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# Renewable Okra Bast Fiber Reinforced Phenol Formaldehyde Resin Composites: Mechanical and Thermal Studies.

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## ABSTRACT

Okra bast fiber (OBF) reinforced thermoset phenol formaldehyde (PF) resin composites were prepared by compression molding methods. In order to found better wetting of filler and matrix, OBF was treated with NaOH. The properties of composites were studied by mechanical tests, thermal methods and water uptake. The mechanical properties such as tensile strength (TS), Young's modulus (YM), tensile elongation, flexural strength (FS) and flexural modulus of composites were varied with NaOH concentration, treatment time and fiber loading. TS and FS were found to increase for fiber loading upto 30% and then decreased whereas YM, FM and tensile elongation were increased with increase of weight fraction. About 21% more TS and 85% more FS was found for 10% alkali treated fiber composite than untreated fiber composite. Treated fiber composites also showed greater thermal stability and lower water absorption property.

## INTRODUCTION

Increasing environmental awareness is encouraging scientific research to produce cheaper, environment friendly, and more sustainable packaging and construction materials. Natural fiber reinforced thermoplastics composites are strong, stiff, light weight, recyclable, and have the potential to meet this requirement. However, from two thousands fiber containing plants species, fewer have economical value. Due to the low cost, low density, and excellent mechanical properties of natural fibers such as sisal, jute, hemp, flax, banana, PALF, coir, palm etc. are promising reinforcement with polymer composites [1,2,3,4]. Okra (*Abelmoschus esculentus*) bast fiber (OBF) is high quality fiber which is now consider as an agricultural wastage. The chemical composition of OBF is 60-70% α-cellulose, 15-20% hemicellulose, 5-10% lignin, 3-5% pectin, fatty and others materials [5]. Though it contains higher percent of cellulose, it may have potentiality to make good quality composite with thermoplastics/thermosets resin. The favorable mechanical properties of OBF however have yet been transferred successfully to thermoplastic composite materials [6]. Though thermoplastic composite used widely for recyclability, its processing temperature is too high. Many times fiber became degraded during composite fabrication. On the other hand, thermoset resins have good dimensional stability, are easily manufactured, and limit moisture absorption. But no report has been found in the literature of the use of thermosets for the fabricated OBF. Therefore, short OBF and thermoset phenol formaldehyde resin has been chosen for the present investigation.

Hand lay-up and compression molding process for fabrication of short fiber/thermoset composites are proficient and economic process [7]. In compression molding fibers are well distributed onto matrices which are most important for composite properties. Besides, fiber content, fiber length, void content, fiber-matrix bonding etc. are very important parameter for natural fiber reinforced composites [8]. Void possesses in composites is reduced mechanical properties. Again a weak interface drastically reduces the off axis strength, the flexural strength and the compression strength. An increase in interfacial strength leads to a substantial increase in tensile strength and modulus of short fiber composites. Since the fiber and matrices are chemically different, strong adhesion at their interfaces is needed for an effective transfer of stress and bond distribution throughout and interface. Weyenberg

et al. [4] reported that alkali treated flax fiber gave improve interfacial bonding of composites. Alkali treatment can remove impurities and produce a rough surface topography. Moreover, alkali treatment leads to fiber fibrillation i.e. breakdown the fiber bundle into smaller fibers. This increases the effective surface area available for wetting by matrix resin. Hence increasing the fiber aspect ratio caused better fiber-matrix interface adhesion and increase of mechanical properties. In the present investigation, the main objective is to determine the suitability of OBF as reinforcement in the PF resin matrix. Systematic studies of the mechanical and thermal properties of the composites as a function of fiber loading and alkali treatment have been done.

## EXPERIMENTAL

### Materials

Okra plant was collected from the local agricultural field in Kushtia, Bangladesh. The fiber was extracted from okra plant by 15-20 days water retting. The OBF were chopped into 2-3 mm of length. PF resin was purchased from Abelin Polymers Ltd., Mumbai, India. The properties of the OBF and PF resin are given in Table 1.

**Table 1: Properties of raw materials**

| Properties | OBF | PF resin |
|---|---|---|
| Diameter (μm) | 218±27 | - |
| Moisture content (%) | 4-6 | - |
| Tensile strength (MPa) | 52±23 | 8-12 |
| Young's modulus (MPa) | 1700±700 | 180-220 |
| Elongation at break (%) | 6.2±2.4 | 0-0.5 |
| Flexural strength (MPa) | - | 5-10 |
| Flexural modulus (MPa) | - | 1.8-2.0 |

### Fiber surface treatment

Short OBF was treated with 1-15 wt% aqueous NaOH solution (solution: fibers weight ratio 50:1) for 1, 5, and 10h at 30°C. The treated fiber was washed with water, with dilute acetic acid solution and distilled water up to pH 7. The fiber was then air dried in room, and in an oven at 50°C for 3 h.

### Composites Preparation

Short dried OBF with different weight fraction (10, 20, 30 and 40 wt%) is initially mixed thoroughly with PF resin in a stainless steel close mould. A releasing agent PAT 607/PCM was sprayed onto a laboratory tissue and smeared evenly onto the surface of the mould. Composites are made using a stainless steel mould at 150°C and 50KN pressure. The curing is completed at room temperature for 24h keeping the constant pressure 10 KN. The specimens for tensile (dimensions: 148.4 mm × 19.7 mm × 4.1 mm) and flexural (dimensions: 79 mm × 10 mm × 4.1 mm) tests were made using cutting machine.

### Composite testing

Tensile tests were conducted according to ASTM D 638-01 using a Universal Testing Machine (Hounsfield UTM 10KN). The tests were performed at a crosshead speed of 5 mm/min. Three-point flexural tests of composites were carried out using Hounsfield UTM 10KN according to the standard method used for flexural properties (ASTM D790-98). The speed for flexural test was set at 5 mm/min. All the results were taken as the average value of 10 samples. Water absorption tests were conducted according to ASTM D 570-99 using cold and hot water. The duration of the test was 24 h.

The thermogravimetry of virgin PF resin and composites were conducted by thermal gravimetric analyzer (TG/DTA 6300, Seiko Instrument Inc., Japan). 20 mg of each type of sample was taken for analysis. The samples were heated up steadily at a rate of 20 K/min from 25 to 550°C in nitrogen atmosphere. To get perfection, analysis was carried out two times for each sample.

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## RESULTS AND DISCUSSIONS

### Effect of fiber loading

The mechanical properties (TS, YM, tensile elongation, FS and FM) of PF resin, treated and untreated OBF composites, as a function of fiber loading, are presented in Table 2. It is evident that the TS and FS of the composites increased with the increase in fiber loading upto 30 wt%. The 30 wt% filled OBF composites showed 95%, 168%, 200%, 111% and 159% higher TS, YM, tensile elongation, FS and FM respectively than unreinforced PF resin.. This behavior is primarily attributed to the reinforcing effect of the fibers, leading to a uniform stress distribution from a continuous polymer matrix to a dispersed fiber phase. However, it was observed that with the increase in fiber loading after 30 wt%, both TS and FS were deteriorated. This decrease in the mechanical properties at high fiber loading is probably due to incompatibility of the fibers within the matrix, which promoted microcrack formation at the interface as well as non-uniform stress transfer due to fiber agglomeration in the matrix [9]. Conversely, YM and FM were increased with increasing fiber loading, as shown in Table 2. This may have been due to the fact that crystallites have a much higher modulus compared to amorphous regions and can increase the modulus contribution of the composite. Tensile elongation also increased with fiber loading. Since OBF exhibits higher elongation against tensile stress, the overall elongation at break increased with increase fiber fraction.

**Table 2: Effect of fiber loading on mechanical properties of untreated fiber composites**

| Fiber loading (wt%) | Untreated fiber composites | | | | |
|---|---|---|---|---|---|
| | TS (MPa) | YM (GPa) | Tensile elongation (%) | FS (MPa) | FM (GPa) |
| PF resin | 8.2 | 180 | 0.1 | 7.0 | 0.85 |
| 10 | 9.0 | 217 | 0.6 | 10.1 | 1.92 |
| 20 | 13 | 339 | 1.2 | 12.7 | 2.03 |
| 30 | 16 | 482 | 2.1 | 14.8 | 2.07 |
| 40 | 15 | 492 | 3.4 | 13.2 | 2.09 |

### Effect of alkali treatment

The variation of TS, YM, tensile elongation, FS and FM of alkali treated fiber composites as a function of NaOH concentration and treatment time for 30 wt% fiber loading is presented in Table 3. From the table, it is evident that the TS, YM, tensile elongation, FS and FM of the composites increased with the increase in NaOH concentration from 1% to 10%. The increment of TS, YM, tensile elongation, FS and FM after 1h treatment with 10wt% NaOH are 21%, 29%, 119%, 85% and 6% respectively then untreated fiber composites. The enhanced properties due to the higher concentration of alkali more hemicelluloses removed from fiber which give more surface roughness. Again alkali treatment fiber surface became rougher which may be gives better interlocking with matrix. Similar investigations have been reported by Gassan et al. [10]. However, at very high alkali concentration (15%) and longer time (5 and 10h) treatment causing deterioration in the mechanical strength of the composites. This may be due to cellulosic chain scission at long time treatment as well as high concentrate alkali, resulting in loss of strength of the fibers.

**Table 3: Effect of NaOH on mechanical properties of 30wt% fiber loaded composites**

| NaOH Conc. (wt%) | Treatment time (h) | TS (MPa) | YM (GPa) | Tensile elongation (%) | FS (MPa) | FM (GPa) |
|---|---|---|---|---|---|---|
| 1 | 1 | 10.2 | 320 | 1.1 | 15.0 | 2.00 |
| 3 | 1 | 14.4 | 498 | 2.6 | 17.3 | 2.05 |
| 5 | 1 | 16.1 | 540 | 3.5 | 20.2 | 2.08 |
| 8 | 1 | 18.5 | 590 | 4.1 | 25.8 | 2.16 |
| 10 | 1 | 19.4 | 620 | 4.6 | 27.4 | 2.20 |
| 15 | 1 | 18.0 | 570 | 2.4 | 18.1 | 2.06 |
| 10 | 5 | 17.6 | 560 | 4.0 | 18.4 | 2.02 |
| 10 | 10 | 12.4 | 500 | 3.1 | 17.3 | 2.02 |

### Water absorption

PF resin and OBF-PF resin composites were subjected to cool water (20°C) and hot water (50°C) for 24h. Water diffuses through the composites by capillary action along the fiber matrix interface, followed by diffusion from the interface into the bulk resin [11]. This results in the development of shear stress at the interface, thereby leading to debonding, delamination, and loss of structural integrity in the composites [12]. The extent of water absorption in the composites is presented in Table 4. However, the treated OBF composites at 30 wt% fiber loading exhibited lower water uptake, in comparison to the untreated OBF composite under the similar conditions. This phenomenon

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is probably due to efficient wettability of the fibers within the matrix that reduced water accumulation in the interfacial voids.

**Table 4: Water absorption of PF resin, untreated and treated OBF-PF resin composites (30 wt%) after 24h**

| Fiber loaded (wt%) composites | Untreated OBF composites | | Treated OBF composites | |
|---|---|---|---|---|
| | In cool water | In hot water | In cool water | In hot water |
| PF resin | 0.50 | 0.75 | 0.50 | 0.52 |
| 10 | 1.87 | 2.64 | 1.32 | 1.68 |
| 20 | 1.26 | 1.82 | 1.08 | 1.52 |
| 30 | 2.98 | 3.64 | 2.30 | 3.21 |
| 40 | 1.55 | 2.88 | 1.61 | 2.87 |

## Thermo gravimetric analysis

The thermogravimetric (TG) curves of virgin PF resin, untreated and treated OBF composites are presented in Figure 1. The curve of PF resin indicates that first decomposition takes place at temperature of 390°C, and nearly 100% decomposition occurred at 490°C. On the contrary, TG curves reveal a comparatively lower thermal stability of the PF resin matrix with the incorporation of fibers. The minor decomposition peak observed at 340 and 358°C in case of untreated and treated (Figure 1) OBF composite respectively which correspond to degradation of OBF. The minor degradation is possibly due to degradation of dehydrocellulose. Comparing the weight loss at 360°C, about 40% for the untreated OBF composite and 26.2% for the treated OBF composite was noted. This shows a higher thermal stability in the treated OBF composite, thus confirming the presence of intermolecular bonding between the fibers and the matrix due to the formation of ester linkage.

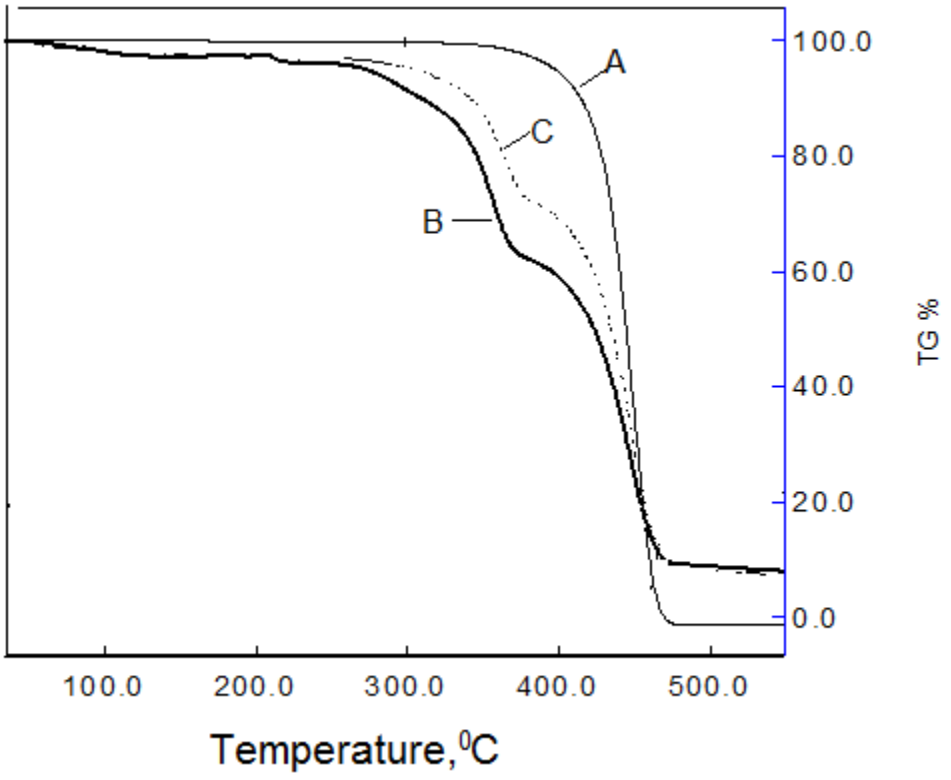


**Figure 1: TG curves of (A) PF resin, (B) untreated (30 wt%) OBF-PF resin composite and (C) 10% NaOH treated (30 wt%) OBF-PF resin composite**

## CONCLUSION

The mechanical properties of PF resin–OBF composites have been investigated. It was observed that the composites prepared at 30 wt% of fiber loaded PF resin composite showed optimum mechanical strength. Alkali treated OBF composites (with 10% NaOH for 1h) showed higher mechanical properties. Alkali treated composite also showed better water absorption and thermal properties than untreated OBF-PF resin composite. Though thermal stability of OBF composites was lower than virgin PF resin, it has higher TS and FS. So, OBF can be used as a potential filler of thermoplastic composites.

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