



Effects of nano-silica on the strength development of geopolymer cured at room temperature



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HIGHLIGHTS

- Effects of nano-silica on strength and microstructure of geopolymers were studied.
- Early-age strength of ambient-cured fly ash geopolymers increased by nano-silica.
- Dense microstructure with better interlocking morphology was resulted by nano-silica.
- Use of OPC or GGBFS with nano-silica and fly ash yielded CSH or CASH and NASH.

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ABSTRACT

Use of nano-silica is gaining wider attention due to its significant effect on the microstructural and mechanical properties of Portland cement based binders. Effects of the incorporation of nano-silica in fly ash based geopolymer binders have been investigated in this study. Low-calcium fly ash was used as the principal source of aluminosilicate and it was blended with either blast furnace slag or Portland cement at small percentages in order to accelerate the curing at room temperature. Nano-silica was used at a rate up to 3% of the total binder in order to understand its effect on the strength and microstructural development. The experimental results show that the strength and microstructural properties could be further developed with inclusion of nano-silica in geopolymer mixes. Compressive strength increased with the increase of nano-silica content up to 2%. Scanning electron microscopy (SEM) images showed denser microstructures with well-connected interlocking morphology for the optimum nano-silica dosage. The strength increase is contributed by densification of the microstructure with the addition of nano-silica.

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1. Introduction

Geopolymerization is a process where a large amount of amorphous aluminosilicate phase is transformed into compact cement by hydrothermal polycondensation [1,2]. Previous researchers revealed that geopolymer binder may be viewed as an amorphous analogue of zeolites or zeolitic precursors [3,4] that follows the same formation process as in zeolites [5,6].

The main reaction product in geopolymers is an aluminosilicate gel where tetrahedral Al^{3+} sites charge-balanced by alkali metal cations. Fly ash, ground granulated blast furnace slag, calcined clay and metakaolin are the common source materials used for geopolymers [7,8]. Most commonly used activators are alkali silicates, hydroxides, carbonates or combinations of these chemi-

cals selected based on the chemical compositions of the source material [9,10].

Fernandez and Palomo [3] studied the reactivity of fly ash in alkali solutions where reactive silica, alumina content and amorphous phase content played a vital role in the geopolymerization process. Other researchers [2,11] showed that higher amount of reactive silica resulted in the formation of higher amount of geopolymeric gel and consequently developed higher mechanical strength. Criado et al. [11] found that products of higher strengths consisted of higher soluble silica content when compared the reaction of fly ash with pure NaOH and sodium silicates with moduli up to 1.27.

Other researchers indicate that particle size distribution has a significant impact on the reactivity of fly ash. Higher amount of fine particles will result in higher surface area, and therefore higher reactivity, resulting in higher compressive strength [12]. The roles of other factors such as molarity of NaOH, alkaline content, curing

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conditions, composition of binder materials [13–16] and post-curing chemical treatments [11] were also investigated by other researchers. Results indicated that higher molarity with low alkaline content and elevated curing temperature at early ages enhanced the high early strength of geopolymer systems. The presence of calcium compound in the source material plays a vital role because the Ca^{2+} is capable of acting as a charge-balancing cation within the geopolymeric binder [17] and forms Ca–Al–Si amorphous structures [18]. The Ca^{2+} available in blast furnace slag and OPC are able to create a coexistence of geopolymeric gel and C–S–H that leads to the increase of mechanical strength [19,20] of geopolymer materials.

The microstructural analysis of geopolymer paste has been highlighted to a microporous framework with the high amount of aluminosilicate gel in the matrix. The characterizations of the micro and nanostructures of geopolymer pastes are complex and generally consist of amorphous gels and different unreacted crystalline phases in the activated systems [1,2,11]. It was shown that the water released during geopolymer formation causes discontinuous nanopores throughout the hydrated geopolymer matrix [18]. Moreover, Bakharev [20] found that the micrographs of geopolymer materials are heterogeneous and contain a large proportion of unreacted fly ash surrounded by aluminosilicate gel. The XRD patterns of alkali-activated fly ashes show that the original crystalline phases are not changed by the activation reactions [21]. However, the formation of hydration products depends on the source materials such as fly-ash, metakaolin, OPC and slag [22–24,34,37].

The ingress of fluid to the interior of geopolymer paste primarily takes place through its pore systems. It was observed that the amount of small pores proportionately increases with the higher molarity of the alkaline activator solution [25]. However, KOH based geopolymer mortars were shown to have lower amount of total porosity than NaOH based geopolymer mortars [26].

Despite the fact that geopolymer mechanism was developed over a couple of decades ago and has many advantageous characteristics, it has not yet been used in concrete as extensively as Portland cement. Widespread use of low calcium fly-ash geopolymers is hindered due to some of its limitations such as slow setting, high porosity and subsequent slow strength development. Improvement of these properties is essential in order to increase the use of geopolymers in concrete as an alternative to energy-intensive OPC binders. The effects of nano-silica in OPC systems are widely investigated and it was found as an effective additive towards the mechanical and microstructural development [32]. However, research on the effects of nano-silica on the properties of geopolymers is scarce in literature. Therefore, this study aimed to investigate the effect of nano-silica on the properties of fly ash geopolymers. In order to determine the optimum quantity of nano-silica in the different types of geopolymers, experimental work was carried out on OPC and GGBFS blended fly ash based geopolymer binders with 0–3% nano-silica contents. The strength and microstructural development of geopolymer paste samples containing different proportions of nano-silica and subjected to room temperature curing were studied.

2. Experimental work

2.1. Materials

Class F fly ash (FA), nano-silica (NS), ground granulated blast-furnace slag (GGBFS) and ordinary Portland cement Type I (OPC) were used in this study. Nano-silica with average particle diameter of 15 nm was obtained from a commercial supplier. The chemical composition and physical properties of the fly-ash, OPC, GGBFS and nano-silica are presented in Table 1. The alkaline activator was a combination of sodium hydroxide and sodium silicate solutions. Sodium hydroxide solution of 8 M concentration was prepared by mixing 97–98% pure pellets with tap water. The mass ratio of SiO_2 to Na_2O of the sodium silicate solution was 2.61 ($\text{SiO}_2 = 30.0\%$, $\text{Na}_2\text{O} = 11.5\%$ and water = 58.5%).

Table 1

Chemical composition and physical properties of fly ash, OPC, GGBFS and Nano-silica.

Chemical analysis	Class F fly ^a (wt.%)	OPC (wt.%)	GGBFS (wt.%)	Nano-silica (wt.%)
SiO_2	46.69	21.1	29.96	99.5
Al_2O_3	29.14	4.7	12.25	0.001
Fe_2O_3	13.81	2.7	0.52	0.001
CaO	3.29	63.6	45.45	–
MgO	1.4	2.6	–	–
MnO	0.16	–	–	–
K_2O	0.72	–	0.38	–
Na_2O	0.86	–	0.31	–
P_2O_5	1.63	–	0.04	–
TiO_2	1.34	–	0.46	–
SO_3	0.43	2.5	3.62	–
Specific gravity	2.40	3.15	2.9	–
LOI ^b	0.68	2.0	2.39	–

^a ASTM C 618.

^b Loss on ignition.

2.2. Geopolymer mixes containing nano-silica

The binder compositions of the geopolymer paste mixes were proportioned based on the previous works on geopolymers cured at room temperature [14,16]. Twenty-one geopolymer paste mixes in 3 series (fly ash only, GGBFS blended fly ash and OPC blended fly ash) were mixed in the laboratory. The mix proportions are given in Table 2. In the fly ash only series, fly ash was used as the main binder with 0–3% nano-silica. In the GGBFS blended fly ash series, 0–3% nano-silica was included with 15% GGBFS and 82–85% fly ash. The binder composition of the OPC blended fly ash series was 0–3% nano-silica, 10% OPC and 87–90% fly ash. The GGBFS and OPC contents of 15% and 10% respectively were used since these proportions were found in previous studies [14,16,24] as the optimum blending with low-calcium fly ash for reasonable setting times. The alkaline liquid content of each mix consisted of the same proportion of alkaline solution (40%) with a sodium silicate to sodium hydroxide ratio of 2.0. The concentration of the NaOH solution was 8 M. The samples containing different binders were named according to the type of binder used in the mix. For example, FA-PC-NS3 represents a geopolymer paste mix having 3% nano-silica (NS) in the Portland cement (PC) blended fly ash based geopolymer paste. Similarly, the mix designations of FA-NS and FA-S-NS represent the fly ash only and GGBFS blended fly ash series respectively.

2.3. Preparation, casting and curing of test specimens

The alkaline solution was prepared in the laboratory by mixing sodium silicate and sodium hydroxide solutions at the required ratio. Usually, nano-silica may not get dispersed well after wetting and add microbubbles to the mix during mixing. A uniform dispersion of the nanoparticles is essential to avoid agglomeration and

Table 2

Mix proportions of geopolymer pastes.

Mix id	NS (wt.%)	Si/Al	Mix components (kg/m ³)			
			Fly-ash	OPC	GGBFS	NS
FA-NS0.0	0.0	1.29	400	–	–	0
FA-NS0.5	0.5	1.30	398	–	–	2
FA-NS1.0	1.0	1.31	396	–	–	4
FA-NS1.5	1.5	1.32	394	–	–	6
FA-NS2.0	2.0	1.34	392	–	–	8
FA-NS2.5	2.5	1.35	390	–	–	10
FA-NS3.0	3.0	1.36	388	–	–	12
FA-PC-NS0.0	0.0	1.34	360	40	–	0
FA-PC-NS0.5	0.5	1.35	358	40	–	2
FA-PC-NS1.0	0.5	1.37	356	40	–	4
FA-PC-NS1.5	1.5	1.38	354	40	–	6
FA-PC-NS2.0	2.0	1.40	352	40	–	8
FA-PC-NS2.5	2.5	1.41	350	40	–	10
FA-PC-NS3.0	3.0	1.43	348	40	–	12
FA-S-NS0.0	0.0	1.34	340	–	60	0
FA-S-NS0.5	0.5	1.36	338	–	60	2
FA-S-NS1.0	1.0	1.37	336	–	60	4
FA-S-NS1.5	1.5	1.39	334	–	60	6
FA-S-NS2.0	2.0	1.40	332	–	60	8
FA-S-NS2.5	2.5	1.42	330	–	60	10
FA-S-NS3.0	3.0	1.43	328	–	60	12

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