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Synthesis and characterization of mechanical properties in cotton fiber-reinforced geopolymer composites

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ABSTRACT

Geopolymers are inorganic aluminosilicate materials that possess relatively good mechanical properties and desirable thermal stability but they exhibit failure behavior similar to brittle solids. This limitation may be remedied by fiber reinforcement to improve their strength and toughness. This paper describes the synthesis of cotton fiber-reinforced geopolymer composites and the characterization of their mechanical properties. The effects of cotton fiber content (0–1.0 wt.%) and fiber dispersion on the mechanical characteristics of geopolymer composites have been investigated in terms of hardness, impact strength and compressive strength. A fiber content of 0.5 wt.% was observed for achieving optimum mechanical properties in these composites.

1. Introduction

Inorganic aluminosilicate Portland cements are used in many building and construction applications because of their good mechanical performance. However, the emission of greenhouse gases associated with their manufacture is a serious problem. In recent years, a new class of environment-friendly and sustainable inorganic aluminosilicate polymers (also known as geopolymers) have emerged as an alternative to cements. These inorganic compounds can be cured and hardened at near-ambient temperatures to form materials that are effectively low-temperature ceramics with the typical temperature resistance and strength of ceramics [1,2]. However, despite their many desirable attributes such as relatively high strength, elastic modulus and low shrinkage, geopolymers suffer from brittle failure like most ceramics. This limitation may be readily overcome through fiber reinforcement as in high performance polymer-matrix composites. As in thermosetting polymers, the low synthesis temperatures of geopolymers renders them particularly suitable as matrices for a range of fibers including organic fibers, with setting times and mechanical properties comparable to Portland cement [3]. Hitherto, the most common fiber reinforcement used in geopolymer composites is based on carbon, basalt and glass fibers, but other inorganic fibers such as silicon carbide, alumina, mullite or boron can be utilized [4–6]. Maximum flexural strengths of >500 MPa have been reported by several authors for unidirectional carbon fiber-reinforced geopolymer composites [4,7] and desirable non-brittle fracture was observed when short carbon fibers were used [8].

Current concerns over the environment and climate change have also given rise to an increasing interest in replacing the synthetic fibers currently used in geopolymer composites or other brittle matrices with natural plant fibers. Plant fibers cost less, have low density and display good mechanical properties when compared with industrial fibers [9–11]. Investigations on natural fibers such as bamboo, sisal, jute and cellulose have revealed desirable effects on the mechanical and physical properties of brittle organic and inorganic matrices. For instance, the mechanical and fracture properties of epoxy resin have been significantly improved as a result of cellulose fiber reinforcement [12–14]. Similarly, Rahman et al. [15] found that bamboo fibers are effective in improving the flexural strength of concrete, and Lin et al. [16] observed a similar desirable effect in wood fiber-reinforced concrete. In another study, Li et al. [17] found that hemp fibers enhanced the toughness of concrete. Wool fibers have also been successfully used in reinforcing geopolymer composites with concomitant improvements in mechanical and fracture properties [18]. However, the use of cotton fibers as reinforcement for geopolymers has not been investigated. The use of cotton fibers has several advantages, which

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include low cost, renewable, and low weight when compared with synthetic fibers.

In this paper, we have synthesized geopolymer composites reinforced with short cotton fibers and characterized their mechanical properties in terms of hardness, compressive strength and impact strength. The effect of fiber contents (0.3, 0.5, 0.7 and 1 wt.%) and their dispersion on mechanical properties were investigated. Scanning electron microscopy (SEM) was used to examine the microstructures of fly-ash and the resultant composites.

2. Experimental procedure

2.1. Materials

Low calcium fly-ash (ASTM class F) [19] collected from the Collie power station in Western Australia was used as the source material to prepare the geopolymer composites. The chemical compositions of fly-ash are given in Table 1. Alkali resistant cotton fibers with an average length of 10 mm, average diameter of 0.2 mm, density of 1.54 g/cm^3, tensile strength of 400 MPa, and Young's modulus of 4.8 GPa were used to reinforce the geopolymer composites. The alkaline activator for geopolymerization 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 is Na_2O 14.7%, SiO_2 29.4% and water 55.9% by mass.

2.2. Sample preparation

To prepare the geopolymer composites, an alkaline solution to fly-ash ratio of 0.35 was used and the ratio of sodium silicate solution to sodium hydroxide solution was fixed at 2.5. Four samples of geopolymer composites reinforced with 0.3, 0.5, 0.7 and 1 wt.% cotton fibers were prepared. Additional water was added to improve the workability and dispersion of cotton fibers in the composites.

An 8 M concentration of sodium hydroxide solution was prepared and it was combined with the sodium silicate solution 1 day before mixing. The fibers were added slowly to the dry fly-ash in a Hobart mixer at a low speed until the mix became homogeneous at which time the alkaline solution was added. This was mixed for 10 min on low speed and for another 10 min on high speed. The walls of the mixing container were scraped down to ensure consistency of the mix. This procedure was followed for all the four test specimens. The mix was cast in 25 rectangular silicon molds of 80 mm × 20 mm × 10 mm and placed on a vibration table for 5 min. The specimens were covered with a plastic film and cured at 105 °C for 3 h, then rested for 24 h before demolding. They were then dried under ambient conditions for 28 days.

2.3. Synchrotron radiation diffraction (SRD)

The Powder Diffraction beamline at the Australian Synchrotron was used to collect the diffraction patterns of fly-ash, cotton fibers and the geopolymer composites. The diffraction pattern of each sample was collected using an incident angle of 3° and wavelength of 0.11267 nm or photon energy of 11.0 keV over the 2θ range of 10°–40°.

Table 1
Chemical composition of fly-ash.

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	LOI
50%	28.25%	13.5%	1.78%	0.89%	0.38%	0.32%	0.46%	1.64%

LOI: Loss on Ignition.

2.4. Scanning electron microscopy (SEM)

A Zeiss Evo 40XVP scanning electron microscope was used to examine the microstructures of fly-ash and geopolymer composites. The specimens were mounted on aluminum stubs using carbon tape, and then coated with a thin layer of platinum to prevent charging before the observation.

2.5. Rockwell hardness

The hardness of geopolymer composites was measured using an Avery Rockwell hardness tester at hardness scale *H*. Before measurement, the surfaces of test samples were polished using a Struers Pedamat polisher finishing with 10-μm grade diamond paste.

2.6. Compressive strength

The measurement of compressive strength testing was conducted using the methodology of ASTM C39 for concrete specimens. Cylindrical samples with a 2:1 height to diameter ratio were cut with a precision diamond blade such that the ends were perpendicular to the sides. A minimum of five samples were tested. Following demolding, the samples were air dried for 1 day before the compressive test. An EZ50 (Lloyd Instruments Ltd., West Sussex, UK) was used to apply a constant stress rate of 0.25 MPa/s, after a 50 N preload, until failure.

The compressive strength (C) of a sample was calculated using the following formula:

$$C = \frac{P}{A} \tag{2}$$

where P is total load on the sample at failure and A is calculated area of the bearing surface of the specimen.

2.7. Impact strength

Rectangular bars with dimensions 80 mm × 20 mm × 10 mm were prepared for Zwick Charpy impact testing to evaluate the impact strength of geopolymer composites. A pendulum hammer with 1.0 J was used during the test to break the samples. Unnotched samples were used to compute the impact strength (σ_i) using the following formula:

$$\sigma_i = \frac{E}{A} \tag{1}$$

where E is the impact energy to break a sample with a ligament of area A.

3. Results and discussion

3.1. Synchrotron radiation diffraction

The synchrotron radiation diffraction (SRD) patterns of commercial fly-ash, cotton fibers and prepared geopolymer reinforced with 0.3, 0.5, 0.7 and 1.0 wt.% of cotton fibers are shown in Fig. 1. The diffraction pattern of cotton fibers shows typical characteristic peaks, indicating the presence of cellulose. Fly-ash displays peaks due to the presence of quartz and mullite as well as other crystalline phases. These crystalline phases are not involved in the geopolymerization reaction, but the amorphous phase generated by coal combustion is actively involved in geopolymerization reactions [20]. Rickard et al. [21] have recently shown that amorphous aluminosilicates in fly-ash are reactive during the formation of a geopolymer.

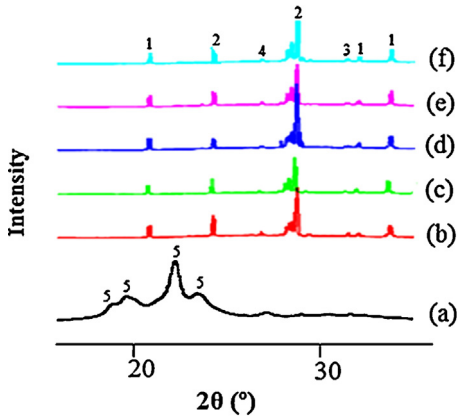

Fig. 1. Synchrotron radiation diffraction patterns of (a) cotton fibers (CF), (b) fly-ash, and geopolymer composite with (c) 0.3 wt.% CF, (d) 0.5 wt.% CF, (e) 0.7 wt.% CF, and (f) 1.0 wt.% CF. [Legend: 1 = mullite, 2 = quartz, 3 = maghemite, 4 = hematite, 5 = cellulose].

Comparing the SRD spectra of the original fly-ash with those of the hardened geopolymeric materials (see Fig. 2) indicates that the crystalline phases (quartz, mullite, etc.) originally existing in the fly-ash have apparently not been altered by the activation reactions; hence they do not participate in the geopolymerization reaction. The diffraction patterns of geopolymer reinforced with 0, 0.3, 0.5, 0.7 and 1 wt.% cotton fibers showed the sharp peaks of the crystalline phases from fly-ash, thus confirming that these phases are neither reactive nor involved in geopolymerization but are simply present as inactive fillers in the geopolymer network.

3.2. SEM observation

The SEM micrographs of fly-ash and geopolymer composites loaded with fiber content of 0.5 wt.% are shown in Figs. 2 and 3. Fig. 2 shows the microstructure of the original fly-ash before being activated with the alkaline activator. As seen in the figure, the fly-ash consists of spherical particles of different sizes. Some particles may contain smaller particles in their interior [22]. The surface texture of fly-ash particles appears to be smooth [23]. The surface of the fly-ash includes the existence of some quartz particles or some vitreous unshaped fragments [24].

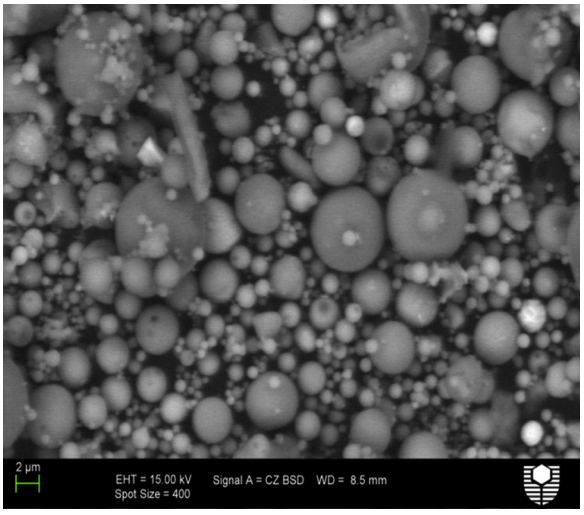

Fig. 2. SEM micrograph showing the typical microstructure of as-received fly-ash. SEM, Scanning electron microscopy.

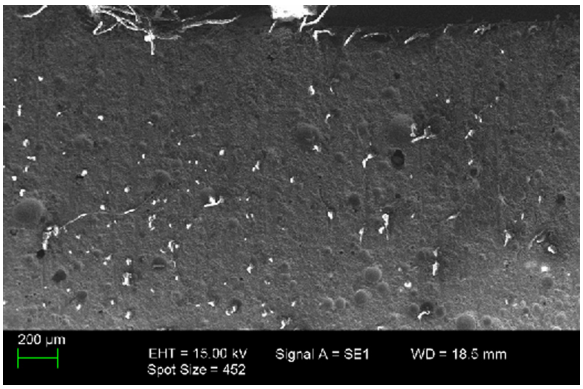

Fig. 3. SEM micrograph showing the typical microstructure of geopolymer composite reinforced with 0.5 wt.% cotton fibers. SEM, Scanning electron microscopy.

Fig. 3 shows that at 0.5 wt.% cotton fiber, the fibers are distributed homogeneously within the matrix. The uniformity of cotton fiber distribution in the matrix plays crucial roles in governing the properties of the composites. To gain advantageous properties, the following factor should be considered during fabrication of cotton fiber-reinforced geopolymer composites.

3.3. Hardness of geopolymer composites

The effect of cotton fiber content on the hardness of the cotton fiber-reinforced geopolymer composites is presented in Fig. 4. The hardness of geopolymer reinforced with 0.5 wt.% cotton fiber increased from 70 to 93 Rockwell hardness H (HRH) relative to the neat geopolymer. This significant enhancement in hardness is due to distribution of the test load on the fibers, which decreased the penetration of the test ball to the surface of the composite material and consequently raising the hardness of this material [25].

However, the hardness decreased with increasing fiber content due to the poor dispersion of cotton fibers in the slurry. The addition of 0.7 and 1.0 wt.% cotton fibers resulted in a reduction in the consistency of the matrix as well as low wettability between the fibers and the paste, and the fibers could be separated from the paste easily. This had to be compensated for by an increase in the water content of the mix. Increasing water content to overcome such a problem may lead to low hardness. The research conducted by Kunal [26] revealed that higher water content results in samples with low hardness. Because a higher than normal water content was needed for the samples to be flexible, the strength of the samples was reduced.

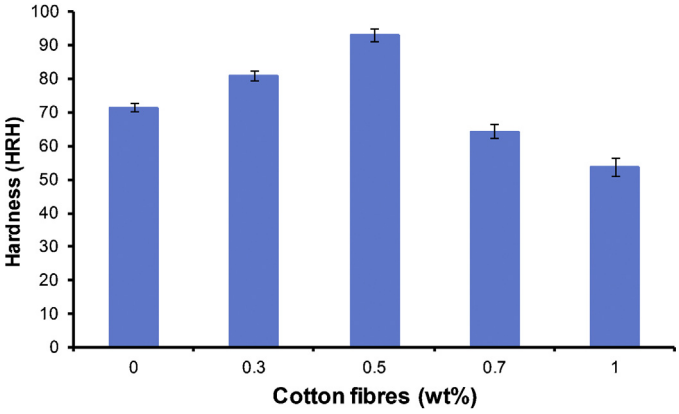

Fig. 4. Hardness of geopolymer composites as a function of fiber content.

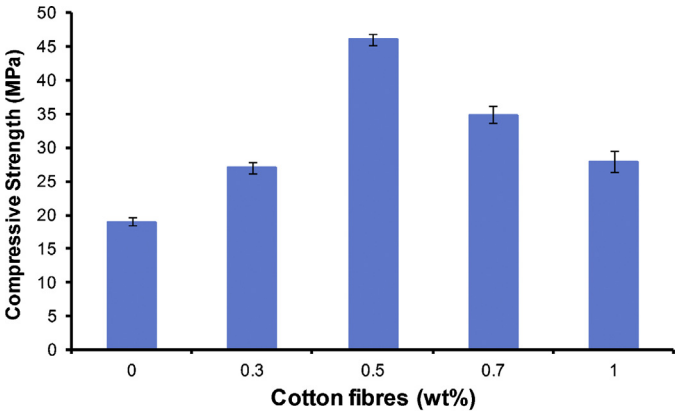

Fig. 5. Compressive strength of geopolymer composites as a function of fiber content.

Similarly, this decrease has been reported by other researchers when dealing with natural fiber based composites. Anup [27] reported that with increasing flax fiber content, the hardness value of high-density polyethylene/flax fiber composites and polypropylene/flax fiber composites decreased. Khairaih and Khairul [28] also reported decreasing hardness values with increasing fiber content when they worked on polyurethane and empty fruit bunch blend composites. They concluded that the decrease was due to the inability of the matrix to encapsulate the fiber strands.

3.4. Compressive strength of geopolymer composites

The 28-day average values of compressive strength of the composites are given in Fig. 5 and their corresponding stress/strain curves are shown in Fig. 6. It can be seen that geopolymer composite with 0.5% cotton fibers had the highest compressive strength. This is attributed to the possibility that the higher loads transferred from the matrix to the fibers, thus resulting in a higher load carried by the fibers. Another reason for such favorable behavior could be good dispersion of cotton fibers throughout the matrix that increases the bonding strength between the fiber and the matrix. From the stress–strain curves in Fig. 6, it is interesting to note that geopolymer composites displayed some non-linearity during fracture whereas a linear fracture behavior was observed for geopolymer. This implies the feasibility of using cotton fibers to mitigate the brittle failure in geopolymers.

However, the geopolymer composites cast with cotton fiber in the amount of about 0.7 and 1% fiber content by weight yielded a weak compressive strength. The reason for the reduction in compressive strength instead of an improvement with the addition of cotton fibers may be attributed to a greater probability of these fibers balling together and leaving voids in the matrix [29].

Other reasons for this weakness may be that the cotton fibers had absorbed too much water, denying the geopolymer around the fibers enough water for geopolymerization, which in turn decreased the bonding strength between the fiber and the matrix.

Similar results were reported by Li et al. [17], who investigated the compressive properties of hemp fiber-reinforced concrete. They found that compressive strength improves slightly when the fiber content by weight is lower than 0.6%, and continuously decreases when the fiber content is greater than this value.

In the present study, the compressive strength of the neat geopolymer paste increased from 19.1 to 46.0 MPa after the addition of 0.5 wt.% cotton fibers. However, adding more cotton fibers (0.7 and 1.0 wt %) led to a reduction in compressive strength.

3.5. Impact strength of geopolymer composites

The impact strength of fiber-reinforced polymer is governed by the matrix fiber interfacial bonding, and the properties of the matrix and the fibers. When the composites undergo a sudden force, the impact energy is dissipated by the combination of fiber pull out, fiber fracture and matrix deformation [30].

The effects of fiber content on the impact strength of cotton fiber-reinforced geopolymer composites are plotted in Fig. 7. It can be seen that the impact strength of the composites increases with an increase in cotton content of up to 0.5 wt.%, and then it decreases thereafter. The enhancement in impact strength may be ascribed to the good dispersion of cotton fibers throughout the matrix, which helps to increase the interaction or adhesion at the matrix/cotton fiber interface. In addition, the increases in impact strength as fiber content increases are due to the increase in fiber pull out and fiber breakage [31]. Hence, this permits the optimum operation of stress-transfer from the matrix to the cotton fibers, thus resulting in an improvement of strength properties.

However, the impact strength of composites decreases when fiber content increases to >0.5 wt.%. This reduction in impact strength at higher content of cotton fiber was due to the formation of fiber agglomerates and voids as a result of increased system viscosity due to the presence of the cotton fiber, which in turn reduced the fiber matrix adhesion.

The impact strength of the neat geopolymer paste increased from 1.9 to 4.5 kJ/m^2 after the addition of 0.5 wt.% cotton fibers. However, adding more cotton fibers (0.7 and 1.0 wt %) led to a reduction in strength.

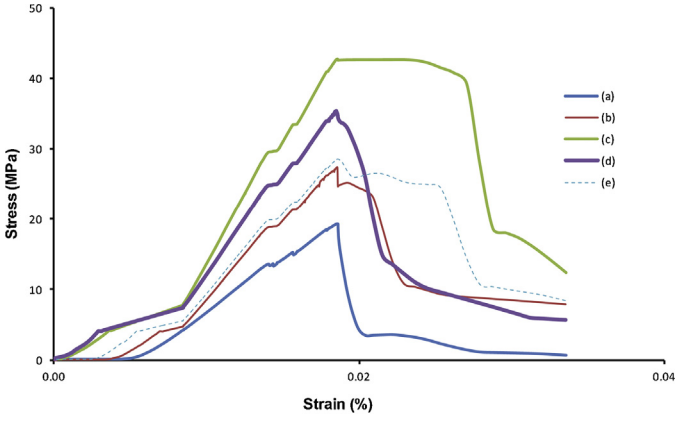

Fig. 6. Typical stress–strain curves of geopolymer composites with various cotton fiber contents (a) 0 wt.%, (b) 0.3 wt.%, (c) 0.5 wt.%, (d) 0.7 wt.%, and (e) 1.0 wt.%.

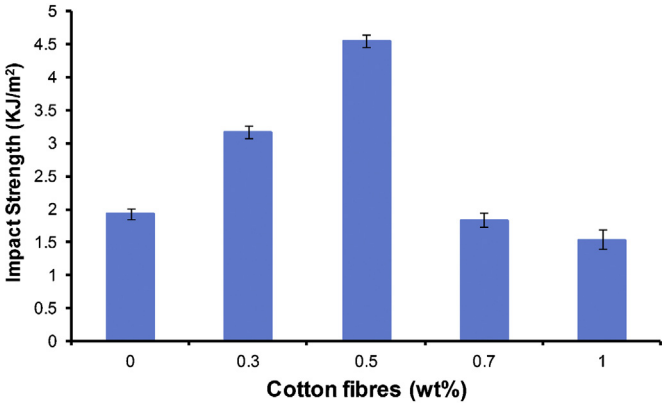

Fig. 7. Impact strength of geopolymer composites as a function of fiber content.

4. Conclusions

Geopolymer composites reinforced with cotton fibers have been fabricated and characterized. Optimum enhancements in hardness, compressive strength and impact resistance were achieved for composites containing up to 0.5 wt.% cotton fibers. However, further increase in cotton fiber content beyond 0.5 wt.% led to fiber agglomerations with a concomitant reduction in mechanical properties by virtue of increased viscosity, voids formation and poor dispersion of fibers within the matrix.

Conflict of interest statement

The authors declare that there are no conflicts of interest.

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