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Influence of mixing methods of nano silica on the microstructural and mechanical properties of flax fabric reinforced geopolymer composites

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HIGHLIGHTS

- Geopolymers are prepared using the wet-mix and dry-mix methods.
- Nanosilica improves the microstructure of geopolymer.
- Flexural and compressive strengths of samples are improved by nanosilica.
- Samples prepared by dry-mixing have better physical and mechanical properties.

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ABSTRACT

This paper presents the effects of two mixing methods of nanosilica on physical and mechanical properties of flyash-based geopolymer matrices containing nanosilica (NS) at 0.5, 1.0, 2.0, and 3.0 wt%. Comparison is made with conventional mechanical dry-mix of NS with fly-ash and wet-mix of NS in alkaline solutions. The influence of NS on the flexural toughness of flax fabric (FF) reinforced geopolymer nanocomposites has also been reported. Physical and microstructural properties are investigated using X-ray diffraction, scanning electron microscopy and energy dispersive X-ray spectroscopy. Results show that generally the addition of NS particles improves the microstructure and increases flexural and compressive strengths of geopolymer nanocomposites. However, samples prepared using the dry-mix approach demonstrate better physical and mechanical properties when compared to wet-mix samples.

1. Introduction

Geopolymers are synthesized by activating a solid aluminosilicate source with alkaline solutions, which forms amorphous networks of tetrahedral SiO_4 and AlO_4 connected by sharing oxygen atoms. Molecular geopolymeric network units can be formed depending on the Si:Al ratio. The fundamental geopolymeric chemical networks are poly-sialate (SiO_4-AlO_4), poly-sialate-siloxo (SiO_4-AlO_4-SiO_4) and poly-sialate-disiloxo (SiO_4-AlO_4-SiO_4-SiO_4), which represent the Si:Al ratios of 1, 2 and 3, respectively [1]. One of the most important parameters that influence the physical and mechanical properties of geopolymer matrices is the chemical content of silicon and aluminum elements. The molar chemical ratio Si:Al has been studied extensively and is considered a critical factor that controls the compressive strengths of geopolymers [2]. The optimum mechanical properties exhibited in geopolymers have Si:Al ranging between 1.8 and 2.5 [2–4]. However, these ratios of the aluminosilicate composition must be identified for the reactive geopolymer gel or the amorphous phase, since the crystalline phases are unreactive in the geopolymeric reaction [5,6].

Improving the mechanical properties such as flexural and compressive strengths, and flexural toughness of geopolymers will significantly increase their applications in the construction industry; and this may be accomplished by two ways: one is through enhancement of physical structure of geopolymer matrices by incorporating nanoparticles in the system, and the other is through improving the toughness behavior of the material by adding fibres as fibre-reinforced geopolymer composite. Currently, nanotechnology has several applications in polymer and ceramic research, especially in producing nanocomposites that exhibit superior physical properties [5]. In geopolymers, the incorporation of nanoparticles is a relatively novel field; however, some types of nanoparticle have been added efficiently to geopolymer pastes to improve their mechanical properties. In a previous study, the effect of nano-clay (Cloisite 30B) on the mechanical and thermal properties of geopolymer composites is investigated [7]. Nanoclay

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particles were found to improve the physical structure of geopolymer matrices, producing geopolymer with superior mechanical performance. Also, nano-alumina and nano-silica particles have been integrated successfully into geopolymer matrices, resulting in higher mechanical properties. The alumina and silica acted positively in two ways: physically as a nano-filler and chemically by activating the geopolymeric reaction [8]. In another study, it has been reported that the silica and alumina nano-particles have the ability to reduce the porosity and water absorption of geopolymer matrices [9]. A further study on the effect of adding carbon nanotubes to fly-ash-based geopolymer has demonstrated a significant improvement in the mechanical and electrical properties of geopolymer nano-composites when compared to the control paste [10]. A critical factor, however, that limits the addition of nanoparticles is the good dispersion of the nanoparticles in the matrix. Poor dispersion of nanoparticles could lead to the agglomeration of nano particles, which adversely affects the physical and mechanical properties of geopolymers [11]. Yet, modifying the preparation procedure including the way of mixing nanoparticles could change the nanoparticles level of dispersion in the matrices, which consequently influences the composite's physical properties [12].

Geopolymers like other ceramics also suffer from brittle cracking under mechanical loads. This limitation may be readily overcome with fibre reinforcement as in high performance polymer-matrix composites. Natural fibres have revealed desirable effect on the mechanical properties of geopolymers. For example, wool and flax fibres have been successfully used in reinforcing geopolymer composites with concomitant improvements in mechanical and fracture properties [13,14]. Furthermore, it has been found that cotton fibres enhanced the strength and toughness of geopolymer [15]. In our previous work, fly-ash based geopolymer has been reinforced with flax fabrics, resulting in significant improvement on the mechanical properties of the eco-composites [16]. Flax fibres offer advantages such as low density, low cost, availability, specified properties and low energy consumption throughout the extraction process. Flax fibre (FF) is a main natural fibre that made up of 64.1% cellulose, 16.7% hemicelluloses, 1.8% pectin and 2% lignin. FF also contains minor amounts of waxes, bound water and inorganic component material [17].

In this study, the effects of dry mixing of nano-silica (NS) with fly-ash before adding alkaline solutions and of the dispersion of NS in alkaline solution on the physical and mechanical properties of geopolymers are investigated. XRD analysis and EDS are used to explore the morphology and microstructure of geopolymer nanocomposites. Besides, the effect of different amounts of NS on the flexural toughness of FF-reinforced geopolymer nanocomposites is also evaluated. To the best knowledge of authors, no study has been reported on reinforcing geopolymer with a combination of both NS and FF.

2. Experimental procedure

2.1. Raw materials

Low calcium fly ash (ASTM class F), collected from the Eraring power station in NSW of Australia, and was used as the source material for the geopolymer matrix. The chemical composition of fly ash is shown in Table 1. Nanosilica is obtained from Nanostructured and Amorphous Materials, Inc. of USA with average particle diameter of 18–25 nm (Fig. 1). 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_2O, 29.4% SiO_2 and 55.9% water by mass.

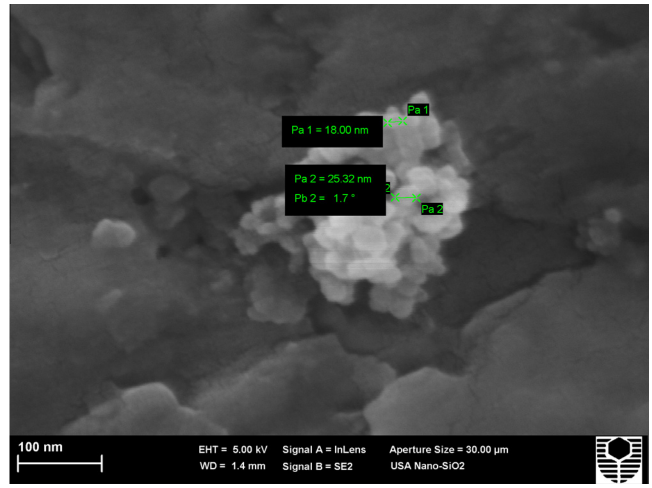

Fig. 1. SEM image of nanosilica (NS) particles.

2.2. Preparation of geopolymer nano-composites

To prepare the geopolymer matrix, 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 it was prepared and combined with the sodium silicate solution one day before mixing.

The geopolymer pastes were prepared by two methods, a dry and wet process (Fig. 2). For dry-mix process, the NS was added first to the fly-ash at the dosages of 0.5, 1.0, 2.0 and 3.0% by weight (Table 2). The fly-ash and NS were dry-mixed for 5 min in a covered mixer at a low speed and then mixed for another 10 min at high speed until homogeneity was achieved. The alkaline solution was then added slowly to the fly-ash/NS powders in the Hobart mixer at a low speed until the mixes became homogeneous, then further mixed for another 10 min on high speed. Similar mixtures dosages were prepared to produce the wet-mix paste. However, the NS powder was first wet-mixed with the alkaline solution mechanically until the dissolution of the NS powder was achieved. Then, the solutions, with different dosages of silica, were mixed with fly ash in the Hobart mixer at the same period of time of the dry-mix process. The resultant mixtures, dry/wet-mixes, were then poured into coated wooden moulds and placed on a vibration table for two minutes to remove any entrapped air inside the pastes.

Similar mixtures were prepared to produce the FF-composites and nanocomposites. The samples prepared by spreading a thin layer of geopolymer paste in a well-greased wooden mould and carefully placing the first layer of FF on it. The fabric was fully saturated with paste by a roller, and the process repeated for ten layers; each specimen contained a different weight percentage of NS. The samples then were left under heavy weight (20 kg) for 1 h to reduce entrapped air inside the samples. All samples (see

Table 1
Chemical composition of fly-ash (wt%).

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