

# Characterisation of mechanical and thermal properties in flax fabric reinforced geopolymer composites

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**Abstract:** This paper presents the mechanical and thermal properties of flax fabric reinforced fly ash based geopolymer composites. Geopolymer composites reinforced with 2.4, 3.0 and 4.1 wt% woven flax fabric in various layers were fabricated using a hand lay-up technique and tested for mechanical properties such as flexural strength, flexural modulus, compressive strength, hardness, and fracture toughness. All mechanical properties were improved by increasing the flax fibre contents, and showed superior mechanical properties over a pure geopolymer matrix. Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) studies were carried out to evaluate the composition and fracture surfaces of geopolymer and geopolymer/flax composites. The thermal behaviour of composites was studied by thermogravimetric analysis (TGA) and the results showed significant degradation of flax fibres at 300 °C.

**Keywords:** geopolymer composites; flax fibre; mechanical properties; thermal properties

## 1 Introduction

Ordinary Portland cements are used in many construction applications because of their good mechanical and durability properties. However, the greenhouse emissions caused by cement based materials have made it necessary to find an eco-friendly alternative. A new group of promising construction material is geopolymer, first introduced and named by Davidovits in 1989, exhibiting good mechanical performance, durability, and fire and acid resistance. It can be cured and hardened at room temperature with 80%–90% fewer CO<sub>2</sub> emissions than Portland

cement [1–5].

Despite their desirable characteristics, geopolymer matrices suffer from brittle failure under applied force and demonstrate low flexural strength ranging between 1.7 and 16.8 MPa [6,7]. Improving their flexural and tensile strengths will significantly increase the application of these materials in the construction and building industries; and this may be accomplished by dispersing inorganic or organic fibres throughout the matrices. Hitherto, the most common fibre reinforcements used in geopolymer composites are based on carbon, basalt, and glass fibres [8–12], but concerns over the environment and non-biodegradability have made renewed interest recently in replacing the synthetic fibres used in geopolymer or other brittle matrices with natural plant

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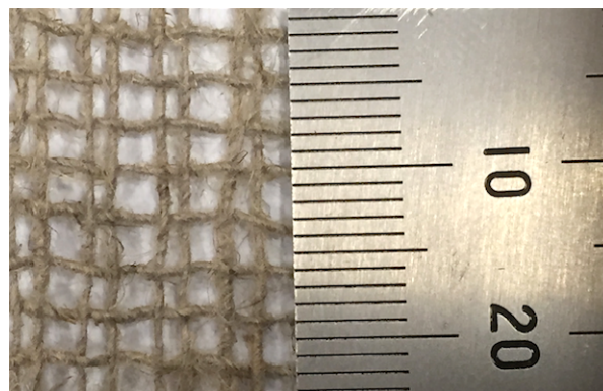
fibres. These include flax, hemp, jute, pineapple, straw, switch grass, kenaf, coir, and bamboo [13,14]. These plant fibres cost less, have low density, and display good mechanical properties when compared with industrial fibres [15]. For example, natural fibres have lower densities than synthetic fibres generally, with many almost 30%–50% less dense than their synthetic counterparts [16]. They are also renewable, recyclable, and biodegradable, and demonstrate excellent mechanical characteristics like flexibility, high specific strength, and high specific modulus [17,18]. For example, wood-derived cellulose can be used for toughening epoxy and other polymers [19–22], and bamboo fibres improve the flexural strength of concrete [23]; the same desirable effect has been observed in wood fibre reinforced concrete [24]. Cotton fabrics also enhance the strength and toughness of geopolymers [25], and wool and flax fibres have been successfully used to reinforce geopolymer composites, with improvements in mechanical and fracture properties [26,27]. However, no study so far has reported the mechanical properties of flax fabric (FF) reinforced fly ash based geopolymer composites despite their advantages of cheapness, ready availability, lack of toxicity, biodegradability, and good tensile strength. The present report describes the development and mechanical properties of new environmentally friendly geopolymer composites reinforced with the readily-available natural flax fibres of Australia, to produce materials with excellent flexural strength and graceful failure properties.

This study considers the viability of developing a green composite material that uses fly ash geopolymer as the matrix and FF as the reinforcement. Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA) are used to investigate the morphology, microstructure, failure mechanisms, and thermal behaviour of geopolymer/flax composites. The effect of different FF contents of 2.4, 3.0, and 4.1 wt% on mechanical properties of the composites such as flexural strength, flexural modulus, compressive strength, hardness, and fracture toughness is also presented in this paper.

## 2 Experimental procedures

### 2.1 Materials

Flax fabric shown in Fig. 1, supplied by Pure Linen



**Fig. 1** Structure of the flax fabric.

Australia, was used as reinforcement in the fabrication of geopolymer composites. The structure and physical properties of the flax fabric are shown in Table 1. Low calcium fly ash (ASTM class F) collected from the Eraring power station in New South Wales, Australia, was used as the source material for 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 and sodium silicate grade D solution. Sodium hydroxide flakes of 98% purity were used to prepare the solution. The chemical composition of sodium silicate used was 14.7% Na<sub>2</sub>O, 29.4% SiO<sub>2</sub>, and 55.9% water by mass.

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, and was prepared and combined with the sodium silicate solution one day before mixing.

The alkaline solution was added to the fly ash in a Hobart mixer at low speed until the mix became homogeneous, then mixed for another 10 min on high speed with an additional 50 mL of water to improve the workability. This produced a geopolymer matrix of molar composition of SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> = 4.16, Na<sub>2</sub>O/SiO<sub>2</sub> = 0.37, and H<sub>2</sub>O/Na<sub>2</sub>O = 11.43.

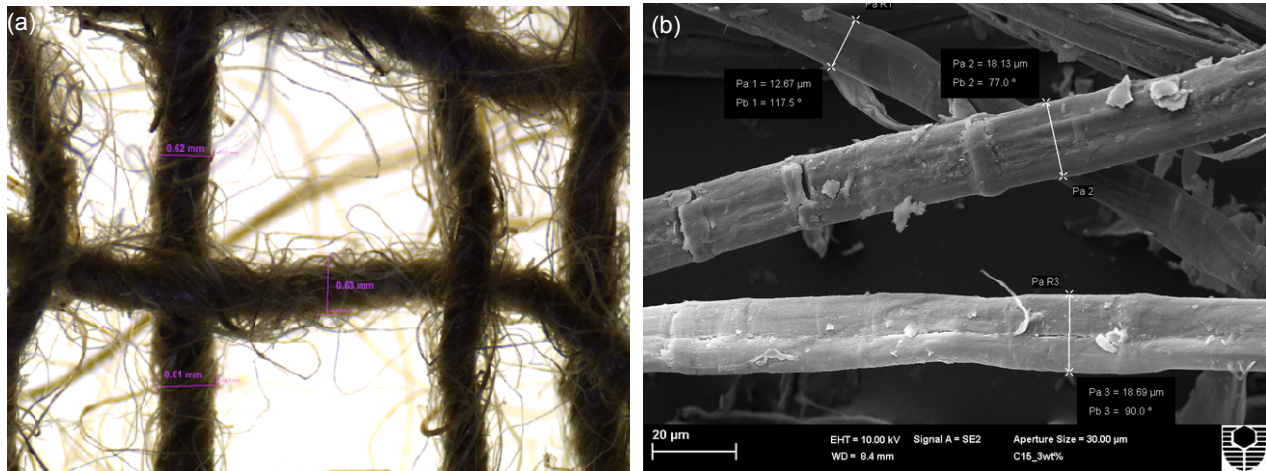
**Table 1** Structure and physical properties of the flax fabric

Fabric thickness (mm)	0.6
Fabric geometry	Woven (plain weave)
Yarn nature	Bundle
Bundle diameter (mm)	0.6 (see Fig. 2(a))
Filament size (mm)	0.01–0.02 (see Fig. 2(b))
Opening size (mm)	2–4
Fabric density (g/cm <sup>3</sup> )	1.5
Modulus of elasticity (GPa)	39.5
Tensile strength (MPa)	660

**Table 2 Chemical composition of fly ash**

(Unit: wt%)

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	MgO	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	TiO <sub>2</sub>	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. 2** Diameters of the (a) flax bundle and (b) flax fibres.

Three samples of geopolymer composites reinforced with 2.4, 3.0, and 4.1 wt% FF were prepared by spreading a thin layer of geopolymer paste in a well greased wooden mould and carefully laying the first layer of FF on top. The fabric was fully saturated with paste by a roller, and the process repeated for the desired number of layers; each specimen contained a different number of 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, then covered with plastic film and cured at 80 °C for 24 h in an oven before demoulding. They were then dried under ambient conditions for 28 days. This procedure of preparing geopolymer composites is reported by Alomayri *et al.* [25].

**2. 2 Mechanical properties**

A LLOYD material testing machine (50 kN capacity) with a displacement rate of 0.5 mm/min was used to perform the mechanical tests. Rectangular bars of 60 mm × 18 mm × 15 mm with a span of 40 mm were cut from the fully cured samples for three-point bend tests to evaluate the mechanical properties. All samples

**Table 3 Formulation of samples**

Sample	Fly ash (g)	NaOH solution (g)	Na <sub>2</sub> SiO <sub>3</sub> solution (g)	Fabric layers	FF content (wt%)
1	1000	214.5	535.5	0	0
2	1000	214.5	535.5	5	2.4
3	1000	214.5	535.5	7	3.0
4	1000	214.5	535.5	10	4.1

were aligned horizontally to the applied load in all mechanical tests. Ten samples of each composite were used to evaluate the flexural strength and the flexural modulus according to the standard ASTM D790 [28]. The values were recorded and analysed with the machine software (NEXYGENPlus) and average values were calculated. The flexural strength ( $\sigma_F$ ) was determined using the equation:

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

where  $p_m$  is the maximum load;  $S$  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 the equation [29]:

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

A crack with a length to width ratio ( $a/W$ ) 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 [29]:

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

where  $a$  is the crack length, and  $f(a/W)$  is the polynomial geometrical correction factor given by [29]:

$$f\left(\frac{a}{W}\right) = 3(a/W)^{1/2} [1.99 - (a/W)(1 - a/W) \times$$

$$\frac{(2.15 - 3.93a/W + 2.7a^2/W^2)}{[2(1 + 2a/W)(1 - a/W)^{2/3}]}$$
(4)

The compressive strength of the geopolymer composites was tested according to ASTM C109 [30], but instead of using the recommended 50 mm cube specimens, 20 mm cubes were used. The compressive strength (*C*) of the sample was calculated using the following formula:

$$C = P / A$$
(5)

where *P* is maximum load on the sample at failure and *A* is the surface area of the specimen.

The hardness of geopolymer composites was measured on the Rockwell H scale using an Avery Rockwell hardness tester. Before measurement, five samples were polished with emery paper to achieve flat, smooth surfaces.

### 2.3 Characterisation

An FTIR spectrum was performed on a Perkin Elmer Spectrum 100 FTIR spectrometer in the range of 4000–500 cm<sup>-1</sup> at room temperature. The spectrum was an average of 10 scans at a resolution of 2 cm<sup>-1</sup>, corrected for background.

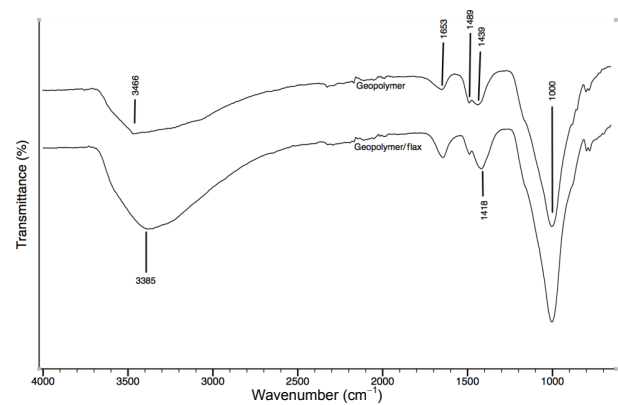
The microstructures of geopolymer composites were examined using a Zeiss Neon focused ion beam scanning electron microscope (FIB–SEM). The specimens were mounted on aluminium stubs using carbon tape and then coated with a thin layer of platinum to prevent charging before the observation.

A thermogravimetric analyser (TGA) was used to examine the thermal behaviours of the composites. Solid samples of 25 mg were placed in an alumina crucible and tests were carried out in an argon atmosphere with a heating rate of 10 °C/min from 25 to 800 °C.

## 3 Results and discussion

### 3.1 FTIR observation

FTIR spectra of both pure geopolymer and flax/geopolymer composite are shown in Fig. 3. The strong peak at ~1000 cm<sup>-1</sup> is associated with Al–O and Si–O asymmetric stretching vibrations and is the fingerprint of the geopolymerisation [31]. The FTIR spectra show a broad peak in the region at 3466 cm<sup>-1</sup> corresponding to the hydroxyl (OH) stretching vibration of free and hydrogen bonded –OH groups [32,33], and

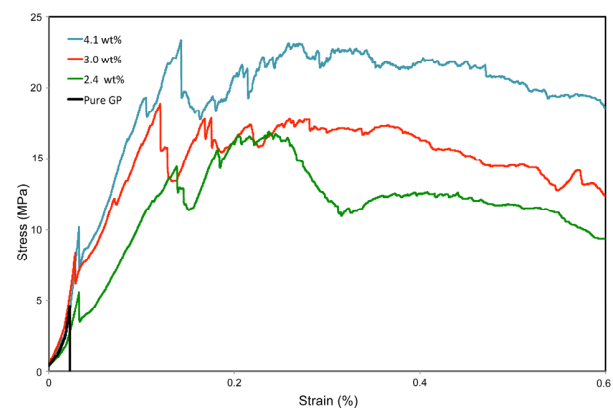


**Fig. 3** FTIR spectra of pure geopolymer and flax/geopolymer composite.

the absorbance peak around 1653 cm<sup>-1</sup> is attributed to the bending vibration of absorbed water [34,35]. The presence of bands in the regions 1440–1490 cm<sup>-1</sup> is an indicator of the atmospheric carbonation on the surface of the matrix where it reacts with carbon dioxide [34]. The presence of flax fibres in the composites can be recognised by the peak at 1418 cm<sup>-1</sup>, which is attributed to the CH<sub>3</sub> bending vibration of cellulose [32]. The intensity of the bands at 3385 and 1653 cm<sup>-1</sup> increases in response to the existence of absorbed water in the cellulose fibres.

### 3.2 Flexural strength and modulus

Generally, flexural tests are used to characterise the mechanical properties of layered composites as they provide a simple means of determining the bending response. This provides useful information on the performance of layered fabric based composites [36]. The effect of FF contents on the flexural stress–strain curves of the geopolymer composites is presented in Fig. 4.



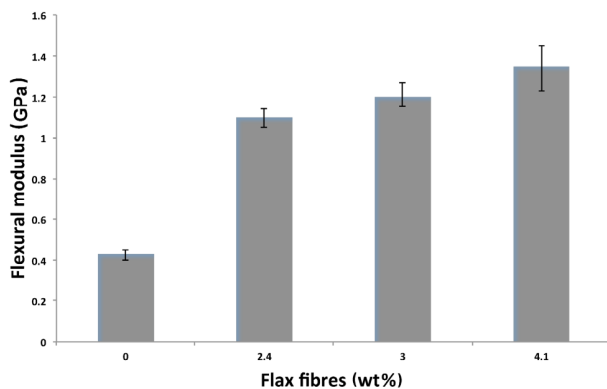
**Fig. 4** Typical stress–strain curves of pure geopolymer and geopolymer composites with various FF contents.

It can be seen that, the composite containing 4.1 wt% FF shows the highest flexural strength among all composites. The flexural strength of the composites improves from 4.5 MPa in the pure geopolymer to about 23 MPa with 4.1 wt% FF. This result is comparable with that of short flax fibre reinforced geopolymer composites reported by Alzeer and MacKenzie [27]. Both studies show that increasing the content of flax fibres leads to a significant improvement in the flexural strength of the composite. This can be explained by the fact that the number of reinforcement layers controls the flexural strength. The lower weight of flax fabrics allows multiple layers of fabric in the composite to resist the shear failure and contribute in sustaining the applied load to the composites. This permits greater stress transfer between the matrix and the flax fibres, resulting in improved flexural strength [37].

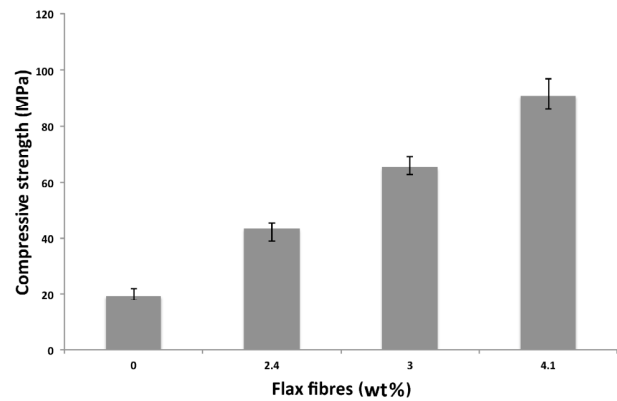
The flexural modulus of geopolymer composites, shown in Fig. 5, also indicates that the addition of FF to the matrix improves the flexural modulus over that of a pure geopolymer matrix. Flexural modulus is the measure of resistance to deformation of the composite in bending. It was observed that none of the reinforced specimens were completely broken at peak load. This could be attributed to crack bridging of the long continuous flax fibres under load, which makes the flexural modulus higher than that of pure geopolymer. Long fibres are able to withstand a higher load and are capable of supporting multiple cracks throughout the loading process, consequently preventing brittle failure of the geopolymer.

### 3.3 Compressive strength

The results presented in Fig. 6 show that the compressive strength of the composites containing FF



**Fig. 5** Flexural modulus of geopolymer composites as a function of fabric content.

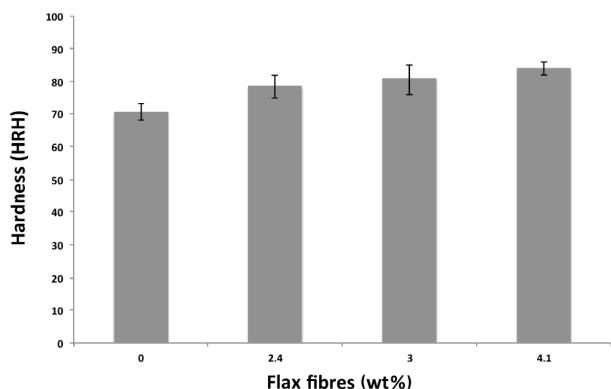


**Fig. 6** Compressive strength of geopolymer composites as a function of fabric content.

increases with increase in fibre contents. The increase in compressive strength with fibre loading may be due to the ability of the flax fibres to absorb stress transferred from the matrix. The compressive strength of the neat geopolymer paste increases from 19.4 to 91 MPa after the addition of 4.1 wt% flax fibres. This significant enhancement of compressive strength is due to the fact that the interface between the fabric and the matrix is not exposed to any shear loading, which in turn reduces the possibility of fabric detachments or delamination from the matrix at high loads. Similar remarkable improvements in compressive strength have also been reported by Alomayri *et al.* [38] in the case of cotton fibre reinforced geopolymer composites. They concluded that the increase is due to the ability of horizontally laid cotton fabric to directly absorb and distribute a load uniformly throughout the cross-section.

### 3.4 Hardness

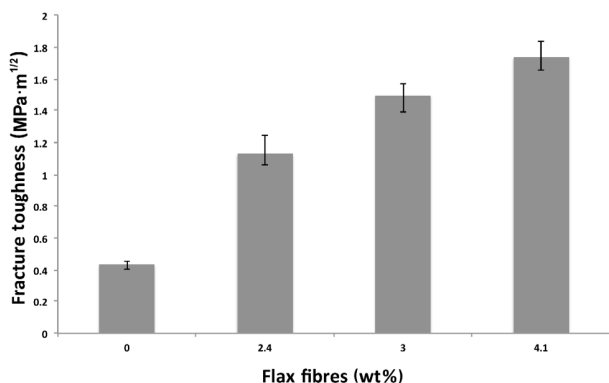
Hardness measurement enables the ability of a material to resist plastic deformation under indentation to be determined. The hardness values of FF reinforced geopolymer composites are shown in Fig. 7. The results show that the hardness of composites increases with the addition of high number of flax fabrics to the geopolymer composite. This enhancement in hardness is due to the uniform distribution of the load on the flax fibres, which reduces the penetration of the test ball at the surface of the composite. A similar increase has been reported by other researchers studying natural fibre reinforced geopolymer composites: for instance, Alomayri *et al.* [25] reported that with increasing cotton fibre content, the hardness value of cotton fibre reinforced geopolymer composites increases.



**Fig. 7** Hardness of geopolymer composites as a function of fabric content.

### 3.5 Fracture toughness

Generally, fibres’ ability to resist crack deflection, debonding, and to bridge cracks, slows down crack propagation in fibre reinforced composites and increases the fracture energy [39–42]. Figure 8 shows the influence of FF content on the fracture toughness of geopolymer composites. It can be seen that the composites containing FF show significantly higher fracture toughness than pure geopolymer matrix, and the higher the FF content, the higher is the fracture toughness. The greatest improvement in fracture toughness was obtained from about 0.4 MPa·m<sup>1/2</sup> in the pure matrix to about 1.8 MPa·m<sup>1/2</sup> with 4.1 wt% FF reinforcement. This extraordinary enhancement is due to the unique ability of flax fibre to resist fracture resulted in increased energy dissipation from crack-deflection at the fibre–matrix interface, fibre-debonding, fibre-bridging, fibre pull-out and fracture, clearly shown in the SEM images (see Figs. 9(a)–9(f)). It can be seen in these images that small pieces of geopolymer paste attached to the fibre surface of the composites: such retention of the matrix on the fibre surfaces shows good adhesion between fibres and



**Fig. 8** Fracture toughness of geopolymer composites as a function of fabric content.

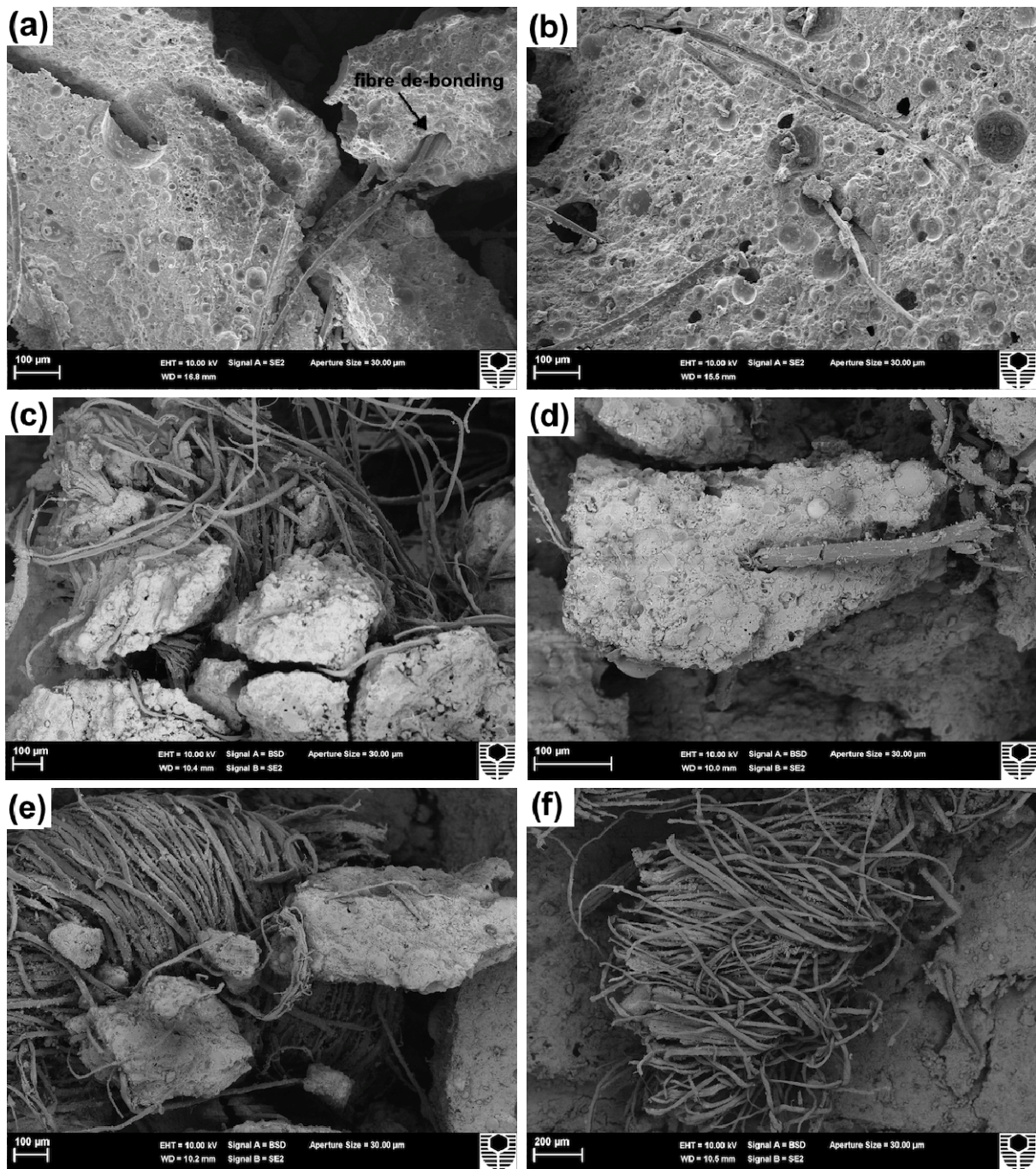
matrix. It was observed that the composites with fibres do not completely break into pieces, as the close spacing of woven FF leads to crack-bridging by fibres and enhancing the resistance to their propagation. The effect of fibre content on the fracture surface can be seen by observing the difference between the matrix region and the fibre region. In Figs. 10(a) and 10(b), composites filled with lower fibre contents (2.4 and 3.0 wt%) show an increase in matrix-rich regions, which means there are insufficient fibres to transfer the load from the matrix. Due to this reason, the geopolymer composites with low fibre content exhibit low fracture toughness and mechanical properties. However, Fig. 10(c) illustrates the fracture surfaces of the geopolymer composites with higher fibre content, which means higher fibre-rich regions of composites with 4.1 wt% of FF. An increase in fibre-rich regions leads to greater stress-transfer from the matrix to the FF thereby resulting in improvement of fracture toughness.

### 3.6 Thermal stability

The thermal stability of samples was determined using thermogravimetric analysis (TGA). In this test, thermal stability was studied in terms of the weight loss percentage as a function of temperature in argon atmosphere. The thermograms (TGA) of FF, neat geopolymer, and FF reinforced geopolymer composite are shown in Fig. 11.

The thermogram of flax fibres shows degradation in three steps. The first transition occurs from 25 to approximately 240 °C, with the release of free water evaporation. Then, the largest weight loss occurred between 240 and 365 °C is due to the decomposition of cellulose. This result is in agreement with Alzeer and MacKenzie [27], where the highest weight loss of short flax fibres under flowing air is in the range of 240–340 °C. The final stage occurs above 365 °C, when the fibres start to decompose but display a lower rate of weight loss, and all volatile substances are dispelled.

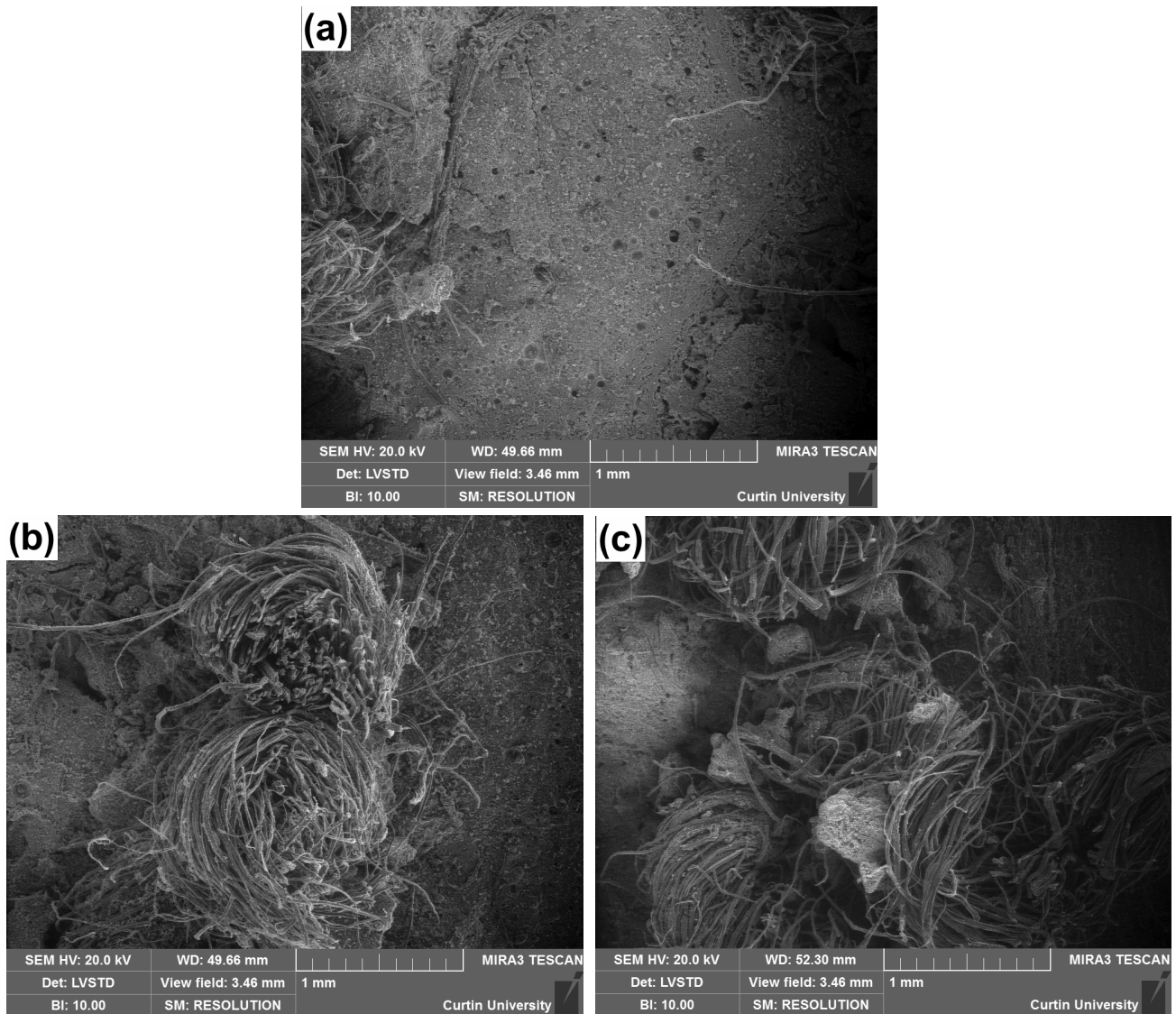
The pure geopolymer shows weight loss occurring from 25 to 300 °C, caused by the evaporation of physically adsorbed water. Above 300 °C, weight loss is attributed to the dehydroxylation of the chemically bound water. The FF reinforced geopolymer composite shows a weight loss of 10.5% up to about 260 °C, which is due to the evaporation of physically absorbed water. Above 260 °C, the composite shows further weight loss because of the degradation of the fibre content in the sample. The porosity of geopolymer matrix allows the



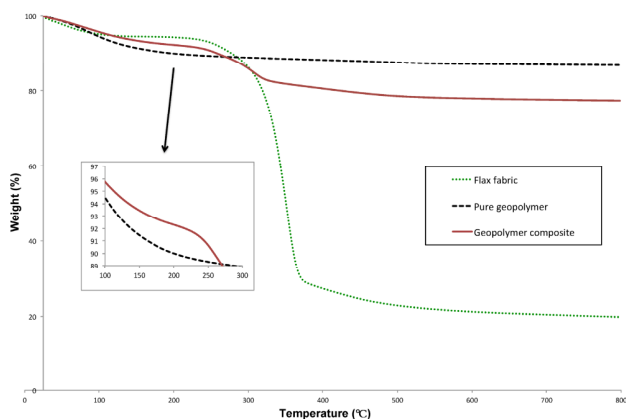
**Fig. 9** SEM images of the fracture surface for geopolymer composites reinforced with flax fibres show (a) fibre debonding, (b) fibre imprint and pull-out, (c) fibre bridging cracks ((d) and (e) show the adhesion between fibre and matrix), and (f) fibre fracture.

oxygen to enter and cause degradation of the flax fibres at high temperatures. The composite shows a total weight loss of  $\sim 15\%$  at  $300\text{ }^{\circ}\text{C}$  which indicates further degradation of fibres inside the composite. At this temperature a substantial amount of fibre degradation has occurred. Therefore, it could be concluded that this composite system is only suitable for service below  $250\text{ }^{\circ}\text{C}$ . It is worth mentioning here that the TGA micro-sample is not necessarily representative of the

whole composite sample because the distribution of flax fibres is not uniform within the geopolymer matrix. Therefore, the fibre content of the TGA micro-sample will be highly dependent on the position it is taken from the composite sample. However, TGA test can provide a good estimation of the thermal stability of a composite when compared to the thermal stability of its components.



**Fig. 10** Low magnification SEM images of the fracture surface for geopolymer composites reinforced with (a) 2.4, (b) 3.0, and (c) 4.1 wt% of flax fibres.



**Fig. 11** TGA curves of the flax reinforced geopolymer composite, the matrix, and the flax fibres.

#### 4 Conclusions

This paper presents the mechanical and thermal properties and microstructural characterisation of FF reinforced geopolymer composites. It shows that the presence of FF in geopolymer composites remarkably increases flexural and compressive strength, hardness, and fracture toughness compared to neat geopolymer. These significant enhancements are 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 are the major factors contributing to the



enhanced mechanical properties of FF reinforced geopolymer composites. Thermogravimetric analysis of the samples indicates that the FF reinforced geopolymer exhibits higher net weight loss than pure geopolymer due to the degradation of flax fibres.

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