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EFFECTS OF DOLOMITE GEOPOLYMER FILLER ON MECHANICAL PROPERTIES OF GLASS FIBRE REINFORCED EPOXY COMPOSITE

The effect of the incorporation of dolomite based geopolymer on the tensile and flexural properties of glass fibre reinforced epoxy composite were investigated. Composites containing different weight percentages of fillers (2.5, 5.0 and 7.5 wt.%) were fabricated using hand lay-up and vacuum bagging techniques. The experimental results showed that the dolomite based geopolymer contributed to the detrimental effect on tensile strength of the composite with 2.5 wt.% incorporation of the filler contributed to the least detrimental effect. 2.5 wt.% incorporation of dolomite based geopolymer meanwhile improved flexural strength by 13.04%.

Keywords: Fibre reinforced composites; glass fibre; dolomite; geopolymer; geopolymer fibre reinforced composites

1. Introduction

Due to exceptional qualities like high-specific rigidity and strength, large damping, great resistance to corrosion, and inferior thermal expansion, glass fibre reinforced epoxy composite, one of fibre reinforced polymer (FRP) composite, reveals various advantages in engineering fields like buildings, aeroplanes, land and water vehicles, chemical businesses, defence, medical uses, biomechanics, equipment of sport, robots, and machinery [1,2]. The matrix alters the applied loads to the fibres, protects the fibres from mechanical wear and damage from the environment, and provides the composite with a stable form [3,4]. The matrix generally behaved as a load transfer medium between the fibres; hence, the ability of the composites relied on the performance of the matrix to transmit the load through the interface [5]. On the other hand, the fibre fulfils the structural role, enhancing a number of composite properties [4]. The advantages of glass fibre are its cost-effectiveness and tensile strength with the range of 2.415 to 4.890 GPa. Glass fibre is especially intriguing because of its resilience to chemicals and low thermal conductivity [6-11]. Epoxy, on the other hand, is frequently used as a resin material and it is a flexible resin system that allows for an extensive range of properties as well as processing capabilities, excellent adhesion to numerous substrates as well as fibres, and remarkable effectiveness against solvents and chemicals [11,12]. Over the years, many efforts have been made to increase the mechanical properties of FRP composite by adopting a variety of techniques, such as the insertion of filler into the matrix. The physical, mechanical, thermal, and tribological characteristics of composites are improved by particulate filler.

Geopolymer filler was first used by Hashim et al. [13,14] to enhance the mechanical characteristics of FRP composite. The filler was used by the author to improve the compressive properties of filament wound glass reinforced epoxy pipe. Fly ash-based geopolymer was incorporated to the composite pipe in amounts ranging from 10 to 40 wt.%. The strength of the composite has increased with the addition of geopolymer at 10 wt.%. Because of the low NaOH molarity (4 M) used during the synthesis of the geopolymer filler, the strength of the composite pipe began to slightly decline as the geopolymer loadings increased from 20 to 40 wt.%. At higher NaOH molarities (8 and 12 M), the increase in geopolymer loadings up to 30 wt.% gave the composite more strength. In addition, 12 M NaOH contributed to highest compressive strength on the composite pipe. In addition,

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Hashim et al. [15,16] synthesized white clay-based geopolymer to be incorporated from 10 to 40 wt.% into the filament wound glass reinforced epoxy pipe. The incorporation of geopolymer mostly improved the strength of composite with the optimum loading at 30 wt.%. Additionally, 8 M of NaOH contributed to better compressive strength on the composite than that of 4 M of NaOH.

Geopolymer is a kind of inorganic amorphous material that is produced by the interaction of aluminosilicate sources such as kaolinite, fly ash, and metallurgical slag, with water-soluble alkali metal silicates under strong alkaline conditions [17-19]. This reaction results in a three-dimensional lattice of SiO_4 and AlO_4 tetrahedra connected by corner-shared O atoms. Geopolymer can be synthesised at room temperature or above through the geopolymerisation process [20,21]. The three significant chemical reactions that make up the mechanism of this geopolymerisation are the dissolution of the aluminate-silicate-oxygen bonds in the source material by the OH^- ion in the alkaline activator, the formation of dimers by the alumino-silicate monomer, that further reacts with the other monomer to form polymer chains during polycondensation, and the precipitation of reaction products together with crystallisation [17,22].

Meanwhile, due to its Al, Si, and Ca levels, dolomite is one of the natural resource that has the potential to be utilised as a raw material for geopolymers [23]. Anhydrous carbonate minerals like dolomite are made of calcium, magnesium, and carbonate, preferably $CaMg(CO_3)_2$. Besides, the term dolomite is utilised to define the sedimentary carbonate rock, which is mostly made up of the mineral dolomite which is also known as dolostone [24].

To the best of our knowledge, little has been written on the impact of dolomite based geopolymer on thermoset polymer (such as epoxy) based fibre composites in terms of mechanical properties, particularly on tensile and flexural. Therefore, the main goal of this research is to find out how the filler materials, dolomite based geopolymer, affect the mechanical characteristics of glass/epoxy composites. Tensile and flexural tests were performed on the composite samples, as well as the findings were compared with those of the reference sample with no additives.

2. Materials and method

2.1. Materials

Diglycidyl ether of bisphenol A (DGEBA) epoxy resin (EpoxAmite 100) and amine curing agent (103 slow hardener) were purchased from Komposit-Innovation, Research and Design (KIRD) Enterprise. 160G plain weave glass fibre was purchased from Mecha Solve Engineering. Dolomite samples used in this research were supplied by Perlis Dolomite Industries Sdn. Bhd. Chemical reagents such as sodium hydroxide, NaOH flakes (99% purity) was supplied from Formosa Plastic Corporation while sodium silicate, Na_2SiO_3 solution was purchased from A.R. Alatan Sains Sdn. Bhd.

2.2. Experimental procedure

The mixture of NaOH and Na_2SiO_3 solution was used as the alkaline activator solution. NaOH flakes were dissolved in distilled water to produce 20 M NaOH solutions. The NaOH solution was left to cool down at room temperature for 24 h prior to mixing with Na_2SiO_3 solution. Dolomite based geopolymer was produced by mixing the dolomite with the alkaline activator at the ratio of 2.5:1 by weight. The alkaline activator used was the mixture of Na_2SiO_3 and 12 M of NaOH (ratio of 2.5:1). The geopolymer was then cured at 80°C for 24 h. The cured geopolymer was then crushed using dry mill blender and sieved until the particle size obtained in the range of 75-100 μm.

The weighted dolomite based geopolymer filler (2.5, 5.0 and 7.5 wt.%) was mixed with epoxy for 60 minutes by using an overhead mixer at 300 rpm until a homogeneous mixture was obtained. The hardener was added to the homogeneous mixture of epoxy resin and geopolymer filler, and was carefully stirred to avoid the formation of air bubbles. The weight ratio of the resin to hardener was 3.521:1, following the manufacturer recommendation. Hand lay-up method was applied to impregnate eighteen layers of glass fibre with the resin while extreme care taken on the orientation of the fibre. A roller was used to remove the air bubbles between the fibre and the resin. Vacuum bagging technique was then applied on the resultant laminates in order to clamp the composite by using atmospheric pressure. Then, the curing was carried out at room temperature for 24 h. The reference glass/epoxy composites were also prepared using the same method. All composite laminates were cut into small specimens for mechanical testing. TABLE 1 represent the composition of resin, filler and fibre reinforcement in this research.

TABLE 1

Composition of resin, filler and fibre reinforcement

Epoxy Resin/ Hardener (wt.%)	Dolomite Geopolymer Filler (wt.%)	Glass Fibre (wt.%)
50.0	0	50.0
47.5	2.5	50.0
45.0	5.0	50.0
42.5	7.5	50.0

The tensile test of the composite samples with the dimension of 125×13×3.4 mm^3 was performed according to ASTM D3039. 50 kN Shimadzu universal testing machine has been used to perform the test. The crosshead speed of 2 mm/min was selected as the test condition. For flexural test, the composite samples with the dimension of 125×13×3.4 mm^3 was conducted according to ASTM D790, by using 250 kN Shimadzu universal testing machine. The crosshead speed of 1.45 mm/min and 54.4 mm supporting span length were selected as the test conditions. The morphology of the fractured cross-section of composites after tensile tests were analysed using Hitachi TM3000 tabletop Scanning Electron Microscope (SEM). All samples were coated beforehand with a conductive layer of platinum before the analysis.

3. Results and discussions

3.1. Tensile Strength

The tensile properties of all composite samples are summarised in Fig. 1 and TABLE 2. The introduction of dolomite based geopolymer particulate filler at 2.5 wt.% loadings provided a detrimental effect on the tensile strength of glass fibre reinforced epoxy composite. This might be a result of the fillers interfering with the interfacial bonding between the matrix and the fibre reinforcement [25]. Higher interfacial bonding will improve tensile stress transfer and lead to decreased stress concentration [26]. Besides, this is probably due to the particulate fillers, which altered the composite's behaviour and rendered it brittle, reducing the tensile strength of the composites [5].

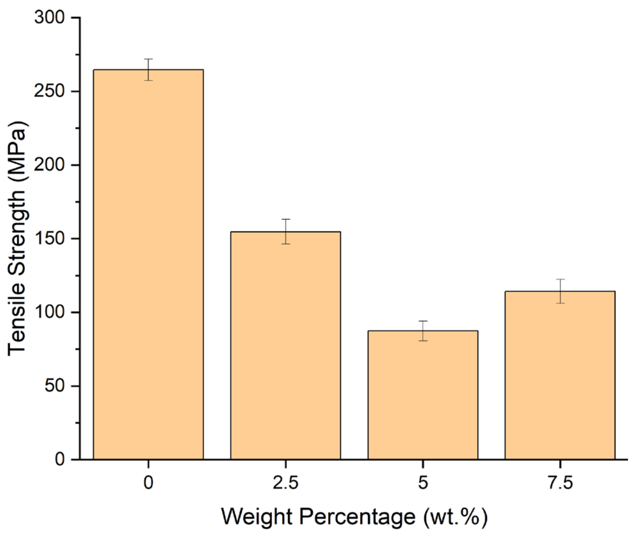

Fig. 1.

TABLE 2

Tensile properties of glass fibre reinforced epoxy composite with 0-7.5 wt.% geopolymer filler loading

Weight Percentage (wt.%)	Tensile Strength (MPa)	Tensile Strain (%)
0	268.88	5.89
2.5	165.38	4.70
5.0	89.29	3.07
7.5	115.50	3.61

The decreasing trend of the strength continues with the increase of the weight percentage of filler. The decrease can be attributed to the resin's higher viscosity with the addition of the fillers, which led to a reduction in the interfacial bonding between the resin and the fibres [27]. Hashim [28] has found that the viscosity of the resin increased as the weight percentage of fly ash based and white clay based geopolymer filler increased in the epoxy matrix. Another reason for the decrease in the tensile strength might be due to the agglomeration of filler particles which reduces the interfacial adhesion between polymer and reinforcement [29]. The homogeneity of the microstructure affects the GFRP's tensile strength [30]. Besides, the decrease of the tensile strength might be due to the voids that may have been produced by the entrapment of air by the filler [31]. In addition, cracks might be created on the surface of the matrix when the filler is added in significant amounts, which are then likely to spread more quickly and lead to early failure [29].

However, the tensile strength of the composite started to increase with the incorporation of the filler at 7.5 wt.%. The mechanical properties of the filler based composites are mainly influenced by the interfacial adhesion between the fillers and the resin material [32]. The increase may be due to better filler dispersion and stronger matrix/filler interface adhesion for effective stress transfer of the composite with 7.5 wt.% loadings than that of 5.0 wt.% loadings [33,34]. In order to obtain more accurate conclusion on the behaviour of the composite after the incorporation of the filler, the effect of higher weight percentage of the filler must be examined in the future work.

Fig. 2 illustrates the scanning micrographs of the fractured cross-sections of reference composite and composite with fillers, under tensile test. The fractured surface of the composite with 2.5 wt.% of fillers shows more fibre pull-out compared to that of the reference sample, indicating the lower adhesion between fibre and matrix due to the lower interfacial bonding between the resin and glass fibre in the presence of all type of fillers. This lower adhesion then gave the lower tensile strength. The fibre pull-out was also observed to be increased at 5 wt.% of filler loadings but reduced at 7.5 wt.%.

3.2. Flexural Strength

The composite's flexural properties are determined by its homogeneous structure. Particle dispersion, wetting, and infiltration of molten polymer into the particles are all factors that impact flexural properties [25,35]. The flexural properties of all composite samples are summarised in Fig. 3 and TABLE 3. The ability of the matrix material to transmit the load due to strong interface bonding might be the cause of the improved flexural strength at 2.5 wt.% of dolomite based geopolymer incorporation [33]. Additionally, the excellent dispersion of the microfiller in the matrix might contribute to extra load-bearing capacity of the matrix and provide flexural strength improvement [36].

TABLE 3

Flexural properties of glass fibre reinforced epoxy composite with 0-7.5 wt.% geopolymer filler loading

Weight Percentage (wt.%)	Flexural Strength (MPa)	Flexural Strain (%)
0	145.56	1.36
2.5	164.54	1.60
5.0	112.27	0.97
7.5	139.93	1.19

Further addition of these fillers has reduced the flexural strength of the composite. The decrease in the flexural property

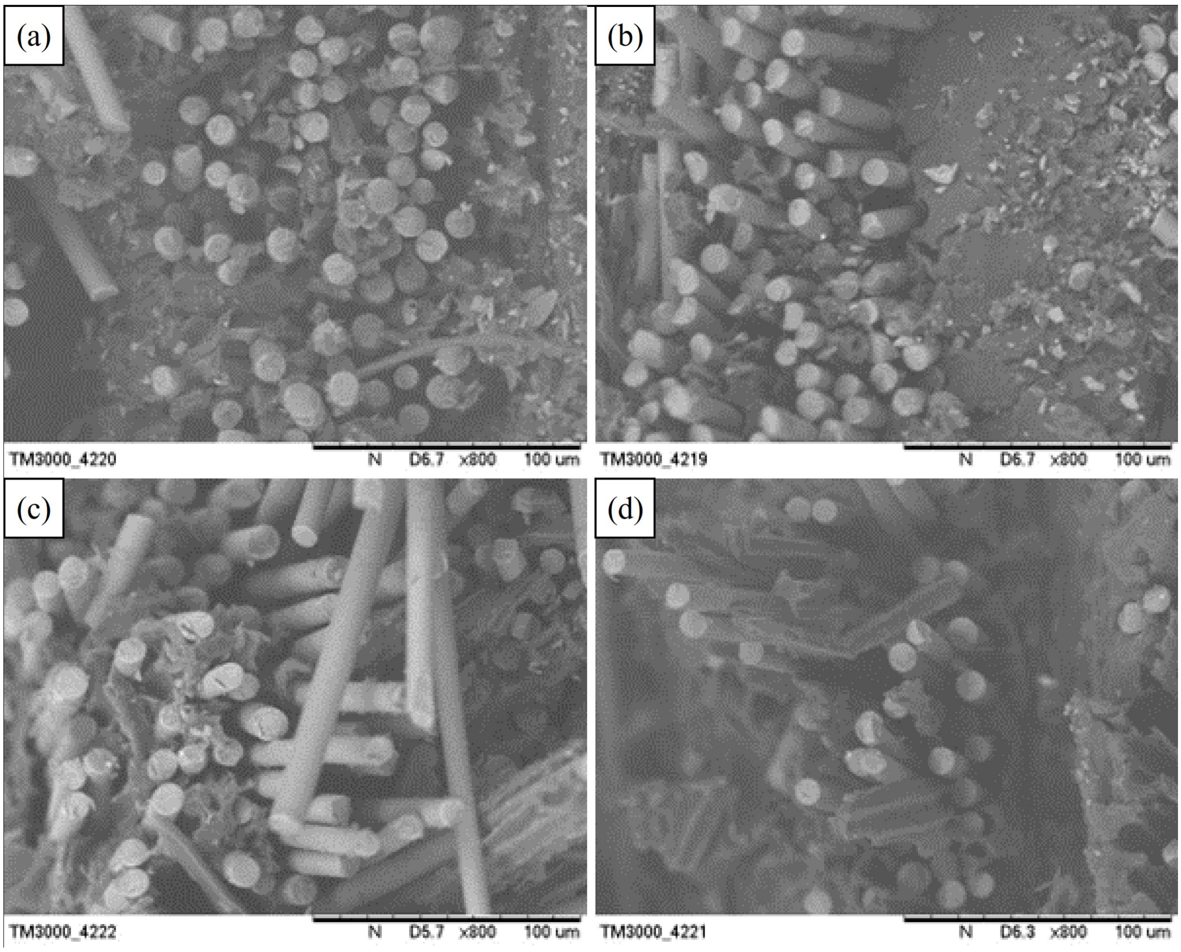

Fig. 2.

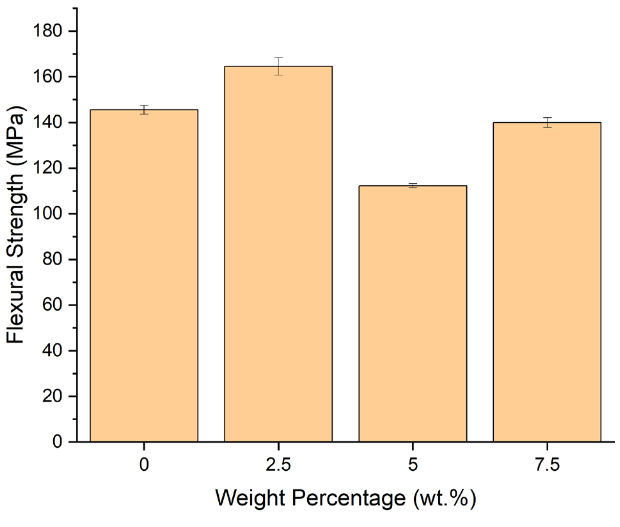

Fig. 3.

might be due to the fact that in three-point bending, the fibres are harmed by the accumulating filler material that comes out from the interface, reducing the load carrying capacity of the composite [29]. Besides, this degradation of strength at higher loading of filler may be due to the chemical reaction between filler particles and resin at the interface which is too weak to withstand the stresses acting at the same time [37]. Another reason for the decrease in flexural strength at 5 wt.% of filler might be caused by the agglomeration of microfillers, which raises the probability of matrix cracking [36].

However, the flexural strength of the composite started to increase back when incorporating 7.5 wt.% of dolomite based geopolymer. A possible reason is due to better dispersion of filler at 7.5 wt.% than that of 5.0 wt.% making the matrix homogeneity of the former better than the latter. This then increase the load-bearing capacity of the matrix. The increase of flexural strength at 7.5 wt.% loadings of dolomite based geopolymer may be explained by the effective interfacial adhesive bonding between the fillers and the matrix [32].

4. Conclusions

In this study, it was found that geopolymer can be utilised as a filler in the FRP application through conventional hand lay-up technique. Glass fibre reinforced epoxy composite filled with dolomite based geopolymer was developed with different weight percentage of the geopolymer material. The experimental result shows the performance of the product through the tensile and flexural tests. Based on the results, the tensile strength of the composite is reduced with the incorporation of the geopolymer particulate filler with 5.0 wt.% incorporation of the filler contributing to the lowest tensile strength. Meanwhile, the flexural strength of the composite is improved with certain loadings of the filler. At 2.5 wt.% incorporation, the filler contributed to the highest flexural strength.

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