FABRICATION AND STUDY OF LIGNOCELLULOSIC *HIBISCUS* SABDARIFFA FIBER REINFORCED POLYMER COMPOSITES

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Fabrication of polymer composites reinforced with lignocellulosic materials has increased considerably during the last few years. This work reports the synthesis of natural fiber reinforced phenolformaldehyde (PF) resin matrix based polymer composite using a compression molding technique. Initially the PF resin was prepared by varying the concentration of formaldehyde with a fixed weight of phenol. Polymeric resin of different P: F ratios were subjected for optimization of their mechanical properties. The sample ratio of 1:1.5 (P: F) was found to possess maximum mechanical strength. Then reinforcing of this optimized resin was done by taking different ratios of Hibiscus Sabdariffa (HS) fiber in short form (3mm) to prepare green polymer composites. Polymer composite materials thus prepared were subjected to evaluation of their mechanical properties such as tensile strength, compressive strength, flexural strength, and wear resistance, etc. Optimum mechanical properties were obtained with a fibre loading of 30%. Thermal (TGA/DTA/DTG) and morphological studies (SEM) of the polymeric resin, and composites thus synthesized have also been studied. The results obtained suggest that these fibers can be a superior candidate for the reinforcement of high performance polymer composites.

Keywords: Lignocellusic fibers; Composites; Reinforcement; Mechanical properties

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INTRODUCTION

The development of polymer composites from renewable raw materials has been increased considerably during the last few years (Singha et al. 2008 a-d; Debasish et al 2004; Jacob et al. 2006; Bledzki et al. 1996, 1998, 1999). Natural fiber-based lignocellulosic materials are now considered as potential substitutes for toxic synthetic fibers for the preparation of reinforced composites. The advantages of natural fibers include easy availability, renewability, biodegradability, non toxicity, low cost, low specific gravity, high toughness, acceptable specific strength, and enhanced energy recovery, etc. (Chauhan et al. 1999, 2000; Singha et al. 2002, 2005, 2008 a-d). Synthetic fibers can cause health problems and are on the whole difficult to utilize, since composites with high synthetic fiber content have low energy values and high ash content (Bledzki et al. 1999). It has been observed that effective utilization of lignocellulosic fibers as reinforcement in polymer composites provides positive environmental benefits with respect to ultimate disposability and most excellent utilization of raw material (Kaith et al. 2007, 2008).

Currently a variety of studies are ongoing to find ways to use lignocellulosic natural fibers in place of synthetic fibers in various applications (Singha and Shama 2007; Panthapulakkal et al. 2006; Chauhan et al. 1999, 2000). Compression molding is one of the most suitable techniques to process lignocellulosic fibre-based polymeric material (Singha et al. 2008 a-d). The properties of bio fibres mainly depend on the source, age, and separating techniques of the fibre. These properties generally vary from plant to plant according to climatic conditions, etc. These fibers consist of cellulose, hemicelluloses, lignin, and extraneous components. Cellulose is principally responsible for the strength of natural fiber because of its specific properties such as high degree of polymerization and linear orientation. Lignocellulose fiber based polymer composite materials have made a primary contribution to the well-being and technological development of humankind (Bledzki et al. 1996, 1998, 1999; Nabi et al. 1999; Nakagaito et al. 2004). The most important factors in the development of natural fiber reinforced composites are fiber dispersion and fiber–matrix interaction (Bledzki et al. 1999; Chauhan et al. 1999; Hagstrand et al. 2001; Singha et al. 2008a-b).

The commercial importance of polymers have derived intense applications in the form of composites in various fields viz. in aerospace, automotive, marine, infrastructure, the military, etc. Among various polymer matrices phenol-formaldehyde (PF) resin is a widely used thermosetting adhesive for exterior-grade natural fiber reinforced composites. It is one of the dominant resins used in oriented strand board (OSB) and plywood production. There have been some concerns regarding the emission of formaldehyde from wood-based composites bonded with formaldehyde-containing resins. Tests have shown that formaldehyde emissions from phenolic bonded products are not significant enough to be regulated (Aierbea et al. 2000).

The Himalayan region of India is blessed with vast resources of lignocellulose fibers. Because of inaccessibility to these hilly areas this precious wealth is not still exploited commercially for various end uses. Among the available types of lignocellulose fibers, *Hibiscus Sabdariffa* (HS) fibers have high potential as a reinforcing material in polymer composites. A literature survey has shown scant information regarding utilization of this fiber as reinforcing material, and hence a comprehensive research programme has been started in our laboratory to synthesize lignocellulosic fiber-reinforced polymer composites. This work reports the synthesis and study of fiber reinforced *Phenol-Formaldehyde* (*P-F*) resin based composites.

EXPERIMENTAL

Materials

Phenol, formaldehyde solution, and sodium hydroxide of Qualigens make were used as received. *Phenol-Formaldehyde* (PF) resin was used as novel polymer matrix for preparing composites. *Hibiscus Sabdariffa* fibers collected from local resources were used as reinforcing material. These fibers, after washing with detergent powder, were soaked in hot distilled water for 7 h and air dried for 60 h, followed by oven drying at 105-110 °C for 12 hours. The fibers were then chopped into dimensions of 3 mm.

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Synthesis of Phenol - Formaldehyde Resin

Phenol-formaldehyde resin was synthesized by the standard method used in the laboratory (Singha and Thakur 2007c; 2008e). Phenol and formaldehyde were taken in different molar ratios (1.0:1.0, 1.0:1.5, 1.0:2.0, 1.0:2.5, and 1.0:3.0) by weight, in the reaction kettle and were mixed with the help of mechanical stirrer. NaOH solution was added slowly with constant stirring and heating, to the Phenol- formaldehyde solution until a pH of 8.5. Since the reaction is exothermic, proper care was taken to maintain the temperature between 50- 60 °C, for an initial 1 hour. Afterwards, the temperature was increased to 70-75 °C and the mixture was heated at this temperature until the resinification started. The reaction mixture was condensed at this temperature for 3-4 hrs until the complete resinification and pH was adjusted to 8.0 for the preparation of resin sheets. Polymeric resin formed as a result of polymerization reaction has been found to be deep brown in colour, having a viscosity of 607 cPs at room temperature with a solid content of 55%. Heating was stopped after polymerization, and the resin was cooled and transferred to specially made moulds. Resin sheets of size 150 mm x 150 mm x 5.0 mm were prepared by a closed mould method as described elsewhere (Singha et al 2005; Singha and Thakur 2007b,c). The mould was then closed and kept under pressure (4.0 MPa) until the resin was set into a hard mass. All the specimens were post-cured at 140°C for 7 hr. An optimized sample of ratio of PF (1:1.5) was used for further fabrication of fiber reinforced polymer composites.

Synthesis of Polymer Composites

Hibiscus Sabdariffa fibers of dimension 3 mm were mixed thoroughly with PF (1:1.5) resin, using a mechanical stirrer with different loadings (10, 20, 30, and 40 %) in terms of weight. Then the above mixture was poured into specially made moulds. The surface of moulds was coated on the inside with oleic acid to avoid adhesion of the mixture and to allow easy removal of the composites. The mixture was then spread equally on the surface of the mould. Composite sheets of size 150 mm x 150 mm x 5.0 mm were prepared by compression moulding technique (Singha and Thakur 2008c,d). During the fiber-composite fabrication process, the resin undergoes polymerization reaction with itself and chemical reaction with cellulosic under various environmental conditions, which comprise temperature, moisture content (MC), relative humidity (RH), and water vapor pressure, etc. These variables could significantly affect the resin curing, along with bonding behavior, and consequently affect the final performance of polymer composites. The longer curing time is required for complete conversion of resin into an insoluble three-dimensional network along with the formation of mechanical bonds between the fiber and resin matrix.

Mechanical Testing

Tensile, compressive, flexural, and wear tests were performed on specimens cut from the above synthesized polymer composite material. Testing of samples for tensile, compressive, and flexural strengths were done on Computerized Universal Testing Machine (HOUNSFIELD H25KS). Wear testing was done on a Wear & Friction Monitor (DUCOM- TR-20L). Seven specimens of each sample were used for the measurement of the above mechanical properties at ambient laboratory environment and average results have been reported.

Analysis of Mechanical Properties of Samples

Tensile strength test

The specimens of dimension 100 mm \times 10 mm \times 5 mm were used for analysis. The tensile test was conducted in accordance with ASTM D 3039 method. The test was conducted at the constant strain rate of 10 mm/min. Force was applied until the failure of the sample and load-elongation curve was obtained.

Compressive strength test

The compression test was conducted in accordance with ASTM D 3410 method. Composite sample was held between the two platforms, and the strain rate was fixed at 10 mm/min, whereas the total compression range was 7.5 mm. The compression stress was applied till the failure of sample. Total compression per unit force was noted.

Flexural strength test

The three-point bend flexural test was conducted in accordance with ASTM D 790 method.

Wear Test

Wear resistance of composites was carried out as per ASTM D 3702 method. Weight loss due to abrasion was calculated, and this weight loss was used as the measure of wear.

SEM Analysis of Samples

Scanning electron microscopy (SEM) was used to study the surface morphology of the composites, the fibers, and the matrix. The excitation energy used was 5 keV. To achieve good electric conductivity, all samples were first carbon-sputtered, followed by sputtering a gold-palladium mixture before examination. These micrographs clearly show the difference between unloaded and loaded PF matrix.

Thermal Analysis of Samples

Thermal analysis of natural and synthetic polymers gives us good account of their thermal stability. Thermogravimetric analysis (TGA), differential thermal analysis (DTA), and differential thermogravimetric (DTG) studies of samples were carried-out in nitrogen atmosphere on a thermal analyzer (Perkin Elmer) at a heating rate of 10 °C /min. TGA is used to characterize the decomposition and thermal stability of materials under a variety of conditions, and to examine the kinetics of the physico-chemical processes occurring in the sample. Basically in this method a change in thermal stability is examined in terms of percentage weight loss as a function of temperature. At the same time DTA involves comparing the precise temperature difference between a sample and an inert reference material, while heating both. DTG is a type of thermal analysis in which rate of material weight change upon heating versus temperature is plotted and is used to simplify reading of weight–versus temperature thermo gram peaks that occur

close together. DTG peaks are characterized by the peak maximum (T_{max}) and the peak onset temperature (T_e) . The area under a DTG curve is proportional to the mass change, and the height of the peak at any temperature gives the rate of the mass change at that temperature. DTG curves are frequently preferred when comparing results with DTA curves because of the visual similarity.

RESULTS AND DISCUSSION

Polymers based on phenol-formaldehyde (PF) resin are generally synthesized by the condensation of phenol with formaldehyde (Grenier et al 1996). Reaction between phenol and formaldehyde is normally catalyzed by acids and bases. The nature of the product formed as a result of the polymerization reaction primarily depends upon the types of catalyst and the molar ratio of the reactants (Singha and Thakur 2008e). It has been observed that the reaction between phenol and formaldehyde in a basic medium yields polymeric materials that possess good fire resistance properties. In the present work PF resin was synthesized by the reaction of phenol with different ratios of formaldehyde in a basic medium, using sodium hydroxide.

Optimization of Phenol – Formaldehyde Resin

Optimization of PF resins with different ratios was done by taking into consideration the optimum mechanical properties such as tensile strength, compressive strength, flexural strength, and wear resistance (Singha and Thakur 2008c,d).

Tensile strength

It was observed that PF samples of ratio 1.0:1.5 were able to sustain more load as compared to samples of other ratios (Singha and Thakur 2007c). This ratio (1.0:1.5) could bear a maximum load of 471.8 N with an extension of 2.7 mm (Fig. 1A). On the other hand, samples of other ratios bore low loads.

Compressive strength

It is evident from Fig. 1B that the samples of ratio 1.0:1.5 could bear a maximum load of 1597 N at a compression of 2.5 mm.

Flexural Strength

It is evident from Fig. 1C that the samples of ratio 1.0:1.5 could bear a maximum load of 397 N at a deflection of 2.17 mm

Wear resistance

It was observed that wear rate of samples of ratio 1.0:1.5 was less as compared to any other samples (Fig. 1D). The wear resistance results also support the tensile, compressive, and flexural results, as brittle materials have higher wear rate and lower tensile, compressive and flexural strength. The ratio (1.0:1.5) of phenol and formaldehyde, showing optimum mechanical properties, was taken for further preparation of biomass-reinforced phenol- formaldehyde composites.



Fig. 1. Load elongation/deformation/deflection and wear resistance curves of PF resin (A, B, C, and D).

Effect of Reinforcement on the Mechanical Properties of PF Based Composites

Tensile strength

It was observed that tensile strength of composites increased on reinforcement with *Hibiscus Sabdariffa* fibers. Composites with 30% wt. loading bore maximum load, followed by 40%, 20%, and 10% loadings (Fig. 2A). It was observed that polymer composites with 10; 20, 30, and 40 percent loading bore loads of 1275, 1529, 1782, and 1671 N, respectively.

Compressive strength

The compressive properties of the composites as the function of load and deformation are presented in Fig. 2B. From the graph it is evident that first compressive force increased with the increase in fiber content up to 30% and then decreased with

further increase in fiber content. It is clear from the figure that composites with 10, 20, 30, and 40 percent loading bore loads of 3387.5, 3925.5, 5225.0, and 4701.0 N, respectively.



Fig. 2. Load elongation/deformation/deflection and wear resistance curves of polymer composites with 10, 20, 30, and 40% loading (A, B, C, and D).

Flexural Strength

Similar trends as obtained in tensile and compressive strength tests were also observed for flexural strength results. The flexural properties of samples as a function of force (in terms of load) and deflection are shown in Fig. 2C. It is clear from the figure that composites with 10; 20, 30, and 40 percent loading bore loads of 980.0 N, 1242.0 N, 1525.0 N, and 1402.0 N, respectively.

Wear test

It is evident from Fig. 2D that the wear rate of PF matrix decreased appreciably as reinforcement with *Hibiscus Sabdariffa* fibers, whereas maximum wear resistance

behaviour was shown by a composite with 30% loading, followed by 40, 20, and 10% fiber loading.

Stress-Strain Behaviour

Tensile Stress-Strain Curve

Tensile stress-strain curves were constructed from the load elongation measurements. The plots of stress vs. strain for fibers reinforced polymer composites are shown in Fig. 3A. From figure it is clear that in the elastic region stress was linearly proportional to strain. When the load exceeded a value corresponding to the yield strength, the specimen underwent gross plastic deformation. It was permanently deformed if the load was released to zero. Different parameters derived from the stress-strain curve are shown in Table 1.

Compressive stress-strain curve

Compressive stress-strain curves also were constructed from the load deformation measurements. The plots of compressive stress vs. strain for polymer composites are shown in Fig. 3B. Different parameters derived from the stress-strain curve are shown in Table 2.

Flexural stress-strain curve

Plots of flexural stress vs. strain for fibers reinforced polymer composites are shown in Fig. 3C. Different parameters derived from the stress-strain curve are shown in Table 3.

Fiber (Wt. %)	Loading	Ultimate Tensile Stress MPa	Yield Strength MPa	Fracture Stress MPa	Standard Deviation	Tensile Modulus MPa
PF		9.4	8.1	8.7	0.72	207
10%		25.5	19.4	22.3	0.94	501
20%		30.5	22.2	27.4	0.99	604
30%		35.6	27.8	31.8	1.07	705
40%		33.4	25.2	29.5	1.21	661

Table 1. Parameters Obtained from Tensile Stress-Strain Curve

Fiber Loading (Wt. %)	Ultimate Compressive Stress MPa	Yield Strength MPa	Fracture Stress MPa	Standard Deviation	Compressive Modulus MPa
PF Resin	31.9	26.0	29.1	0.87	771
10%	67.7	57.8	62.5	1.17	1478
20%	78.5	67.3	74.2	1.20	1712
30%	104.	93.7	99.8	1.28	2280
40%	94.0	86.3	90.0	1.45	2051

Table 2. Parameters Obtained from Compressive Stress-Strain Curve

 Table 3. Parameters from Flexural Stress-Strain Curve

Fiber Loading (Wt. %)	Ultimate Flexural Stress MPa	Yield Strength MPa	Fracture Stress MPa	Standard Deviation	Flexural Modulus MPa
PF Resin	119	107	114	0.75	3582
10%	294	279	288	0.92	8507
20%	372	361	368	1.10	10742
30%	457	443	452	1.15	11572
40%	420	407	416	1.20	10997



Fig. 3. Stress strain curves under tensile, compressive, and flexural tests (A, B, C).

From the foregoing results it is clear that in order to achieve good fiber reinforcement, interfacial bonding between the fiber and polymer matrix is the most essential factor. For a polymer composite to be an effective load bearing system, the fibers and matrix must cooperate. This coupling between the fibers and the matrix will not exist without the presence of contact at the interface. The interfacial bonding depends on the surface topology of the fiber. The interface acts as a 'binder' and transfers load between the matrix and the reinforcing fibers. Interfacial bonding is a result of good wetting of the fibers by the phenolic (PF) matrix as well as the formation of a chemical bond between the fiber surface and the PF matrix. The chemical bonding accounts for the adhesion between PF resin and cellulose of fiber. The higher bond strength obtained for the PF resin matrix is attributed to the possible reaction between the methylol groups of the resin with the hydroxyl group of cellulose. It was observed that mechanical properties increased up to 30% fiber loading and then decreased. This behaviour can be explained due to fiber-to-fiber contact at higher fiber loadings. Further, when fiber-reinforced PF composites were subjected to a load, the fibers acted as carriers of load, and stress was transferred from the matrix along the fibers, which resulted in composites with good mechanical properties. The uniform distribution of stress was dependent on the population and orientation of the fibers. At low levels of fiber loading, the orientation of fibers was poor, the fibers were not capable of transferring load to one another, and stress accumulated at certain points of the composite, which led to lower mechanical properties. At 30% levels of loading the population of the fibers was just right for maximum orientation and the fibers actively participate in stress transfer and hence maximum results were obtained in case of this loading.

Thermal Behaviour of PF Resin and its Composites

Thermo-gravimetric analysis (TGA) of raw fiber, polymeric resin, and composite with 10% loading was studied as a function of percent weight loss with the increase in temperature. The initial decomposition temperature (IDT) and final decomposition temperature (FDT) of fiber, resin, and composite are presented in the Table 4. These results are consistent with results reported earlier (Singha and Thakur 2007 b, c; 2008 c-e). These studies are further supported by (DTA and DTG) results as shown in Tables 5 and 6. The TG and DTA curves reveal that the lignocellulosic fiber, PF resin, and fiber reinforced composites decomposed in different stages in the temperature range of 199-500°C, 400-1195°C, and 332.7-996°C, respectively. Comparison of the magnitudes and locations of peaks found in the DTA/ DTG curves showed that there was change in the thermal behaviour of the polymer matrix when reinforced with cellulosic fibers (Singha and Thakur 2007 b, c; 2008 c-e).

Sr. No.	Sample Code	Exothermic/Endothermic peaks °C(μ V)
1.	HS	63 [-0.9]; 361 [-1.5]
2.	P-F Resin	164 [9.0]; 421 [6.0]
3.	SF-Rnf-PF	60[-9]; 579 [-17]; 681[-32]

Table 4- Thermo-dravimetric Analysis of PF, HS, and SF-Rhi-PF C	
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Table 5. Differential T	hermal Analysis of PF,	HS and SF-Rnf-PF Composite
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Sr. No.	Sample Code	Exothermic peaks Temperature (mg/min)
1.	HS	56 [0.098]; 293 0.237]; 355[1.126]
2.	P-F Resin	156 [112.8]; 215 [114.9] ;416 [126.1];490[235.5]
3.	SF-Rnf-PF	52[45]; 309[257]; 478[59]; 520[97]

Table 6. DTG analysis of PF, HS and SF-Rnf-PF Composites

Sr. No.	Sample Code	IDT (°C)	% wt. loss	FDT (⁰C)	% wt. Ioss	Final Residue (%)
1.	HS	199	7.65	500	85.7	14.2
2.	P-F Resin	400	15.6	1195	48.5	51.4
3.	SF-Rnf-PF	327	28.2	976	64.3	35.6

Morphological Study of Composites

Morphological results clearly show the difference in the morphology of the polymer composites when compared with the morphology of the fiber and the polymer matrix separately (Figs. 4A-F). Morphological results clearly show that when polymer resin matrix was reinforced with the different loadings of fiber, morphological changes took place, depending upon the bonding between the varying loading of fiber and the polymer resin matrix. In case of lower fiber loading (10 or 20 percent), the content of polymeric resin being higher, there was lower bonding between the matrix and the reinforcement (Figs. 4 C-D), and hence mechanical properties had lower values as compared to 30% fiber loading. In case of 30% fiber loading there was intimate mixing of the reinforcement with the resin matrix (Fig. 4E) and hence resulting polymer composite materials had higher mechanical properties. However at higher fiber content (beyond 30%), agglomeration of fibers took place, which resulted in decreased mechanical properties (Fig. 4F).

CONCLUSIONS

- 1. Phenol-formaldehyde (PF) resin synthesized in the ratio of 1.0:1.5 exhibited optimum mechanical properties.
- 2. The mechanical characterization of PF resin and their composites with lignocellulosic fibers were studied in terms of tensile, compressive, flexural, and wear resistance results. Mechanical properties of fiber reinforced polymer composites were investigated as a function of the chemical nature of matrix polymer and the content of the reinforcing material. The mechanical properties of

reinforced polymer matrix resin based composites were found to be higher than the parent polymer matrix.

- 3. Scanning micrographs of polymer matrix, fiber, and composites showed a clear distinction in their morphology. These micrographs also showed that there was intimate mixing of the fiber with polymer matrix which depends upon the percent fiber loading.
- 4. Various thermal studies revealed that the PF resin was slightly more thermally stable than the corresponding polymer composites.
- 5. From stress–strain behavior results it was found that the composites were more ductile as compared to the PF matrix, which showed that PF resin based composites can be potential candidates for exterior-grade composites.



Fig. 4. SEM images of (A) PF resin, (B) *Hibiscus Sabdariffa fibers,* (C, D, E, and F) composites with 10, 20, 30, and 40% loadings.

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