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Evaluation of Mechanical Properties and Fractograph Analysis of Flax Fiber Composite with the Influence of Varying Expendable Graphite Matrix

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Abstract

Recent research on natural fiber composite validates its utilization, and the significant micromechanical properties developed by different processing methods overlays the attention for its widespread application in many aspiring components of the automobile and aerospace industries. In comparison to synthetic fiber, natural fiber has relatively low weight to strength ration and good mechanical properties. These materials are restricted in automation, marine, and aerospace industries, due to high flammability of natural fiber and lack of durability. This study aimed to learn more about flax fiber composite materials and their mechanical performance and fracture behavior when adding varying percentages of Expandable Graphite (EG). With the addition of EG, there is a disparity in the mechanical properties because of the agglomerations of EG particles and the different modes of fiber pullout. The mechanical properties such as tensile strength, elastic modulus, and flexural strength affect most due to the addition of EG with flax fiber reinforced composite. These properties of the flax fiber composite are useful and overcome different challenging aspects of the aspiring components of modern automobile and aerospace industries.

Keywords

Natural fiber composite, Expandable graphite, Flax fiber, Toughness, Agglomeration

Introduction

Since 1945, polymer composite material has been used in several industrial applications, in the marine, automobile, and aerospace industries, due to its advanced qualities obtained through processing procedures. These industries achieve the mechanical attributes they desire. Metals or non-metals are employed, and their strength-to-weight ratio must be compromised. Natural fiber composites, as opposed to synthetic composites, are more effective in overcoming this challenge because they are lighter and stronger. Natural fiber composites have a high strength-to-weight ratio, which helps to increase the working efficiency of the parts. Metals can be easily replaced by natural fiber composites. Other important characteristics of natural fiber composites include high specific stiffness and excellent damping capability without compromising mechanical qualities [1, 2]. Natural fibers' features such as biodegradability, minimal pollutant emission, and natural sources; as a result of these characteristics, they are widely used as research material [3-5].

According to a group of archaeologists and paleontologists at Harvard University, flax fibers used as a material are deemed archaic, dating back more than 34,000 years [6]. Regardless, flax fiber studies and research do not fully cover its capabilities. The great tensile strength and low density of flax fiber set it apart from other composite reinforcing materials [7]. Additionally, it serves

as an acoustic and thermal insulator, therefore it can take the role of glass fiber [8]. The flax, it is extracted from does not require special conditions for cultivation [9]. It is an excellent alternative to glass fiber since it is also an effective thermal and acoustic insulator. These composites are employed in car bonnets, bumpers, interior and furniture panels, and other automotive elements. Tanks are one type of structural application for which natural fiber composites may be used [6-8]. Flax fiber has higher impact strength and sound absorption capacity than other natural fibers, as well as good mechanical qualities; hence it is commonly utilized as a reinforcement material in composites [9, 10]. When flax fibers are employed as brakes, they have a high friction coefficient. Flax fiber shows low resistance at high temperatures [1].

Materials and Methods

Reinforcement

The core of the flax (*Linum usitatissimum*) stem consists of a wood core and skin. Technical fibers are composed of 10 to 40 elementary fibers that are between 20 and 50 mm long and 0.1 to 0.8 mm thick. A single strand of flax contains many layers of fiber. These layers have cellulose and hemicellulose-containing thin primary wall. There is a secondary wall that aids in the strengthening of fiber as well as the presence of microfibrils. These microfibrils are made up of 30 - 100% cellulose [11]. Mat-type flax fiber was employed for the experimental investigation, and the composite was fabricated with a twill machine-woven mat [12]. The fiber was treated to eliminate dirt, and a residue from its surface was removed using alkali treatment (5% concentrated NaOH solution). The composite material is created using the Hand Lay-up method. Since it is simpler to use and more easily accessible than other techniques, this is the earliest method of material fabrication.

Matrix

Epoxy-LY556 is the name of a thermoset resin that is used to make epoxy materials. The most prevalent type of polymer, epoxy resin, is regularly coupled with glass, carbon, and natural fiber in a variety of applications, including those in the automotive, aerospace, and marine industries. Because of its many advantages, such as its superior adhesion, great chemical resistance, and little cure shrinkage, epoxy resin is also used [13].

Additive

To enhance the thermal and wear qualities of the EG flax fiber composite, a filler material consisting of EG powder of mesh size 2500 and mesh size 5 µm is utilized. To create EG, sulfur and nitrogen are intercalated into the crystal graphite (EG) in graphite, made of layers of carbon which are parallel by the intercalation process, atom such as nitrogen and sulfur, is sandwiched between these parallel layers [14]. The use of EG enhances the thermal insulation capabilities of polymer composites and serves as a flame retardant [15]. The capacity to exfoliate exists in EG when exposed to high temperatures. Low-density vermicular graphite is produced by its rapid, wormlike growth [16]. According to studies that have been documented in the literature, the mechanical properties of the generated material will be seriously reduced if the weight percent (wt%) of the EG goes beyond 5% [17-19]. Table 1 shows the flax fiber composite used in this experiment with varying amounts of EG in the reinforcing matrix.

Table 1: The percentages of matrix, reinforcement, and EG in FC [14].

Composite	Expandable Graphite	Reinforcement	Matrix
Flax Fiber Composite (FC)	0	50	50
2% Expandable Graphite Flax Fiber Composite (2% EGFC)	2	50	49
5% Expandable Graphite Flax Fiber Composite (5% EGFC)	5	50	47.5

Experimental procedure

The Hand Lay-up method is used to prepare the composites for testing. It is a straightforward, affordable, and precise process for the fabrication of composite materials. This open mold technique involves laying out the fiber or reinforcing material in the mold at room temperature, followed by the application of the matrix material (epoxy) with a brush to impregnate the fiber. The procedure is carried out once more till the appropriate composite thickness is achieved. EG is included in the composite at different weight percentages, such 2% and 5% of a matrix weight. Flax fiber was chosen as the reinforced component, making up about 50% of the composite [20]. In order to eliminate dirt and scale and prepare the surface for surface modification, the flax fiber will first undergo a chemical treatment with a 5% NaOH aqueous solution for approximately 30 minutes. Flax fiber is dried for 24 hours at 65 °C in a hot air oven to remove any moisture content. To create fiber composite, a 250 mm x 150 mm x 3 mm open mold is employed.

An organized schematic of composite manufacture is shown in figure 1. A rectangular mold is cleaned with acetone and placed over the acrylic sheet that has been wax-coated for a great surface finish and cured composite plate removed easily. To remove extra moisture, the EG powder is baked for 8 hours at 60 °C to 65 °C. The dried EG is combined with the epoxy using an ultrasonicator set to 1000 revolutions per minute for 30 minutes. The hardener is then added once the hardened EG has been mixed in. After mixing, the epoxy-EG was subjected to a 15-minute exposure in a room-temperature ultrasonicator to eliminate air bubbles with a 50% amplitude and a 24 kHz frequency. In a 10:1 ratio, the hardener HY951 is used with the epoxy LY-556 to hasten the cross-linking of

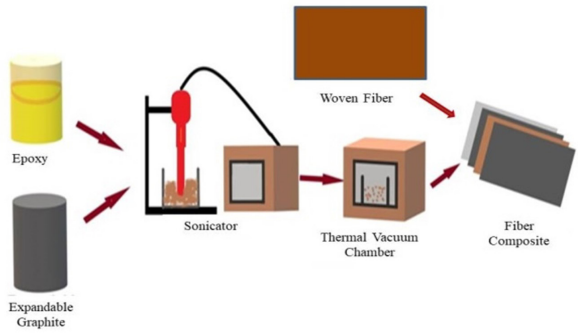

Figure 1: Systematic diagram of the FC fabrication experimental setup [14].

the epoxy. The fiber was spread over the EG-Epoxy mixture in the mold using the same Hand Lay-up method that was used for construction. The composite material was cured by placing a sample under constant pressure of 20 kPa for 24 hours at room temperature, as shown in figure 1.

Tensile strength

The produced flax fiber composites were tensile tested according to the ASTM D3039 standard. Figure 2 shows the standard dimensions of the rectangular-shaped sample, which is 250 mm x 25 mm x 3 mm, with externally attached grasping. The tensile test is carried out on a Bliss Plug n Play tensile test machine (UT-01-0025) with 100 KN load cells, as indicated in figure 3. A crosshead speed of 1 mm/min and a gauge length of 70 mm were implemented in accordance with ASTM D3039. With the mechanical extensometer, tensile moduli were estimated between 0.1 and 0.3% strain. Tensile strength, tensile strain, elastic modulus, and breaking load for the various flax fibers generated for this experimental inquiry were estimated using the tensile test.

Flexural strength

The three-point bend test method was used to assess the flexural properties of the manufactured flax fiber composites. The dimensions of the samples were 120 mm x 15 mm x 3 mm, as specified by the ASTM D790 standard. Figure 4a depicts the sample as per the ASTM D790 standard, while figure 4b depicts the sample's flexural test results on the three-point bend test. With a crosshead speed of 3 mm/min and a supporting span of 48 mm, this test was completed. Flexural stress and flexural elastic modulus values are calculated after the flexural testing.

Results

The tensile and flexural properties of flax fiber and flax fiber composites with additions of 2% and 5% EG mix are evaluated, and the findings are compared to flax fiber composite and the influence of different EG addition in flax fiber composites.

Tensile test

The tensile properties of flax fiber composites and flax fiber with varying amounts of EG were investigated, experimental data was analyzed, and a graph for tensile stress and strain was created for 2% EGFC and 5% EGFC, as shown in figure 5. Tensile strength and elastic modulus for the 2% EGFC and the 5% EGFC were plotted in figure 6. Tensile strength and Young's modulus both drop with the addition of EG particles. However, it has an impact on the hardness of the material, which is enhanced by the inclusion of EG particles.

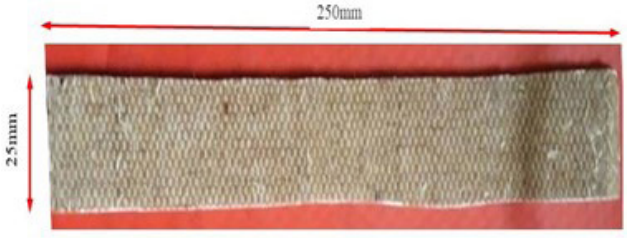

Figure 2: Tensile test sample of FC prepared as per ASTM 3039 standard.

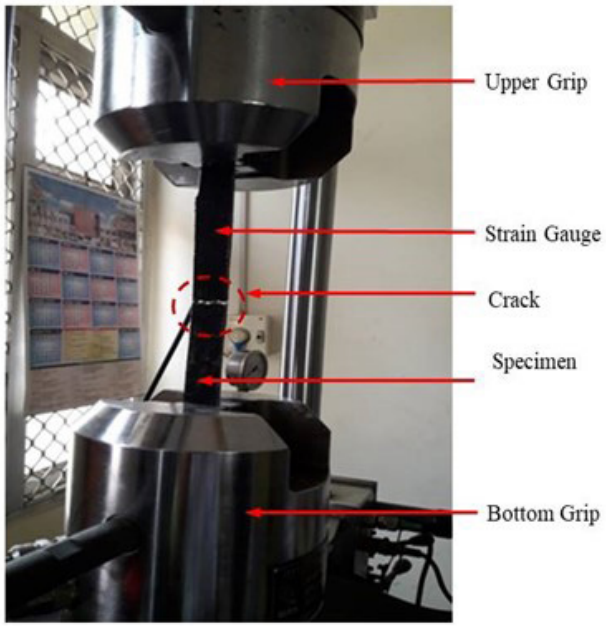

Figure 3: Tensile test in Bliss Plug n Play tensile test machine (UT-01-0025).

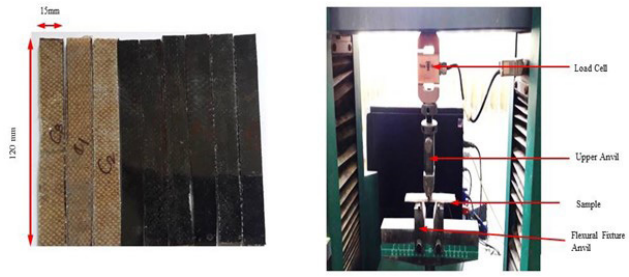

Figure 4: (a) Samples prepared for flexural test as per ASTM D790 standard and **(b)** Sample attached in 3-point bend test setup.

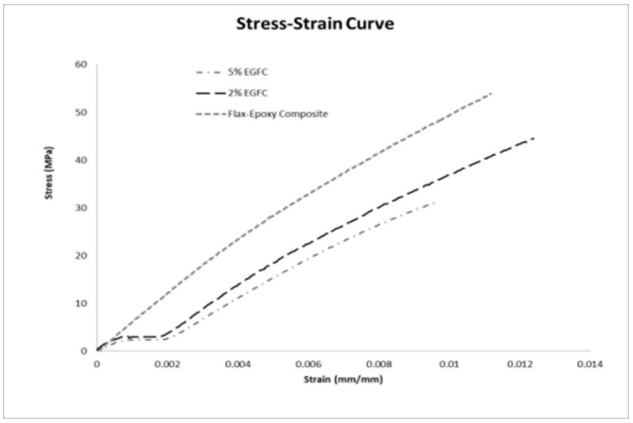

Figure 5: Stress-Strain plot of epoxy FC and different compositions of EGFC.

Figure 5 depicts stress-strain graphs for FC, 2% EGFC, and 5% EGFC, which reveal that when stress rises, so does strain, and follows the behavior of brittle material's stress and strain graph. The stress-strain graph of EGFC composites

shows a decrease as the fraction of expandable graphite particle composition in the composite material increases. Figure 6 indicates that when the percentage of EG particles in the material increases, the tensile strength of the material falls. There is a risk of particle agglomeration in epoxy as a result of the addition of EG particles, which is why the tensile property has decreased. The numerical value of the calculations from the tested data is shown in table 2. According to experimental data, there is a 17.48% decrease in tensile strength when compared to FC in 2% EGFC, but an 11.44% gain in tensile strength when compared to epoxy. Compared to FC, there is a 40.42% reduction in tensile strength for 2% EGFC and a 7.36% rise in tensile strength with 5% EGFC. In comparison to FC, Young's modulus decreases by 27.4% and 47.5% for 2% EGFC and 5% EGFC, respectively. In these compositions, the EG agglomerates within the epoxy cause this reduction. Weak adhesion at the epoxy, EG, and flax fiber interface is thought to be the cause of the lower performance. The trapezoidal method is also used to compute the toughness of the composite material, and it has been discovered that the inclusion of the EG filler increases the toughness of the material.

Surface fracture characteristics typical of brittle fracture can be found in all of the samples analyzed. The EGFC composite has a higher toughness than the FC composite. Toughness has significantly improved. Tensile strength decreases as toughness increases. Composite has a higher load-bearing capacity before it fractures. Due to the disruption of load transfer from of the polymer matrix to the flax fibers caused by EG particles, the reinforcement could not be used to endure tensile stresses and enhance mechanical properties. In comparison to FC and epoxy, EGFC has lower rigidity.

Flexural test

Figure 7 shows the experimental values of flexural parameters such as flexural strength and flexural modulus obtained from flexural load against an extension for FC, 2% EGFC, and 5% EGFC. The FC's flexural load-extension response shows that in the elastic zone, flexural load values grow linearly. The middle span of a three-point bending arrangement experiences the most stress. When the maximum tension is reached, the composite layers delaminate and break.

The fracture mode for the flexural behavior of several composite specimens in the three-point bending test is shown in figure 8. The stress that occurs in the composite specimens is highly intricate, resulting in distinct fracture modes because the specimens are subjected to both tensile and compressive forces during bending. This may be explained by the fact that inadequate interfacial adhesion can result in delamination failure, which in turn reduces the flexural characteristics of flax fiber composites, despite the fact that the flexural properties of flax fiber composites are normally governed by the polymer matrix. This result indicates that strong C-C bonding exists, which is thought to be the cause of the composite's increased flexural strength following carbonization.

Figure 8 displays the FC's average values for flexural strength and modulus, which are 76.48 MPa and 3.921 GPa, respectively. The average flexural strength, flexural modulus,

Table 2: Tensile properties of FC with varying percentages of added EG.

Percentage of EG in FC	Tensile Strength (MPa)	Young's modulus (GPa)	Toughness (J/m³)
FC	53.93 ± 2.7	6.78 ± 0.3	29653.2
2% EGFC	44.50 ± 2.5	5.23 ± 0.2	177354.4
5% EGFC	32.13 ± 1.4	4.31 ± 0.2	150910.5

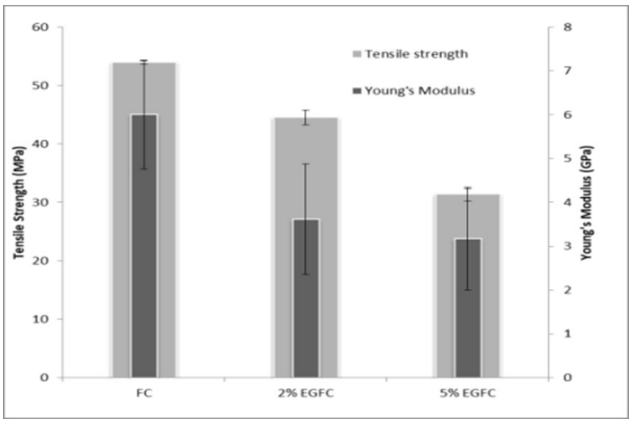

Figure 6: Tensile strength and Young's modulus bar graph for epoxy FC and different compositions of EGFC.

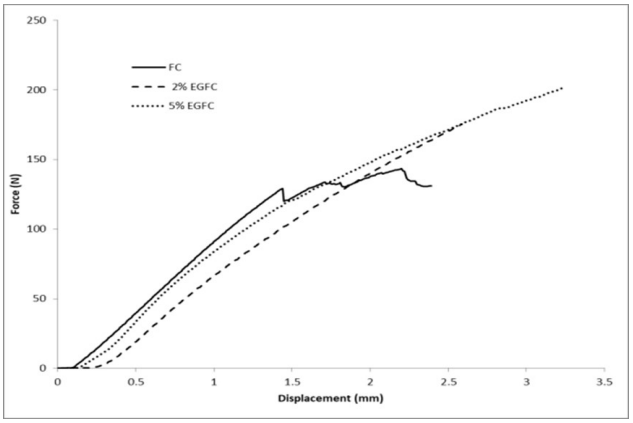

Figure 7: Force and displacement graph pattern of flexural test for FC and 2% EGFC.

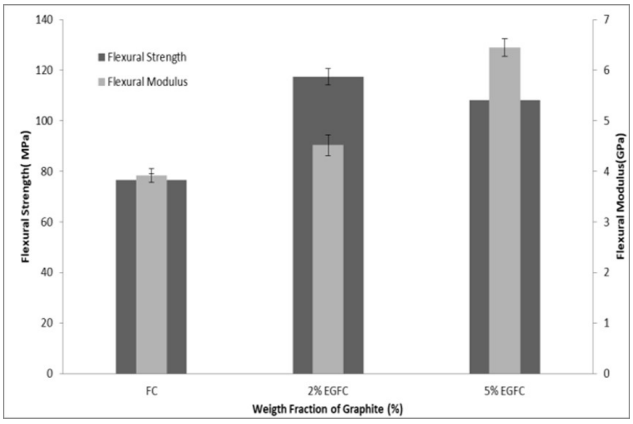

Figure 8: Flexural strength and Flexural modulus bar graph for epoxy FC and different compositions of EGFC.

and flexural load-extension response of the composite material were determined to be 117.33 MPa and 4.516 GPa, respectively. The average flexural strength, flexural load-extension response, and modulus values for the composite material were determined to be 152.26 MPa and 6.4512 GPa, respectively.

Table 3 shows that the addition of 2% EG filler to the flax fiber composite results in a 53.41% flexural strength and a 59% increase in flexural modulus when compared to the FC composite. In comparison to FC, adding 5% EG filler to flax composite increases flexural strength by 41.55% and flexural modulus by 64.54%.

Fractographic analysis

The bonding strength and dispersion of EG and flax fiber into the epoxy are revealed through microscopic analysis of tested tensile samples using a scanning electron microscope (SEM) at various magnifications. Figure 9a and 9b of scanning electron images of epoxy flax fiber at 2000X indicate that the fiber and matrix are well dispersed, resulting in good stress distribution in composites. The fibers are drawn out of the epoxy in the image of figure 9a due to the compatibility of the matrix and fiber. The photos of the cleavages that justify the brittle mode fracture of the samples are shown in figure 9b.

Figure 9c and 9d show photos of 2% EGFC broken samples. Furthermore, because of the low tensile strength of the composite, EG has low compatibility with matrix fibers, which leads to crack propagation. Furthermore, the addition of 5% EG to the flax fiber highlights the weakening of the matrix's bonding, as observed in figure 9e and 9f. A river-like structure between the matrix and the EG filler material can also be seen in the SEM image of 5% EGFC. In addition, as compared to other epoxy flax fiber and 2% EGFC, there is greater fiber material breaking and delamination due to poorer bonding, as demonstrated in figure 9e. As a result of the current examination of this experimental analysis of FC and altering EGFC, the failure of composites is a mix of fiber fracture, fiber pullout, and low matrix-filler-fiber compatibility.

Figure 10 shows the energy-dispersive X-ray spectroscopy (EDS) results of produced composite material with various EGFC percentages. When compared to flax composite, it was discovered that EG filler included a larger amount of carbon and a lower proportion of oxygen. In the % EGFC compared to the FC, the carbon weight% grew from 72.76% to 77.35%, while the oxygen wt.% decreased from 25.52% to 20.82%. When comparing the % EGFC to the % EGFC, the carbon wt.% increased from 77.35% to 84.25%, while the oxygen wt.% decreased from 20.82% to 13.25%.

Table 3: Flexural strength, and flexural modulus of the flax fiber and EGFC.

Percentage of EG in FC	Flexural strength (MPa)	Flexural Modulus (GPa)
FC	76.48 ± 3.2	3.92 ± 0.2
2% EGFC	117.33 ± 4.5	4.51 ± 0.2
5% EGFC	108.26 ± 4.8	6.45 ± 0.3

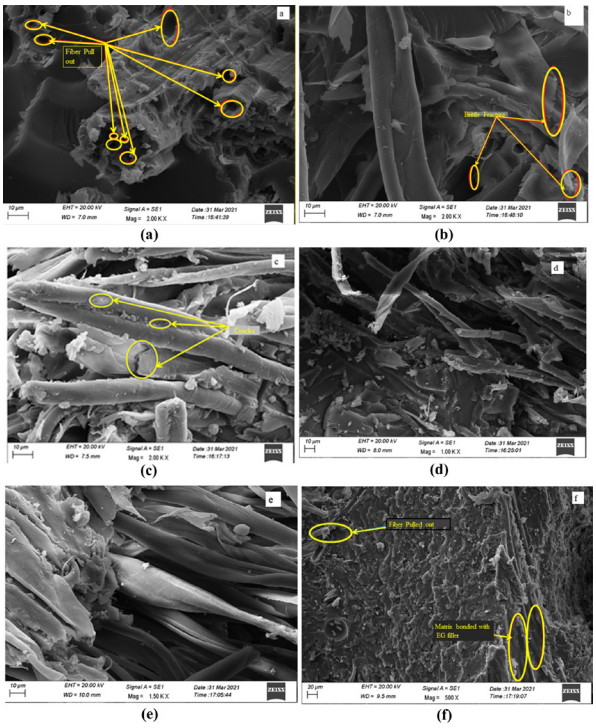

Figure 9: SEM images of **(a** and **b)** tension fractured flax epoxy composite sample, **(c** and **d)** 2% EGFC fractured sample of tensile test, and **(e** and **f)** 5% EGFC fractured sample of tensile test.

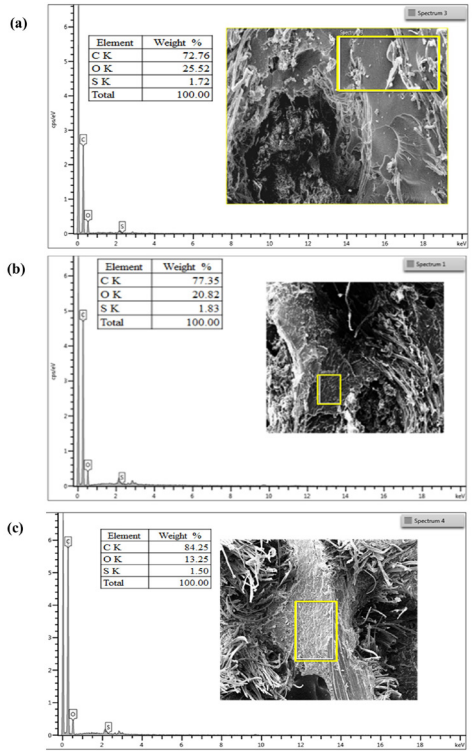

Figure 10: EDS analysis of **(a)** FC, **(b)** 2% EGFC, and **(c)** 5% EGFC at 500 µm.

Discussion

The following significant conclusions were made based on the current experimental examination of epoxy flax fiber with variable amounts of expandable graphite of 2% EGFC and 5% EGFC. As the amount of EG particles in the flax fiber composite increased, the tensile strength decreased. In comparison to 2% EGFC and epoxy flax fiber, 5% EGFC has a high toughness characteristic. The low level of interfacial bonding between EG particles, fibers, and matrix causes this decrease in tensile strength. The flexural properties of the composite increase with the adding of EG. Low adhesion in the fiber matrix interfacial and delamination failure causes the flexural property to vary. Moreover, the analysis of fractography under the SEM of the epoxy flax fiber, 2% EGFC, and 5% EGFC impend the mode of propagation of cracks through fiber pullouts and low compatibility of the matrix and the variation of mechanical properties is vital due to the addition of EG in flax fiber composite. Thus, the overall examination of this work reveals that adding EG to flax fiber alters the mechanical properties of the composite, allowing flax fiber composites to be used in a wider range of applications. Flax fiber composites with EG matrix will undoubtedly answer the demand for future material choices in current manufacturing sectors such as aerospace and car for a variety of competent parts while also meeting our economic and environmental goals.

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Conflict of Interest

The authors declare no conflict of interests that are relevant to the content of this article.

Credit Author Statement

Parul Singh: Experimentation, Data analysis, Writing - original draft preparation; Y. G. Bala: Study design, Supervision. All the authors read and approved the manuscript.

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