

Fly ash-slag interaction during alkaline activation: Influence of activators on phase assemblage and microstructure formation



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HIGHLIGHTS

- Interaction between slag and fly ash during activation and geopolymerization.
- SEM/EDS and XRD elucidate the nanostructure, phase assemblage, and compositions.
- Rietveld semi-quantitative analysis quantifies phase assemblage in binary system.
- Ternary phase diagrams with statistical composition distributions were analyzed.

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ABSTRACT

In this work, the influence of activator on the interaction mechanisms between slag and low-calcium fly ash (FA-F) during alkaline activation was investigated. In particular, the evolution of phase assemblage and microstructure of alkali-activated binary slag and FA-F paste was monitored by means of X-ray diffraction with Rietveld analysis, scanning electron microscopy, and energy-dispersive spectroscopy (SEM/EDS). The results show that the nature of amorphous gels and type of crystalline phases formed are dependent on sample age, initial slag to FA-F ratio, and activator type and dosage. The interaction between FA-F and slag during alkaline activation or geopolymerization is primary demonstrated as: (1) modified nanostructure (e.g. crystallography and lattice parameters) and chemical composition of precipitates, (2) depressing or triggering the formation of some crystalline phases, and (3) spatial heterogeneity of composition in microstructure. The activator is chemically incorporated into interactions between slag and FA-F, and likely affects the dissolution and reaction kinetics.

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1. Introduction

Due to environmental concerns related to cement and concrete production, repurposing of ground-granulated blast furnace slag (GGBFS) and Class F fly ash (FA-F) into infrastructure materials has been gaining wider attention [1,2]. As GGBFS and FA-F are both industrial by-products with remarkable cementitious or pozzolanic activities, alkali-activated binary FA-F and slag materials have become one of most promising alternative binders to ordinary Portland cement (OPC) [3,4]. The combined use of FA-F and slag for preparing alkali-activated binders has several advantages over either pure alkali-activated slag (AAS) or pure FA-F-based geopolymer alone. For instance, addition of small dosages of slag into NaOH-activated FA-F-based geopolymer can potentially resolve its setting problems, since NaOH-activated FA-F typically takes much longer time to harden at room temperature than OPC

[5]. On the other hand, as AAS has considerable chemical and drying shrinkage [6–8], the incorporation of FA-F in AAS binders can enhance its volumetric stability [9]. With respect to further implementation in construction field, the physical and engineering properties of alkali-activated FA-F/slag binders must be known. There is no doubt that the properties of these binders are inherently controlled by the main hydration products formed during the activation process [10].

Previous research has revealed that slag and FA-F, when separately activated, exhibit distinct hydration products [11,12]. Basically, the main hydration product in AAS binders is calcium-alumina-silicate-hydrate (C-A-S-H), while for pure FA-F-based geopolymers, it is rich in alkali-aluminosilicate-hydrate (N-A-S-H) [12]. It has been widely shown that C-A-S-H formed in AAS has a lamellar structure with a strong similarity to the structure of tobermorite [13–15]. The Al^{3+} in C-A-S-H is incorporated in the dreierketten silicate chains and substitutes Si^{4+} , which is charge balanced by alkali or Ca^{2+} cations [16]. In case of N-A-S-H, Si^{4+} and Al^{3+} are arranged in tetrahedral forms to create 3D

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Table 1
Oxide compositions and specific gravity of FA-F and raw slag (mass%).

	CaO	SiO ₂	Al ₂ O ₃	MgO	SO ₃ ^a	S ²⁻	Fe ₂ O ₃	Na ₂ O	K ₂ O	P ₂ O ₅	MnO	TiO ₂	LOI	Specific gravity
Slag	43.83	30.04	12.74	4.79	3.11	0.85	1.16	0.24	0.40	0.08	0.22	–	2.56	2.89
FA-F	2.53	48.67	25.13	0.86	1.46	–	16.08	1.04	1.97	0.26	0.04	1.03	1.58	2.50

^a The element compositions were measured by ICP-AES method, the relatively high SO₃ content in slag is primarily due to the presence of gypsum.

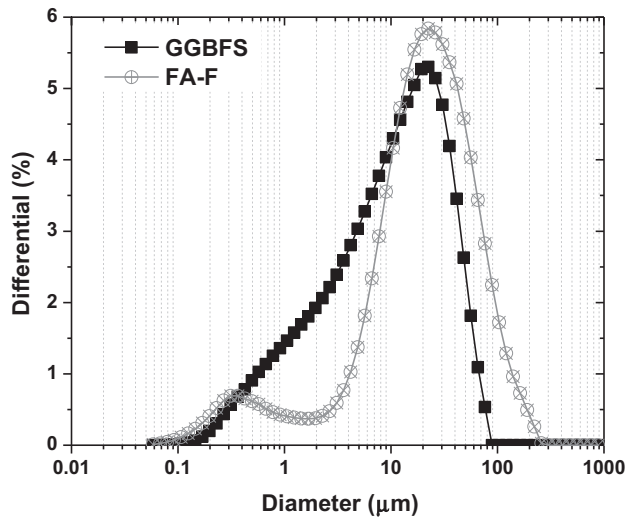


Fig. 1. Particle size distribution of raw slag and FA-F obtained by laser diffraction.

structures, with alkaline cations (e.g. Na⁺) compensating the charge balance associated with Al³⁺. In addition, the Al/Si ratio in N-A-S-H varies and typically ranges from 0.28 to 0.36 [17]. Similarities between N-A-S-H gel structure and the structure of zeolites have been reported in numerous papers [17–19]. For alkali-activated FA-F/slag binary materials, the main hydration product

Table 2
Mixture proportions for alkali-activated binary FA-F/slag-based geopolymer pastes.

Mixture ID	Activator/Binder (by vol.)	NaOH Molarity (M)	Sodium silicate/NaOH (by mass)	Slag/FA-F (by vol.)
FA00S-NS	0.75	6	2	0/100
FA20S-NS				20/80
FA50S-NS				50/50
FA80S-NS				80/20
FA00S-HN		6	0	0/100
FA20S-HN				20/80
FA50S-HN				50/50
FA80S-HN				80/20
FA00S-LN		2	0	0/100
FA20S-LN				20/80
FA50S-LN				50/50
FA80S-LN				80/20

is in a Na₂O-CaO-Al₂O₃-SiO₂-H₂O system, and the hydrated products are reported to contain both N-A-S-H, C-A-S-H, and potentially N-C-A-S-H [20]. Recent study has shown that there may exist chemical interactions between slag and FA-F during alkaline activation and geopolymerization process [21,22]. In addition, it was reported that the nature of hydrated products formed in binary systems are influenced by the FA-F/slag ratio [20,22,23], temperature [24], alkaline activator (e.g. pH, dissolved silica) [25].

Despite an extensive previous investigation, the interaction mechanism between slag and FA-F, as well as the influence of activator on this interaction remains unclear. Recent investigators

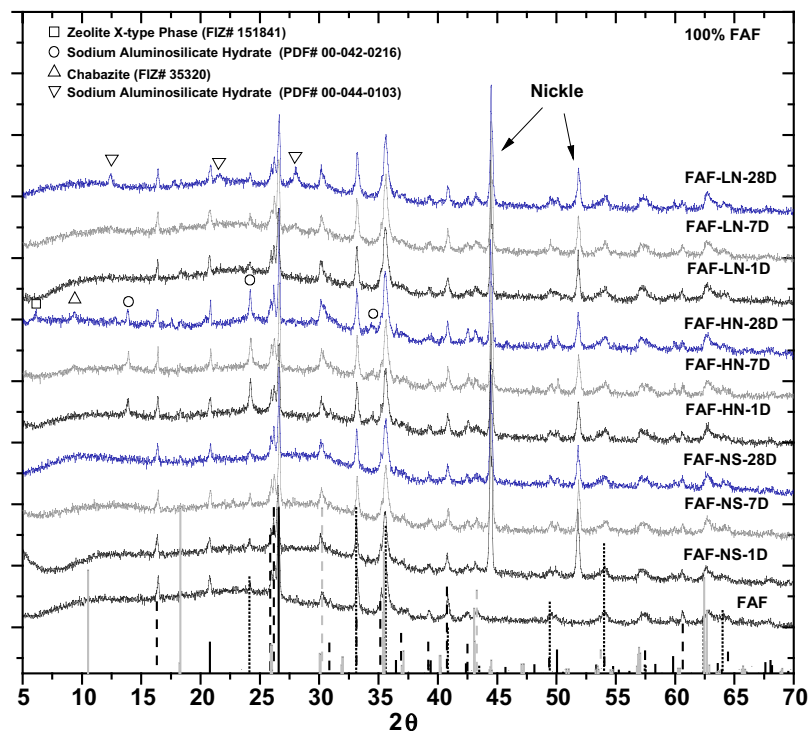


Fig. 2. X-ray diffraction patterns of pure alkali-activated FA-F with various types of activators at different ages (i.e. 1, 7, and 28 days). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

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