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Synthesis and mechanical properties of flax fabric reinforced geopolymer composites

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Abstract. Geopolymer composites reinforced with different layers of woven flax fabric are fabricated using lay- up technique. Mechanical properties, such as flexural strength, flexural modulus and fracture toughness of geopolymer composites reinforced with 2.4, 3 and 4.1 wt% flax fibres are studied. The fracture surfaces of the composites are also examined using scanning electron microscopy. The results show that all the mechanical properties of the composites are improved by increasing the flax fibre contents. It is also found that the mechanical properties of flax fabric reinforced geopolymer composites are superior to pure geopolymer matrix. Micro-structural analysis of fracture surface of the composites indicated evidence of various toughening mechanisms by flax fabrics in the composites.

Keywords: geopolymer composite; flax fibre; mechanical properties

1. Introduction

Ordinary Portland cement concretes are used in many construction applications because of their good mechanical properties. However, the serious greenhouse emissions caused during manufacturing of cement has made it necessary to find an eco-friendly alternative material. A new type of promising materials is the aluminosilicate inorganic polymers (also known as geopolymer). These inorganic compounds exhibit good mechanical performance, durability, inflammability and acid resistance. Furthermore, they can be cured and hardened at room temperature with 80-90% less carbon dioxide emission than Portland cement (Barbosa *et al*. 2000, Li *et al*. 2004, Duxson *et al*. 2007, Pernica *et al*. 2010).

Geopolymers are synthesized by activating a solid aluminosilicate source with alkaline solutions. They are currently attracting widespread attention because of their potential as high performance and environmentally friendly replacements for ordinary Portland cement in many applications (McLellan *et al*. 2011, Pacheco *et al*. 2012). Davidovits (1991) coined the name "Geopolymer" to denote a new material that was prepared by reacting aluminosilicate sources such as fly-ash, volcanic ash, and granulated ground blast furnace slag with alkali silicate solution under highly alkaline conditions. It has been shown that a wide range of waste aluminosilicate materials

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may be converted into building materials, as they acquire excellent physical and chemical properties (Davidovits 1991, Buchwald *et al*. 2005, Lemougna *et al*. 2011, Higgins 2003).

There are many factors influencing the choice of the source material when making geopolymers, such as availability, cost, type of application, and specific demands of the users. Recent research has shown that fly-ash is the most favourable raw material for geopolymer production, as the strength of fly-ash increases with time and its moisture content is less than that of soils (Hardjito *et al*. 2004). Therefore, the present research focuses the use of fly-ash as a source material in geopolymers.

Despite their desirable characterisations, geopolymer compounds, like most ceramics, suffer from brittle failure mode under applied force. The typical values of the compressive strength of geopolymer-based ceramics are around 45MPa (Kriven *et al*. 2003) which are comparable to the strength of Portland cement pastes. However, geopolymer pastes show low flexural strength ranging between 1.7MPa and 16.8MPa (Kriven *et al*. 2003, Lin *et al*. 2008). Improving the flexural and tensile strengths will increase the application of these materials significantly in construction and building. This may be readily accomplished through dispersing inorganic or organic fibres into the geopolymer matrix. Various inorganic fibres such as carbon, basalt, and glass fibres have been utilized for this purpose (Hung *et al*. 2008, Rill *et al*. 2010, Silva and Thaumaturgo 2003, Vijai *et al*. 2012, Zhao *et al*. 2007). However, the environmentally unfriendly and costly methods needed to produce such fibres tend to limit their use in future applications. For these reasons, natural fibre composites have attracted much interest in recent years (Zeng *et al*. 2005).

Natural fibres include flax, hemp, cellulose, jute, pineapple, straw, switch grass, kenaf, coir, and bamboo (Dweib *et al*. 2004, Tanobe *et al*. 2005). As reinforcements that enhance the strength and fracture resistance of polymeric matrices, natural fibres are increasingly used because of their low density, low cost, renewability, recyclability, and excellent mechanical characteristics like flexibility, high specific strength, and high specific modulus (Low *et al*. 1995, Low *et al*. 2007). Furthermore, sustainability, new environmental regulations, growing global environmental awareness, and societal concerns have also given an impetus to develop environmentally friendly composite materials. Non-toxic materials like natural fibres are better for health and safety, rarely being hazardous during handling (Satyanarayana *et al*. 1990, Bessadok *et al*. 2007).

Investigations on natural fibres such as bamboo, sisal, jute and cellulose have revealed desirable effect on the physical and mechanical properties of brittle organic and inorganic matrices. For example, wood derivative cellulose has been used for toughening epoxy and other polymers (Zadorecki and Michell 1989, McGrath *et al*. 2004, Panaitescu *et al*. 2008, Low *et al*. 2009). Similarly, the flexural strength of concrete can be improved effectively if it is reinforced by bamboo fibres (Rahman *et al*. 2011). Also, the same desirable effect has been observed in wood fibre-reinforced concrete (Lin *et al*. 1994). In another study, it has been found that cotton fibres enhanced the strength and toughness of geopolymer (Alomayri and Low 2013). Wool and hemp fibres have also been successfully used in reinforcing geopolymer composites with concomitant improvements in mechanical and fracture properties (Alzeer and MacKenzie 2012, Alzeer and MacKenzie 2013). However, no study so far been reported the mechanical properties of flax fabric (FF) reinforced fly-ash based geopolymer composite. The use of flax fabrics has several advantages, which include low cost, renewable, and low weight comparing with synthetic fibres.

In this study, flax fabrics (FF) have been saturated with geopolymer paste, stacked, and compressed by a roller to force the paste to penetrate the fabric and to remove most of the trapped air. The effect of various contents of flax fabrics on mechanical properties was investigated. Useful

results have been gathered for composites with different FF contents (0, 2.4, 3 and 4.1 wt%) under three-point bending tests. The results show that the addition of FF improves the mechanical properties of geopolymer composites such as flexural strength, flexural modulus and fracture toughness. Scanning electron microscopy (SEM) was used to characterize the microstructure, and failure mechanisms of flax fabrics reinforced geopolymer composites.

2. Experimental investigation

2.1 Materials

Flax fabric was used as reinforcement for the fabrication of geopolymer composites. The flax fabrics of 30×30 cm^2 was supplied by Pure linen Australia. This fabric is made up of yarns with a density of 1.5 g/cm^3, the opening space between the yarns is between 2-4 mm (see Fig. 1). This is sufficient space that allows the geopolymer matrix to penetrate through the opening. The diameter of the fibres is about 10 μm (see Fig. 2). The physical properties of flax fabrics from various references are shown in Table 1. Low calcium fly-ash (ASTM class F), collected from the Eraring power station in NSW, was used as the source material of the geopolymer matrix. The chemical composition of fly-ash is shown in Table 2. The alkaline activator for geopolymerisation was a combination of sodium hydroxide solution and sodium silicate grade D solution. Sodium hydroxide flakes with 98% purity were used to prepare the solution. The chemical composition of sodium silicate used was 14.7% Na_2O, 29.4% SiO_2 and 55.9% water by mass.

Table 1 Mechanical properties of flax fibers from various references.

Young's modulus(GPa)	Tensile strength (MPa)	Strain to failure (%)	Reference
103	690	_	(McMullen 1984)
100	1100	2.4	(Bledzki *et al*. 1996)
52	621	1.33	(Davies *et al*. 1998)

Table 2 Chemical composition of fly-ash

SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	Na_2O	P_2O_5	SO_3	TiO_2	MnO	BaO	LOI
63.13	24.88	2.58	3.07	2.01	0.61	0.71	0.17	0.18	0.96	0.05	0.07	1.45

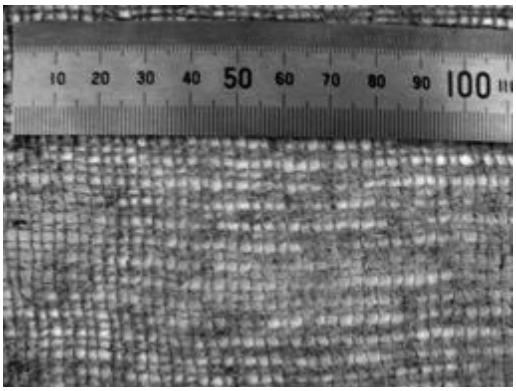

Fig. 1 Structure of the flax fabric

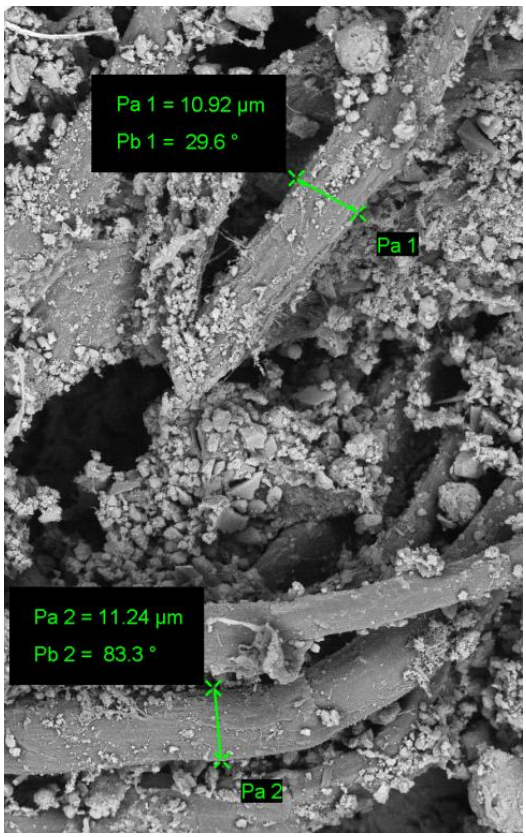

Fig. 2 Diameter of the flax fibres

2.2 Preparation of geopolymer composites

To prepare the geopolymer composites, an alkaline solution to fly-ash ratio of 0.75 was used and the ratio of sodium silicate solution to sodium hydroxide solution was fixed at 2.5. The concentration of sodium hydroxide solution was 8 M, which is prepared and combined with the sodium silicate solution 1 day before mixing.

The alkaline solution was added slowly to the fly-ash in a Hobart mixer at a low speed until the mix became homogeneous, then mixed for another 10 min on high speed with additional 50 ml of water to improve the workability. This procedure produced a geopolymer matrix of molar composition SiO_2/Al_2O_3= 4.16 , Na_2O/SiO_2=0.37 and H_2O/Na_2O= 11.43.

Three samples of geopolymer composites reinforced with 2.4, 3 and 4.1 wt.% FF were prepared. Composite specimens were prepared by spreading a thin layer of geopolymer paste in a well-greased wooden moulds followed by carefully laying the first layer of FF on that layer. Then, the fabric was fully saturated with geopolymer paste by a roller with the process repeated for the desired number of FF layers. Each specimen contained different layers of FF (see Table 3). For each specimen, the final layer was geopolymer paste. The wooden moulds were then placed on a vibration table for 2 min before they were covered with a plastic film and cured at 80◦ C for 24 h in an oven before demolding. They were then dried under ambient conditions for 28 days.

3. Characterization

3.1 Mechanical properties

Rectangular bars of 60×18×15 mm^3 were cut from the fully cured samples for three-point bend tests with a span of 40mm to evaluate the flexural strength. A LLOYD Material Testing Machine (50kN capacity) with a displacement rate of 0.5 mm/min was used to perform the test. Five samples of each group were used to evaluate the flexural strength, flexural modulus and fracture toughness of geopolymer composites. The values were recorded and analyzed with the machine software (NEXYGENPlus) and average values were calculated. The flexural strength (σ_F) was determined using the equation:

$$\sigma_F = \frac{3 p_m S}{2W D^2} \tag{1}$$

where P_m is the maximum load at crack extension, S is the span of the sample, D is the specimen width and W is the specimen thickness.

Flexural modulus (E_f) values were computed using the initial slope of the load displacement curve, $\Delta P/\Delta X$, using as follows (Low *et al*. 2007):

$$E_F = \frac{S^3}{4W D^3}\left(\frac{\Delta P}{\Delta X}\right) \tag{2}$$

A crack with a length to width (a/W) ratio of 0.4 was introduced into the specimen using a 0.4 mm diamond blade to evaluate fracture toughness. The fracture toughness (K_{IC}) was calculated using the equation (Low *et al*. 2007):

$$K_{IC} = \frac{p_m S}{W D^{2/3}} \cdot f\left(\frac{a}{W}\right) \tag{3}$$

where a is the crack length, and $f(a/W)$ is the polynomial geometrical correction factor given by (Low *et al*. 2007):

$$f\left(\frac{a}{W}\right) = \frac{3(a/W)^{1/2}\left[1.99 - \left(\frac{a}{W}\right)\left(1-\frac{a}{W}\right)\times\left(2.15 - 3.93\frac{a}{W} + 2.7\frac{a}{W}\right)^2\right]}{2\left(1+2\frac{a}{W}\right)\left(1-\frac{a}{W}\right)^{2/3}} \tag{4}$$

3.2 Scanning electron microscopy (SEM)

The microstructures of geopolymer composites were examined using a FIBSEM. The specimens were mounted on aluminium stubs using carbon tape and then coated with a thin layer of platinum to prevent charging before the observation.

4. Results and discussion

4.1 Flexural strength and modulus

Generally, the flexural tests are often used to characterize the mechanical properties of layered materials since they provide a simple means of determining the bending response. This provides useful information on the performance of layered fabric-based composites (Abanilla *et al*. 2006). The effect of FF content on the flexural strength of the geopolymer composites is presented in Fig. 3. The composite containing 4.1 wt% FF showed the highest flexural strength among all composites. The flexural strength of the composites improved from 4.5 MPa to about 23 MPa compared to pure geopolymer. This implies that increasing numbers of FF layers lead to significant improvement in flexural strength in the composite. This result can be justified from the fact that the number of reinforcement layers controls the flexural strength. The lower weight of FF permits multiple layers of fabric in the composite, to resist the shear failure and contribute in supporting the applied load to the composites. This allows greater stress transfer between the matrix and the FF, leading to higher flexural strength of the composite (Sim *et al*. 2006).

The flexural modulus of geopolymer composites is shown in Fig. 4 and indicates similar trends to flexural strength values. In fact, the addition of FF in the geopolymer matrix improves the flexural modulus over pure geopolymer matrix. The flexural modulus is a measure of resistance to deformation of the composite in bending. It was observed that none of the reinforced specimens are completely broken at peak load. This could be due to the crack bridging by long continuous flex fibres, which makes their flexural modulus higher than the pure geopolymer. Such fibres are able to withstand a higher load and are capable of undergoing multiple cracks throughout the loading process, consequently preventing brittle failure of the geopolymer. Similar results have also been reported by Low *et al*. (2007) and Alamri and Low (2012) in the case of cellulose fibre-reinforced epoxy laminates. They reported an increase in both flexural strength and modulus as the fibre contents increase.

The increase in FF content was remarkably useful in terms of improving the mechanical properties of geopolymer matrix.

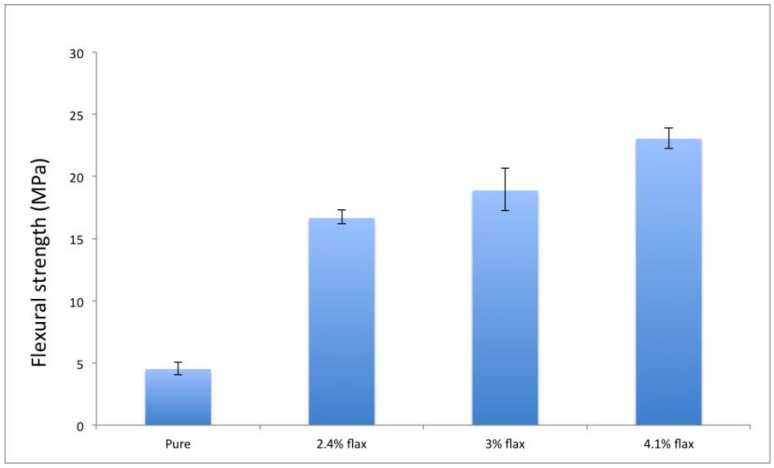

Fig. 3 Flexural strength of geopolymer composites as a function of fabric content

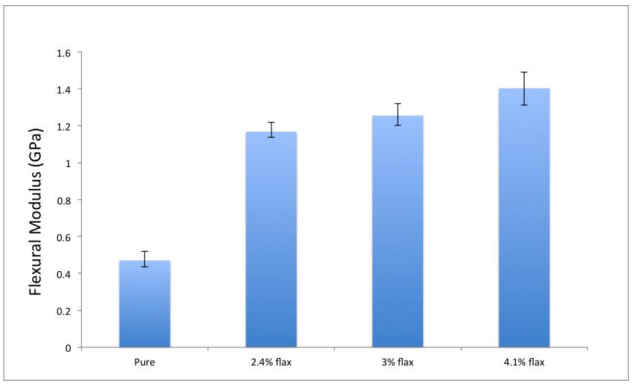

Fig. 4 Flexural modulus of geopolymer composites as a function of fabric content

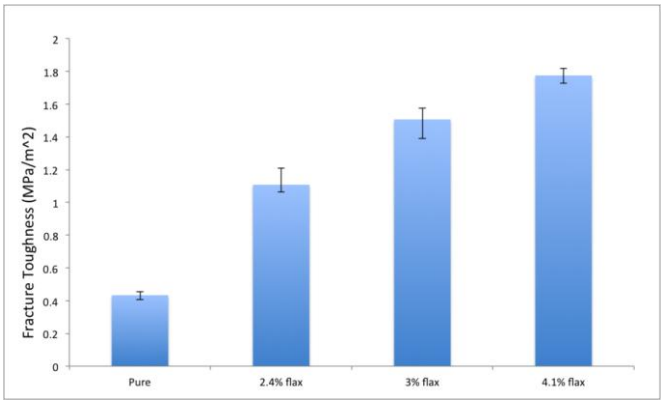

Fig. 5 Fracture toughness of geopolymer composites as a function of fabric content

4.2 Fracture toughness

Generally, crack deflection, debonding and bridging of cracks by fibres slows down the crack propagation in fibre reinforced composites and increase the fracture energy (Reis 2006, Silva *et al*. 2009, Silva *et al*. 2010, Toledo *et al*. 2003). The close spacing of woven FF did not allow the geopolymer composites specimens to completely break into two pieces, which lead the fibres to bridged the cracks and enhanced the crack propagation resistance. This significant enhancement of facture toughness is due to fibre fracture, fibre-bridging and fibre pull-out as clearly shown in the SEM images of Figs. 6B–E.

The influence of FF content on the fracture toughness of the composites is shown in Fig. 5. All composites containing FF showed significantly higher fracture toughness than pure geopolymer matrix and the higher the FF content the higher fracture toughness. The greatest improvement in fracture toughness (from about 0.4 MPa.m^{½} in the pure matrix to about 1.8 MPa.m$^{1/2}$) was obtained with 4.1 wt% FF reinforcement. This enhancement is due to the remarkable properties of FF to resist fracture resulted in increased energy dissipation from crack-deflection at fibre–matrix interface, fibre-debonding, fibre-bridging, fibre pull-out and fibre-fracture. The high values of

fracture toughness obtained in geopolymer composites with FF were due to better interaction between the fibre and the matrix as shown in Fig. 6. The improved interfacial adhesion enabled higher stress transfer between the fibres and the matrix and reduced the chance of fibres de-bonding. Consequently, the load required to break the sample increased as the content of FF is increased. Therefore, the fracture toughness of geopolymer composites increases with increase in wt% of FF.

Fig. 6 shows the scanning electron micrographs of fracture surface of geopolymer composite and describes the fracture toughness behavior. Fig. 6A presents the fracture surface of the pure geopolymer specimen, and unreacted fly-ash particles can be seen on the fracture surface.

The effect of FF content on the fracture surface is clearly seen in Figs. 6 B- D with 2.4, 3, 4.1wt% of FF content, respectively. The lower fibre content means the higher geopolymer regions. This indicates that the composites with richer matrix content are not reinforced by enough fibres. Therefore, there are insufficient fibres to transfer the load from the matrix (Joseph *et al*. 1999). Due to this reason, the composites with higher fibre content showed better fracture toughness and mechanical properties. An increase in fibre regions means greater stress-transfer from the matrix to the FF, thereby improvement of mechanical properties.

Figs. 6 D- F show that small pieces of geopolymer paste were attached to the fibre surface of flax fibres. Hence, retention of the matrix on the fibre surfaces demonstrates the good adhesion between flax fibres and geopolymer matrix. The penetration of geopolymer paste into flax bundle and fibre pull out can be clearly seen in Figs. 6 (G)-(H), respectively.

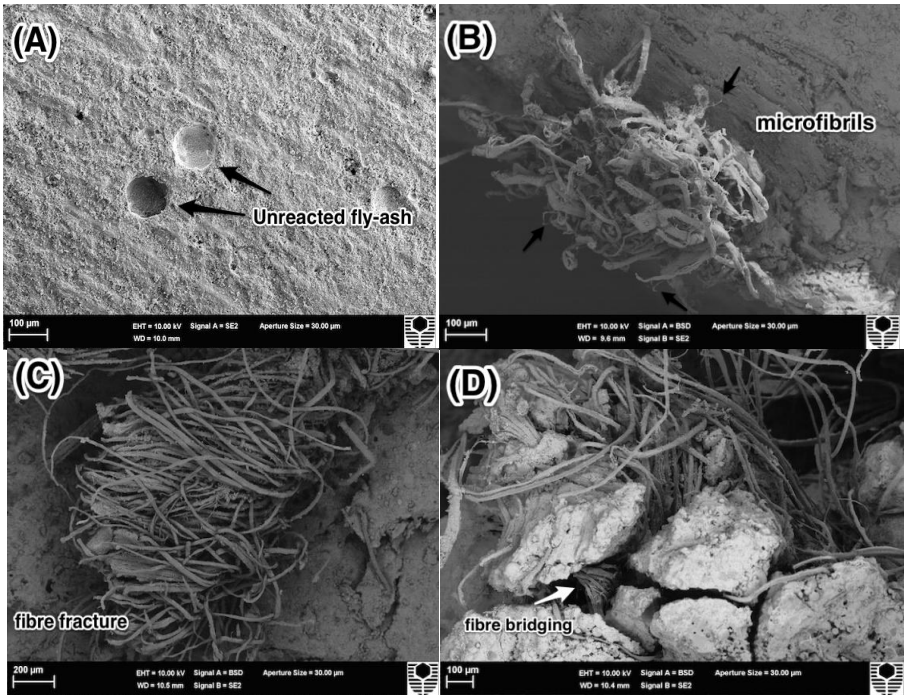

Fig. 6 SEM images of the fracture surface for geopolymer composites reinforced with varying content of flax fibres (A) pure, (B) 2.4 (C) 3 and (D-H) 4.1 wt%. The micrographs of (E and F) the adhesion between fibre and matrix, (G) penetration of the geopolymer matrix into flax fabrics, fibre bridging (D), and (H) fibre pull out

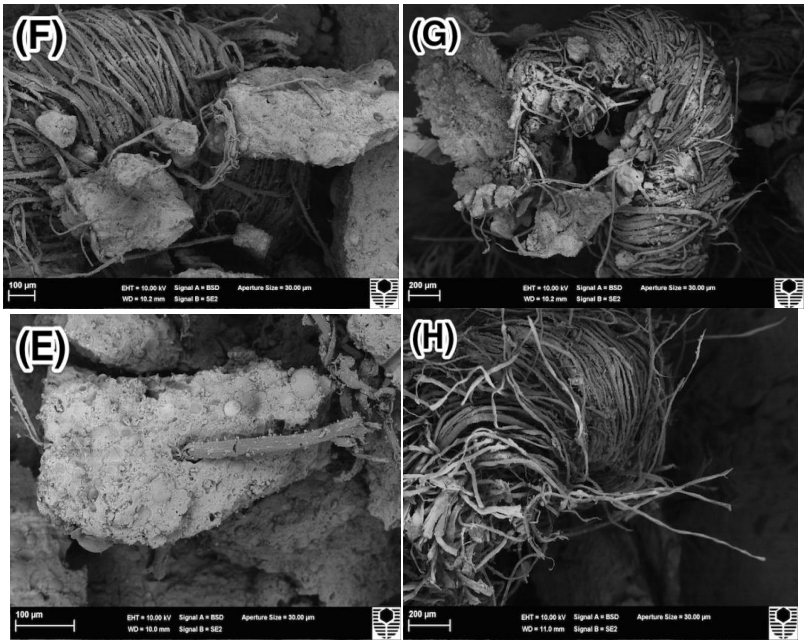

Fig. 6 Continued

5. Conclusion

This paper presents the mechanical properties and microstructural characterization of FF-reinforced geopolymer composite. It has been shown that the presence of FF fabrics in the geopolymer composites remarkably increased the flexural strength, flexural modulus and fracture toughness compared to neat geopolymer. This significant enhancement is due to the unique properties of flax fibres in resisting greater bending and fracture forces than the more brittle geopolymer. SEM micrographs show a number of toughening mechanisms that include crack bridging, fibre pull-out and fibre fracture. These toughening mechanisms are the major factors contributing to the enhanced mechanical properties of FF-reinforced geopolymer composites.

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