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Epoxy-Graphene composite reinforced with bamboo biochar powder: Effect on mechanical properties

Mazlan Mohamed[1,2#], Nor Rohimah Abdullah[1], Jidon Janaun[3], Hasyiya Karimah Adli[2], Mohamad Faiz Mohd Amin[4], Arman Shah[5], Muhammad Iqbal Ahmad[4]

1 Institut Kepintaran Buatan dan Data Raya, Pejabat Timbalan Naib Canselor Penyelidikan dan Inovasi, Universiti Malaysia Kelantann, MALAYSIA.
2 Fakulti Sains Data dan Komputeran Universiti Malaysia Kelantan, Kampus Kota, 16100, Kota Bharu, Kelantan, MALAYSIA.
3 Chemical Engineering Programme, Faculty of Engineering, Universiti Malaysia Sabah, Jalan UMS, 88400, Kota Kinabalu, Sabah, MALAYSIA.
4 Faculty of Earth Science, Universiti Malaysia Kelantan, Jeli Campus, Jeli, Kelantan, MALAYSIA.
5 Faculty of Technical and Vocational, Universiti Pendidikan Sultan Idris, 35900 Tanjong Malim, Perak, MALAYSIA.
Corresponding author. E-Mail: mazlan.m@umk.edu.my; Tel: +6019-5562919

ABSTRACT Epoxy-graphene composites have been widely used in many applications due to their exceptional properties, such as excellent mechanical strength, thermal ability, electrical conductivity, and chemical resistivity. Bamboo is a natural resource that could be regarded as a high-performance material that involves low production costs. It is grown easily and is quicker to harvest than other natural resources. Bamboo biochar (BB) can be produced using the pyrolysis method. Studies on the effects of using biochar as filler in epoxy composites have proven that this approach can increase the mechanical strength of the material. The role of BB has been studied by researchers, but only as fertilizer. Little research has been undertaken on the mechanical properties of BB, especially when used as filler in a composite material. This research aimed to determine the effects of adding BB as filler in epoxy-graphene composites. Using pure epoxy and epoxy-graphene composites (EG) for reference, the mechanical properties of an epoxy-bamboo biochar composite (EB) and an epoxy-graphene-bamboo biochar composite (EGB) were investigated. Thus, the role of BB in enhancing the mechanical properties through its matrix was properly observed and studied. The composites were fabricated using the solution blending method, whereby all the mixtures were mixed homogeneously before being degassed in a vacuum chamber. Curing took place in an oven at 70 °C for 24 hours, and post-curing occurred at room temperature for an hour before demolding. Tensile and three-point bending tests were conducted to analyze the tensile and flexural properties of the composites. The tensile strength of EB showed a 56.216% increment compared to that of pure epoxy, while BB filler increased the tensile strength of EGB by 12.350% compared to that of EG. The flexural strength of EB increased by 0.258% compared to that of pure epoxy, while EGB had 6.535% higher flexural strength compared to EG. Scanning electron microscopy (SEM) was conducted on the sample EB, EG, and EGB at magnifications of 100x, 500x, and 1000x. Graphene agglomeration could be observed on the sample containing graphene. The dispersion of graphene and BB could be observed clearly. The study indicates that bamboo biochar altered the mechanical properties of the epoxy-graphene composite by increasing the tensile strength and stiffness, although it reduced the flexibility of the material.

KEYWORDS: Composites; Bamboo Biochar; Filler Material; Mechanical Properties Enhancement; Tensile Strength
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INTRODUCTION

Nowadays, polymer composites are attracting researchers' attention due to their unique properties, which vary by the types of polymers and reinforcement used. In addition, the presence of additives in its composition can help to alter and improve a composite's properties such that the desired material behavior is achieved (Rosdi *et al*., 2022). Epoxy resin is one of the most appealing polymer materials in the materials industry due to its excellent mechanical strength, resistivity to chemicals, good thermal behavior, and electrical properties, which allow it to be used in a wide range of applications (Berhanudin *et al*., 2017). Graphene is a carbon nanomaterial with excellent mechanical strength, which makes it highly favored as reinforcement in polymer composites. The covalent bonds between the carbon atoms are the main reason for graphene's magnificent strength. Research by Wei *et al*. (2017) showed that the addition of 0.3 wt% graphene into epoxy resin resulted in 64.64 MPa tensile strength and 97.10 MPa flexural strength. This study proved that a small amount of graphene was sufficient to enhance the mechanical properties of the composite (Mohamed *et al*., 2020; Mohamed *et al*., 2021). Bamboo biochar is the product of pyrolysis bamboo culm in an inert environment. The use of biochar as reinforcement has been studied because its high carbon content provides excellent mechanical strength, while it is also low-cost and environmentally friendly. Little research has been conducted on using bamboo biochar as reinforcement in polymer composites. However, considerable research has been undertaken on biochar as a cheap replacement for carbon reinforcement. The addition of Mischantus biochar into an epoxy-agave leave composite produced 63% more ultimate tensile stress (UTS) than the original epoxy-agave leave composite, which was highly impressive (Charfi *et al*., 2020). The solution blending technique has been widely used to fabricate epoxy-graphene composites. The crucial aspect of this technique is to disperse the reinforcement homogeneously in the matrix. The solution is usually stirred using a mechanical stirrer or shear mixer to accelerate the mixing process. However, traditional methods such as mixing the solution manually can still be used with extreme care. Hardener or a curing agent is added at the last stage or when mixing to avoid the composite curing prematurely. Since epoxy has a high cross-linking density, it is easy to cure, especially when exposed to heat. Moreover, much research has been conducted on using the solution blending technique to disperse graphene and bamboo biochar in a polymer matrix. Thus, in this research, bamboo biochar role as reinforcement in epoxy resin matrix was studied, especially in improving the mechanical properties of the materials. The biochar was incorporated with the same technique and composition as graphene incorporation in epoxy resin matrix. Furthermore, epoxy-graphene-bamboo biochar was also fabricated to understand more on mechanical behaviour of each composite composition.

EXPERIMENTAL

Materials

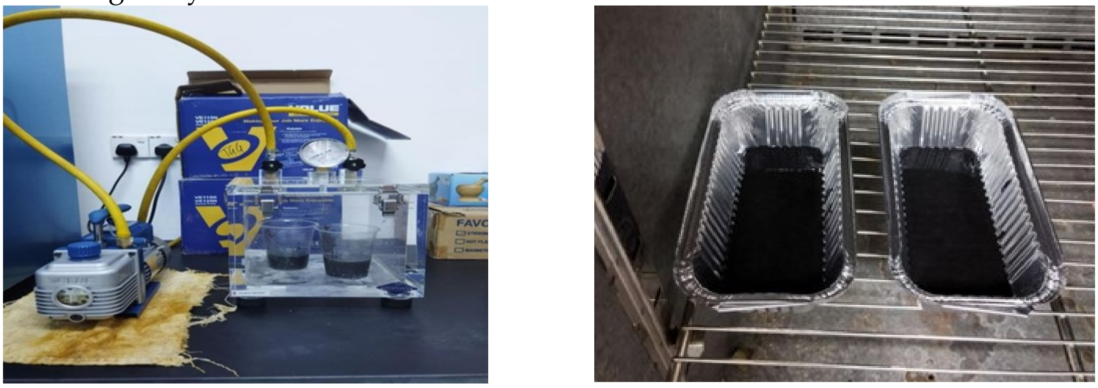
Epoxy resin used as the matrix consist of epoxy and hardener are manufactured by Daewoo Waterproof. The ratio of epoxy: hardener used was 3:1 as instructed by the manufacturer. Four samples were prepared: a pure epoxy sample (EH), an epoxy-graphene composite (EG), an epoxy-bamboo biochar composite (EB), and an epoxy-graphene composite reinforced with bamboo biochar (EGB). In this experiment, 5 g of graphene powder with lateral size 40 µm and 1-2 nm thick was used in sample EG and EGB, bamboo biochar powder (100 µm) that was produced by Ain Biomass Industries Sdn. Bhd. was used as reinforcement in sample EB, and EGB as shown in Table 1.

Table 1. Reinforcement composition in each sample

Sample Name	Content	
	Graphene (wt%)	Bamboo Biochar (wt%)
EH	-	-
EG	5	-
EGB	5	5
EB	-	5

Fabrication of Composites

The solution blending technique was used to fabricate the samples. Four were samples prepared: EH, EG, EGB, and EB. EH, the only sample with no reinforcement, consisted only of epoxy resin and hardener. As instructed by the manufacturer, the pure epoxy sample, EH, was prepared using an epoxy-to-hardener ratio of 3:1. The mixture was then mixed thoroughly by hand. For the EG and EGB samples, the preparation was similar. Graphene powder of 5 wt% was poured into epoxy resin and mixed well. For the EGB sample, 5 wt% bamboo biochar powder was added after the graphene and epoxy resin had been mixed. Hardener was added to the EG and EGB samples before being mixed again until they were homogeneous. All the samples were then degassed in a vacuum chamber at room temperature for 30 minutes (Figure 1). The mixtures were then poured into molds and cured at 70 °C for 24 hours. Post-curing occurred at room temperature for one hour before the samples were removed from the mold. The samples were then cut based on the dimensions required for each testing analysis.



(A) (B)

Figure 1. Experimental Setup and Process. (A) Sample Mixture in Vacuum Chamber, (B) Curing Process in the Oven.

Characterization Analysis

A tensile test was conducted according to ASTM D3039 using a Universal Testing Machine (UTM). The specimens were shaped according to the standard (3 mm x 20 mm x 150 mm). Each sample dimension (length, width, and thickness) was measured using vernier calipers to ensure more accurate measurements. Each dimension value input was needed before the testing could be conducted. The specimens were pulled at 2 mm/min until they showed plastic deformation behavior.

To determine the flexural strength of the samples, tests were carried out using the UTM with the three-point bending fixture, according to ASTM D790. The dimensions of 150 mm x 10 mm x 3 mm were used for the samples. The samples were positioned such that the load was perpendicular to each sample's axis. Load was then applied at a speed of 3 mm/min to analyze the flexural behavior of the specimens.

The surface morphology of samples EG, EGB, and EB was observed using scanning electron microscopy (SEM). Each specimen's fracture area from the tensile test was cut to 5 mm in height before SEM analysis was undertaken. The fracture surface morphology was observed using magnifications of 100x, 500x, and 1000x. This analysis aimed to examine the interaction between each component in the composite and the reinforcement dispersion in the epoxy matrix.

RESULT AND DISCUSSION

Figure 2 shows the tensile strengths of the EH, EB, EG, and EGB samples, as measured according to ASTM D3039. Tensile strength is the ability of a material to contain tension force before it breaks. The tensile strength of EB was 10.19 MPa, which was 56% higher than that of EH (6.52 MPa) and 34% lower than that of EG (13.64 MPa). Meanwhile, the tensile strength of EG was 109% higher than that of EH. Thus, bamboo biochar arguably improved the mechanical strength of the epoxy polymer quite considerably, by up to half the amount of EG. This may have been due to the carbon content in bamboo biochar, which can reach 96.53% (Zhu *et al.*, 2020). Materials with high carbon content also have high strength due to the carbon-carbon bonds in the material. Bonding between carbons is termed covalent bonding, which is known to be one of the strongest intermolecular forces. This explains how the carbon influenced the strength of the materials. In terms of EG, it is widely known that a small addition of graphene can substantially increase the tensile strength of a composite (Zhao *et al*., 2018). Graphene has only carbon atoms in its composition, making it stronger than bamboo biochar, which has mixed elements. The addition of bamboo biochar into epoxy-graphene did not significantly change the tensile strength of the composite, as shown in Figure 2. EGB (15.32 MPa) had a tensile strength that was 12% higher than that of EG, demonstrating that bamboo biochar only marginally increased the strength.

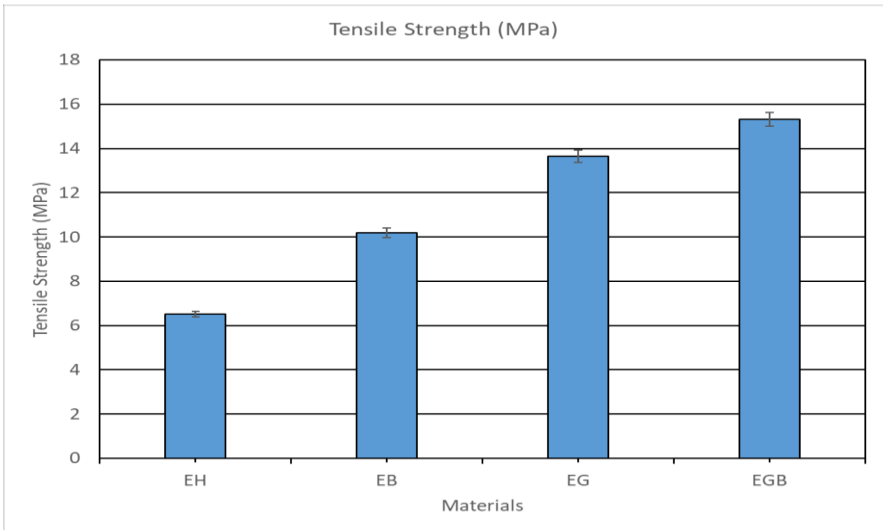


Figure 2. Tensile Strength of the samples (MPa)

Figure 3 illustrates the Young's modulus of the EH, EB, EG, and EGB samples. EB had a Young's modulus of 0.35 GPa, which was higher than that of EH (0.17 GPa) but lower than that of EG (1.16GPa). The major difference between the Young's modulus values of EG and EB was that graphene could resist deformation under tension better than bamboo biochar. The addition of bamboo biochar into the epoxy-graphene composite was observed to increase the Young's modulus value by only 13%, which was not a significant change.

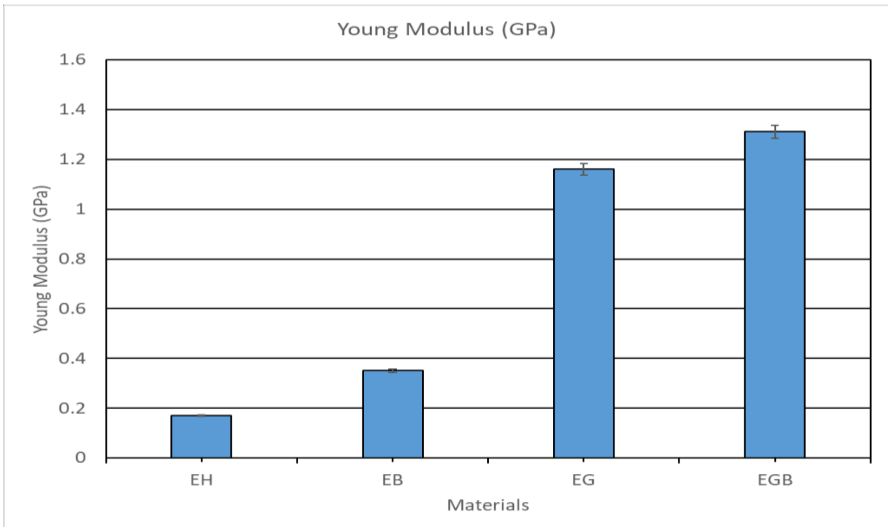


Figure 3. Young's modulus of the samples (GPa)

Figure 4 shows the elongation at break values of the EH, EB, EG, and EGB samples. Elongation at break is the ratio between the change in length after deformation during the tensile test compared to the initial length before the test. It also explains the ability of a material to maintain its shape before deformation. The higher the elongation at break value, the more it sustains breakage. These properties are usually applied to flexible materials such as polymers, rather than stiff materials such as ceramics, which have low elongation at break values. As shown in Figure 4, pure epoxy (EH) had the highest value, compared to EG (2.70%) and EGB (2.40%). The presence of graphene in the composite increased the stiffness of the materials. However, the EB value (10.31%) was greater than those of EG and EGB, indicating that bamboo biochar did not make the composite too stiff and rigid. In addition, when the tensile strength and elongation at break of all the composite samples were compared, EB stood out the most. This was because while the bamboo biochar increased the tensile strength by half the amount of graphene, it did not significantly affect the composite's flexibility.

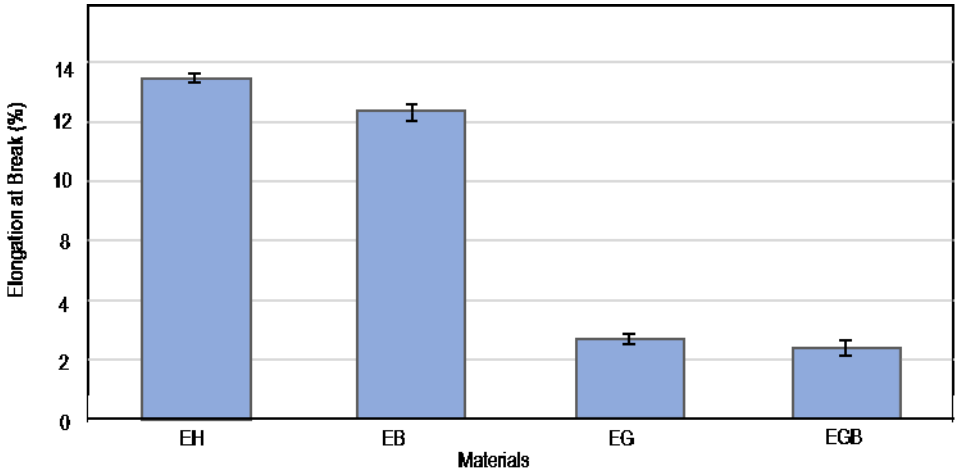


Figure 4. Elongation at break of the samples (%)

Figure 5 illustrates the flexural strength of the EH, EB, EG, and EGB samples. No significant change was observed in the flexural strength of each sample. The EB sample had the highest flexural strength, followed by the EH, EG, and EGB sample, with values of 46.07 GPa, 45.95 GPa, 41.38 GPa, and 38.68 GPa. The three-point bending shows the behavior of materials under both tensile and compressive force. When load is applied, the upper part of the sample experiences compressive force, while the lower part of the sample experiences tensile force. The decrease in flexural strength when adding graphene occurred because it is a 100% carbon-based material, which increases the stiffness of a material. The flexural strength of EB was slightly higher than that of EH, which may have been because the bamboo biochar had better dispersibility in epoxy compared to graphene. Agglomerations were detected in the EG and EGB samples when observed through SEM. When dispersed well in the matrix, graphene increases a composite's flexural strength, as shown in the study by Wei (2015).

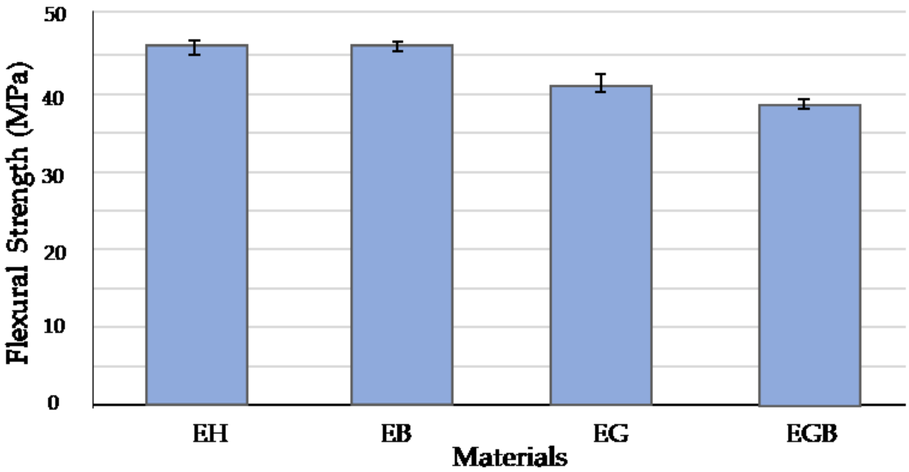


Figure 5. Flexural strength of the sample (MPa)

Figure 6 shows the flexural modulus of the EH, EB, EG, and EGB samples (GPa). EB (7.75 GPa) had a higher modulus of flexibility than EH (3.32 GPa), EG (3.29 GPa), and EGB (7.31 GPa). Comparing the EB and EH samples showed that the presence of bamboo biochar increased the flexural modulus of the materials. This was more obvious when comparing the flexural modulus of the EG and EGB samples. EG had a lower flexural modulus than EH, which had no reinforcement. This showed that graphene lowered the flexural modulus of the composite. In the EGB sample, bamboo biochar increased the flexural modulus of the composite, but the addition of graphene lowered the value, which was proven when the flexural modulus of EGB was compared to that of EB. However, a study by Shivakumar (2020) stated that graphene improves the flexural strength of the composite, whereby the addition of 0.5 wt% and 1.0 wt% of graphene into an epoxy matrix led to flexural modulus values of 2.75 GPa and 2.79 GPa. However, in the same study, the addition of more than 1.0 wt% of graphene content decreased the flexural modulus of the composite, whereby 1.5 wt% and 2.0 wt% of graphene led to flexural modulus values of 2.63 GPa and 2.62 GPa, respectively. Thus, the addition of 5 wt% of graphene into the composite might have made the composite too stiff, compared to the addition of 5 wt% of bamboo biochar.

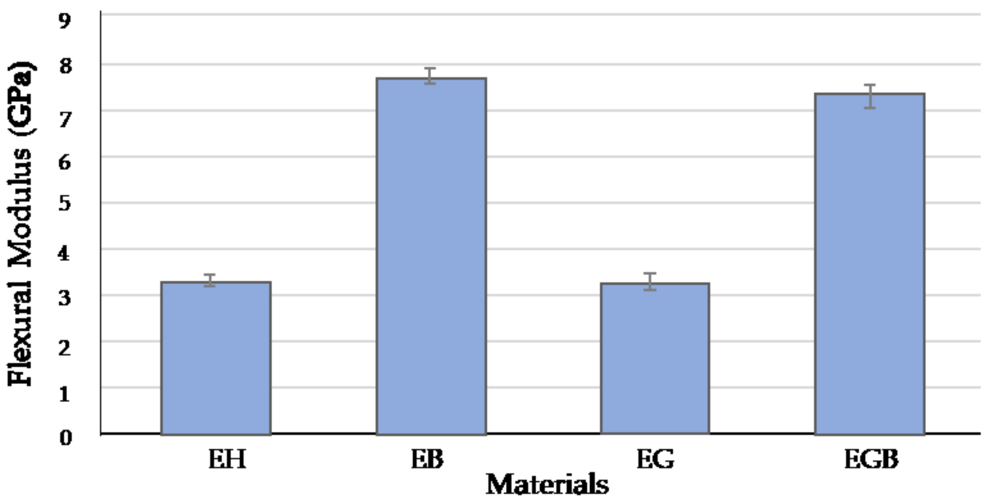
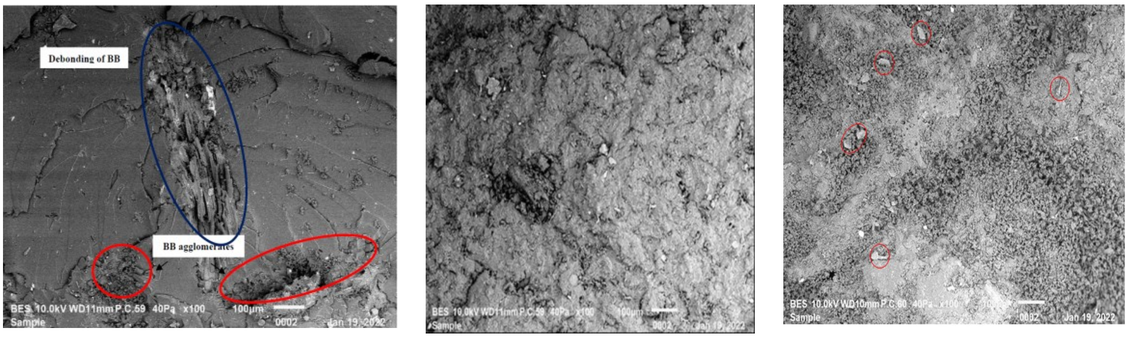


Figure 6. Flexural modulus of the samples (GPa)

Figure 7 shows the fracture surface of the EB sample under SEM. In the figure, bamboo biochar can be observed in various sizes. This may be due to the friction in the bamboo biochar container reducing the size of the biochar. SEM analysis detected less-to-no pores in the sample, which was the result of the degassing procedure in the vacuum chamber. The process helped by removing the air bubbles from the composite solution before curing took place. One disadvantage of using the solution blending technique to incorporate the composite is the non-uniform dispersion of the reinforcement in the matrix, as can be seen in Figures 7. In addition, agglomerations tend to occur when using this technique. The problem can be solved by adding solvent or dispersant to help increase the dispersibility of the biochar in the polymer. However, to preserve the original behaviour of the bamboo biochar in the epoxy matrix, no solvent was used in any of the samples because this might have changed the chemical composition and the mechanical properties of the biochar. Figure 7A illustrates the debonding of bamboo biochar from the epoxy matrix (circled in blue).



(A) (B) (C)

Figure 7. (A) EB sample under SEM at 100x magnification; (B) EG sample under SEM at 100x magnification; (C) EGB sample under SEM at 100x magnification.

CONCLUSION

In conclusion, the mechanical properties of an epoxy-graphene-bamboo biochar composite were investigated. The composite was fabricated using the solution blending technique and without adding any other elements except epoxy and its hardener, graphene, and bamboo biochar. This was a crucial step to achieving a better understanding of bamboo biochar behavior as reinforcement in composites and comparing equally the mechanical properties of biochar and graphene. Based on the data gained from this study, using bamboo biochar as reinforcement increased both the tensile and flexural strength of the composite. There were also increments in the tensile and flexural modulus. However, the elongation at break of EB was lower than that of EH. It was observed that the tensile properties of the graphene-reinforced epoxy were higher than those of the bamboo biochar-reinforced epoxy, but the flexural properties of EG were lower than those of EB. This may have been due to the high graphene composition used in the mixture. Furthermore, although bamboo biochar did not exhibit the same excellent mechanical properties as graphene, it could still be used as a cheaper replacement in structural applications such as transport body structures or load-bearing furniture.

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