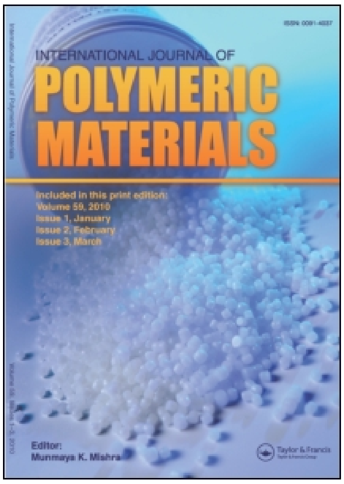

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# Synthesis and Characterization of *Grewia Optiva* Fiber-reinforced PF-based Composites

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# Synthesis and Characterization of *Grewia Optiva* Fiber-reinforced PF-based Composites

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*In this article, the synthesis of natural fiber-reinforced phenol-formaldehyde (PF) resin matrix-based polymer composites has been reported. Initially the phenol-formaldehyde resin was prepared by varying the concentration of formaldehyde with a fixed weight of phenol. Polymeric resins 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* Grewia optiva *fiber in particle form (200 μ) to prepare green polymer composites. The polymer composite materials thus prepared were subjected for evaluation of their mechanical properties such as tensile, compressive, flexural, and wear resistance. It has been observed that optimum mechanical properties were obtained for fiber loading of 30%. Further, the mechanical strength of the composites has been found to be higher than the parent phenol-formaldehyde resin matrix. The morphological and thermal properties of the composites have also been studied.*

**Keywords:** mechanical properties, morphological and thermal behavior, natural fibers, polymer composites

## INTRODUCTION

Natural fibers have been used in the past as reinforcing materials in different types of matrices for increasing the various properties

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of the materials [1–4]. Various examples can be found in domestic life all around the globe, which shows the effective utilization of various natural fibers in a number of applications [5–8]. In recent years greater attention has been paid to their effective utilization as a reinforcing material for polymer matrices. Polymeric materials especially from renewable resources have been vastly used during the last two decades due to environmental concerns [9–11]. The automotive industry has shown more inclination towards these materials because of the advantages offered by them. Natural fibers-based composite materials have made a primary contribution to the well-being and technological development of the human race. These natural fibers offer a number of advantages over synthetic fibers [12–14]. Such as low cost, easy availability, low density, high toughness, good specific properties, biodegradability, and eco-friendliness [15–19]. Natural fibers have generally been classified as bast, leaf or seed-hair fibers. The most important constituents of natural fiber are cellulose, hemicelluloses, lignin and wax. However, the cellulose content in the fiber plays an important role in deciding the physical and chemical properties of the particular fiber. Various natural fibers such as saccaharum cilliare, pinus, hibiscus sabdariffa, flax, cannabis, oil palm, sisal, banana, coconut, and jute have all proved to be good reinforcements in polymer matrices [3–6, 20–22]. The properties of natural fibers generally depend on the source, age and separating techniques of the fiber. All these properties of natural fibers have made them very attractive for various industries currently engaged in searching for products with mechanical properties comparable to those of synthetic fiber-reinforced composites. The most important factors in the development of natural fiber-reinforced composites are fiber dispersion and fiber-matrix interaction.

Himachal Pradesh, being a hilly state, is blessed with vast natural fibers. Because of inaccessibility to these areas this precious wealth is not yet exploited commercially for better end use. Among various types of natural fibers, *Grewia optiva* fibers have high potential as a reinforcing material in polymer matrix-based composites. Since not much information is available in the literature, a comprehensive research program has been started in our laboratory to synthesize *Grewia optiva* fiber-reinforced polymer composites using different polymer resin matrices. In the present article we present the synthesis and study of various properties of *Grewia optiva* fiber-reinforced phenol-formaldehyde (P-F) resin-based biocomposites.

# EXPERIMENTAL

## MATERIALS AND METHODS

### Polymer Matrix

Phenol-formaldehyde solution and sodium hydroxide of Qualigens make were used as received. Phenol-formaldehyde (PF) resin was used as a novel polymer matrix for preparing biocomposites.

### Reinforcing Material

*Grewia optiva* fibers collected from local resources were used as reinforcing material. *Grewia optiva* fibers were first thoroughly washed with detergent powder. 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. After this the fibers were converted into particle form of dimension 200 micron.

### Synthesis of Phenol-Formaldehyde Resin

Phenol-formaldehyde resin was synthesized by the standard method developed in our material science laboratory [23]. Phenol and formaldehyde were taken in different molar ratio (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 a mechanical stirrer. NaOH solution was added slowly with constant stirring and heating to the phenol-formaldehyde solution until pH of 8.5. Since the reaction is exothermic, proper care was taken to maintain the temperature between 50–60°C, for the initial hour. Then the temperature was increased to 70–75°C and the mixture was kept at this temperature, until the resinification started. The reaction mixture was condensed at this temperature for 3–4 h until the complete resinification. Then heating was stopped and the resin was cooled and transferred to specially made molds. Resin sheets of size $150 \times 150 \times 5.0$ mm were prepared by a closed mold method as described elsewhere [4]. The mold 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 h.

### Synthesis of Polymer Composites

Dried *Grewia optiva* fibers of dimension 200 micron were mixed thoroughly with phenol-formaldehyde resin, using a mechanical stirrer, at different loadings (10, 20, 30 and 40% in terms of weight). Then this mixture was poured into specially made molds. The surface of the molds was coated on the inside with oleic acid to avoid adhesion of the mixture and to allow easy removal of the composites. The mixture completely filled the mold. Composite sheets of size 150 × 150 × 5.0 mm were prepared by compression molding technique [4]. Compression molding was performed in a hot press using a mold preheated to 150°C. The material, first placed in a hot open mold, was left for about 7 min, and then the mold was closed. Composite sheets were prepared by hot pressing the mold at 150°C for 30 min. The pressure applied ranges from 3–4 MPa depending on the loading of reinforcing material. All the specimens were then post-cured at 150°C for 12 h.

## CHARACTERIZATION OF POLYMER RESIN AND POLYMER COMPOSITES

### Tensile Strength Test

Tensile strength tests of resin and composite samples were conducted on a Computerized Universal Testing Machine (Hounsfield H25KS). Specimens of dimension 100 × 10 × 5 mm were used for analysis. The tensile test was conducted in accordance with the ASTM D 3039 method. The sample of 10 cm length was clamped into the two jaws of the machine. Each end of the jaws covered 2 cm of the sample. Tensile strength was studied over the rest of 6 cm gauge length. Reading of the tensile strength test instrument for Newton force and extension was initially set at zero. The test was conducted at a constant strain rate of 10 mm/min. Tensile stress was applied until the failure of the sample and the load-extension curve was obtained. Each sample was tested on seven specimens and standard deviation, coefficient of variance and standard error were calculated.

### Compressive Strength Test

Compression strength tests of resin and composite samples were conducted on a Computerized Universal Testing Machine. The test was conducted in accordance with the ASTM D 3410 method. The composite sample was held between two platforms and the strain rate was fixed at 10 mm/min whereas the total compression range was 7.5 mm. Compression stress was applied until the failure of sample. Total compression per unit force was noted.

## Flexural Strength Test

Flexural strength of samples was also determined on the Computerized Universal Testing Machine. The three-point bend flexural test was conducted in accordance with the ASTM D 790 method.

## Wear Test

Wear test of the sample was conducted on a Wear & Friction Monitor (DUCOM- TR-20L). Wear resistance of composites was carried out as per ASTM D 3702 method.

## Stress-Strain Analysis

Stress-strain diagram expresses a relationship between a load applied to a material and the elongation/deformation/deflection of the material, caused by the load. Stress-strain diagram is determined from tensile, compressive and flexural tests. Stress is calculated as usual by dividing the applied load with the initial annular cross-sectional area, while strain is defined as the ratio of the displacement to the initial length.

## Morphological Analysis of Samples

To evaluate changes in the composite surface morphology, fibers and matrix, they were analyzed by scanning electron microscopy (SEM). 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. SEM micrographs of the samples show the morphology of the biocomposites prepared. These micrographs clearly show the difference between unloaded and loaded P-F matrix.

## Thermal Analysis of Samples

Thermal analysis of materials gives us an account of their thermal stability. Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) 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. The mass change characteristics of a material are strongly dependent on the experimental conditions such as sample mass, volume and physical form, the shape and nature of the sample holder, the nature and pressure of the atmosphere in the sample chamber and the scanning rate. 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 vs. temperature is plotted and is used to simplify reading of weight vs. temperature thermogram peaks that occur close together. DTG peaks are characterized by the peak maximum ($T_{max}$) and the peak on set temperature ($T_e$). The area under the 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

Mechanical, morphological and thermal characterizations have been found to be the most trusted tools in determining the overall behavior of polymer composites. Mechanical analysis has been the most significant tool in studying their behavior. In this context we have studied mechanical properties like tensile strength, compressive strength, stress-strain, flexural and wear resistance of fiber-reinforced polymer composites. These static mechanical properties of fiber-reinforced composites depend on the nature of (i) matrix material, (ii) distribution and orientation of the reinforcing material, and (iii) the nature of the fiber-matrix adhesion.

### Tensile Strength

The ability of a material to resist breaking under tensile stress is also one of the most important and widely measured properties of materials used in structural applications. The force per unit area (MPa or psi) required to break a material in such a manner is the ultimate tensile strength or tensile strength at break.

### Compressive Strength

The ability of a material to resist breaking under compression stress is also one of the most important and widely measured properties of materials used in various applications.

The value of uniaxial compressive stress reached when the material fails completely is designated as the compressive strength of that material. The compressive strength is usually obtained experimentally by means of a compressive test. The apparatus used for this experiment is the same as that used in a tensile test.

## Mechanism for Synthesis of Phenol-Formaldehyde Resin

Polymers based on phenol-formaldehyde resin are generally synthesized by the condensation of phenol with formaldehyde. Reaction between phenol and formaldehyde is normally catalyzed by acids and bases. The nature of the product formed as a result of polymerization reaction primarily depends upon the types of catalyst and the molar ratio of the reactants. It has been observed that the reaction between phenol and formaldehyde in a basic medium yields polymeric materials which possess good fire-resistance properties [24]. In the present work phenol-formaldehyde resin has been synthesized by the reaction of phenol with an excess of formaldehyde in the molar ratio of 1:1.5 in a basic medium using sodium hydroxide. The OH-anion contributes to the formation of phenates by abstracting the alcoholic proton. The resulting ortho and para methylol phenols are more reactive towards formaldehyde than the original phenol. These methylol phenols on further reaction with formaldehyde lead to the formation of di- and trimethylol derivatives. The above polymerization reaction on further heating results in the condensation between methylol phenols and methylol phenol; and phenol and methylol phenol, to give products having ether and methylene linkages, respectively. Further heating causes crosslinking among the above products and produces the desired polymeric resin.

## Optimization of Phenol-Formaldehyde Resin

Optimization of phenol-formaldehyde resin was done by taking the mechanical properties such as tensile strength, compressive strength, flexural strength and wear resistance into consideration [3,4,25].

## Tensile Strength

It has been observed that PF samples of ratio 1.0:1.5 bear more load than samples of other ratios [23]. This ratio (1.0:1.5) could bear a load of 471.8 N with an extension of 2.7 mm (Figure 1A).

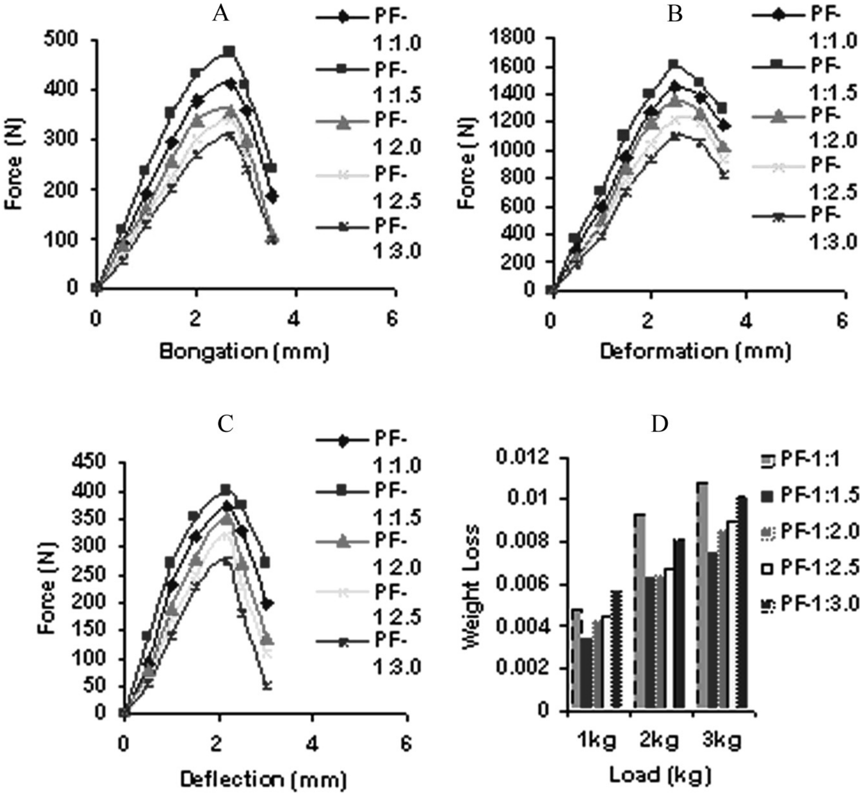

**FIGURE 1** Load elongation/deformation/deflection and wear resistance curve of PF resin (A, B, C, and D).

## Compressive Strength

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

## Flexural Strength

It is evident from Figure 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 has been observed that wear rate of samples of ratio 1.0:1.5 was lower than other samples. Loss of material was due to abrasion and friction of samples with disc (Figure 1D).

The wear resistance results also supports the tensile, compressive and flexural results, as brittle materials have higher wear rate and low tensile, compressive and flexural strength. The ratio (1.0:1.5) of phenol to formaldehyde showing optimum mechanical properties was taken for further preparation of biomass reinforced phenol-formaldehyde biocomposites.

## Effect of Reinforcement on the Mechanical Properties of PF Matrix-based Polymer Composites

### Tensile Strength

It has been observed that tensile strength of composites increases on reinforcement with *Grewia optiva* fiber. Composites with 30% wt loading bear maximum load followed by 40, 20, and 10% loadings Figure 2A. They bear a load of 1893 N, 1787 N, 1619 N and 1387 N, respectively.

### Compressive Strength

Compressive strength of PF matrix has been found to increase with reinforcement with *Grewia optiva* fibers. It has been found that on particle reinforcement compressive strength increases to a greater extent. The compressive properties of the composites as the function of load and deformation are presented in Figure 2B. From the graph it is evident that first the compressive force increases with the increased fiber content, but after 30% fiber content a significant decrease in compressive strength is observed. The strength values are 3997.5 N, 4393.5 N, 5796.0N and 5258.0 N, respectively.

### Flexural Strength

Similar trends as obtained in tensile strength and compressive strength tests have been observed for flexural strength results. The flexural properties of samples as a function of force (in terms of load) and deflection are shown in Figure 2C, with values of 1071.47 N, 1340 N, 1637.41 N and 1528 N, respectively.

### Wear Test

As evident from Figure 2D the wear rate of the PF matrix decreases appreciably as reinforced with *Grewia optiva* fiber. The maximum

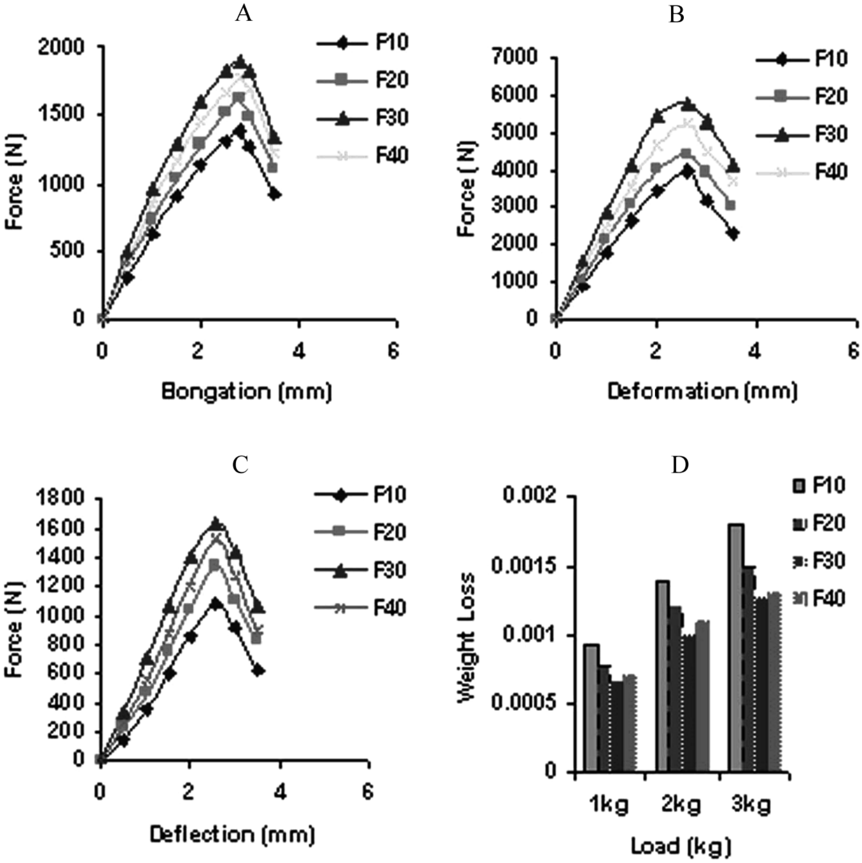

**FIGURE 2** Load elongation/deformation/deflection and wear resistance curve of fiber-reinforced composites (A, B, C, and D).

wear resistance behavior is shown by composite with 30% loading followed by 40, 20 and 10% loading.

## Stress-Strain Behavior

### *Tensile Stress-Strain Curve*

The tensile stress-strain curve is constructed from the load elongation measurements. Tensile stress is obtained on dividing the load by the original area of the cross-section of the specimen. The strain used for the tensile stress-strain curve is the average linear strain, which is obtained on dividing the elongation of the gauge length of the specimen by its original length. Since both the stress and the strain

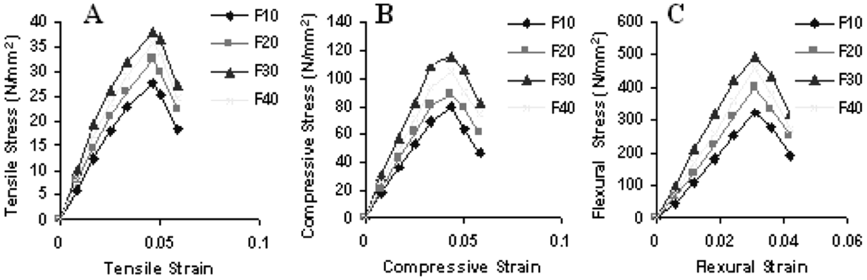

**FIGURE 3** Stress-strain curves of polymer composites under tensile, compressive and flexural tests (A, B, and C).

are obtained by dividing the load and elongation by constant factors, the stress-strain curve has the same shape as the load-elongation curve. The plot of stress vs. strain for the *Grewia optiva* fiber reinforced polymer composites is shown in Figure 3A. From the figure it is clear that in the elastic region stress is linearly proportional to strain. When the load exceeds a value corresponding to the yield strength, the specimen undergoes gross plastic deformation. It is permanently deformed if the load is released to zero. Various parameters derived from the tensile stress-strain curve are shown in Table 1.

## Compressive Stress-Strain Curve

The plot of compressive stress vs. strain for *Grewia optiva* fiber-reinforced polymer composites is shown in Figure 3B. From this figure it is clear that in the elastic region stress is linearly proportional to strain. Various parameters derived from the compressive stress-strain curve are shown in Table 2.

**TABLE 1** Parameters Obtained from Tensile Stress-Strain Curve at Different Loadings

| Fiber loading (Wt.%) | Ultimate tensile stress N/mm$^2$ | Yield strength N/mm$^2$ | Fracture stress N/mm$^2$ | Standard deviation | Tensile modulus N/mm$^2$ |
|---|---|---|---|---|---|
| PF | 9.40 | 8.10 | 8.70 | 0.72 | 207.70 |
| 10% | 27.74 | 21.06 | 25.73 | 1.07 | 590.27 |
| 20% | 32.38 | 25.15 | 29.21 | 1.04 | 681.84 |
| 30% | 37.86 | 29.16 | 33.8 | 0.97 | 801.4 |
| 40% | 35.74 | 28.18 | 31.75 | 1.15 | 761.95 |

**TABLE 2** Parameters Obtained from Compressive Stress-Strain Curve at Different Loading

| Fiber loading (Wt.%) | Ultimate compressive stress N/mm$^2$ | Yield strength N/mm$^2$ | Fracture stress N/mm$^2$ | Standard deviation | Compressive modulus N/mm$^2$ |
|---|---|---|---|---|---|
| PF Resin | 31.94 | 26.07 | 29.14 | 0.87 | 771.00 |
| 10% | 79.95 | 67.15 | 74.78 | 1.70 | 1841.40 |
| 20% | 87.81 | 75.32 | 82.25 | 1.57 | 2072.00 |
| 30% | 115.92 | 99.73 | 107.26 | 1.92 | 26792.81 |
| 40% | 99.00 | 83.37 | 90.23 | 1.45 | 2441.58 |

## Flexural Stress-Strain Curve

Flexural stress-strain curve is also constructed from the load deflection measurements. A plot of flexural stress vs. strain for *Grewia optiva* fiber-reinforced polymer composites is shown in Figure 3C. The parameters derived from the flexural stress-strain curve are shown in Table 3.

In order to achieve good fiber reinforcement, interfacial strength between the fiber and matrix is a most essential factor. For a composite to be an effective load-bearing system, the fibers and matrix must have a strong interface. The interfacial strength depends on the surface morphology 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 PF matrix as well as the formation of chemical bonds between the fiber surface and the PF matrix. Hence the interface plays a key

**TABLE 3** Parameters from Flexural Stress-Strain Curve for Different % Age Loading

| Fiber loading (Wt.%) | Ultimate flexural stress N/mm$^2$ | Yield strength N/mm$^2$ | Fracture stress N/mm$^2$ | Standard deviation | Flexural modulus N/mm$^2$ |
|---|---|---|---|---|---|
| PF Resin | 119.34 | 107.37 | 114.98 | 0.75 | 3582.94 |
| 10% | 321.4 | 307.03 | 316.15 | 1.02 | 9422.86 |
| 20% | 402.00 | 389.52 | 397.37 | 1.17 | 11035.02 |
| 30% | 491.22 | 475.72 | 483.30 | 1.25 | 12920.88 |
| 40% | 458.4 | 447.72 | 453.53 | 1.43 | 11850.84 |

role in controlling the mechanical properties of a composite. It can be concluded that different degrees of reinforcement effects are achieved by the addition of natural fibers to the PF matrix-based polymer. This may be due to the different adhesion strength between matrices and fibers. The adhesion is usually the strongest in polar polymers capable of forming hydrogen bonds with hydroxyl groups available on the fiber surface. The chemical bonding accounts for the adhesion between PF resin and cellulose of fiber. Still higher bond strength obtained for PF resin matrix is due to the possible reaction between the methylol groups of the resin with the hydroxyl group of cellulose.

A decrease in the mechanical properties of PF matrix-based composites is observed when fiber loading reaches beyond the optimum value. It has been observed that mechanical properties increase up to 30% fiber loading and then decrease. The mechanical strength of PF composites levels off at high fiber loading This behavior can be explained as due to fiber-fiber contact at higher fiber loading. Hence lower results are obtained for 40% loading. 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 a lower mechanical properties. At 30% levels of loading the population of the fibers is just right for maximum orientation and the fibers actively participate in stress transfer and hence maximum results are obtained in case of this loading. As discussed, the modulus showed a clear continuous increase up to 30% fiber loading in the case of random orientation. At higher levels of fiber loading, the increased population of fibers led to agglomeration, and stress transfer was partially blocked resulting in lowering of mechanical properties above 30% loading.

## Morphological Study of Biocomposites

Morphological results (Figure 4) show that there is proper intimate mixing of fiber with the resin in the biocomposites thus synthesized. These results evidently demonstrate that when polymer resin matrix is reinforced with different loadings of fiber, morphological changes take place depending upon the interfacial interaction between the varying loading of fiber and the resin matrix.

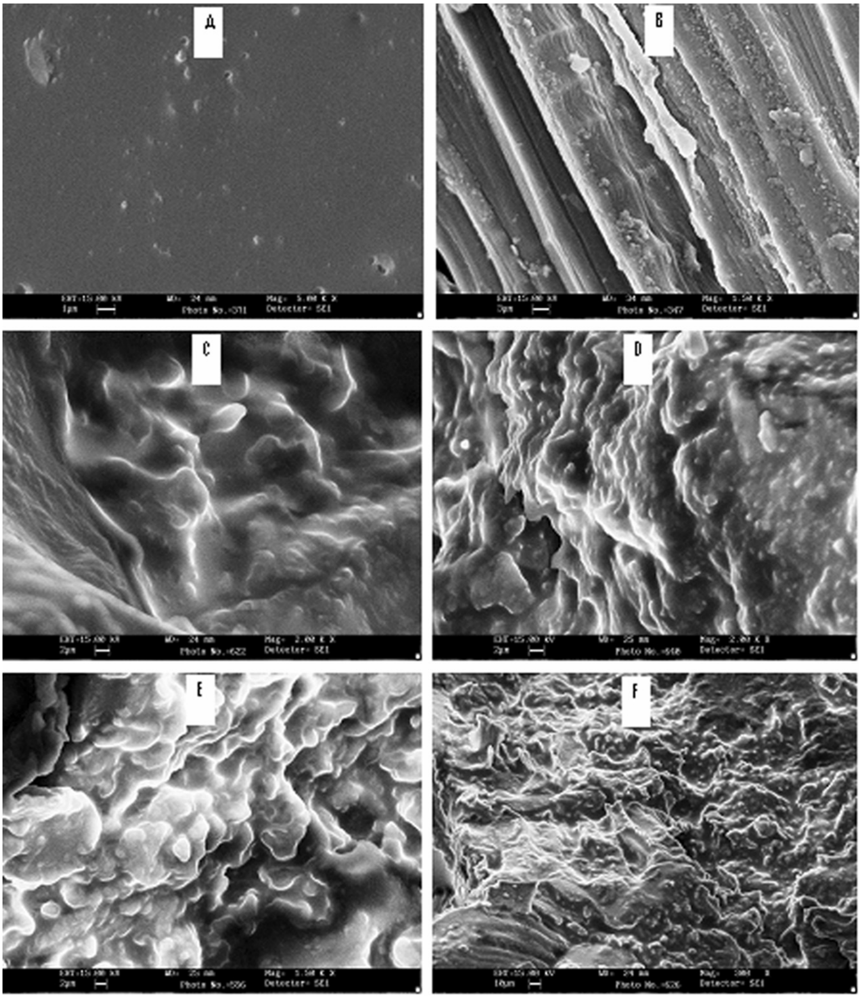

**FIGURE 4** SEM images of (A) RF resin (B) *Grewia optiva* fiber (C, D, E and F) composite with 10, 20, 30 and 40% loadings.

**TABLE 4** TGA/DTA Analysis of PF, GO and P-Rnf-PF Composites

| Sr. no. | Sample code | IDT (°C) | %wt. loss | FDT (°C) | %wt. loss | Final residue (%) | Exothermic endothermic peaks °C(μV) |
|---|---|---|---|---|---|---|---|
| 1 | GO | 200 | 7.25 | 501 | 87.44 | 12.66 | 61 [−2.0]; 361 [−3.3] |
| 2 | P-F Resin | 400 | 15.68 | 1195 | 48.53 | 51.47 | 164 [9.0]; 421 [6.0] |
| 3 | P-Rnf-PF | 337 | 21.39 | 989 | 53.84 | 46.26 | 69 [−4]; 335 [−5]; 695 [−35] |

**TABLE 5** DTG Analysis of G.O., PF and P-Rnf-PF Composites

| Sr. no. | Sample code | Exothermic peaks temperature (mg/min) |
|---|---|---|
| 1 | GO | 61 [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 | P-Rnf-PF | 59 [53]; 179 [67]; 325 [282]; 547 [110] |

## Thermal Behavior of PF Resin and Its Composites

Thermogravimetric analysis (TGA) of raw fiber, polymeric resin and biocomposite with 10% loading was studied as a function of % weight loss with the increase in temperature. In case of raw fiber, depolymerization, dehydration and glucosan formation first took place between the temperature ranges of 191° to 230°C followed by the cleavage of C-H, C-C and C-O bonds. Initial decomposition (IDT) temperature is 200°C and final decomposition temperature is 501°C. However, in the case of phenol formaldehyde resin there is a single stage decomposition, and the initial decomposition temperature is 400°C. The final decomposition of the resin took place at 1195°C. The degradation temperatures for natural fiber-reinforced composites fall between the degradation temperatures for the matrix and the fibers. It has been observed that for biocomposites the initial decomposition temperature is 337.0°C and the final decomposition of the composite took place at 989°C. This indicates that the presence of cellulose fibers affects the degradation process. The initial decomposition (IDT) temperature, final decomposition temperature (FDT) and differential thermal analysis (DTA) of fiber, resin and biocomposite are presented in Table 4. This is consistent with results reported earlier [3]. These studies are further supported by DTG results shown in Table 5.

## CONCLUSIONS

The mechanical properties of *Grewia optiva* fiber-reinforced polymer composites have been investigated as a function of the chemical nature of matrix polymer and the content of the reinforcing material. The mechanical properties of *Grewia optiva* fiber-reinforced composites have been found to be higher than those of the matrix polymer. Further, *Grewia optiva* fiber loading of 30% in a particulate form imparts maximum mechanical strength to the composite material. These properties can make *Grewia optiva* fibers a potential material for the synthesis of a new class of biocomposites.

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