



Compressive strength and microstructure of fly ash based geopolymer blended with silica fume under thermal cycle



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ABSTRACT

This work aims to reveal the effects of silica fume on properties of fly ash based geopolymer under thermal cycles. Geopolymer specimens were prepared by alkali activation of fly ash, which was partially replaced by silica fume at levels ranging from 0% to 30% with an interval of 10%, by mass. Microstructure, residual strength and mass loss of fly ash based geopolymer blended with silica fume before and after exposed to 7, 28 and 56 heat-cooling thermal cycles at different target temperatures of 200 °C, 400 °C and 800 °C were assessed and compared. The experimental results reveal that silica fume addition enhances strength development in geopolymer. Under thermal cycles, the compressive strength of geopolymer decreases, and the compressive strength loss, as well as the mass loss, increases with increasing target temperature. The strength loss is the same regardless of silica fume content after thermal cycles. Microstructure analysis uncovers that pore structure of geopolymer degrades after thermal cycles. The pores of geopolymer are refined by the addition of silica fume. The incorporation of silica fume optimizes the microstructure and improves the thermal resistance of geopolymer. Silica fume increases the strength of the geopolymer and even though the strength loss is the same, the strength after heat cycle exposure is still good.

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1. Introduction

Portland cement, as one of the most important and indispensable building materials, consumes a lot of energy and contributes a lot to carbon emissions in its production process [1–4]. Apart from the environmental issue, certain durability problems of cement concrete are difficult to solve. In order to solve the durability issues, extensive research have been undertaken recently. Previously published papers have proved that the supplementary cementitious materials (SCMs) including silica fume (SF), fly ash (FA), metakaolin (MK) and granulated blast-furnace slag (BFS) were promising in terms of sustainable development and durability aspects of concrete [5–10].

Often the high volume replacement level of SCMs in cement will cause changes in performance including particle size distribution, specific surface area, oxide composition and phase composition, which are closely related to the durability of concrete. Due to large

specific surface area and high porosity, SCMs such as silica fume and nano-scale additives have detrimental effects on workability due to increasing water demand, which may limit their application [11–16]. Furthermore, SCMs are believed to decrease the early strength due to dilution of cement even though they increase late strength through the pozzolanic reaction. These trends are consistent for most of SCMs including metakaolin [17–20], fly ash [21], limestone powder [22–24], rice husk ash [25–28] and ground-granulated blast furnace slag (GGBFS) [29–32]. Published literatures also concluded that SCMs increased porosity of concrete when more than 10% metakaolin was added [17,22]. Sotiriadis et al. [33] observed slower deterioration in limestone cement concrete stored in chloride-sulfate solution compared to the severe deterioration observed in fly ash, natural pozzolan, metakaolin and GGBFS concretes. This was attributed to the delayed sulfate induced deterioration by chlorides in limestone cement concrete. Additionally, a high content of calcined MK (25%) accelerated the carbonation of concrete [18].

According to the shortages of SCMs in cement system, new cementitious materials must be explored urgently to replace the traditional cement to enlarge the utilization of SCMs including fly

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ash, slag, metakaolin, etc. The promising exploratory alternative is the geopolimer system or alkali activation of slag, fly ash, metakaolin and other aluminosilicate materials.

Geopolymer is prepared by combining supplementary cementitious materials (SCMs) or industrial by-products with strong alkali solutions such as sodium hydroxide (NaOH) and sodium silicate (in most cases) to form a three-dimensional amorphous aluminosilicate network with properties similar to or better than that of cement. Research work has been carried out widely on geopolymer. Duan et al. [34] investigated durability and microstructure of fly ash and metakaolin based geopolymer. They concluded that geopolymer presented better durability and denser microstructure compared to ordinary Portland cement when exposed to elevated temperatures and acid attack. Nematollahi and Sanjayan [35] reported that fly ash paste activated by multi-compound activator ($\text{Na}_2\text{SiO}_3/\text{NaOH} = 2.5$) had higher relative slump and compressive strength with reference to the NaOH-activated fly ash paste. Ranjbar et al. [36] presented the effects and adaptability of palm oil fuel ash as a replacement material in fly ash based geopolymer mortar. They found higher early compressive strength in fly ash based geopolymer compared to palm oil fuel ash added geopolymer. Sarker et al. [37] carried out high temperature exposure experiment up to 1000 °C for fly ash based geopolymer and ordinary Portland cement. The geopolymer concrete specimens were found to suffer less damage than the OPC concrete specimens after high temperature exposure. Furthermore, the geopolymer concrete specimens were found to have higher tensile strength and bond strength than OPC concrete [41,42].

Ground granulated blast-furnace slag [38], nano-SiO₂ and nano-Al₂O₃ [39] were all proved to be efficient for the enhancement in strength, and the bonding strength of steel bar and geopolymer pastes were found to be slightly higher than that of control concrete by Songpiriyakij et al. [40].

Analysis of published literatures indicates that fly ash based geopolymer receives more attention and interest from researchers due to sustainably high mechanical strength, high temperature resistance, higher durability and other characteristics. Additionally, silica fume has been widely used to improve compressive strength [43,44], durability [45–47] and to optimize microstructure [48–50] of cement and concrete. However, study on fly ash based geopolymer blended with silica fume is scant. Only Chindaprasirt et al. [51] used silica fume to improve the strength and resistance to sulfate and acid of fly ash based geopolymer. The geopolymer with 3.75% silica fume showed the optimum strength with 28-day compressive strength of 17.0 MPa. The compressive strengths of geopolymer with 3.75% silica fume immersed in 5% magnesium sulfate solution and 3% sulfuric acid solutions were substantially higher than the control.

Furthermore, few references were related to properties of geopolymer under thermal cycle, relative work carried out in the area about properties of geopolymer exposed to thermal cycle was mainly collected by Rashad [52–56]. However, thermal properties of fly ash-silica fume geopolymer were not introduced in the previously published work. Study on properties of fly ash based geopolymer blended with silica fume under thermal cycle is still uninvestigated.

Therefore, this study aims to investigate microstructure, residual strength and mass loss of alkali activated fly ash geopolymer blended with silica fume after exposed to 7, 28 and 56 heat-cooling thermal cycles at different heating temperatures of 200 °C, 400 °C and 800 °C when fly ash was partially replaced by silica fume at levels ranging from 0% to 30% with an interval of 10%, by weight.

2. Experimental

2.1. Materials

Fly ash and silica fume were provided by Shenhua Junggar Energy Corporation in Junggar, Inner Mongolia, China. Their chemical compositions were evaluated by X-ray fluorescence (XRF) spectrometry and were provided in Table 1.

The micrographs of fly ash and silica fume are provided in Fig. 1. Scanning electron microscopic study reveals some interesting features related to the morphology of fly ash samples. Fig. 1(a)-(b) indicate the geometrical properties, it can be seen that shape is usually irregular and appears to be porous in a potential state of fragmentation with small and rough particles. Fly ash shows the presence of very fine particles with size of approximately 10 μm being perceived as an agglomeration of particles.

Fly ash was employed as source material of alkali activation. The original Blaine surface area is about 230 m²/kg. Fly ash particles were ground into smaller particles to reach a Blaine surface area of 400 m²/kg with higher chemical activation, which is helpful for the preparation of geopolymer with high strength and high thermal stability.

Silica fume, as regular spherical particles with particle size less than 150 nm shown in Fig. 1(c)-(d) were used in this study. The particle size analysis of fly ash and silica fume were carried out using a laser diffraction particle size analyzer (MASTERSIZER S, Malvern, U.K.). The characteristic particle diameters D_{10} , D_{50} and D_{90} of fly ash and silica fume particles have been tabulated in Table 2. A wide variation in particle size was observed. The span for fly ash and silica fume was 3.39 and 1.44, respectively.

Alkali activator solution was prepared by dissolving solid sodium hydroxide (99.2% NaOH, analysis reagent purity) in the industrial water glass. The liquid portions in the mixture are 10 M sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) with 14.51% Na₂O, 33.39% SiO₂, and 48.53% H₂O. The modulus Ms. (molar ratio of Na₂O to SiO₂) of liquid alkali activator is 1.5.

2.2. Sample preparation

In this work, geopolymer specimens were synthesized by alkaline-activation of fly ash with varied content of silica fume in liquid alkali activator. The liquid/solid (L/S) mass ratio was kept constant at 0.5, the liquid materials consisted of liquid alkali activator and extra water, and the solid materials consisted of fly ash and silica fume.

The workability of fly ash geopolymer blended with silica fume was measured by using a flow table to check the consistency of fresh pastes before casting. It indicates only 75 mm flow diameter when the liquid/solid (L/S) mass ratio was kept constant at 0.5

Table 1
Chemical compositions of raw materials by XRF analysis (mass, %).

Composition	Fly ash	Silica fume
SiO ₂	27.35	95.72
Al ₂ O ₃	50.85	0.09
Fe ₂ O ₃	2.01	0.63
MgO	0.28	0.37
CaO	5.41	0.23
Na ₂ O	0.04	0.09
K ₂ O	0.33	0.26
MnO	0.02	0.02
TiO ₂	2.12	0.01
SO ₃	2.35	0.01
P ₂ O ₅	1.05	0.04
LOI	7.74	2.53

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