



Compositional, microstructural and mechanical properties of ambient condition cured alkali-activated cement



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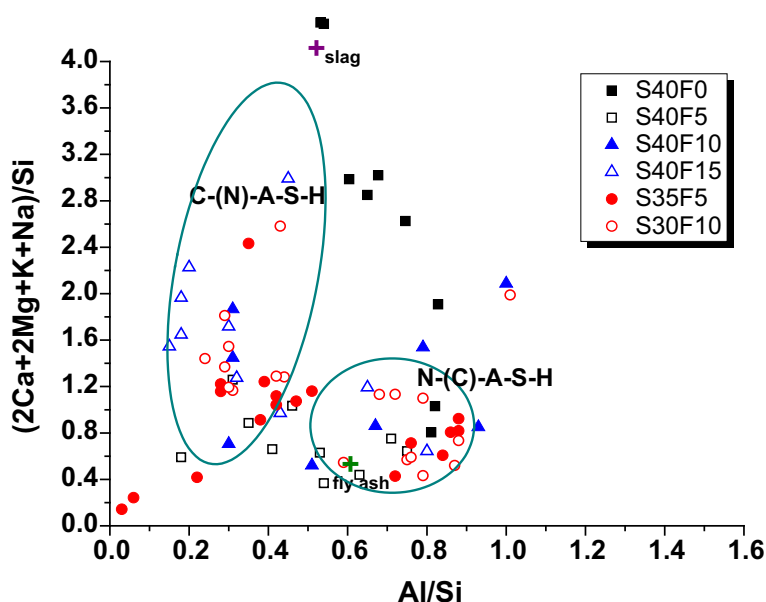
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HIGHLIGHTS

- Alkali-activated cement (AAC) by fly ash, slag and silica fume.
- AACs are manufactured and aged at ambient conditions for 1 year.
- Gels in AACs are heterogeneous hybrid N-(C)-A-S-H and C-(N)-A-S-H.
- No clear compositional boundary to separate the two hybrid gels.
- Porosity is important in determining strength, like in PC system.

GRAPHICAL ABSTRACT

Atomic $(2Ca + 2Mg + K + Na)/Si$ versus Al/Si ratios of the gels in the AACs at 1-year of ageing at ambient conditions. The solid materials are slag (S) mixed with fly ash and silica fume (F).



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ABSTRACT

As an alternative binder, alkali-activated fly ash/slag blend cement is attractive in practice due to controllable setting behaviour at ambient temperature conditions. This study investigates the mineralogical and elemental compositions in six alkali-activated cements (AACs) prepared by sodium silicate-activated ternary systems of fly ash, slag and silica fume at ambient conditions. X-ray diffractometry (XRD) and scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS) show the poorly crystalline and extremely heterogeneous nature of reaction products at 1 year. Around residual fly ash particles N-(C)-A-S-H gels present with relatively high Al/Si ratios, 0.6–1.0, and probably high polymerization degree. The Al/Si ratio decreases gradually in the gel towards residual slag particles at micrometre scale, and becomes C-(N)-A-S-H around slag particles. There is no clear

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compositional boundary to separate the two hybrid gels. Comparing with the composition of the gel, the porosity and pore structure are more relative and determining to the compressive strength development of the AACs.

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1. Introduction

Alkali-activated cement (AAC) based on fly ash has been considered as “a cement for the future” [1,2]. The development of this type of materials has at least three benefits: (1) significant reduction of CO₂ emissions compared to manufacturing Portland cement; (2) added value into fly ash, a by-product or waste material from coal combustion; and (3) less landfilling and environment protection costs by converting this potentially harmful by-product into construction material. Despite the fact that many aspects regarding to the chemistry of reaction are still unclear, the generally accepted reaction mechanisms of alkali activation involve these four steps: (1) dissolution of vitreous/amorphous aluminosilicate under high pH conditions; (2) precipitation of the released aluminate and silicate species into oligomers, which may take place promptly after being dissolved; (3) formation of alkaline aluminosilicate hydrate gel ($M_2O \cdot Al_2O_3 \cdot nSiO_2 \cdot mH_2O$, M is Na and/or K); (4) transition of gels into Si-rich phases by incorporating more Si as the reaction progresses, or into more ordered production by crystallization under certain reaction and curing conditions [2,3]. The product based on alkali-activated fly ash has shown chemical resistance and stability against corrosive solutions [1,4–6], attracting wide interests in the development of high performance and durable construction materials. However, the relatively low reactivity of fly ash, particularly for Class F grade with low concentrations of calcium and alumina, usually leads to slow setting, which in many cases takes more than 24 h at ambient conditions. Therefore, to achieve useful setting time and early strength, the activation is usually performed an elevated curing temperature between 40 and 90 °C [7]. This becomes one of the problems barring the industrial application as the existing practise on Portland cement performs at ambient conditions. It is extremely important to remember that, as highlighted in [8], research on avoiding high-temperature curing is essential for the purpose of reducing CO₂ emissions and to move towards industrial application.

The partial replacement of ground granulated blast furnace slag for fly ash has been proven to be a practical approach to accelerate the setting and hardening of alkali-activated systems at ambient temperatures. Activating with waterglass mixed with sodium hydroxide solution, when the blending slag content increased from 10 to 30 wt.%, the initial and final setting times of the pastes can be shortened from 100 and 180 min to 31 and 60 min respectively as measured at 17 °C; by increasing the testing temperature to 28 °C the setting times were shortened by 3–5 times [8]. A similar trend was found in alkali activated slag-fly ash-limestone systems manufactured at 20 °C [9]. This means that setting behaviour can be controlled by careful design of the solid raw materials and activator according to the environment temperature. In practise, the AAC concretes available on Australian market (commercial name is called earth friendly concrete, EFC) are produced with fly ash and slag blends.

In terms of mechanical properties, the 1 to 90-day compressive strengths of sodium hydroxide activated fly ash/slag blends generally increased with the increase of slag proportion [10]. It had been assumed that the mechanical development of alkali activated slag/fly ash blends is due to the formation of two different products, independently of curing conditions: a low-crystalline calcium

silicate hydrate (C-S-H) formed mainly by the alkali activation of slag and an amorphous alkaline aluminosilicate as a type of zeolitic gel from activation of fly ash [11]. However, energy dispersive spectroscopy (EDS) analysis of alkali activated slag/fly ash blends showed that there was no evident difference in the composition of products [12]; the average Ca/Si and Al/Ca ratios of two different detected areas of the reaction products ($1 \times 1 \mu\text{m}$ in [12], not specified in [10]) mainly depend on the slag dose. It has been hypothesized that the “relatively homogeneous” reaction products around fly ash and slag particles were a type of Al-substituted calcium sodium silicate hydrates (C, N)-A-S-H, with structure between the crosslinked N-A-S-H gel and linear C-S-H gel [12]. A more recent study [13] showed again that the gels formed in the alkali activated fly ash/slag blends were depending on the raw materials. According to the analysis in [13], the compositions of the gels at 28, 90 and 180 days in fact did not distinguish with each other with clear borders but overlap and distribute randomly within certain ranges. These gels are defined as “hybrid (C, N)-A-S-H” either from calcium substitution into N-A-S-(H)-type gels or from sodium sorption or substitution in chain-to-crosslink silicate C-A-S-H-type gels [3]. In terms of structural ordering of the gel, it is understood that high humidity, high temperature and the use of NaOH (instead of sodium silicate) favour high crystallinity, either for alkali-activated fly ash or alkali-activated slag [5,14]. The blending of slag introduces calcium source in alkali silicate activated fly ash and results in more disordered gels that even hard to be transferred into crystalline phases by hydrothermal process [15].

All the findings above raise the interest of the nature of the gel in alkali-activated slag/fly ash blend binders. The composition seems to depend on the quick release, interaction and gelation of calcium, alumina and silica. An in-depth investigation of these gels will be necessary and important for understanding and predicting the long term properties. This study investigates the compositional and microstructural properties of gels formed through alkali-activated fly ash-slag-silica fume ternary system. The reason of selecting the three supplementary cementitious materials as raw materials is because that they have been specified in cement and concrete standards and widely used in industry. For the development and application of AACs, using these materials is possibly the most practical choice. X-ray diffractometry (XRD) and scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDS) are used to focus on the examination of the gels. The porosity and true density of the alkali-activated products are also investigated and related to the mechanical properties. The understanding of long-term ambient condition cured AACs in this study will provide useful information and confidence of future commercial-scale development.

2. Experimental programme

2.1. Materials

Fly ash obtained from Queensland Millmerran power station (Australia) was used as the aluminosilicate source materials. Ground granulated blast furnace slag was supplied by Cement Australia Pty Ltd. Densified silica fume used in this study was supplied by ECOTEC. The compositions of the three materials including loss on ignition at 1000 °C in air as determined by X-ray fluorescence (XRF, ARL-9800) are given in Table 1. Alkaline activating solution was formulated by blending a commercial sodium silicate solution (Na₂O = 14.7 mass%, SiO₂ = 29.4 mass%, D-Grade

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