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Characterization of alkali activated slag–fly ash blends containing nano-silica

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HIGHLIGHTS

- The nano-silica addition reduces the flowability and slightly retards the early age reaction.
- The gel structure remains stable regardless of nano-silica content and slag/fly ash ratio.
- The addition of nano-silica slightly increases the chemically bound water content.
- A nano-silica content of around 2% presents the optimum strength and porosity.

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ABSTRACT

Applying nano-technology/modifications in construction and building materials has shown inspiring results. This paper investigates the effects of nano-silica incorporation on an eco-friendly alkali activated slag–fly ash blends. The fresh behaviors, reaction kinetics, gel structure, porosity and strength of samples with different nano-silica contents and slag/fly ash ratios are analyzed. The results indicate that as the nano-silica content increases, the slump flow is significantly reduced, and the reaction process is slightly retarded according to the setting time and isothermal calorimetry results. The microstructure analysis carried out by FTIR and TG/DSC shows that the addition of nano-silica slightly increases the chemically bound water content. The main reaction product is a chain structured C-A-S-H type gel, regardless of the slag/fly ash ratio and nano-silica content. Increasing the nano-silica content up to around 2% benefits the compressive strength and contributes to a reduced porosity, but further higher nano-silica contents show a negative effect on the strength and the pore refinement becomes less significant. It is suggested that the nano-silica benefits the microstructure and strength by providing additional reactive silica source and the filler effect. Furthermore, the slag content exhibits a dominant role on setting times, early age reaction, compressive strength and porosity in this blended alkali system.

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1. Introduction

In order to reduce the negative environmental impacts of cement industry, great attention has been paid to apply alkali activated materials as alternatives to Portland cement. This type of material generally exhibits superior mechanical properties [1], durability [2,3], thermal resistance [4] together with significantly reduced energy costs and carbon emissions [5,6]. Based on the chemical composition of the raw materials, two types of binding systems can be classified: one is the alkali activated slag (Si + Ca) system, having a C-A-S-H type gel with a low Ca/Si ratio and a high Al incorporation as the main reaction product [7]; the other is the alkali activated class F fly ash/metakaolin (Si + Al) system, having N–A–S–H type gels with three-dimensional network as the major reaction products [8]. Both systems exhibit distinct behaviors regarding alkaline and curing demands, setting and hardening processes, strength development due to their differences in reaction mechanism and gel characteristics.

Recently, growing interests have been focused on the blended alkaline system (Na_2O–CaO–Al_2O_3–SiO_2 system) that is prepared by mixing calcium enriched precursors and low calcium aluminosilicates. Compared to either of the individual system, the blended system shows a better control of setting times [9], as well as modified workability [10], shrinkage [11], mechanical properties and durability [12]. Micro-scale analysis reveals that the reaction products are stably coexisting C-(A)-S-H and N-A-S-H type gels with higher degree of cross-linking [13–15], indicating a desirable formation of gel structure. Furthermore, the effects of activator type and dosage, raw materials' composition and curing conditions

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on reaction kinetics, gel characters, mechanical properties and durability issues were also intensively investigated [16–21]. The recent progresses in understanding the blended system provide solid basis for further investigations and those modified properties demonstrate a promising future for the application of alkali activated materials.

On the other hand, nano-silica (NS) has recently been utilized in Portland cement based systems as an effective pozzolanic binder for the improvement of mechanical properties and microstructure [22]. Due to its considerably fine particle size, nano-silica has been observed to accelerate the hydration process at early ages [23], refine the pore structure [24,25] and enhance the mechanical properties even at small levels of replacement [26]. Moreover, nano-silica also exhibits ideal pozzolanic activity owing to its amorphous nature and high specific surface area [27,28], which leads to the formation of additional C-S-H gel by reacting with calcium hydroxide and results in a denser matrix [29–31]. Meanwhile, attention has also been paid to apply nano-silica in alkali activated systems. Tanakorn et al. [32] studied the effect of nano-silica on fly ash based geopolymers, showing that increasing the nano-silica content results in the decrease of setting time and the increment of mechanical properties. Microstructure analysis revealed that a nano-silica content up to 2% increases the reaction product and densifies the matrix, but higher nano-silica contents show negative effects. Gao et al. [33] applied nano-silica up to 3% by mass in metakaolin based geopolymers, increased reaction products due to nano-silica addition were observed and an optimum nano-silica content of 1% in terms of strength and porosity was reported. Adak et al. [34] investigated the effect of nano-silica on strength and durability of fly ash based geopolymers, the used nano-silica content was up to 10% by mass. The results showed that a nano-silica content of 6% exhibits appreciable mechanical properties under ambient temperature, as well as less water absorption and reduced charge passed in rapid chloride ion penetration test. As can be noticed, the previous studies showed the superiority of applying nano-silica in alkali activated low calcium systems. However, there are limited mechanism studies and performance evaluations concerning the effect of nano-silica on the blended alkaline system (Na_2O–CaO–Al_2O_3–SiO_2).

The purpose of this study is to understand the influence of nano-silica on fresh behavior, early age reaction kinetics, mechanical properties, reaction products and microstructure of alkali activated slag–fly ash blends. The flowability, setting times, porosity and compressive strength of alkali activated slag–fly ash blends with nano-silica addition are identified. Furthermore, micro-scale analyses are carried out by using isothermal calorimetry, thermogravimetry/differential scanning calorimetry (TG/DSC) and Fourier transform infrared spectroscopy (FTIR).

2. Experiment

2.1. Materials

The solid precursors used in this study were ground granulated blast furnace slag (GGBS, provided by ENCI B.V., the Netherlands) and a commercial Class F fly ash. Their major chemical compositions were analyzed by X-ray fluorescence and are shown in Table 1. The detailed particle size distributions of solid raw materials and nano-silica, measured applying a light scattering technique using Mastersizer 2000, are given in Fig. 1. The used slag has a median particle size (d_{50}) of 12.43 μm and a specific density of 2.93 g/cm^3; while the fly ash has a d_{50} of 22.06 μm and a specific density of 2.30 g/cm^3. A commercially available nano-silica slurry is selected, the slurry has a solid content of 50 wt.%, a d50 of 0.12 μm and a density of 1.4 g/cm^3. The chemical composition of nano-silica provided by the supplier is listed in Table 1. Concerning the alkaline activators, commercial sodium silicate solution (27.69% SiO_2, 8.39% Na_2O and 63.92% H_2O by mass) and analytical level of sodium hydroxide pellets (99 wt.%) were used. The desired activator modulus (Ms, SiO_2/Na_2O molar ratio) was achieved by adding the appropriate amount of sodium hydroxide pellets into the sodium silicate solution. Distilled water was added in order to reach the desired water/binder ratio. The mixed activator solution was cooled down to room temperature prior to further use.

Table 1
Major chemical composition of slag, fly ash and nano-silica.

Oxides (wt.%)	Fly ash	Slag	Nano-silica
SiO_2	54.62	34.44	98.68
Al_2O_3	24.42	13.31	0.37
CaO	4.44	37.42	0.09
MgO	1.43	9.89	–
Fe_2O_3	7.21	0.47	–
Na_2O	0.73	0.34	0.32
K_2O	1.75	0.47	0.35
SO_3	0.46	1.23	–
LOI	2.80	1.65	–

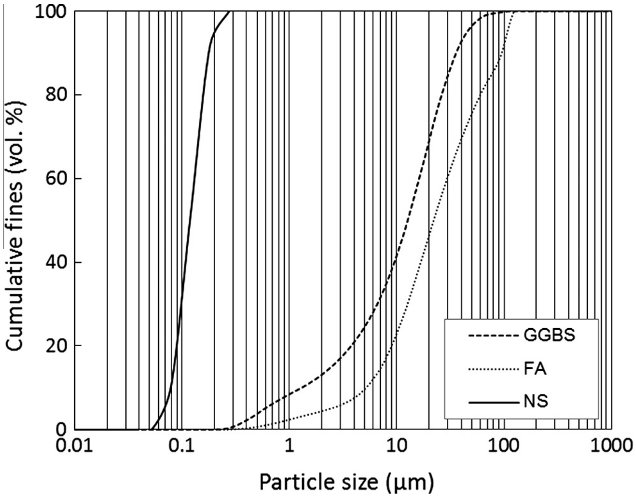

Fig. 1. Particle size distributions of raw materials.

2.2. Sample preparation

The activator used in this study has an equivalent sodium oxide (Na_2O) content of 5% by mass of the binder and an activator modulus of 1.4 for all mixes. The target activator modulus was reached by mixing sodium silicate solution and sodium hydroxide pellets with a solution/pellets mass ratio of 6.41. The water/binder ratio was kept constant at 0.35 in all mixtures. The water consisted of the added distilled water, the water contained in the activator solution and nano-silica slurry. The chosen Na_2O content and water/binder ratio were preliminarily determined that would provide sufficient alkalinity without efflorescence and satisfying flowability, respectively [21,35]. The slag/fly ash ratios of 70/30 and 30/70 by mass and nano-silica replacement from 0% to 3% by mass were used. The detailed information of mix proportions is listed in Table 2. The paste samples were prepared in a laboratory mixer; firstly the solid raw materials were added into the mixer and mixed for 1 min, followed by the nano-silica slurry and the activating solution; then their mixtures were mixed at a slow speed for 30 s and rested for 30 s before another 120 s at a medium speed. The fresh paste/mortar was poured into plastic molds of 40 × 40 × 160 mm^3 and vibrated for 1 min, then covered with a plastic film on the top surface for 24 h; finally all specimens were demolded and cured at a temperature of 20 °C and a relative humidity of 95% until the testing age.

2.3. Testing methods

The workability of paste samples were tested by the mini spread-flow test applying the Hägermann cone according to EN 1015-3 [36]. Fresh samples were transferred into a standard conical ring and a free flow without jolting was allowed. Two diameters that are perpendicular to each other were determined and the mean value was recorded as the slump flow. The initial and final setting times were measured by a Vicat needle method as described in EN 196-3 [37]. The determined values were an average of three samples.

The reaction kinetics was investigated by an isothermal calorimeter (TAM Air, Thermometric). Solid raw materials were firstly mixed with activating solution externally for around 1 min vibrated by an electrical vibrator, then the mixed paste was injected into the ampoule and sealed by a lid, and loaded into the calorimeter. All measurements were conducted for 72 h under a constant temperature of 20 °C.

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