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Experimental investigation on jute/snake grass/kenaf fiber reinforced novel hybrid composites with annona reticulata seed filler addition

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Abstract
Natural fiber composites are hybridized nowadays to explore the synergetic effect of more fibers used in the properties of the composites. The natural fiber hybrid composite made with filler material has excellent wear resistance characteristics. This research work examined the mechanical properties, namely tensile, flexural, interlaminar shear strength, impact strengths, and hardness of the natural fiber reinforced hybrid composite that uses jute, snake grass, and kenaf fibers as reinforcement with various fiber volumes. Further, the wear behavior of the hybrid composites was enhanced by using Annona reticulata (custard apple) seed powder as a filler material. This study revealed that the sample has an equal proportion (12.5% of each) of snake grass and kenaf fiber (without filler) has excellent mechanical strengths. The wear behavior of the sample with 5 wt% filler shows a lower wear rate than other samples.

1. Introduction

Natural fiber-reinforced composites (NFRCs) are gaining the focus of researchers and scientists due to the diversity in their properties. NFRCs found their application in defense, aerospace, and automobile industries by replacing metal structures. NFRCs have a high strength-weight ratio, extended fatigue life, corrosion resistance, and eco-friendliness [1]. But, the massive production of NFRCs is a challenge due to the variation in their properties due to the manufacturing methods, origin, and quality of natural fibers, and lack of knowledge in machinability and parameter settings [2]. Natural fibers are also called lingo-cellulosic fibers, constituted by lignin, cellulose, and hemicellulose.

Lingo-cellulosic fibers are hydrophilic and incompatible with thermoplastics, resulting in a weak bond with the resin matrix. Therefore, the fiber surface is modified using chemicals to make the fibers less hydrophilic [3]. The alkaline chemical agent NaOH was used for cleaning the surface ad altering the cellulosic fiber’s structure, and the process is known as alkalization [4]. The surface morphology, thermal properties, and the crystallinity index of the lingo-cellulosic fibers, namely sisal, jute, kapok, and hemp fibers, were treated chemically with NaOH at 20°C for 2 days, and the properties were compared with the untreated fibers. This work proved that the chemically treated fiber has better bonding with resin, leading to enhanced mechanical and thermal properties [3]. The alkalization of lingo-cellulosic fibers with 5% NaOH for 60 min registered the optimum properties with eliminated lignin and hemicellulose [6].

The NFRCs can be fabricated by the compressed mold technique in which split mold holds the arrangement of matrix and reinforcement at high pressure for a specific time with or without heat. The range of pressure and heat is based on the size and shape of the composite fabricated [4]. The composite fabricated using jute fabric with a bio pool showed improved adhesion properties and thus registered higher mechanical properties [7]. The effect of alkaline (NaOH and KOH) treatment of kenaf fiber as that of untreated kenaf fibers as reinforcement in the epoxy composites on mechanical properties were compared experimentally, and the optimum fiber content was reported to be 40% in weight. They also added that the NaOH-treated kenaf fiber showed 26.8%, 37.2%, and 25.8% higher tensile, flexural, and impact strengths than KOH-treated fibers [8].
Mechanical properties of the NFRC depend on various factors like the type of fiber, fiber maturity, orientation, alignment, distribution and chemical treatment of fiber, weight or volume fraction, processing methods and additives used, etc [9–13]. A jute fiber reinforced epoxy resin composite using cenosphere filler material by hand layup technique showed increased hardness, flexural strength, impact strength, and shearing of lamina for the addition of fiber up to 35%. The water-absorbing capacity of the composite was reduced while adding the filler material and was increased with the addition of fiber content [14]. The mechanical characterization of jute fiber reinforced epoxy resin composite fabricated by vacuum-assisted infusion showed higher strength and stiffness. But, the henequen fiber reinforced epoxy composite fabricated by the same method showed higher strain values than the former [15]. In addition to the NFRCs, natural fibers reinforced hybrid composites NFRHCs were prepared using two or more fibers as reinforcement to obtain diversified properties.

The NaOH-treated snake grass fibers reinforced epoxy composites were analyzed for mechanical and thermal properties and compared with snake grass fiber and sisal fiber RCs, concluding that the 4% of SiC in the resin matrix with hybrid reinforcement has improved mechanical properties and thermal properties. Further, the water absorption was also reduced [16]. The study on the flexural strengths of sisal, jute, and flax fiber-reinforced polymer hybrid composite has optimized the parameters, namely type of fiber, type of chemical treatment, volume fraction of fiber, and duration of chemical treatment by response surface methodology. This study concluded that flexural behavior was improved by adding jute fiber [17]. The effect of rape short fiber reinforcement in HDPE (high-density polyethylene), PS (polystyrene), and POM (polyoxymethylene) were studied for mechanical and thermal properties and reported that the maximum flexural strength was registered in HDPE reinforced with 30% wt% of the rape–reinforced composite. The TGA and DTA examination showed that the reinforcement started to burn before the matrix began, and thus burning was delayed [18]. The reinforcement of sisal, ramie, and curious fibers and their hybrid in epoxy resin confirmed that the mechanical properties of the sisal–based composite were improved by hybridization, and hybridization has no effect on the thermal stability of the composite [19]. The doum fiber reinforcement of 30 wt% in the LDPE matrix has 145% of mechanical properties as a neat polymer. Also, it showed 135% of Young’s modulus in 20 wt% of fiber and 97% of torsion modulus.

Further, the study stated that the added doum fiber reduced the thermal properties [13]. 5% of date palm fiber with epoxy resin showed thermal conductivity of about 6 W/mk. Further addition of fiber reduced the thermal conductivity as the date palm fiber is a good insulator. Also, the presence of voids and the homogeneity of fiber and matrix reduced the thermal conductivity [20].

A study of the past literature ensures that the natural fiber composite is now potentially used for various applications [21–23]. There is no study on the properties of epoxy resin composites hybridized with jute, snake grass, and kenaf fibers. Various fillers of derived from waste organic seeds from the plants such as Tamarindus indica, palm, false Ashoka, or other minerals such as red mud, and coconut shell ash have been added to the matrix to reduce the material costs with excellent wear properties [24–29]. This research work aimed to explore the possibility of using novel Annona reticulate seed powder as filler material in the epoxy matrix for the development of partially biodegradable material for erosive applications.

2. Materials and methods

2.1. Materials

**Jute (J) fiber** is lignocellulose, partially wood and textile fiber, a product from the bast of the plant genes corchorus of the *Malvaceae* family. A typical jute fiber has 64% cellulose, 12% hemicelluloses, 11.8% lignin, 10% water, water-soluble 1%, 0.5% wax and 0.2% of pectin. Several cells formed a layer of lignin and hemicelluloses, and multiples of these primary and secondary cell walls formed a multiple-layer composite [30].

**Snake grass (SG) fiber**, also known as *Sansevieria Ehrenbergii*, can be found in the rural area of Tamilnadu, India. A typical SGF, has 80% of cellulose, 11.25% of hemicelluloses, 10.55% of moisture, 7.8% of lignin, 0.6% of ash and 0.45% of pectin [31]. Kenaf (K) fiber has great potential to replace synthetic fibers. Kenaf fiber has 58% of cellulose which enhances biodegradability and eco-friendliness. Also, it has hemicelluloses, lignin, and ash, about 22%, 17.5%, and 2.4% [32]. The fibers used for this research were procured from Gogreen products, Chennai. The fibers used in this work are shown in figure 1. Physical properties of the selected fibers are listed in the table 1.

**Epoxy resins (LY 556) were used as a matrix as they are superior in mechanical behavior, make a strong bond with fibers, and highly resist environmental degradation. Also, high resistance to water as it does not have an ester group. Epoxy was purchased from Covai seenu & Company, Coimbatore, Tamilnadu.**

**Annona reticulata** were collected from the matured fruit seeds. The seed is dried under hot sun for 7 days. Then the dried seeds are ball-milled with the help of planetary ball mill. The ball-to-powder weight ratio is taken as 15:1 with the motor speed of 60 rpm for the milling time of 16 h and the stearic acid is used as controlling agent. Furthermore, ball milled AR seed size maintained in micro level.
2.2. Composite preparation

The samples were prepared as per the ratio presented in table 2. The weight percentages of epoxy resin and jute fiber were kept constant at 70 and 5. The remaining 25 wt% was shared by snake grass fiber and kenaf fiber.

The composites were prepared using the compression molding technique. A cleaned and well-lubricated mold was filled with the first layer of resin 2 mm thick. A homogeneous mixture of weighed fibers was distributed evenly on the resin layer over which another resin layer was formed. The mold was closed, and a hydraulic press applied 3 MPa pressure for 24 h. The pressure was released gradually, and the composite plate was ejected from the mold. The samples prepared are shown in figure 2.

The mechanical behaviors of all five samples of J/S/SG/K FRC (Jute/Snake grass/Kenaf fiber reinforced composites) of various proportions were studied, and the superior sample was identified. Three samples were prepared for each test and average values are recorded. To improve this fabricated sample’s thermal and wear behavior, the Annona Reticulata seed powder was added as filler material in the (J/S/SG/K/AR RC) in various proportions, as presented in table 3 fabricated. The microstructures of these samples were studied, and wear behavior was analyzed. Figure 3 is the sample prepared with filler material.

2.3. Tests

2.3.1. Mechanical properties

The tensile test measures the force needed to break a specimen of standard size 250 mm × 25 mm × 2.5 mm, and the gauge length was 150 mm as per the ASTM D3039/D3039M. The test specimen was gripped firmly, and

Table 1. Physical properties of the fibers [33, 34].

| Property               | Jute fiber | Snake grass fiber | Kenaf fiber |
|------------------------|------------|-------------------|-------------|
| Density (g/c.c.)       | 1.3        | 0.887             | 1.4         |
| Tensile strength (MPa) | 393–773    | 240–450           | 930         |
| Young’s modulus (GPa)  | 26.5       | 6.42              | 20          |
| Elongation at break (%)| 1.5–1.8    | 2.87              | 1.6         |
The tensile force was applied with a 1 kN load cell at a rate of 2 mm per minute in a universal testing machine (Test Bench, Dak Systems Inc.).

The flexural strength test induces compressive and tensile stresses on the specimen’s concave and convex sides. According to the ASTM 790 standard, the test specimen with the dimension of $150 \times 12.5 \times 2.5$ mm was held on a support span of 100 mm, and the load was applied at the center of the specimen to produce three-point bending [24]. A load cell of 20 kN was used with a 2 mm per minute crosshead speed.

**Table 2. Composition of composite samples.**

| Sample No. | Epoxy resin wt% | Jute fiber wt% | Snake grass fiber wt% | Kenaf fiber wt% |
|------------|------------------|----------------|-----------------------|-----------------|
| 1          | 70               | 5              | 5                     | 20              |
| 2          | 70               | 5              | 10                    | 15              |
| 3          | 70               | 5              | 12.5                  | 12.5            |
| 4          | 70               | 5              | 15                    | 10              |
| 5          | 70               | 5              | 20                    | 5               |

**Table 3. Composition of samples with filler material.**

| Sample | Epoxy resin wt% | Jute fiber wt% | Snake grass fiber wt% | Kenaf fiber wt% | Annona reticulata wt% |
|--------|------------------|----------------|-----------------------|-----------------|-----------------------|
| A      | 70               | 5              | 11.25                 | 11.25           | 2.5                   |
| B      | 70               | 5              | 10                    | 10              | 5                     |
| C      | 70               | 5              | 8.75                  | 8.75            | 7.5                   |
| D      | 70               | 5              | 7.5                   | 7.5             | 10                    |

**Figure 2.** J/SG/K FRC samples.
Interlaminar shear strength is the force required to shear the layers (laminates) in the composite. It was found from the relationship between flexural strength and the cross-section of the specimen. Interlaminar shear strength is calculated according to equation (1).

\[ ILSS = \frac{3F}{4BT} \]  

Where, ILSS—Inter-laminar shear strength, F - Flexural strength, B - Breadth of the specimen, T - Thickness of the specimen.

The impact test measures the sudden (impact) force required to break the composite sample; the same was found by following the ASTM D256. The specimen of 65 mm \( \times \) 12.7 mm with a 45° notch of 2 mm was broken by a weighted pendulum to study the energy absorbed.

Shore hardness is a measure of soft, flexible materials’ hardness by measuring a specific indenter’s indentation depth. This test was conducted based on ASTM D1957 using The Yuzuku shore D durometer.

2.3.2. Wear test
The test to find the specific wear rate \( (W_{sp}) \) of the composite samples was determined by a pin-on-disc method as per the ASTM G99 standard. Specific wear rate is the volume of material removed when sliding a particular distance with a specific load. The tribometer (wear test equipment) was allowed a dry run with a loaded counter face pin made of hardened EN 31 steel (69 HRC, 8 mm thick and 0.6 μm) at a constant speed of 575 rpm in a track radius of 50 mm to trace 1000 m. The load was applied using a pulley setup of the equipment as 10N, 20 N, and 30N. The digital unit in the tribometer shows the testing conditions like time, rotating speed of the disc, and temperature every second. The specific wear rate was found by the difference in the weight of the specimen before and after wear [24]. Also, the friction forces developed at the pin disc interface were noted at various externally applied loads. The values of specific wear rates were calculated according to equation (2).

\[ W_{sp} = \frac{\Delta V}{NS} \]  

Where,
\[ \Delta V \]—Volume loss of material in mm³
\[ N \] - Applied load in N
\[ S \] - Sliding distance in m

The image captured by scanning electron microscope was also examined to understand the bonding and mechanical properties of the prepared sample with filler material.
3. Discussions

3.1. Mechanical properties of J/SG/K RC

The tensile strength of sample 1 with 5% of SGF (snake grass fiber) showed as 20.3 Mpa for an ultimate load of 2257.7N with an elongation of 1.2%. Further addition of SGF and the corresponding reduction in kenaf fiber increased the tensile strength to 4325.6N, almost double the former sample. Maximum tensile strength registered by sample 3 as 50.12 MPa with an elongation of 2.81%. This sample has 12.5% of SGF and KF.

Further addition of SGF and a corresponding reduction of KF reduced the tensile strength; the same was observed from samples 4 and 5. Adding KF to the composite increases the tensile strength as KF has higher tensile strength than SGF. This happens up to a 12.5% composition of KF. Decreasing the KF content below 12.5% leads to a decrease in the tensile strength due to poor bonding with epoxy. The tensile strengths of the J/SG/K FRCs are given in Figure 4. Apart from that, the cross-linking network between the fiber and the matrix is also a reason resulting in higher tensile strength in sample 3.

The flexural strengths of the samples also followed the same pattern as the tensile strength. The lower flexural strength was shoed by sample 1, about 60.32 MPa. The increased composition of SGF and a corresponding reduction of KF in the composited improved the flexural strength to 91.3 MPa, and the maximum strength was registered by sample 3 (12.5% if SGF and KF) at about 132.53 MPa. The samples (4 and 5) with SGF content greater than 12.5% and corresponding KF showed lesser flexural strength than sample 3. Since the bonding between fiber and epoxy is good, the flexural strength increases up to 12.5% of KF. But, in the composite with above 12.5% of KF, the bonding is poor and shows lower flexural strength. The flexural strengths of the J/SG/K FRCs are shown in Figure 5. It was to be noted that the flexural strength of sample 3 was approximately 120% high. The increased flexural strength of sample 3 is attributed to the improved resistance to crack initiation and propagation. The inter-laminar shear strength of the J/SG/K FRCs has also followed the same pattern of flexural strength as it was found from the flexural strength. The variation of inter-laminar shear strength in the samples of J/SG/K FRC is shown in Figure 3. Here, the ratio of highest to lowest inter-laminar shear stress was 3.6. As already mentioned, samples 4 and 5 have a weak bond between fiber and epoxy.

Overall, the mechanical properties of the NFRCs fabricated for this work showed well in sample 3. Sample 3 has an equal proportion of SGF and KF. Other samples have either higher content of SGF or KF, which were registered with lesser strengths. However, sample 5 shows some increased value to sample 4 may be due to the anisotropic behavior of tested specimens. Because these fibers are randomly oriented.
The impact strengths found using the Izod test with the samples of J/SG/K FRCs of 12.7 mm × 5 mm are shown in figure 6. Sample 4 had broken with lower energy absorption at about 1.15 J. The added SGF with corresponding KF increased the impact strength from 1.25 J to 2.23 J, observed in samples 1 to 3. Again, the increase in the SGF content reduced the impact strength. The impact strengths of the J/SG/K FRCs are given in figure 5.

Figure 5. Flexural strengths of the J/SG/K FRCs.

Figure 6. Inter-laminar shear strengths of the J/SG/K FRCs.
figure 7. Interlaminar shear strength also depends on the bonding strength of the composite. Samples 1, 2, and 3 have good bonding strength with increasing content of KF. So, the inter-laminar strength increases to 12.5% and above, and even though the KF content is high, the poor bonding strength reduces the strength. Compared to sample 3 other samples shows the lowest impact strength because of reduced stress transfer.

The hardness of the samples was not varied drastically as observed in the other mechanical properties as it mainly depends on the epoxy used and not on the fiber-reinforced. All the samples have the same amount of epoxy and registered a similar range of hardness as shown in figure 8. But, small variations observed might be
caused by noise and other external factors. The hardness (Shore D) of the developed samples is high mainly due to the high rigidity and stiffness. High hardness leads to an increase in the brittle behavior of the material is one of the major concerns. Similar mechanical findings have been reported earlier by Prabhu et al. [35].

3.2. Frictional force and specific wear rate

Figure 9 shows the variation of frictional force developed during the rubbing of a pin with samples 3, A, B, C, and D. It is known that the frictional force developed is directly proportional to an externally applied load. The chart reveals the same. The increased applied load develops more friction at the pin disc interface. The friction force developed at 10N load on the pin is much less in all four samples. Sample 3 with no filler material shows a very low frictional force of 3.8N at 10N applied load condition and gradually increases with applied load. The maximum load of 30N develops 13.5N frictional force in composite sample 3. Among the four samples of J/SG/K/AR FRC, B shows higher frictional force than other samples almost at all applied loads. Other samples, namely A, C, and D, show slight variation in the developed frictional force in all load levels. Sample C registered lower frictional force up to 22 N of the applied load. Beyond that load, comparatively more friction was developed. In the range of 25N to 30N of applied load, sample D shows less friction, followed by samples A and C. These variations may be registered by the uneven distribution of AR seed powder filler on the surface of the composite samples.

The plot of the specific wear rate for all applied loads on the four samples of J/SG/K/AR FRCs was given in figure 10. The specific wear was noted for tracing 1000m at a speed of 575 rpm. Figure 8 reveals that the specific wear
rate is increasing with applied load in all five samples A, B, C, and D. Sample 3 has no filler material that registered a wear rate of 0.0064 mm³/Nm at 10N load and 0.0032 mm³/Nm at 30N load. It is to be noted that the AR filler reduced the wear rate of the samples. This may be due to the hardness of the filler powder that enhances the wear properties. Among these four samples with AR filler (A, B, C, and D), higher wear rates were registered by sample A in all applied loads. This sample shows a higher wear rate at higher load conditions. The amount of increase in wear increases with an increase in load. It shows the highest wear rate of 0.006 mm³/Nm at 10N load and the lowest wear of 0.0036 mm³/Nm at 25N load. Sample B registered a lower wear rate among the four samples in all loads. The minimum wear rate at 10N was found to be 0.003 mm³/m at 10N load, and the highest specific wear rate was found to be 0.019 mm³/m. The specific wear rate of samples C and D lies between samples A and B at all load levels. In general, the wear rate decreases with an increase in applied load up to 25N and tends to flatten with a further increase in applied load.

The samples of J/SG/K FRCs have registered good mechanical properties by the samples that have equal weight percentages of SGF and KF. Similarly, all the samples of J/SG/K/AR FRCs have equal proportions of SGF and KF. But, the variation in the weight percentage of the AR powder differs. This influences the wear behavior pattern of the prepared samples. AR powder filler imparts wear resistance in the composites up to 5%, and further addition of AR in the composite samples reduces the specific wear rate. Figure 11 shows the
microstructure of the worn-out portion of samples 1 (without filler), A, B, C, and D. Figure 11 (1) shows the fiber and pulled-out broken fiber in the trace of the pin during the wear test. It has voids due to the improper packing of the resin during the molding process. It is evident from the image that the optimum ratio of fibers and epoxy resin provided good boning among them. So, it has good mechanical properties. Because of reducing the wear rate, AR seed powder was added to the composite as a filler material. Figure 11 (A) shows the twisted fibers and AR filler powder present in composite sample A. The voids present in this sample are comparatively larger than the voids in the other samples.

Figure 11 (B) shows sample B’s filler powder, voids, and fibers, which have a less specific wear rate. Comparatively, it has fewer voids and the proper ratio of fibers and is filled with epoxy resin that enhances the better wear properties. Figures 11 (C) and (D) are the corresponding SEM images of samples C and D. It depicts the presence of more AR filler powder. But, these samples have significantly fewer voids compared to all other samples.

4. Conclusions

The natural fiber composites prepared using jute, snake grass, and kenaf fiber with epoxy are mechanical properties, namely tensile strength, flexural strength, inter-lamina shear strength, impact strength, and shore hardness resin successfully. The wear properties of this NFRC were enhanced by successfully adding Annona reticulate seed powder to the composite. The conclusions derived from this study are listed as follows.

- The J/SG/K FRC has good mechanical properties, particularly the composite with equal proportions of SG and K fibers in weight percentage.
- The tensile, flexural, inter-lamina, and impact strengths of the composite with an equal amount of SG and K fibers were 50.12 MPa, 132.53 MPa, 1.952 MPa, and 2.23 J, respectively.
- The maximum hardness of the sample was found to be 82.58 on the Sg scale.
- The maximum frictional force was found in sample B, which has 5 wt% of AR filler; the minimum was found in sample C in the lower range of load and sample D in the high load range (above 25N).
- The lower specific wear rate was given by sample B (5 wt% of AR) in all load conditions. Also, it developed a higher frictional force comparatively.

Data availability statement

No new data were created or analyzed in this study.

Conflicts of interest

The authors declare that there is no conflict of interest regarding the publication of this article.

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