The Microstructural and Mechanical Properties of Glass Fiber Reinforced Geopolymer Composites

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Abstract

Geopolymers are inorganic aluminosilicate materials that possess excellent mechanical properties and good thermal resistance properties. However, the limitation with brittle behaviour and shrinkage effect is there. One of the best solutions to address the limitation is by incorporation of fibers to a brittle matrix as to improve the compressive and flexural strength of geopolymers. This study broadly evaluates the effects of glass fiber content at 0.2 to 2.0wt% with length of 6mm and 12mm on microstructural and mechanical properties of geopolymer composites. The compression and flexural strength of composites was reported, as the morphological structure of fracture surface and fiber-matrix interface was examined using Scanning Electron Microscopy (SEM). Results show a porous surface and partially reacted fly ash were observed in the microstructural analysis of all geopolymers. The incorporation of glass fiber up to 1wt% into the geopolymer matrix reduces the shrinkage and enhances the flexural and compressive strength of the composites. Microstructural images demonstrated the glass fibers act as filler for pores within geopolymer matrix, producing denser geopolymer composite, thus improving the mechanical properties. Further increase the fiber content up to 2wt%, resulted lower level of compression and flexural strength due to shrinkage effect.

Keywords: Fiber, Geopolymer, Microstructural

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Introduction

The usage of coal in electric power industry plants such as in Kapar, Manjung, Sejingkat, Jimah and Tanjung Bin has given a serious impact not only to the health but also to the environment. Millions of tons of coal ash are produced each year from coal combustion. Practically, the coal combustion product knows as coal ashes are required only to store in landfills and disposal site [1].The improper disposal process of coal ash will cause soil pollution that indirectly leads to water solution. The ecological cycles and environment hazards will be disrupted. Concerning to that issue, researchers start to find the best alternatives that can be implemented as to reduce that such effects. Started with 1970s, Davidovits and teams found that waste product of coal ash can are turn into valuable products which can be applied in many fields such as in cement industry, road materials, agriculture fields, and the latest as a coating material. In fact, fly ash is also widely used in Ordinary Portlant Cement (OPC) concrete as it gives many benefits and able to enhances the performance of concrete in both state wither fresh or hardened. Fly ash in concrete can enhance the strength and durability of hardened concrete. When fly ash is added to concrete, the amount of OPC may be reduced, significantly will value to the money.

The terms of "geopolymer" being introduced by Davidovits as the product of chemical reactions between coal ash and alkaline activator solution [2], [3]. Geopolymer is a synthetic green "polymeric" material that formed by chemical reaction of rich aluminosilicates reactive materials gained from power plants such as coal fly ash, mineral clays (metakaolin), and/or the slag form metallurgy. An alkaline and/or alkaline silicate activator solution was used as accelerators in the development of geopolymer [4]. This highly potential material gained an attention among the researchers likes in in the 1980 – 1990, in North America a hybrid concrete called "pyrament" that having superior early strength was commercialized using geopolymer. However, the existing of quasi-brittle and shrinkage behavior gives the limitation to the geopolymer performance. One of the best methods to reduce shrinkage issue is by adding a reinforcement agents, such as a fiber into the geopolymer matrix, which later known as geopolymer composite [5].

Davidovits in 1991 was first introduced the fiber reinforced geopolymer composites for the plastics processing industry in fabrication of molding tools [4]. There is a various type of fibers that have been used as a reinforcement agent in geopolymer. Natali et al. proved that by incorporated polyvinyl alcohol (PVA) to metakaolin based geopolymer composites had increased by at least 30% and up to 70% of flexural strength with 1 wt% of PVA [6]. Yan et al. [7] found the addition of 1 wt% graphene gave 17% improvement in fracture toughness as compared to plain geopolymer. In 2016, a same group used reduce graphene oxide/leucite nanocomposite, and observed the toughness was increased to the maximum value of 75.4 MPa [8]. Lin et al.[9] used a sheet-like carbon fiber as to strengthen a geopolymer of the type poly(sialate-siloxo). The results show a maximum flexural strength of 91.3 MPa was obtained using 7mm fiber length. Yuan et al. [10] studied the effects of short SiC fibers on the microstructure and mechanical properties of the geopolymer composites. The highest flexural strength of 94MPa was obtained using 2 vol% of fiber with 5mm length. Korniejenko et al. [11] studied the mechanical properties of geopolymer composite reinforced with natural fibers such as cotton, sisal, raffia and coir. The results of compressive and flexural strength tests show that the addition of cotton, sisal and coir fibers improves the strength. However, the lack of cohesiveness between the fibers and geopolymer matrix was observed with the addition of the raffia fibers. A comprehensive study of the polypropylene fiber reinforced geopolymer composite was conducted by Ranjbar et al. [12]. The results indicate

that the workability of the composites is reduced significantly by increasing the percentages of fiber inside because of higher shear resistance to flow. Addition of PP fibers up to 3 wt% enhances the energy absorption of the composites. The behaviour of basalt and carbon reinforced geopolymer was studied by Faiz and Sharani [13]. The findings show that the geopolymer composites with addition of carbon fibers offered better mechanical properties than that basalt fibers. The volume of pores was higher in basalt reinforced geopolymer composites, resulted to low strength of compressive and flexural. Bhutta et al. [14] studied the mechanical behaviour of geopolymer composites reinforced with steel macro fibers. Three different types of steel were tested; length-deformed, end-deformed and straight. The results show that the length-deformed one is the least favorable as reinforcement agent due to the highest matrix-fiber adhesion that cause matrix failure prior to fiber pull-out effect.

Though so many materials have been tested as a reinforcement agent in geopolymer as to produce fiber reinforced geopolymer composite, there are only few studies discussed with the potential of the glass fiber. Glass fiber is a low cost synthetic material which easily available in the market comes with high tensile strength. It is an amorphous silicate with additives that function as intermediates and modifiers network formers. Sankar [15] studied the flexure and properties tensile of metakaolin-based geopolymers reinforced with glass fiber. The average tensile and flexural strengths were 39.3 ± 7.2 MPa and 25.6 ± 4.8 MPa, respectively for sodium geopolymer reinforced with glass fiber. Study by Nematollahi and Sanjayan [16], found the inclusion of glass fiber in the geopolymer matrix lead to the poor workability of the composite. The latest studied conducted by Samal [17] show the glass fiber had expanded in the matrix, thus creating a cage like structure. This kind of structure prepared the fiber from sliding effect in between of the matrix, considerably maintains the strength of composite though at elevated temperature.

Based on the existing literatures, it can be summarized that glass fiber manages to improve the strength and improve the other mechanical properties of geopolymer composite. However, there was a little study discussed on the relationship of glass fiber reinforced geopolymer composite's morphology towards mechanical properties. Therefore, this study was carried out as to address the effect of glass fiber on the mechanical properties of geopolymer through morphological point of view. A variant of the glass fiber mass between 0.2 to 2.0 weight% with two different lengths of 6mm and 12mm was incorporated into geopolymer matrix (fly ash based). The adhesive and compressive strength of composites were measured for 28 days curing period.

Materials and Methods

Materials

The glass fiber chopped strands, shown in Fig. 1 as a reinforcement agent was imported from JN Technologies Pvt. Ltd with the average diameter of 14µm. The type of glass fiber used in present study was AR glass fiber. (AR: alkali resistant) glass fibers are specially designed for concrete construction as they are effective to prevent concrete cracking. It is ideal for applications with high acidic medium and to add strength of the structure. Two different fiber lengths were chosen; i.e 6 mm and 12mm. Glass strands are mainly composed of SiO_2, Al_2O_3, CaO, MnO, SO_3, MgO, Na_2O, Fe_2O_3, K_2O, TiO_2 and P_2O_5. The properties of glass fiber were summarized in Table 1. Low calcium of fly ash (classified as class F), as shown in Fig.2 obtained from the Manjung power station in Perak, Malaysia was used as the source material to prepare geopolymers. The major composition of fly ash was listed in Table 2. Prior to

develop geopolymer paste, 10M concentration of alkaline activator of sodium hydroxide (NaOH) solution was prepared by dissolving NaOH pellets in distilled water and was stored 24 hours in air tight DURAN® GLS 80® wide neck glass bottle to prevent changes in the composition of the solutions due to contamination, and so that the solutions were stable and uniform.

Table 1: Properties of glass fiber

	Tensile strength(Mpa)	Modulus of elastic-ity(Gpa)	Water absorption	Alkali resistance	Corrosion resistance	Colour
Glass fiber	1700	72	<1%	high	high	white

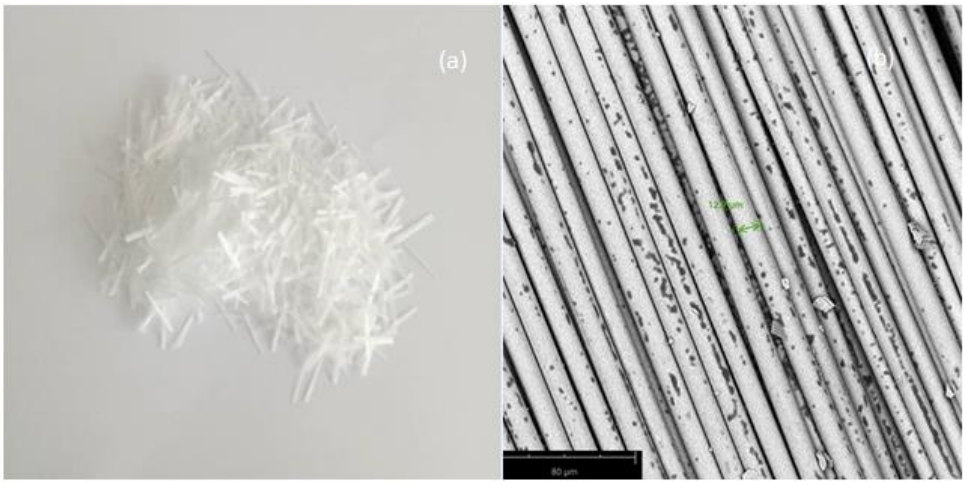

Fig. 1: Image of glass fiber strands (a); SEM image of glass fiber strands at 500X(b)

Table 2: Chemical composition of fly ash

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	SO_3	Na_2O	TiO_2	P_2O_5	BaO	SrO	MnO	other	LOI
43.25	20.59	12.49	11.11	3.76	1.96	1.45	0.92	0.89	0.32	0.18	0.12	0.11	5.96	2.84

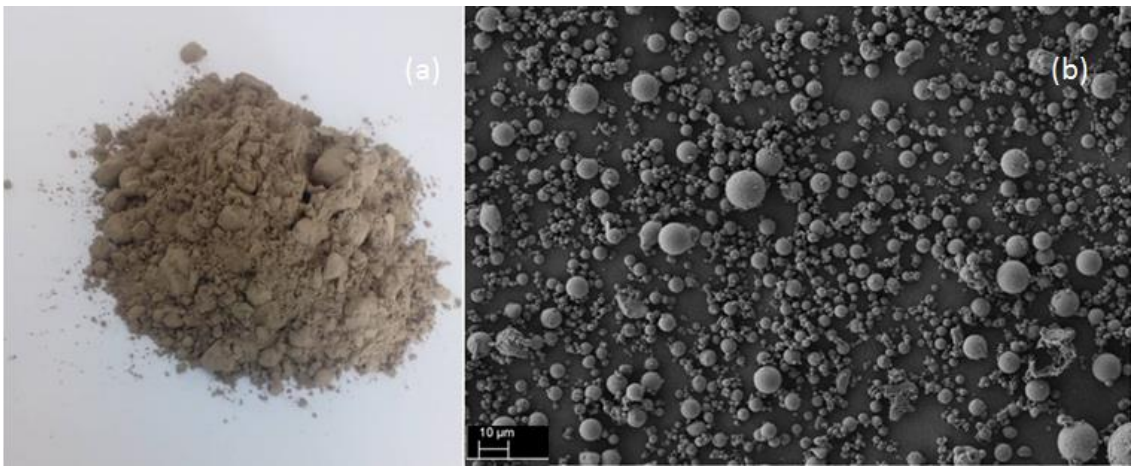

Fig. 2: Image of fly ash (a); SEM image of fly ash (b)

Preparation of geopolymer composite

Twenty different geopolymer composites with one plain sample were prepared. Their mix design is shown in Table 3.To prepare the geopolymer composite, fly ash and glass fiber were thoroughly mixed with the alkali activator solution of NaOH using an overhead mechanical stirrer at a stirring speed of 1000 rpm for 5 minutes to ensure homogeneity. The mass of glass fiber varied from 0.2 to 2.0 wt%, with two different fiber lengths at 6mm and 12mm. Water: solid and Na: Al ratios were fixed at 0.33 and 1.0, respectively. The Si: Al ratio of 1.78 was used as ready as its actual composition of fly ash. Prior to the mechanical testing, all the samples were cured in an oven at 60°C up to 28 days of curing period. The geopolymer acronyms, $GC_{x,y}$ reflected to the fiber length and mass percentage. For instance, $GC_{6,0.2}$ indicates the geopolymer composite with 6mm glass fiber length with 0.2 wt% of fiber mass present. While, G_{neat} represent the control geopolymer without presence of glass fiber.

Table 3: Mix design of the investigated geopolymer samples

Sample	Fiber length(mm)	Fiber content (wt %)	Na:Al	Water:solid
G_{plain}	-	-	-	-
$GC_{6,0.2}$	6	0.2	1	0.33
$GC_{6,0.4}$	6	0.4	1	0.33
$GC_{6,0.6}$	6	0.6	1	0.33
$GC_{6,0.8}$	6	0.8	1	0.33
$GC_{6,1.0}$	6	1	1	0.33
$GC_{6,1.2}$	6	1.2	1	0.33
$GC_{6,1.4}$	6	1.4	1	0.33
$GC_{6,1.6}$	6	1.6	1	0.33
$GC_{6,1.8}$	6	1.8	1	0.33
$GC_{6,2.0}$	6	2	1	0.33
$GC_{12,0.2}$	12	0.2	1	0.33
$GC_{12,0.4}$	12	0.4	1	0.33
$GC_{12,0.6}$	12	0.6	1	0.33
$GC_{12,0.8}$	12	0.8	1	0.33
$GC_{12,1.0}$	12	1	1	0.33
$GC_{12,1.2}$	12	1.2	1	0.33
$GC_{12,1.4}$	12	1.4	1	0.33
$GC_{12,1.6}$	12	1.6	1	0.33
$GC_{12,1.8}$	12	1.8	1	0.33
$GC_{12,2.0}$	12	2	1	0.33

Determination of flexural strength

Prior to the testing, the samples were hand-formed and poured into 13 mm x 3 mm x 127 mould and then subjected to vibratory removal of air bubbles. The samples were heated in the oven at 60° C for 28 days curing period. The samples were unmolded prior to the flexural test. In flexural test, the samples were subjected to three-point bending tests using Gotech Al-7000 S universal testing machine (50 kN capacity) with a displacement rate of 1.0 mm/min. The flexural strength (F_s) was determined using the following equation [18]–[20]:

$$F_s = \frac{3}{2}\frac{P_m s}{BW^2} \quad \text{Eq. 1}$$

Where, *Pm* is the maximum load at crack extension, *S* is the span of the sample, *B* is the specimen width and *W* is the specimen thickness or depth.

Determination of compressive strength

The standard EN 12390-3-2009 was a guideline to conduct a compression test [21]. A cubical sample with the measurement of 50mm x 50mm x50mm was prepared prior to the testing. The testing was carried out with Liangdong compression testing machine.

Results and discussion

Chemical analysis of fly ash

Based on the data in Table 2 clearly show that ash used in this work was class F [22]. If the total of constituting of SiO + Al_2O_3 + Fe_2O_3 greater than 70% its mean that ash is belong to the class F classification [23]–[25] The presence of iron oxide will affect to the dissolution process of aluminosilicate and thus it will consider as a detrimental effect on the system. In a meanwhile, the presence of calcium oxide gives cementitious properties to the geopolymer during curing process [26]. The loss on ignition (LOI) value refers to the mass of unburned carbon that remains in fly ash.

Flexural strength

The effect of fiber content on the flexural strength of geopolymer composites was shown in Fig. 3&4, as for 6mm and 12mm fiber length, respectively. Noticeable on both systems, greater flexural strength was obtained in reinforced geopolymer composites compared to the neat geopolymer, regardless the fiber length. A similar trend was shown in both systems; flexural strength is directly proportionate to the fiber content up to 1 wt%. In 1 wt% of the glass fiber content, the highest flexural strength of 11.4 MPa and 8.8 MPa was obtained for the 6mm and 12 mm systems, respectively. A strong adhesion between the fiber and matrix was obtained due to the well homogenous distribution of fiber. This kind of distribution helps in minimizing the pores size and volume between the matrix. Such well dispersion and less pores in the composite structure is believed to permit an optimal stress-transfer operation from matrix to fibers, significantly improving the composite properties[27], [28].

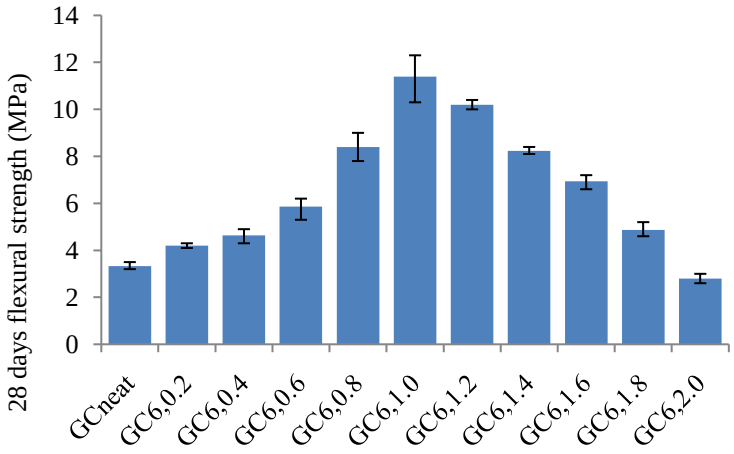

Fig. 3: Flexural strength of geopolymer composites as a function of glass fiber content of 6mm length.

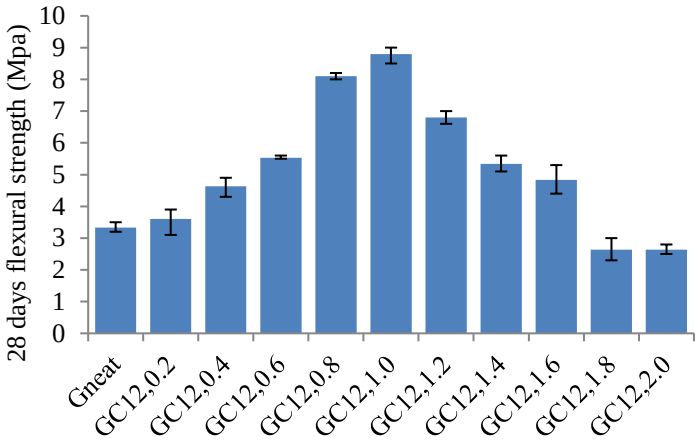

Fig. 4: Flexural strength of geopolymer composites as a function of glass fiber content of 12mm length.

However, further increasing fiber content beyond the optimal weight, a detrimental effect may happen to the flexural strength of composite. It can be seen in both figures, the flexural strength starts to decrease after fiber content exceeds 1 wt%. With too much presence of fiber, i.e. 2 wt% glass fiber in the system resulted weaker composite with similar behaviour to the neat geopolymer. Lowest flexural strength of 2.8 and 2.6 MPa was obtained from 6mm and 12 mm fiber length system, respectively. The decrease in the flexural strength above the optimal weight of fiber can be attributed to a weak interfacial bond between the fiber and matrix, probably due to the fibers is ball to each other in the paste. The interfacial bond plays a significant role in the geopolymer matrix as to transfer the stress and elastic deformation from the matrix to the fibers. Having a weak bond, the fibers were unable to carry the external load applied. It only can happen by having a strong interfacial bond between the fiber and matrix, ultimately increases the yield strength of the geopolymer composites [29]. Lower flexural strength was observed in 12mm fiber length systems. Poor distribution of fibers leads to the agglomeration effect in geopolymer matrix, producing a brittle composite with low strength

Compressive strength

The addition of different concentrations of glass fibers to geopolymer composites affects the resulting material's compressive strength. The addition of the glass fiber led to improvements in compressive strength on both 6mm and 12mm fiber length systems, as shown in Fig. 5&6. The highest compressive strength in the geopolymer composites was achieved with the addition of 1wt% glass fiber, found to be 89% greater than that of the neat geopolymer, using 6mm glass fiber and 69% by using 12mm glass fiber. A 34 MPa and 30MPa of compressive strength were obtained from 6mm and 12mm glass fiber system, respectively. Three mechanisms can be related to the enhancement in compressive strength; firstly, the bridging effect of fibers into pores, produce a compact and dense geopolymer structure than that neat geopolymer. Secondly, is the potential of the fiber to delay propagation of micro crack, indirectly improve the energy absorption and ductility of the composite through a bridging effect, leading to a greater load carrying capacity. Thirdly, is the well dispersion of fiber into geopolymer matrix, significantly create a strong bonding at the fiber / matrix interface. An optimal transferring of stress from matrix to fibers will be occurred by having a good interface interaction, thus significantly improve the compressive strength of composites [24], [30].

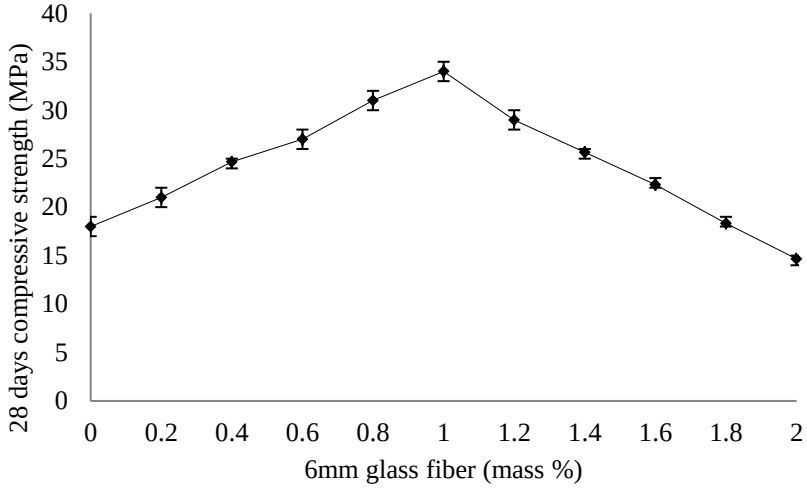

Fig. 5: Compressive strength of geopolymer composites as a function of 6mm glass fiber content

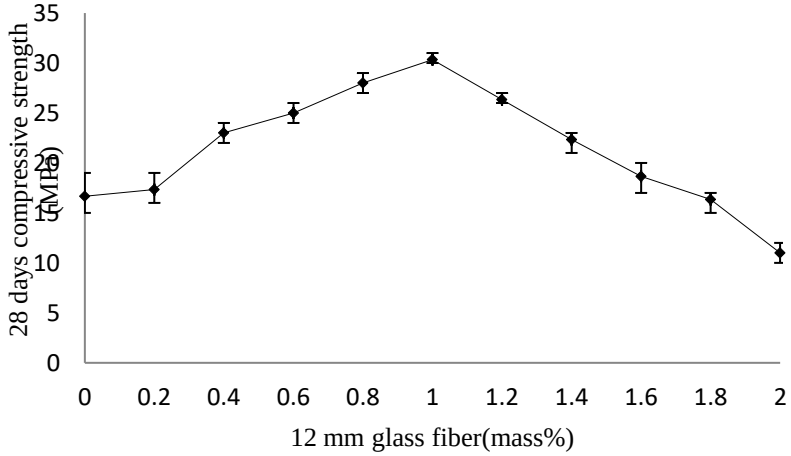

Fig. 6: Compressive strength of geopolymer composites as a function of 12mm glass fiber content

However, there was a gradually decreased in compressive strength with a further increment of the fiber weight (after 1wt% onwards) and length. The results indicate with too much fiber in the system, lead the compressive strength as low as to neat geopolymer. The higher fiber content suggests a greater chance for fibers to ball together. Such balling effect may lead to the existing of voids in the matrix, resulting in poor of the interfacial bonding between fibers and matrix [31]. Furthermore, the presence of voids and balling effect creates a stress concentration when the composite is carried load; therefore the compressive strength is reduced. The effectiveness of the fiber in bridging the cracks is determined by the interfacial bonding between the fiber and matrix [32]. Decreasing in compressive strength by 12mm fiber length could be explained by balling effect among the fibers, resulting poor distribution of fiber through the matrix. This turns in lack of stress transferring between the fiber and matrix.

Microstructure evaluation

To evaluate the mechanisms reinforcement of glass fiber reinforced geopolymer composites, the fracture surfaces of compressive test samples were examined by SEM. Fracture surfaces of those composites, together with neat sample are shown in Fig. 7 (a)-(g). Partially reacted of fly ash with existing pores in between the matrix, was observed in Fig. 7 (a). This kind of structure show there was fragmentation mechanisms happen from larger particles into small particles during geopolymerization process. A composite with brittle characteristic was produced, that required little amount of energy to break it.

A significant changes to the morphological structure of geopolymer was observed by adding the fiber into geopolymer matrix; Fig. 7(b) – (g). As compared to the neat geopolymer, the presence of pores manages to be reducing by reinforcing the fiber into the matrix. The compactness of structure was observed at 1 wt% of fiber, regardless 6mm or 12 mm fiber length, shown in Fig. 7 (d&e). The fiber was strongly adhered to the matrix interface, significantly closed the pores. This structure produces dense material which that can sustain more load applied. These types of morphology, suggesting that toughening mechanisms may come by crack bridging and fiber pullout effect. These strengthening mechanisms are believed as an important parameter contributes to the greater fracture energy of glass fiber reinforced geopolymer composites. The concentration exists in the composite, being transferred to the glass fiber which then controlled by shear process between interlayer of fiber and matrix at crack propagation area.

Despite this, Fig. 7 f & g clearly indicates the glass fiber is ball together and loses the adhesion between the matrix after 2 wt% fiber addition. It was sparse to each other and poor embedded into the paste. These morphology images suggest that the presence of too many fibers lead to increase the growth rate of crack due to the crack propagation at the interface between fibers and matrix. The large gap between the matrix and fiber interface lead to the strength losses because of debonding situation between fiber and matrix. The results proved that interfacial bonding is better in geopolymer composites with 1 wt% fiber content, contrasting to those with 2 wt%.

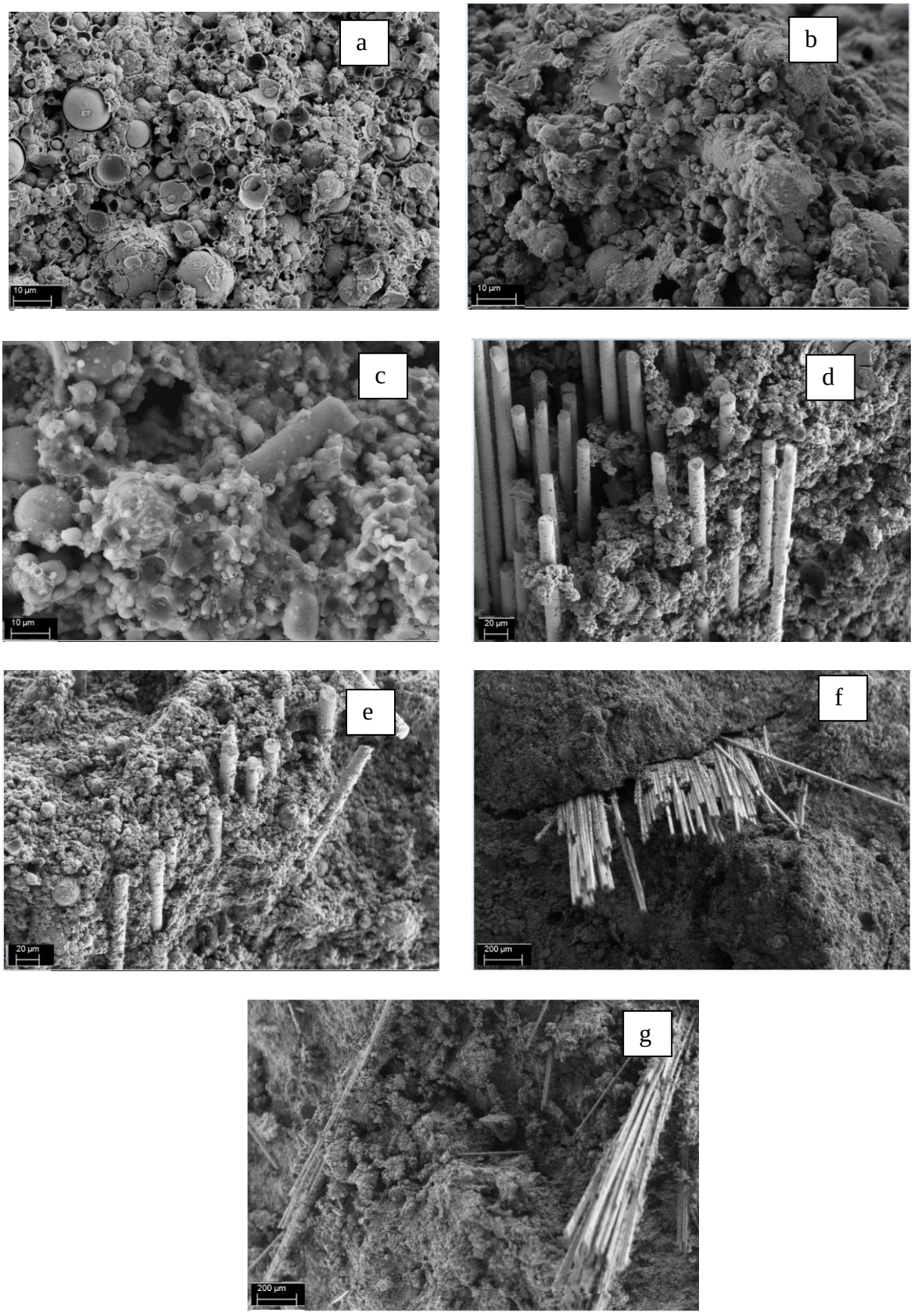

Fig.7: SEM images of fracture surface for geopolymer composite reinforced with glass fibers (a)0 wt%, (b) 0.2 wt%, 6mm, (c) 0.2 wt%, 12mm, (d) 1 wt%, 6mm, (e) 1 wt% , 12mm (f) 2 wt%, 6mm, (g) 2 wt% , 12mm glass fiber

Conclusion

The findings indicate the optimum content of glass fiber that should be incorporated in producing higher strength of composite is by 1 wt%. FESEM images show there were only small amount of pores presence in geopolymer with 1 wt% of glass fiber. However, contrast structure morphology were observed when fiber content exceeds 1 wt%. It can be seen, poor dispersion of fiber in matrix happen that cause to the agglomeration consequence. The existing of pore act as stress concentrator that generates a weak area for fracture starts to initiate. It can be concluded that glass fibers is one of the appropriate materials that can used as a reinforcement agent to develop a geopolymer composite, owing to it superior mechanical properties.

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Author Contributions

Experiments were largely designed by M.Y. with input from S. and M.A. and conducted and analyzed by M.Y., S., and R. The manuscript was drafted by M.Y. and reviewed by all authors.

Disclosure of Conflict of Interest

The authors declare no competing financial interests.

Compliance with Ethical Standards

This chapter does not contain any studies with human participants or animals performed by any of the authors.

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