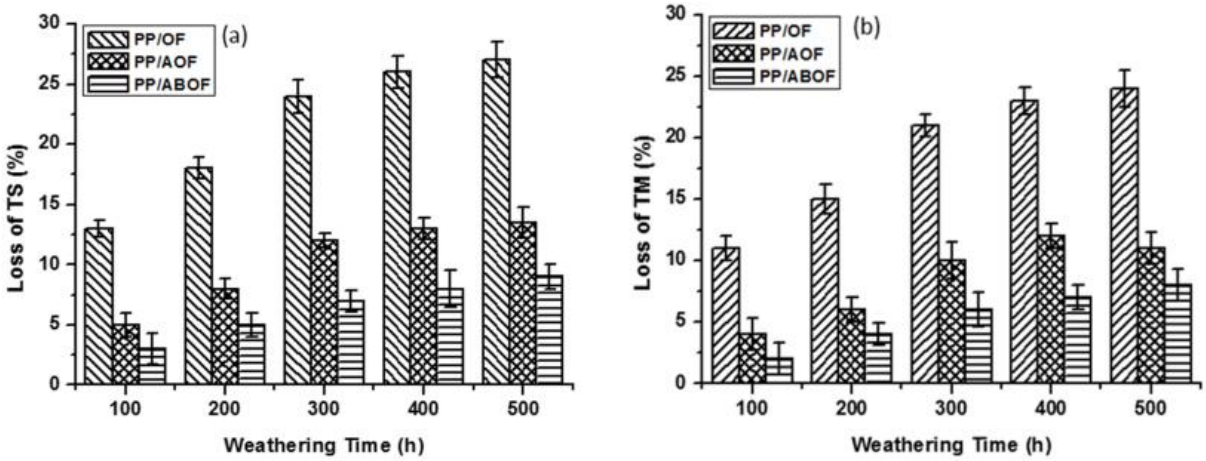

Figure 7: (a) Loss of tensile strength of the untreated and treated composites due to weather simulation; **(b)** Loss of tensile modulus of the untreated and treated composite due to weather simulation.

Conclusion

In this experiment, the effect of alkaline and benzoylation treatment of OFs on the basic features of PP/OF composites was investigated and analyzed. Based on the experimental outcomes, the following conclusions can be drawn from the current investigation. First, mechanical features such as the tensile strength, tensile modulus, impact strength, and hardness of the PP/CF composites

were increased to optimal fiber content of 40 wt%. However, 50 % fiber load composites have lower tensile features than 40 % fiber load composites. Second, the mechanical features of PP/AOF and PP/ABOF composites were higher than those of untreated composites. Third, SEM investigation of the fractured surfaces of the treatment composite revealed a rich bond between PP and OFs. Fourth, the water absorption behavior of the treated specimens showed a remarkably lower tendency than that of the untreated specimens. Weather studies have shown that the loss of TS and TM in treated specimens may be less than that of untreated specimens in terms of their decay time.

Disclosure Statement

The author(s) did not report any potential conflict of interest.

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