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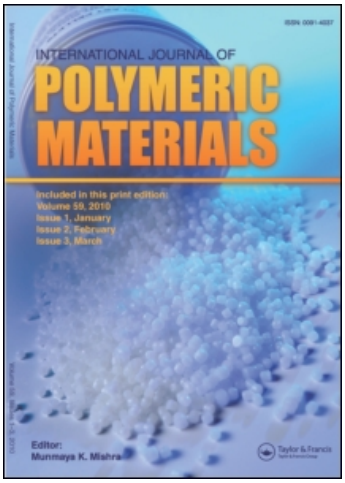

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Physical, Chemical and Mechanical Properties of *Hibiscus sabdariffa* Fiber/Polymer Composite

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Physical, Chemical and Mechanical Properties of *Hibiscus sabdariffa* Fiber/Polymer Composite

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Tensile, compressive, flexural and wear resistance properties of Hibiscus sabdariffa fiber-reinforced phenolic (Resorcinol Formaldehyde) resin matrix-based composites were evaluated to assess the possibility of using these fibers as a new eco-friendly material in engineering applications. Polymer composite samples were fabricated by a compression-molding technique developed in our laboratory. The effect of fiber dimension on mechanical properties was evaluated. The interfacial bonding between Hibiscus sabdariffa fiber and the polymer matrix has been found to affect the mechanical properties of the resorcinol formaldehyde resin matrix. It has been observed that particle-reinforced polymer composites exhibit better mechanical properties as compared to short and long fiber-reinforced polymeric composites. These composites were further subjected to an evaluation of morphological, thermal, physical (swelling and moisture absorption) and chemical properties.

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Keywords: mechanical, natural fiber, physical and chemical properties, polymer composite, reinforcement

INTRODUCTION

In recent years emphasis is being focused on fiber-reinforced polymers (FRPs) as high-performance materials in various fields because of their unique properties [1,2]. These properties include excellent

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mechanical strength, high corrosion resistance, dimensional stability, low assembly costs, and light weight [3,4]. Polymer composite materials are being used in aerospace, automotive, marine, infrastructure, military, leisure boats, aircraft industry, sport equipment, and industrial fields. Traditionally, polymers and polymeric composites have been derived from petroleum. However, with the rapid growth of industry, the applications of polymeric materials have increased several times. To cope with the requirements of industry, alternate and newer sources of polymer materials are being investigated. Due to the wide variety of polymer matrices and reinforcement materials available, the design possibilities for the fabrication of polymer composites are unlimited [5–7]. Natural fiber reinforced composites have considerable potential to replace conventional materials like metal in structural and nonstructural applications, especially in the furniture industry [8–11]. The matrix, often a polymeric material, surrounds the reinforcement and keeps it in place. The reinforcing fibers are chosen to improve the properties of the matrix, e.g., stiffness and strength. As compared to traditional synthetic fibers, natural fibers are able to impart the polymer composite with properties like low cost, nonabrasive effect, low health hazards, low specific gravity, enhanced mechanical properties, biodegradability, high degree of flexibility, good acoustic and thermal insulation, less machine wear and good availability [12–15]. In fact, lignocellulosic natural fibers have become the most sought material for the fabrication of natural fiber-reinforced composites [16,17].

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A review of the has literature revealed that very little work has been done on the effective utilization of *Hibiscus sabdariffa* fibers as reinforcing material in polymer composites [5–10]. In the present work, therefore, we have reported some of our investigations on mechanical, morphological, thermal, and swelling behaviors of *Hibiscus sabdariffa* fiber-reinforced resorcinol-formaldehyde polymer matrix-based composites.

EXPERIMENTAL

Materials

Resorcinol, formaldehyde solution and sodium hydroxide supplied by Qualigens Chemicals Ltd. were used as received. In the present research work we have used phenolic resin such as resorcinol-formaldehyde (RF) as polymer matrix and lignocellulosic *Hibiscus sabdariffa* fibers as reinforcing material. *Hibiscus sabdariffa* fibers were collected from local resources of the Himalayan region.

Purification of Fiber

Hibiscus sabdariffa fibers were thoroughly washed with detergent powder and water. After this, these fibers were soaked in hot distilled water for 7 h, dried for 60 h in air at room temperature followed by drying in a hot air oven at 105–110°C for 12 h. These fibers were then used in three different forms:

Particle Reinforcement

Hibiscus sabdariffa fibers were ground to a powder and filtered through a sieve of pore size 200 microns.

Short Fiber Reinforcement

Hibiscus sabdariffa fibers chopped into 3 mm size were used as short fibers for the fabrication of polymer composites.

Long Fiber Reinforcement

Hibiscus sabdariffa fibers chopped into 6 mm size were used as long fibers for the fabrication of Resorcinol Formaldehyde matrix-based polymer composites.

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Various Testing Instruments Used

Weights of the samples were taken on Shimadzu made electronic balance (LIBROR AEG-220). Curing of samples was done on a compression-molding machine (Santech India Ltd). Thermal studies were carried out on a thermal analyzer (Perkin Elmer) and SEM micrographs were taken on (LEO 435VP). Tensile, compressive and flexural strength tests were performed on Computerized Universal Testing Machine (Hounsfield H25KS). Wear test was performed on Wear & Friction Monitor (DUCOM- TR-20L).

Synthesis of Resorcinol-Formaldehyde Resin

Resorcinol-Formaldehyde resin was synthesized by the standard method developed in our laboratory [6,14]. Suitable conditions of temperature, acidity of the medium and pH were maintained while carrying out the reaction as reported earlier [14]. The resin synthesized was then transferred to a specially made mold. Resin sheets of size 150 mm × 150 mm × 5.0 mm were prepared by a closed-mold method described else where [5–9]. The cured samples were then subjected to various mechanical, thermal and morphological characterizations.

Fabrication of Polymer Composites

The *Hibiscus sabdariffa* fibers of different dimensions were mixed thoroughly with resorcinol-formaldehyde resin using a mechanical stirrer with suitable loadings (1.0:0.01) in terms of weight [8]. The above mixture was poured into specially made molds. The surfaces of molds were 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 mold. Composite sheets of size 150 mm×150 mm×5.0 mm were prepared by compression-molding technique. Compression molding was performed in a hot press using a mold preheated to 50°C. Composite sheets were prepared by hot-pressing the mold at 50°C for 30 min. The pressure applied ranges from 3 to 4 MPa depending on the loading of reinforcing material. All the specimens were then post-cured at 50°C for 12 h.

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Mechanical Properties

Tensile Strength

The tensile strength test was conducted on a Computerized Universal Testing Machine. Specimens of dimension 100 mm × 10 mm × 5 mm were used for analysis. The tensile test was conducted in accordance with ASTM D 3039 method. The test was conducted at a constant strain rate of 10 mm/min. Force was applied untill the failure of the sample and load – elongation curve was obtained.

Compressive Test

The compression test was also conducted in accordance with ASTM D 3410 method. The test was conducted at a constant strain rate of 10 mm/min whereas the total compression range was 7.5 mm.

Flexural Test

The three-point bend flexural test was conducted in accordance with ASTM D 790 method. The test was conducted at a constant strain rate of 2.54 mm/min.

Wear Test

The wear test of the sample was conducted on a wear and friction monitor. Wear resistance of composites was carried-out as per ASTM D 3702 method.

Swelling, Moisture Absorbance and Chemical Resistance

While studying the development of a natural fiber-reinforced polymer matrix-based composite, particular interest lies in its behavior against

weathering conditions [10, 14–17]. In fact, the effect of environmental conditions on the polymer biocomposites has been a subject of much debate and many studies have been reported in the literature. The commercial viability of the newly fabricated natural fiber-reinforced resin matrix composites lies in their physical and chemical properties. Therefore, a comprehensive study on swelling behavior in different solvents, moisture absorbance at different humidity levels and chemical resistance behavior against 1 N HCl and 1 N NaOH of resorcinol–formaldehyde polymer matrix composites has been carried out.

Swelling Behavior

Swelling behavior of the composite samples was evaluated by studying the swelling in different solvents such as methanol, isobutanol, carbon tetrachloride and water. For the swelling test, the specimens were dried in an oven for a specified time at a particular temperature and then cooled in a dessicator. Known weight (W_i) of the initial samples were immersed in 100 ml of different solvents at room temperature for 15 days. The samples were filtered and the excess solvent was removed with the help of filter paper, patted dry with a lint-free cloth and then the final weight (W_f) was noted. The percent swelling was calculated from the increase in initial weight in the following manner:

$$\text{Percent swelling } (P_S) = \frac{W_f - W_i}{W_i} \times 100$$

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Moisture Absorbance

Moisture absorbance studies on different samples were carried out at various humidity levels (varying from 20 to 100%). Moisture absorbance was found out by placing the known weight (W_i) of dry samples in a humidity chamber (Swastika make) which was set at a particular humidity level for about 12 h and then the final weight (W_f) of the samples exposed at a particular relative humidity (RH) was taken. The percent moisture absorbance was then calculated in the following manner:

$$\%\text{Moisture absorbance}(\%M_{abs}) = \frac{W_f - W_i}{W_i} \times 100$$

Chemical Resistance

For the chemical resistance test, the dried specimens were immersed in 100 ml of 1 N NaOH and 1 N HCl for different time intervals

(24–144 h). After this, the samples were filtered out, dried and weighed. The percent chemical resistance (Pcr) was calculated in term of weight loss in the following manner:

$$\text{Percent chemical resistance } (P_{cr}) = \frac{Ti - W_{aci}}{T_i} \times 100$$

where Ti = initial weight and W_{aci} = weight after certain interval.

Morphological and Thermal Studies

Changes in the surface morphology of the composite prepared with different loadings were studied by scanning electron microscopy (SEM). An excitation energy of 5 keV was used. To achieve good electric conductivity all samples were first carbon sputtered followed by sputtering a gold palladium mixture before carrying out the SEM analysis. SEM micrographs of the samples show the morphology of the biocomposites prepared. These micrographs show clear distinction between the different resorcinol-formaldehyde polymer matrix and *Hibiscus sabdariffa* fiber-reinforced composites.

Thermal analysis of natural and synthetic polymers gives us good account of their thermal stability. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) studies of samples were carried out in a nitrogen atmosphere on a thermal analyzer (Perkin Elmer) at a heating rate of 10°C/min.

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RESULTS AND DISCUSSION

Mechanism for Fabrication of Resorcinol-Formaldehyde Resin

Resorcinol is a reactive compound and readily combines with formaldehyde to form methylene derivative [6,14]. In this reaction the methylol group occupies either the position ortho to both hydroxyl groups, or ortho to one and para to other. Since resorcinol is very reactive towards formaldehyde, the reaction is carried out in a neutral medium. And proper care must be taken. In the polymerization reaction, methylolated resorcinol condenses with formaldehyde molecules and other resorcinol molecules to form a polymeric structure. Where resorcinol nuclei are joined together through methylene bridges.

Optimization of Resorcinol-Formaldehyde Resin

It has been observed that a resorcinol-formaldehyde resin in the ratio 1:1.5 exhibits optimum mechanical properties [6–14]. This ratio (1.0:1.5) could bear a load of 250.8 N with an extension of 2 mm, a load

TABLE 1 Tensile, Compressive, Flexural and Wear Resistance Results of Fiber-Reinforced Polymer Composites

Composite sample*	Tensile test force (N)/elongation (in mm)	Compression test force (N)/deformation (in mm)	Flexural test force (N)/deflection (in mm)	Wear test load (Kg)	Weight loss (Kg)
RF Resin	250.80/1.2	1370.2.17	117.80/1.0	3	0.0091
P-Rnf	690.00/2.23	3193.50/2.54	321.47/1.6	3	0.002275
SF-Rnf	660.47/2.27	3053.50/2.58	310.00/1.62	3	0.002527
LF-Rnf	637.00/2.31	2953.00/2.61	293.00/1.67	3	0.002676

*P = Particle, SF = Short Fibers, LF = Long Fibers, Rnf = Reinforced.

of 1370 N at a compression of 2.17 mm in compressive test and a maximum load of 117.8 N at a deflection of 1.0 mm in flexural test. Further, the sample of ratio 1.0: 1.5 exhibits maximum wear resistance. Hence it was taken here for the preparation of polymer composites.

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Mechanical Properties of Resorcinol-Formaldehyde-Based Composites

Tensile Strength

It has been observed that *Hibiscus sabdariffa* fiber-reinforced polymer composites with particle reinforcement showed highest tensile strength, followed by short fiber and long fiber-reinforced composites (see Table 1).

Compressive Strength

Compressive strength of RF resin matrix has been found to increase when reinforced with *Hibiscus sabdariffa* fiber. It has been found that on particle reinforcement, compressive strength increases to a much higher extent than short and long fiber reinforcement (Table 1).

Flexural Strength

Flexural strength of PF resin matrix has been found to follow the same trend as is observed for tensile and compressive strength. It has been found that with particle reinforcement, compressive strength increases to a much higher extent than short and long fiber reinforcement (Table 1).

Wear Test

As evident from Table 1, the wear rate of the RF matrix decreases appreciably when it is reinforced with *Hibiscus sabdariffa* fibers.

TABLE 2 % Swelling Behavior of Polymer Composites in Different Solvents

Sample	Water	Methanol	Isobutanol	Carbon tetrachloride
RF Resin	0.14	0.12	0.09	0.05
P-Rnf	3.24	3.11	3.01	2.92
SF-Rnf	3.39	3.22	3.16	3.08
LF-Rnf	3.55	3.34	3.29	3.22

It was observed that particle reinforcement decreases the wear rate to a lesser extent than short and long fiber reinforcement.

Swelling, Moisture Absorbance and Chemical Resistance Behavior of Polymer Composites

Hibiscus sabdariffa fiber-reinforced polymer composites with different dimensions shown in Table 2, different swelling behavior in different solvents and follows the trend: $H_2O > CH_3OH > C_4H_9OH > CCl_4$. The swelling behavior of polymer composites increases with increase in fiber length due to greater ease of water penetrating deeper into the matrix of the composite.

The moisture absorbance behavior at different humidity levels as a function of fiber length is shown in Table 3. It has been found that moisture absorbance (Mabs) increases with increase in humidity level ranging from 20 to 100% with increased fiber length (Particle $<$Short $<$ Long fiber).

In case of chemical resistance behavior, it has been observed that resistance towards chemicals decreases with the increase in exposure time (Table 4A–B). This is similar to the above observation.

Morphological and Thermal Analysis of Polymer Biocomposites

Morphological investigations (Figure 1A–D) clearly indicate that proper mixing of *Hibiscus sabdariffa* fiber with the resorcinol-formaldehyde

TABLE 3 % Moisture Absorption Behavior of Polymer Composites at Different Relative Humidity Levels

Sample	Hum = 20	Hum = 40	Hum = 60	Hum = 80	Hum = 100
RF Resin	0.00012	0.00025	0.00034	0.00052	0.00069
P-Rnf	0.00024	0.00037	0.00054	0.00068	0.00084
SF-Rnf	0.00029	0.00044	0.00059	0.00075	0.00091
LF-Rnf	0.00035	0.00051	0.00065	0.00079	0.00098

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TABLE 4 Chemical Resistances (In terms of % wt. loss) of Polymer Composites at Different Time Intervals against (A) 1 N HCl and (B) 1 N NaOH

Sample	24 Hrs	48 Hrs	72 Hrs	96 Hrs	120 Hrs	144 Hrs
(A)						
RF resin	1.1	2.17	3.12	4.25	4.71	5.07
P-Rnf	3.39	4.61	5.41	6.34	7.49	9.16
SF-Rnf	3.43	4.62	5.47	6.41	7.57	9.17
LF-Rnf	3.48	4.72	5.52	6.44	7.62	9.24
(B)						
RF resin	1.52	2.59	3.12	4.62	4.77	5.45
P-Rnf	3.75	4.88	5.64	6.81	7.81	9.36
SF-Rnf	3.72	4.83	5.56	6.81	7.78	9.32
LF-Rnf	3.69	4.78	5.52	6.72	7.71	9.26

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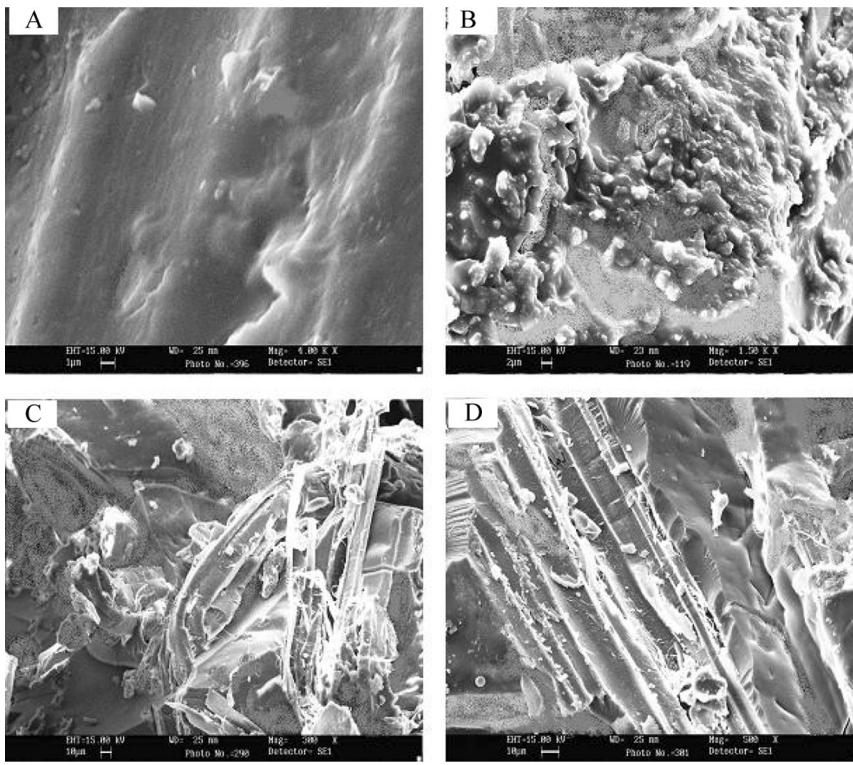

FIGURE 1 SEM images of (A) RF resin (B, C and D) composite with particle, short and long fiber reinforcement.

TABLE 5 Thermogravimetric Analysis of *Hibiscus sabdariffa* Fiber, Resorcinol-Formaldehyde Resin and LF-Rnf Composites

No.	Sample code	IDT (°C)	% wt. loss	FDT (°C)	% wt. loss	Final residue (%)
1.	*Hibiscus sabdariffa fiber*	199	7.65	500	85.71	14.29
2.	Resorcinol-formaldehyde resin	299	22.64	990	51.78	48.22
3.	LF-Rnf Composites	235	25.37	937	67.51	32.41

resin takes place. These micrographs also show the distinction in the morphology of the polymer composites of different fiber dimensions and their parent polymer matrix (resorcinol-formaldehyde) (Figure 1A–D). Thermogravimetric analysis (TGA) of raw *Hibiscus sabdariffa*, polymeric RF resin and biocomposites was studied as a function of % weight loss with the increase in temperature. In the case of raw *Hibiscus sabdariffa* the initial decomposition temperature (IDT) has been found to be 199°C [6], and the final decomposition temperature (FDT) to be 500°C [Table 5]. On the other hand, for RF resin, the observed initial decomposition temperature (IDT) is 299.0°C and the final decomposition of the resin took place at 990.0°C (Table 5). It is observed that for biocomposites with long fiber reinforcement the initial decomposition temperature and final decomposition temperature were 235.0 and 937.0°C, respectively (Table 5). These values lie between the degradation temperatures observed for matrix and the fiber. This indicates that the presence of cellulose fibers affects the degradation process of the biocomposites. Similar behavior is expected for particle and short fiber reinforcement [5–9]. These studies are further supported by differential thermal analysis (DTA) (Table 6). The TGA and DTA curves reveal that the *Hibiscus sabdariffa* fiber, RF resin and fiber-reinforced composites decompose in different stages in the temperature range of 199–500°C, 299–990°C and 235–937°C, respectively. Comparison of magnitude and location of peaks (exothermic/endothermic) found in the TGA/DTA curves shows that there is a change in the thermal behavior of

TABLE 6 Differential Thermal Analysis (DTA) of *Hibiscus Sabdariffa* fiber, Resorcinol-Formaldehyde Resin and LF-Rnf composites

No.	Sample code	Exothermic/endothermic peaks °C
1.	*Hibiscus sabdariffa* fiber	63; 361
2.	Resorcinol-formaldehyde Resin	65; 244; 280
3.	LF–Rnf composites	72; 79

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the polymer matrix when reinforced with cellulosic *Hibiscus sabdariffa* fiber fibers [5–9, 14].

From the above discussion it is evident that the tensile strength of *Hibiscus sabdariffa* fiber-reinforced resorcinol-formaldehyde based composites basically depends upon the strength and modulus of lignocellulosic *Hibiscus sabdariffa* fibers, effectiveness of the bonding strength between the polymer matrix and fibers in transferring stress across the interface along with the strength and chemical stability of the matrix [5–8]. It has been observed that the mechanical properties of *Hibiscus sabdariffa*-reinforced resorcinol-formaldehyde matrix composites decreases with increase in fiber dimension [8]. This behavior can be explained as due to fiber ndash;fiber contact at higher fiber length. Indeed, during the fabrication of polymer composites with *Hibiscus sabdariffa* fiber, these lignocellulosic fibers acted as carriers of load and transferred stress from the matrix along the reinforcement which resulted in composites with good mechanical properties.

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CONCLUSION

Mechanical properties of polymer composites increase with the incorporation of fiber into the polymer matrix due to the transfer of stress from the matrix to the fiber. However mechanical properties of polymer composites showed a slight decrease with the increase in the dimension of lignocellulosic fiber and excellent results are obtained when the *Hibiscus sabdariffa* fibers used were in the particle form. The results of swelling, moisture and chemical resistance behavior of composites show that the composites are sensitive to swelling and moisture absorption along with reduced chemical resistance due to the hydrophilic behaviour of the lignocellulosic fiber. In spite of these limitations the *Hibiscus sabdariffa* fibers can be a suitable alternative to synthetic fibers as a green reinforcing material for the preparation of various industrially useful polymer matrix-based composites.

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