Experimental Study of Thermal and Mechanical Behaviour of Graphite-Filled UJF Composite

K. Manohar Reddy,1 D. Harsha Vardhan,1 Y. Santhosh Kumar Reddy,1 Gujjala Raghavendra,2 and Ramesh Rudrapati3

1Department of Mechanical Engineering, PVKK Institute of Technology, Ananthapuramu, Andhrapradesh, India
2Department of Mechanical Engineering, National Institute of Technology, Warngal, Telangana, India
3Department of Industrial Engineering, Bule Hora University, Bule Hora, Ethiopia

Correspondence should be addressed to Ramesh Rudrapati; rameshrudrapati@gmail.com

Received 8 June 2021; Revised 28 June 2021; Accepted 5 July 2021; Published 13 July 2021

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The advancement of composites mixed with natural fibers and with fillers has become the most supportable alternative material for engineering applications, especially in industries such as automotive and aerospace. Natural fibers are renewable, cheap, biodegradable, and ecological materials. In the present work, already used woven jute fibers, which are extracted from gunny bags with the same grams per square meter (GSM), were used, and then, woven jute fibers were chemically treated to improve their characteristics. Graphite powder-filled used jute fiber reinforced epoxy composite (UJF) are prepared by using the hand-layup technique. Tests such as tensile, flexure, impact, and thermo-gravimetric analysis (TGA) were conducted. These tests were according to ASTM standards to evaluate the effect of graphite filler content on hybrid epoxy jute composites. The composite material is prepared by changing the content by weight of the filler by 3%, 6%, 9%, and 12%. The experimental results reveal that 6% of the graphite composites showed the maximum tensile strength and modulus. With the increase in the filler content, there is a decrease in the flexural properties. The impact resistance increases slightly up to 6% of the filler content. The study of thermal decomposition showed that the lowest mass loss was found at 9% by weight of the filler content. Morphological analysis performed by FE-SEM showed that the addition of filler content improved the binding of the fiber and matrix up to 6% by weight of the filler content. It should be noted that these hybrid composites are a promising material at low cost for lightweight structural applications.

1. Introduction

The usage of naturally produced plant fibers to reinforce polymeric composites for the production of inexpensive engineering materials has attracted considerable interest in recent years [1]. The latest environmental regulations, as well as market demand, have led the manufacturing sectors (in particular, the automotive [2, 3], construction [4], and packaging [5]) to seek alternative products that can be replaced by existing nonrenewable reinforcing materials such as synthetic fibers [6]. The natural properties provided by the natural fibers help in great thermal and acoustic insulation such as lowering density and giving a traditional cell structure [7–9]. Scientists across the world started to work on finding out an alternative energy in the scenario of a growing crisis of energy. The fibers traditionally used were carbon, glass, and others. The plastics reinforced with sisal [10, 11], jute [12, 13], bamboo [14], and porcelain fibers [15], amongst the natural fibers, have attracted attention for their low cost compared to man-made fibers [16, 17]. Graphite is a pure carbon in crystalline form, very similar to mica sheets formed tightly by bound atoms [18, 19]. However, at normal temperatures, graphite behaves like perfect dry lubricant due to its porosity [20]. This consists of a structure where the carbon atoms are linked. This link is created by covalent bonds with the other carbons in the same plane by the Van
Der Waals force acting between sequential layers [19]. For the effective use of graphite as stuffing in a polymeric composite, the layers should be detached and spread in the polymeric matrix [21], jute fibers were extracted from the jute substrate, which has a bidirectional orientation [22] and the flexibility to improve the properties of natural fibrous composites by modifying the graphite filler matrix [23]. TGA is a heating method that is widely used to describe both inorganic and organic materials, including polymers (e.g., cellulose). It offers quantitative data on the weight loss of a material as a function of temperature [24]. In our previous work for the optimization of the chemical treatment carried out, K\textsubscript{3}\text{MnO\textsubscript{4}} was obtained with a concentration of 30% and a treatment time of 40 minutes, and the same methodology has been adopted in the present work.

In that perspective attempt, the influence of micro-graphite filler on the thermal and mechanical properties of used jute natural fiber composite fabricated through hand-layup technique is studied. The FE-SEM analysis was used to analyse the fractured surface. The results were compared with nontreated used jute fiber composite and treated used jute fiber composites. The results revealed that the graphite filler has a significant impact on the material characteristics.

2. Materials and Methods

2.1. Matrix and Fibers. The Araldite LY 556 (Epoxy) and HY951 (Hardener) in 10:1 ratio is used as a matrix [25]. Used jute woven fiber has been extracted from gunny bags, as shown in Figure 1, and woven jute fibers of 350 ± 10 gram per square meter (GSM) was taken for the study.

2.2. Filler. Graphite filler powder of 10–20 microns is used in the present work. The filler content is varied by weight percentage in the matrix by 3 wt.% to 12 wt.% in the 3 wt.% step sizes. The powder is having a density of 1980 kg per cubic meter and a molecular weight of 12.011 g/mol.

2.3. Chemical Treatment. Initially, the fibers were soaked in methanol and Benzene in a 1:1 ratio for 24 hours to remove wax, paint, and foreign contaminants. From the weight loss of jute (fabrics) by the N\textsubscript{a}OH treatment, it was observed that considerable hemicelluloses content was dissolves. To strengthen the fiber, further treatment was done using alkaline, benzyl chloride, and permanganate solutions, respectively. The optimal chemical treatment for used jute fiber is treating with 30% concentration for 40 minutes and post-treating with K\textsubscript{3}MnO\textsubscript{4} [23], for treated composites named in the present work as 30N40K composites, i.e., 30, for 30%, N for NaOH, 40 for 40 minute treatment time, and K for K\textsubscript{3}MnO\textsubscript{4}. For the composite, adding filler after treatment was named as 30N40K + percentage of filler.

2.4. Preparation of Composite. In the development of hybrid composites, the hand-lay-up technique was used. Varying proportions of a weighted amount of graphite powder and epoxy resin were mixed and later the hardener (HY-951) was mixed to begin the reaction. With diverse weight fractions of graphite filler (3%, 6%, 9%, and 12%) in the matrix, specimens were prepared. Filler volume was restricted to 12% in the matrix because of the increase in the filler concentration up to 12%, and the mixture became dense losing its fluidity as of the occurrence of gelation. A mould has been prepared for preparing the composites. Moreover, a spray release the mould has been used to ease the cast plate release. With the help of a mechanical jig-saw cutting machine, the dimensions required were marked and sized using the plate. Composites were prepared and tested as per the ASTM standards.

2.5. Testing. Mechanical testing such as tensile, flexural, and impact testing were carried out as per ASTM Standards of D638, D790, and D256. Tensile and flexural tests were carried out using computerised universal testing machine made by Instron. Test is conducted at a cross head speed of 2.5 mm/min. The impact test was carried out with an impact hammer striking at the rate of 3.2 m/s of mass 1.3 kg.

3. Results and Discussion

3.1. Tensile Strength. Figure 2 shows the differences in the strength of the tensile of the laminated composites with graphite filler and without graphite filler. The tensile test was done on the used jute fiber composite and graphite-filled jute fiber composite samples. This was done so to understand the tensile strength. In addition, to know the contribution made by the fillers, the addition of filler influences the variations in the tensile strength of the laminated composites, and this is due to the good dispersion of the filler in the matrix obtained by mechanical stirring.

Figure 3 shows that the failure of treated jute epoxy is due to fiber pull-out, and it is very clear that there is weak bonding between the matrix and the fiber due to which fibers slipped from the matrix and the scars are observed on the matrix. The untreated used jute epoxy composite has tensile strength of 68 MPa; after chemical treatment, it has enhanced by 34% and reached to 96.28 MPa. The tensile strength of graphite-filled jute fiber composites is higher when compared to that of unfilled composites. This might be because of the restriction of the movement and matrix deformability with the introduction of mechanical restraint and the filler particle size.
From Figure 2, the results show that 6 wt.% graphite added epoxy composite with jute fiber as reinforcement yielded the maximum tensile strength value of 111.98 MPa. From Figure 4, it is very clear that, for 6 wt.% filler content, there is strong bonding between the fiber and the matrix. The morphology result shows the splitting of fibers and initiation of matrix cracks. Initially, the matrix fails; then, fiber failure occurs at this percentage. Due to the doping of filler and matrix first failure, the specimen, modulus of composite is reduced, but the strength is enhanced.

Subsequently, it is noticed that because of the increase of filler from 9% to 12%, it is observed that the gradual decrease of the tensile strength and the tensile modulus shows slopes down. At 12 wt.% of graphite, tensile strength is observed as 101.14 MPa and modulus is as 2.41 GPa. From Figure 5, it is evident that failure of the composite is due to the slipping of fibers from the matrix. This phenomenon is due to more amount of filler in the matrix that allows the fiber to slip over the graphite as it is a solid lubricant.

It is obvious that the adding of graphite after a certain amount worsens the tensile strength and the elastic modulus of the composite. It is interesting to note that the increase content of the filler alone and/or 3 wt.% graphite to the fiber shows an enhancement in the vital Longitudinal tensile load-bearing capacity, i.e., tensile strength and the elastic modulus of the epoxy composites [20].

3.2. Flexural Strength. Additionally, the study demonstrates that when graphite content is increased to 9 wt. percent of filler, and the flexural strength increases from 162 MPa to 212 MPa, as shown in Figure 6. This may be due to better adherence of fillers between the matrix and the fiber and also with enriched internal bonding load bearing capability enhanced. Furthermore, flexural strength decreases with the increase of graphite filler due to the agglomeration of graphite filler in the composite. This is due to the high hardness of the filler material [26]. The flexural modulus decreases with an increase in filler content, i.e., graphite percentage decreases.

3.3. Impact Strength. Impact test is conducted to analyse the impact capability of the diverse specimens. Based on the reading obtained from the Charpy impact machine, the energy loss is identified. The impact toughness (strength) is identified by the energy loss of the pendulum, or it can be identified by accurately measuring the height loss in the pendulum’s swing. The changes in impact toughness for jute/epoxy laminated composites according to the filling percentage were shown in Figure 7. The percentage of filling is effective on the impact behaviour of jute/epoxy composite material. Among various filling percentages of jute/epoxy composite which was reinforced with 6% filling percentage of graphite, filler had the highest improvement in impact toughness. Graphite filling into jute epoxy up to 6% increased the impact toughness of the material. After 6% filled percentage, these values decreased. This is because the strength in impact loading reduced with the increasing filler density. This occurs as a result of the material’s decreased elasticity and the inclusion of fillers, limiting the matrix’s deformability and ability to absorb deformation energy. The chemical treatment has greater effect on impact resistance. The filler percentage does not show much effect on impact resistance; however, after 6 wt.%, there is slight reduction in resistance. The more the material’s impact energy, the greater its toughness, and vice versa [27].

3.4. Thermal Properties. The thermo-gravimetric analysis (TGA) results of used jute epoxy composite at various filler content are shown in Figure 8. The results revealed that the mass loss was initiated at 100°C and continued up to 410°C. Majority of the mass loss was observed between the temperature range 350–410°C. Up to 100°C, the moisture present in the sample will be evaporated, and then, the matrix
thermal erosion started. From results, it is very clear that the composite specimen without filler initiated mass loss after 100°C, but adding the filler to matrix graphite filler enhanced the stability to resist high temperatures. The matrix degradation was continued up to 350°C.

After 350°C with complete combustion of the matrix, the fibers were exposed and caused by rapid mass loss in temperature range of 50°C. Natural fiber has less temperature resistance capability. After 400°C, the mass loss was minimum. Almost 70 percent of the composite was completely burnt. The remaining residue slowly degraded with the increase in temperature. After 500°C, there is no observable change in the mass. The residual was very less without filler composite specimen. The residual was
Figure 6: Flexural strength of UJE polymer composites.

Figure 7: Impact strength of UJE polymer composites.
increasing with the increase in filler content, as the melting point of graphite filler was very high. The residual will increase with an increase in filler content. At the bottom line, the graphite filler enhanced the thermal stability of the composite material and residual.

4. Conclusions

In the present research, by using hand-layup technique, a variety of hybrid composites were developed through the used jute and graphite fillers, which were strengthened with the epoxy thermoset matrix. Evaluation was done on thermal and mechanical properties. The following conclusions were made by this study:

1. The results show that 6 wt.% graphite-added epoxy composite with used jute fiber as reinforcement yielded the maximum tensile strength values of 111.98 MPa. The addition of filler influences the variations in the tensile strength of the laminated composites and this is due to the good dispersion of the filler in the matrix attained by mechanical stirring.

2. The 9 wt.% graphite added epoxy composite with used jute fiber as reinforcement yielded the maximum flexural strength value of 212 MPa.

3. The 6 wt.% of graphite filler composite had the highest improvement in impact toughness. Graphite filling in used jute epoxy up to 6% increased the impact toughness of the material.

4. The TGA curve indicated that the graphite filler with 3 wt.% composite shown better thermal stability than the chemically treated used jute fiber composite.

5. Graphite filler enhances the mechanical property, creating a mechanical lock between the fiber and the matrix up to 6 wt.% of filler; beyond that, due to the domination of filler content in the matrix, the graphite filler exhibits its natural solid lubricant behaviour, leading to fragile composite.

Data Availability

The data used to support the findings of this study are included within the article.

Disclosure

It was performed as a part of the employment of Bule Hora University.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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