Investigation on the Mechanical and Thermal Properties of Jute/Carbon Fiber Hybrid Composites with the Inclusion of Crab Shell Powder

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Abstract: In recent years, natural fiber-reinforced hybrid composites have been utilized in many applications because of their lower cost, biodegradability, and low density. The aim of this research is to convert crab shell waste into an effective reinforcement in jute/carbon fiber hybrid composites. Various weight percentages of crab shell powder (0%, 1%, 3%, 5%, and 7%) were used to prepare crab shell powder hybrid composites. The performance of the crab shell powder hybrid composites was evaluated by preparing the specimens to conduct tensile, flexural, impact, and hardness tests as per ASTM standards. The results show that the inclusion of 5% crab shell powder displayed a better enhancement in the properties of the hybrid composite compared to other weight percentages. The tensile, flexural, and impact strengths of the 5% crab shell powder hybrid composite increased by 21%, 52%, and 50%, respectively. Also, the hardness of the hybrid composite was enhanced by 33%. Scanning electron microscopy (SEM) tests were conducted on the tensile-fractured specimen surfaces, and their morphology and structure confirmed the presence of a well-bonded interface between the fiber and matrix. Differential Scanning Calorimetry (DSC) and Thermogravimetry (TG) analysis have shown that the crystallization behavior and thermal stability of the composite were enhanced with the inclusion of crab shell powder. The presence of crab shell powder in the hybrid composite was identified using SEM with Energy-Dispersive X-ray Spectroscopy (EDS).

Keywords: crab shell powder; jute/carbon fiber; hybrid composites; mechanical properties; thermal properties; SEM with EDS

1. Introduction

Every year, huge quantities of seafood waste are created, which mainly consist of fish scales, crab shells, lobsters, etc., and are dumped on wastelands. These wastes create pollution in the environment and have harmful effects on society. Furthermore, their proper disposal involves significant investment. Instead, if these wastes are recycled and collected, they can be used in the development of biodegradable materials, which will help in the sustainable growth of the economy.

Global urbanization produces 7–10 billion tons of waste residues every year, and there is a need for recycling [1,2]. Oladele and Isola [3] have developed goat bone-reinforced epoxy composites for biomedical applications. The EDX data of the goat bone reinforcement showed calcium and phosphorus as the main elements. The epoxy composite with 16% goat bone reinforcement has improved mechanical properties such as tensile strength, flexural
strength, and hardness. In agriculture-based countries like India, cows have become part of people’s lives. Therefore, researchers have focused on the usage of cow dung in medical, agricultural, and industrial applications [4,5]. Olabisi [6,7] developed composites using cocoa bean shell and agro-waste residue to enhance the mechanical properties of aluminum. Palm kernel shell was another agro-waste residue produced globally that was reinforced with aluminum alloys to prepare a composite with enhanced performance characteristics [8–10]. Petrovic et al. [11] have developed a composite using walnut-shell ash as a reinforcement to improve the strength and hardness of Al_2O_3. Tile et al. [12] have investigated the effect of the inclusion of groundnut shell particulates on the hardness, yield strength, and ultimate tensile strength of Al-Mg-Si composites. The results showed an enhancement in properties with an increase in the weight percentage of groundnut shell particulates up to 10%. Eggshell waste particles, consisting of 95% CaCO_3 ceramic particles, have also proved to be desirable reinforcements for the composites [13,14].

The poultry, fishery, meat, and leather industries are major sources of animal waste. Especially seafood wastes discarded at sea generate toxic gasses, resulting in the pollution of water, air, and soil [15–18]. Arulvel et al. [19] have investigated the role of crab shell wastes in coating applications by examining their thermal stability, surface properties, and stability. Ahmed et al. [20] have conducted studies to enhance the mechanical and thermal properties of chitosan-based nanocomposite films used as food packaging materials. The blend of crab shell chitosan and graphene oxide nanosheets has improved the tensile properties and transition temperature of the composite films due to the more compact network structure between crab shell and graphene oxide. This was ascertained from the FTIR and SEM analyses of the composite films. Subaer et al. [21] examined the effect of chitosan on the mechanical, thermal, and anti-bacterial properties of geopolymer pastes used in the manufacturing of hybrid composites. The Differential Scanning Calorimetry (DSC) and bending test results of the composites showed high thermal resistance and better flexural strength. Also, the addition of 1.5 wt. % of chitosan powder reduced the growth of the bacteria, as observed from Total Count Plate (TCP) tests. Singaravelu et al. [22] compared the thermal stability, fade rate, and recovery rate of brake pads developed using chemically treated crab shell powder and thermally processed crab shell powder. Thermogravimetric analysis results showed better thermal stability for the thermally processed crab shell powder-based brake pads, whereas the fade rate and recovery rate were better for the chemically treated crab shell powder-based brake pads when tested using the Chase test following IS2742 Part 4 [23]. Gadgey and Bahekar [24] have investigated the mechanical behavior of the crab shell in different forms. The stress–strain curves obtained for crab cuticle exhibited low strain discontinuity, indicating brittle failure. For the crab exoskeleton, tensile tests were conducted in longitudinal and normal directions on wet and dry samples. The results showed the anisotropic nature of mechanical properties along the longitudinal direction, i.e., no permanent deformation, whereas deformation exists in the normal orientation. Also, the hardness of the outer layer is twice that of the inner layer of the crab shell. Kumaran et al. [25] have developed jute fiber epoxy composites reinforced with treated and untreated Portunus sanguinolentus shell powder. The mechanical characterization of these composites was compared with that of neat jute fiber epoxy composites for their hardness, tensile strength, flexural strength, impact, compression strength, and thermal stability. Soundhar et al. [26] investigated the fatigue behavior and damage progress of chitosan/sisal/glass fiber (CS/SF/GF)-reinforced epoxy composites under cyclic stress. The fatigue behavior of the composite was evaluated based on the fatigue endurance limit of the composite. Also, XRD, FTIR, and SEM-EDS analyses were carried out to determine the ability of the composite to form a new apatite layer on the surface. Ismail et al. [27] compared the crystal phase, crystal weight percentage, crystal size, crystal system, and elemental composition of calcium carbonate extracted from green mussel and crab shells with commercially available calcium carbonate. It was observed that green mussel CaCO_3 is nearly identical to commercial CaCO_3, whereas crab shell CaCO_3 contains a number of components other than Ca, C, and O. Joseph et al. [28] developed PLA-reinforced fish
scale powder filament for 3D printing. The test specimens were printed based on fused deposition modeling with three different combinations (10%, 20%, and 30%) of fish scale powder with PLA. Mechanical characterizations of the 3D-printed specimens exhibited the best performance in tensile and flexural strengths with a 20% fish scale powder composite. Cheng et al. [29] contributed to providing insights for future research to bridge the gaps between advanced process and continuous fiber-reinforced composite lightweight structures (CFRSs) multi-level design and fully explored the potentials of 3D-printed CFRSs for a wide range of applications. In the past, many researchers have reported the inclusion of carbon nanoparticles as filler materials in polymer composites, which has improved their mechanical and thermal properties. In recent years, researchers have attempted to study the behavior of polymer composites when they are added to natural filler materials like plant, animal, and sea waste. One such naturally available filler material is crab shell. The present research aims to study the mechanical and thermal properties of a jute/carbon fiber epoxy composite reinforced with crab shell powder for lightweight applications. The composite laminates were fabricated and tested for mechanical loading to determine the influence of crab shell powder on the tensile, flexural, and impact strengths, as well as the hardness of the composite. DSC and TGA tests were performed, and thermal properties were extracted and analyzed. The morphology of the fractured surface of tensile test specimens was studied using the SEM technique. The optimal weight percentage of crab shell powder was estimated, for which the performance of the hybrid composite was enhanced in terms of mechanical and thermal properties.

2. Materials and Methods

2.1. Materials

The hybrid composites used in this study consist of jute and carbon fibers as the reinforcement, along with a matrix comprising epoxy resin mixed with crab shell powder and hardener. The physical properties of the jute and carbon fibers are listed in Table 1. The epoxy resin and hardener were mixed in a weight ratio of 10:1. The crab shells procured from the local market were properly cleansed for the removal of meat and appendages left in the shells. Figure 1a shows the crab shells after cleaning. The shells were then dried and ground into coarse particles, as shown in Figure 1b. The particles were then processed in a ball mill to make them into a fine powder, as shown in Figure 1c.

Table 1. Specifications of jute and carbon fiber.

<table>
<thead>
<tr>
<th>Property</th>
<th>Jute Fiber</th>
<th>Carbon Fiber</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strength</td>
<td>380 g/m²</td>
<td>300 g/m²</td>
</tr>
<tr>
<td>Thickness</td>
<td>0.6 mm</td>
<td>0.4 mm</td>
</tr>
<tr>
<td>Density</td>
<td>1.5 g/cm³</td>
<td>1.8 g/cm³</td>
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Figure 1. (a) Cleaned crab shells; (b) coarse particles of crab shells; (c) crab shell powder.

2.2. Fabrication of Crab Shell Powder Hybrid Composites

A hybrid composite is a combination of synthetic and natural fibers and is developed for the improved mechanical behavior of two reinforcements. In this study, carbon and jute fibers were chosen as the synthetic and natural reinforcements of the hybrid composite, respectively, along with crab shell powder as the natural filler material.
The fabrication of the hybrid composite was given in the form of a flowchart, as shown in Figure 2. The jute and carbon fibers were placed in alternate layers during the manufacturing of the hybrid composite in a weight ratio of 67:33, and the weight proportion of reinforcement to the matrix was 54:46. The fine powder of crab shell was mixed into epoxy resin in different weight percentages, as given in Table 2. Figure 3 shows the composite laminates fabricated using the hand lay-up process. The specimens for tensile, flexural, and impact tests were cut from the composite laminates as per the dimensions of ASTM standards, shown in Figure 4.

**Figure 2.** Flowchart for the composite preparation.

**Table 2.** Designation and composition of the developed composites.

<table>
<thead>
<tr>
<th>Designation</th>
<th>Composite Composition</th>
</tr>
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<tbody>
<tr>
<td>Pure epoxy</td>
<td>100% Epoxy</td>
</tr>
<tr>
<td>1%</td>
<td>1% Crab shell powder + 99% Epoxy</td>
</tr>
<tr>
<td>3%</td>
<td>3% Crab shell powder + 97% Epoxy</td>
</tr>
<tr>
<td>5%</td>
<td>5% Crab shell powder + 95% Epoxy</td>
</tr>
<tr>
<td>7%</td>
<td>7% Crab shell powder + 93% Epoxy</td>
</tr>
</tbody>
</table>

**Figure 3.** Fabricated crab shell powder hybrid composites.
2.3. Mechanical Testing

The hybrid composites were produced with varying weight percentages of crab shell powder and tested for mechanical properties such as tensile strength, flexural strength, impact strength, and hardness. The tension test was conducted using a 5-ton (Fuel Instruments and engineers make) ultimate tensile machine at a crosshead speed of 4 mm/min. The average of three values of the tensile test was considered for the same weight percentage of crab shell powder in order to confirm the tensile strength results. Ultimate tensile strength and elongation were determined from the tension test. A three-point bend test was conducted to determine the flexural strength of the composites. The specimen bends and breaks when the load is applied at its center. An Izod impact test was conducted using an XJJU-5.5 model impact test rig made in China, on which a specimen was mounted and a ram was allowed to swing and break the specimen. The energy needed to fracture the specimen was determined using the impact test. A Micro Vickers hardness tester from HDNS-Kelly Instruments made in China was utilized for finding the hardness value, where the loading range was 10 g to 1 kg. Scanning electron microscopy, a Carl Zeiss instrument made in Germany, was used to record the fractured surface of the tested specimens in order to analyze the morphology and structure of the crab shell powder and the matrix.
2.4. Thermal Testing

The thermal properties of the composite material were analyzed using the Differential Scanning Calorimetry (DSC) technique. DSC analysis was performed using the Themys One+ apparatus made in France, which has the capacity to heat up to 1600 °C at a rate ranging from 0.01 to 100 °C/min. The amount of heat absorbed or released by the composite sample was estimated in this analysis. The thermal stability of the polymer composites will depend on their heat resistance and thermal oxidation capability. Heat resistance measures the material’s ability to remain unaffected by heat, whereas thermal oxidation capability is used to evaluate its resistance to degradation. A Thermogravimetric analyzer (TGA) was utilized to investigate the heat resistance and thermal degradation of the composites. In this analysis, the thermal stability of the composite sample was determined when it was subjected to high thermal loading.

2.5. Characterization Testing

SEM with EDS was employed to identify the major elements available in the composites. Similarly, the atomic percentage of the elements was also estimated to understand the mapping analysis of the content and the performance.

3. Results and Discussion

3.1. Tensile Behavior of the Crab Shell Powder Hybrid Composites

The tensile properties of the crab shell powder hybrid composites were used to analyze the effect of crab shell powder on the tensile behavior of the hybrid composites. Figure 5 shows the load–deflection curves obtained from the tension tests for hybrid composites with varying weight percentages of crab shell powder. It clearly shows that the tension test specimens of the hybrid composites exhibited failure due to their brittle nature. For the pure epoxy composite, the fracture occurred at a load of 3355 N with an elongation of 1.25 mm. Similarly, for the crab shell powder hybrid composites with 1%, 3%, 5%, and 7%, fracture occurs at 3428 N, 3640 N, 4118 N, and 3844 N, with elongations of 2.32 mm, 2.7 mm, 2.64 mm, and 2.53 mm, respectively, as shown in Figure 6. Therefore, it was confirmed that the load-carrying capacity of the crab shell powder hybrid composite was enhanced with the addition of crab shell powder.

![Figure 5. Load vs. deflection curve for the crab shell powder hybrid composites.](image-url)
The stress–strain curves obtained for the crab shell powder hybrid composites are shown in Figure 7, and information about the tensile strength of the crab shell powder hybrid composites is represented in a bar chart, as shown in Figure 8. It shows an upward trend in the tensile strength of the composite up to a 5% weight of the crab shell powder, and further increases in the crab shell powder weight percentage showed a decline in tensile strength. When compared with all the other weight percentages of the crab shell powder hybrid composites, maximum tensile strength was observed at the 5% weight of crab shell powder. The reason for increased tensile strength in the 5% crab shell powder hybrid composite was strong bonding between the matrix and reinforcement fibers. Beyond this point, the tensile strength decreases due to the agglomeration of crab shell powder in the epoxy resin. The tensile strengths for the 1%, 3%, 5%, and 7% crab shell powder hybrid composites are 7.8%, 14.5%, 20.9%, and 5.5%, respectively, compared to the pure epoxy hybrid composites. From the results, it can be confirmed that the tensile strength of jute/carbon fiber hybrid composites was enhanced with the addition of crab shell powder.
3.2. Flexural Behavior of the Crab Shell Powder Hybrid Composites

The flexural behavior of the crab shell powder hybrid composites was analyzed by conducting a series of three-point bending tests on 1%, 3%, 5%, and 7% crab shell powder hybrid composite specimens, and the test results are shown in Figure 9. It shows an upward trend in the flexural strength of the hybrid composites as the amount of crab shell powder is increased. This enhancement in flexural strength is mainly due to the good bonding between the epoxy and crab shell powder. Also, during the flexural test, the outer layer of the fiber experiences tensile load, while the inner layer experiences compressive load, which acts as the carriers of the load and helps in the uniform distribution of stress within the matrix.

3.3. Impact Strength of the Crab Shell Powder Hybrid Composites

The capability of the material to resist fractures due to suddenly applied loads is termed impact strength. The impact behavior was studied in terms of energy absorbed during fracture caused by suddenly applied loads. Figure 10 shows the effect of crab shell powder on the impact strength of the hybrid composite. As the weight percentage of the crab shell powder increases up to 5%, the impact strength of the hybrid composite also increases, but further increases in crab shell powder decrease the impact strength.

Figure 8. Ultimate tensile strength of the crab shell powder hybrid composites.

Figure 9. Flexural strength of the crab shell powder hybrid composites.

Figure 10. Impact strength of the crab shell powder hybrid composites.
3.4. Hardness of the Crab Shell Powder Hybrid Composites

The shore D hardness values for the crab shell powder hybrid composites are presented in Figure 11. The inclusion of crab shell powder in the hybrid composite improved the hardness value from 71.5 to 94.9, with the maximum hardness value obtained at 5% crab shell powder. The main reason for the improved hardness was that fewer internal pores developed in the composite due to the inclusion of carbon shell powder.

3.5. Tensile Fractured Surface Morphology

The tensile-fractured samples were analyzed to understand the fracture mechanisms of the composites using SEM micrographs. Figure 12 shows the fractured surfaces of the crab shell powder hybrid composites under tensile load. For the pure epoxy hybrid composites, the nature of fracture exhibited in the tension test was brittle. This is due to the matrix damage in the epoxy resin, as shown in Figure 12a. In the case of the 1% crab shell powder hybrid composites, the tensile-fractured samples displayed poor adhesion between the fiber and matrix, accompanied by matrix damage, as shown in Figure 12b. Figure 12c shows the tensile-fractured samples of the 3% weight crab shell powder hybrid composites, in which the fracture was due to both fiber bundle and matrix damages [30]. Figure 12d shows the fracture sample of the 5% crab shell powder hybrid composites,
which displayed good interfacial bonding between the fiber and matrix. Also, the crab shell powder eliminated matrix damage and enhanced the mechanical properties of the hybrid composites. Similar kinds of bonding can be seen for the 7% crab shell powder hybrid composites, except for fiber pullouts, as shown in Figure 12e. The SEM image showing the dispersion of crab shell powder within the epoxy matrix is shown in Figure 12f, and the crab shell powder size obtained was found to be 260 µm. It was confirmed that crab shell powder at weights below 5% have poor fiber matrix interfacial bonding and poor load-carrying capacity in the composites.

Figure 12. SEM images of the hybrid composites with (a) pure epoxy; (b) 1% crab shell powder; (c) 3% crab shell powder; (d) 5% crab shell powder; (e) 7% crab shell powder; and (f) Dispersion of crab shell powder within the epoxy matrix.
3.6. Differential Scanning Calorimetry (DSC) Analysis

To investigate the effect of crab shell powder on the crystallinity and melting point temperature of the hybrid composites, DSC analysis was conducted. A sample of 30 mg was collected from each specimen and placed on a pan made of Alumina (Al$_2$O$_3$). The samples were characterized under an inert gas atmosphere during heating to a temperature range of 0 to 400 °C. Figure 13a explains that the pure epoxy hybrid composite displayed a melting point temperature of 212.03 °C. Figure 13b,c displayed the changes in the peak temperatures of crystallization for the hybrid composites with 3% and 5% crab shell powder. The rate of heat flow was observed closely when the temperature reached the melting point of the hybrid composites.

![DSC analysis plots](image)

Figure 13. (a) DSC plot for the pure epoxy hybrid composites; (b) DSC plot for the 3% crab shell powder hybrid composites; (c) DSC plot for the 5% crab shell powder hybrid composites.

The crystallization temperatures measured for the hybrid composite with varying weight percentages of crab shell powder exhibited an interesting development. At lower
proportions of crab shell powder, as concentration increases, the crystallization temperature of the composite increases. However, after a certain limit of crab shell powder weight percentage, the crystallization temperatures began to decrease. For example, the crystallization temperature of the pure epoxy hybrid composite lowers from 212.03 to 223.47 °C with the addition of 5% crab shell powder. This outcome shows that with the inclusion of crab shell powder, the crystallization behavior of the hybrid composite changes, starting with an increase in crystallization temperature for the low weight percentages of crab shell powder and ending with a decrease in crystallization temperature for the high weight percentages of crab shell powder.

It was also observed that the jute/carbon fiber hybrid composite showed a maximum heat flow of 5094 mW (Figure 13a), but with the reinforcement of a 5% weight fraction of crab shell powder, the hybrid composite showed a maximum heat flow of 5532 mW (Figure 13c). Hence, it can be concluded that the heat flow capacity of the hybrid composite increased by 8.6% upon reinforcement with crab shell powder.

3.7. Thermogravimetry Analysis (TGA)

To evaluate the effect of crab shell powder on the thermal stability of the hybrid composite, thermogravimetric analysis was performed. The samples were characterized under an inert atmosphere during heating to a predetermined temperature of 820 °C. Figure 14a shows the breakdown curve for the pure epoxy hybrid composite, and Figure 14b,c show the breakdown curves for hybrid composites with 5% and 7% crab shell powder. It can be observed that the thermal stability has improved prominently due to the inclusion of crab shell powder as reinforcement in the hybrid composites.

The initial temperature for thermal stability of the pure epoxy hybrid composite was found to be 395.07 °C. However, the inclusion of crab shell powder in the hybrid composite led to an enhancement in thermal stability. For example, the starting temperature of the hybrid composite with 5% crab shell powder was found to be 419.57 °C, compared to 395.07 °C of the pure epoxy hybrid composite, signifying an improvement in thermal stability. This improvement in thermal stability was observed for crab shell powder weight percentages up to 5%, followed by a slight decrease at the 7% crab shell powder weight percentage. It was also demonstrated from the experiments that the temperature rise occurred in the crab shell powder hybrid composites, where the highest breakdown rate occurred. This behavior of crab shell powder hybrid composites led to good thermal characteristics and high thermal stability.

![Graph showing thermal stability](image)

**Figure 14. Cont.**
Figure 14. (a) TGA curve for the pure epoxy hybrid composites; (b) TGA curve for the 5% crab shell powder hybrid composites; (c) TGA curve for the 7% crab shell powder hybrid composites.

It is also possible to verify from the TGA curves of crab shell powder hybrid composites, in comparison to the pure epoxy hybrid composite, that the initial and maximum decomposition temperatures of crab shell powder hybrid composites were improved. This enhancement in thermal stability may indicate better interface adhesion between the crab shell powder and epoxy matrix.

3.8. Characterization of the Composites Using SEM with EDS Data

The variation in the chemical composition caused by the inclusion of crab shell powder was studied using EDS. Figure 15a–c display the SEM images and EDS of pure epoxy and crab shell powder (3% and 5%) hybrid composites. The presence of CaCO$_3$ was confirmed from the EDS data of the crab shell powder hybrid composites, and its atomic percentage increased with the increase in weight percentage of the crab shell powder. The Ca content of the crab shells was determined to be 0.8 wt% and 1.02 wt% for 3% and 5% crab shell powder hybrid composites, as shown in Figures 15b and 15c, respectively.
Figure 15. (a) SEM-EDS evaluation of pure epoxy hybrid composites; (b) SEM-EDS evaluation of 3% crab shell powder hybrid composites; (c) SEM-EDS evaluation of 5% crab shell powder hybrid composites.

4. Conclusions

In summary, the jute/carbon fiber composites were fabricated using the hand-layup method with the reinforcement of crab shell powder. The effect of crab shell powder on the tensile, flexural, and impact loadings, along with the hardness, was determined for the composite. The composites were characterized by DSC and TG to assess their heat flow.
capacity and thermal stability. SEM analysis was carried out to study the morphology of the tensile fracture surface. The following are the conclusions drawn from this research:

1. The 5% crab shell powder hybrid composite showed enhancements in tensile, flexural and impact strength by 21%, 52%, and 50%, respectively, compared to other weight percentages.
2. The inclusion of 5% crab shell powder enhanced the hardness of the hybrid composite by 33%.
3. The heat flow capacity of the 5% crab shell powder hybrid composite was improved by 8.6%.
4. The thermal stability of the hybrid composite was improved as the initial decomposition temperature was raised from 395.06 °C to 419.57 °C with the inclusion of 5% crab shell powder.
5. SEM images of the crab shell powder hybrid composites showed good interfacial bonding between the fiber and matrix.
6. The SEM with EDS data showed the presence of crab shell powder in the hybrid composites.


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Conflicts of Interest: The authors declare no conflicts of interest.

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