In-plane shear strength analysis of Basalt Fiber-Reinforced Epoxy Laminates with Biowaste Catalyst Free Carbon

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Abstract. This study aims to analyse the effects of carbon nanospheres (CNSs) on the in-plane shear properties of basalt fiber-reinforced epoxy composite laminate (BFR). The CNSs were obtained from an economical fibrous residue attained from the sago palm tree, which is known as biowaste sago bark. Hand lay-up method was used to fabricate the unidirectional basalt fiber-reinforced epoxy composite laminates. The epoxy resin was mixed with carbon nanosphere particles (i.e., 0.6 wt% - 1 wt%). In-plane shear tests have been conducted as per ASTM standards. In addition, emphasis on the microstructural investigation using Scanning Electron Microscopy (SEM) is given, in order to study the fracture surfaces of the composite laminates. The results demonstrated significant improvement in in-plane shear strength when carbon nanosphere particles were included in the basalt fiber-reinforced epoxy composite laminate. The best result was obtained at 1.0 wt% CNSs. It displayed an increment of 37.1% in in-plane shear strength, and 36.4% increment in modulus of rigidity, respectively, in comparison to neat basalt fiber-reinforced epoxy composite laminate. The improved accomplishment of CNSs/ basalt fiber-reinforced epoxy composite laminate is due to good distribution of CNSs particles in the epoxy matrix.

Keywords. Shear strength, fiber reinforced, biowaste

1. Introduction
Fiber-reinforced polymer composites (FRCs) have been used extensively in many applications, especially in areas such as construction, aerospace, recreational equipment, automotive, marine and other high performance applications. This is due to the advantages of having high stiffness to weight ratio and their ease of processing [1, 2]. To enhance the overall properties of the composite laminate, fibers, for instance carbon fiber, glass fiber, and basalt fiber are used as filler for epoxy-based laminates. Recent research demonstrated the advantages of utilizing natural fibers in the reinforcement of polymers in order to improve the environmental safety [3]. As a waste material, the use of natural fibers as the reinforcement for the composite of the polyester is a good way move towards eco-reusing [4]. Furthermore, the benefits of natural fibers over glass fibers are in terms of the cost, density, strength-to-weight ratio, resistance to breakage during processing, energy content and recyclability [5].

Among the natural fibers, basalt fiber (BF) has been proven as a potential reinforcement of composite materials because of its good mechanical properties. Basalt fibers originated from basalt rocks through melting process. The basalt rocks can be broken down into smaller particles to produce fibers characterized by high strength, excellent fiber/resin adhesion, ability to be easily processed using conventional processes and equipment [6], sound insulation properties, low water absorption [7], good resistance to chemical attack, ecologically friendly, free from carcinogens and health hazards, high operating temperature range, and can be produced at considerably lower cost than carbon fiber [8-
In addition, BFs do not contain any other additives in the production process, which makes them cost effective. Furthermore, BFs are known to have higher tensile strength than E-glass fiber and they have larger strain to failure than carbon fiber [9]. The excellent heat resistance and low water absorption capability make them suitable as thermal insulation materials and hot fluid conduits. BFs also have advantages over the glass and asbestos fibers in terms of environmental cleanliness [12]. Moreover, the basalt fiber has high chemical stability and sound mechanical properties as well as being non-toxic and non-combustible [13].

Basalt fibers which are extracted from the volcanic rock are preferably used for reinforcing polymer matrices [14]. The chemical composition of BF is approximately identical to glass; the basic components of both elements are SiO₂, Al₂O₃, CaO, MgO, K₂O, Na₂O, Fe₂O₃ and FeO [15]. It is also important to highlight the melting temperature of the basalt fibers which ranges between 1350 and 1700°C. Basalt will solidify as a partial crystalline structure when it undergoes slow cooling process. In fact, basalt fibers can be used from 200 to 600°C without any significant losses of the mechanical properties. Basalt fibers can be applied for structural strengthening of material based on the advantages that they possess. Besides that, they also have great potential as transportation and construction materials. Before using fiber-reinforced polymer (FRP) materials as a strengthening material, preferably the mechanical and durability issues of the material are tested first.

Similarly, carbon-based nanomaterials have potential function in ultracapacitors [16-18], nanoelectronics, catalysis, microelectrical devices, sensors, and electrochemistry. Among the various forms of carbon nanomaterials, carbon nanospheres are getting more attention. This is because, in its spherical form, they are usually unclosed shells with little waving flakes that follow the curvature of the sphere. Many studies found that the mechanical, electrical and thermal properties of polymer composites are enhanced when CNSs are used as fillers in the resulting composites.

In the present study, carbon nanosphere particles will be used to enhance the properties of basalt fiber/epoxy composite laminates. The carbon nanospheres (CNSs) are obtained by a simple environmentally benign catalyst-free pyrolysis procedure from biowaste sago bark, which is a low-priced fibrous residue acquired cheaply from the local sago palm tree. The sago bark is rich in cellulose and hemicellulose, contain a small percentage of lignin and have a porous structure. The basalt fiber/epoxy laminates dispersed with 0.6–1.0 wt% carbon nanosphere particles will be fabricated by hand lay-up method. To the best knowledge of the authors, no one has yet reported on the use of carbon nanospheres particles for the reinforcement in basalt composite laminates. The present work aims to study the new approach to investigate the effect of the incorporation of inexpensive carbon nanosphere particles in a basalt fiber-reinforced-epoxy matrix on the mechanical properties (in-plane shear properties) of the composite laminates. The fractured surfaces will then be characterized by scanning electron microscopy (SEM).

2. Experimental Investigation

2.1 Materials

Basalt fiber (300 g/m² unidirectional 0°), was supplied by Suretex Composite International China. The epoxy used was epocast (Bisphenol) and the curing agent was amine based epoharden (cycloaliphatic amine). Both epocast and epoharden were supplied by Portal Trading Malaysia. The resin hardener mix ratio was 2:1 by weight. The prepared carbon nanospheres were in powder form. Scanning electron microscopy (SEM) was used to present the structure of the carbon nanospheres particles. Figure 1 shows the formation of carbon nanoparticles. The average particle size was 45-60 nm.
2.2 Fabrication of composite laminates

First, the epoxy resin was dispersed in the acetone (10 wt% to resin). The CNSs powder was then mixed with the epoxy acetone solution (without hardener). The suspension was mixed for 24 hours using automatic mixer. After that, the hardener was added to the solution. The solution was stirred manually for 15 minutes to homogenise it. The mixture was then degassed for 1 hour in a desiccator to remove the entrapped air bubbles. CNSs powder concentrations added varied from 0.6 wt% to 1 wt% with respect to the mixture of the epoxy/hardener. The basalt fiber reinforced composite laminates were fabricated by the hand lay-up method. A total of four plies of basalt fibers fabric with a size of 300 mm x 300 mm were stacked together. Three laminates were fabricated for this work. First laminate was of neat basalt, second laminate was of basalt with 0.6 wt% of CNSs particles, and third laminate was of basalt with 1.0 wt% of CNSs particles. The laminates were cured at room temperature for 24 hours. Then the specimens for in-plane shear test were cut according to the ASTM standards from the prepared laminates. The schematic layout of fabrication of BFRP laminates is shown in Figure 2.

Figure 1. The particle size distribution of CNSs using SEM

Figure 2. Schematic layout of the preparation and fabrication process of the BFRP and composite laminates.
2.3 Material testing

In-plane shear test was carried out to determine the shear strength of the laminate composites. The in-plane shear tests were performed at room temperature under standard laboratory conditions (23±5°C and 50±10% relative humidity) according to ASTM D7078, respectively. The average in-plane shear stress was defined by the ratio of maximum applied load $P$ to the cross section area $A$ ($w \times t$) between the two notches, where $w$ is the distance between upper and lower notch tip and $t$ is the thickness of the specimen.

$$\tau_{xy} = \frac{P_{\text{max}}}{A}$$  \hfill (1)

Scanning electron microscopy (SEM, JEOL JSM-5900) was utilized to identify the morphological structure of the carbon nanospheres particles and the cross-section of the fracture surface of the composite laminates.

3. Results and Discussion

In-plane shear tests were performed to observe the effect of CNS particles on the stress concentration. It is due to the existence of the geometry and material discontinuities at the free edge notches of the composite laminates. The obtained stress-strain curves are presented in Figure 3. The obtained results for in-plane shear tests are reported in Table 1. The in-plane shear strengths of the composite laminates increased by 24.3% and 37.1% for basalt with 0.6 wt% CNS and basalt with 1.0 wt% CNS, respectively. The results also show significant improvement in the shear modulus. The modulus of neat basalt was 2.8 GPa, but the modulus of composite laminates increased to 3.14 GPa and 3.82 GPa for basalt with 0.6 wt% CNS and basalt with 1.0 wt% CNS, respectively. Similar to the results in tensile test, basalt with 1.0 wt% CNS showed the highest improvement in in-plane shear properties among all the conditions. This indicates that the increase of the mechanical properties of the composite laminates is caused by the homogenous dispersion and the strong interactions between epoxy matrix and CNS particles, and basalt fiber-epoxy interfacial interaction. This brings to the efficient stress-transfer from epoxy to CNS particles and basalt fibers. A good dispersion of filler particles is necessary for an effective load transfer of polymer matrix to filler particles. Studies showed that the load-transfer mechanism happens due to the mobility of filler particles. The particles orient themselves under tensile stress, forming temporary cross-links with polymer matrix, and creating a local region of enhanced strength. The improvement of in-plane shear modulus of the basalt fiber-epoxy composite laminates is because of the increase in shear modulus of the nanospheres matrix.

![Figure 3. Typical in-plane stress-strain curves of the laminate](image-url)
Table 1. Summary of the in-plane shear properties of BFRP and CNSs

| Sample            | In-plane shear strength (MPa) | % Gain in strength | Modulus (GPa) | % Gain in modulus |
|-------------------|------------------------------|--------------------|---------------|-------------------|
| Neat Basalt       | 14                           | 0                  | 2.8           | 0                 |
| Basalt with 0.6wt% CNS | 17.4                       | 24.3               | 3.14          | 12.1              |
| Basalt with 1.0wt% CNS | 19.2                       | 37.1               | 3.82          | 36.4              |

3.1. Fractography

Figure 4 shows the SEM images of the prepared samples after in-plane shear test. The detailed fracture process such as matrix debonding and fiber pull outs were observed in each of the samples. In the case of neat basalt fiber laminates, fiber pull out was observed as shown in Fig 4 (a). The fracture surfaces showed improvement with the inclusion of CNS particles (see Fig 4 (b) and 4 (c)). Some single fibers were seen on the surface rather than completely pulled-out fiber bundles, as shown in Fig. 4 (b) and 4 (c). Basalt with 0.6 wt% CNS (Fig 4 (b)) displays matrix debonding when the in-plane shear test was carried out.

![SEM images](image)

**Figure 4.** Representative SEM images of the fracture surfaces after in-plane shear test of: (a) Neat basalt, (b) Basalt with 0.6 wt% CNS, and (c) Basalt with 1.0 wt% CNS.

4. Conclusion

In this study, we have demonstrated that in-plane shear strength and modulus of rigidity are enhanced by reinforcing the basalt fiber with carbon nanospheres particles. To summarise, this paper examined the result of carbon nanospheres particle-loading of 0.6 wt% and 1.0 wt%, on the mechanical properties of basalt fiber-reinforced epoxy composite laminate. The composite laminates with different CNS loading showed better in-plane shear properties compared to the neat basalt fiber-epoxy composite (BFRP). The best result was found for 1.0 wt% addition of CNS particle, where the in-plane shear strength increased by 24.3% and 37.1% with the addition of 0.6wt% and 1wt% of CNS particles.
respectively. Furthermore, the modulus of rigidity increased by as high as 36.4% when added with 1.0 wt% of CNS particles.

SEM images displayed good epoxy-fiber bonding with carbon nanospheres particles. The improvement in the mechanical properties is associated to the good dispersion of CNS particles in the epoxy matrix. This paper indicates that the In-plane shear strength of neat basalt fiber can be improved by the homogeneous dispersion of inexpensive CNS particles obtained from the biowaste sago bark in the epoxy matrix. With proper dispersion of micro/nano fillers, it will enhance the mechanical properties of the basalt composite laminate. This paper indicates that the In-plane shear strength of neat basalt fiber can be improved by the homogeneous dispersion of inexpensive CNS particles obtained from the biowaste sago bark in the epoxy matrix. With proper dispersion of micro/nano fillers, it will enhance the mechanical properties of the basalt composite laminate. The applications of basalt fiber in the industries would benefit from this study and could contribute to more knowledge on the exploitation of micro/nano particles to further improve the properties of epoxy-based composite materials.

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