



# Physical–mechanical and microstructural properties of alkali-activated fly ash–blast furnace slag blends

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## Abstract

This paper investigated physical–mechanical and microstructural properties of alkali-activated binders based on blends of fly ash (FA) and blast furnace slag (BFS). FA–BFS blends were alkali-activated with sodium silicate (water glass) solution. The synthesis of alkali-activated binders was conducted at 95 °C during 24 h, with different FA–BFS mass ratios (100:0; 75:25; 50:50; 25:75; 0:100), different moduli of alkali activator ( $\text{SiO}_2/\text{Na}_2\text{O}$ : 0.5; 1.0; 1.5) and different alkali activator concentration (%  $\text{Na}_2\text{O}$ : 4; 7; 10). The influence of alkali activation conditions on the flexural and compressive strengths, setting time, drying shrinkage and the microstructure of synthesized binders was investigated. It was found that the compressive strength mostly depended on the composition of the FA–BFS blends and the water/binder ratio. The setting time highly depended on the activator concentration, while the drying shrinkage was mostly affected by the curing temperature. The blend comprising FA 25%–BFS 75%, activated with the activator of modulus 1.0 and the activator concentration of 10%  $\text{Na}_2\text{O}$  yielded optimal mortar regarding investigated physical–mechanical characteristics. Microstructural examination showed that the chemical composition of the binding phase varied as a function of the blend composition. Predominance of FA alkali reaction products in the binding phase positively influenced the flexural strength, while the predominance of BFS alkali reaction products positively affected the compressive strength. Optimal characteristics of alkali-activated binder were related to the following chemical composition of the binding gel:  $\text{Ca}/\text{Si}=0.34\text{--}0.50$ ,  $\text{Al}/\text{Si}=0.15\text{--}0.24$ ,  $\text{Mg}/\text{Si}=0.07\text{--}0.16$  and  $\text{Na}/\text{Si}=0.21\text{--}0.37$ . Empirical values of optimal gel composition could serve as a basis for tailoring the properties of alkali-activated binders based on different industrial waste precursor material.

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## 1. Introduction

Alkali-activated binders are getting increasing attention due to their excellent properties and environmentally friendly nature. These binders are formed by mixing the alkali activator solution with solid precursor, usually calcium silicate or aluminosilicate material. As activators, most often used are the solutions of alkali hydroxides and/or alkali silicates such as NaOH and  $\text{Na}_2\text{SiO}_3$ , while as solid precursors metakaolin, fly ash from thermal power plants, blast furnace slag and other materials can be used [1–5]. Among various industrial waste materials used

for alkali activation, the most popular choices and the most extensively studied are fly ash and blast furnace slag.

FA is a waste material generated by the coal combustion in thermal power plants. Binders obtained by alkali activation of FA (geopolymers) in general are known of having good strength, good durability in aggressive environments, low shrinkage and good thermal resistivity [3,6–8]. The main product of alkali activation of FA is alkaline amorphous aluminosilicate gel, or N–A–S–H gel (N— $\text{Na}_2\text{O}$ ; A— $\text{Al}_2\text{O}_3$ ; S— $\text{SiO}_2$ ; H— $\text{H}_2\text{O}$ ). However, the limiting factor for wider use of FA for geopolymer synthesis is its low reactivity and consequent low reaction rate and low strength gain when cured at room temperature [9–12]. The reactivity of FA in the reaction of alkali activation can be improved by its mechanical

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activation [13,14] as well as by the appropriate choice of the reaction conditions, of which the curing temperature is one of the most important [3]. Elevated curing temperature (above 60 °C) during alkali activation reaction accelerates the reaction rate, contributing to better strength gain in FA geopolymers [10,12,15–18].

On the other hand it is known that binders based on alkali-activated slag, a byproduct of pig iron production in steel plants, show high mechanical strength and good durability in corrosive environments [2,12,19–23]. The main product of alkali activation of slag is calcium silicate hydrate with Al in the structure, or C–A–S–H gel (C—CaO; A—Al<sub>2</sub>O<sub>3</sub>; S—SiO<sub>2</sub>; H—H<sub>2</sub>O). Along with high strength, binders based on alkali-activated slag are known of having some problematical properties such as poor workability, fast setting and substantial dry shrinkage [24–26]. In previous efforts to overcome these setbacks, some authors studied the effects of various admixtures (superplasticizers and shrinkage reducing admixtures) developed for Ordinary Portland Cement (OPC) binders, on rheological and physical–mechanical properties of binders based on alkali-activated slag [27–29]. It was observed that most admixtures used make no significant contribution to workability of alkali-activated slag paste. Moreover, workability improvement is usually achieved along with the decrease in mechanical strength [27,29]. According to Palacios and Puertas [30], prolonged mixing time can lengthen the setting and affect the shrinkage reduction in water glass-activated slag pastes. One of the most efficient ways for reducing the drying shrinkage is elevated curing temperature [31–33]. It was determined that after 6 h of curing at 70 °C, the shrinkage of Na-silicate activated slag concrete monitored within the period of 360 days, was similar to the shrinkage of OPC concrete (cured at room temperature) monitored within the same period [31]. Aydin and Baradan [32] managed to effectively reduce the shrinkage of Na-silicate activated slag mortar after autoclave curing at the temperature of 210 °C and pressure of 2.0 N/mm<sup>2</sup> for 8 h.

In recent times the synthesis of alkali-activated binders based on FA and BFS blends has become very attractive. Besides implying the use of different waste materials, joint activation of FA and BFS can counterbalance the disadvantages that each of the precursor materials exhibit when alkali-activated separately. Primarily, binders based on alkali-activated FA–BFS blends show better compressive strength comparing to the strength of sole alkali-activated FA. The dominant factor in the strength gain of the FA–BFS binders is the amount of BFS in the blend. Increasing the BFS content in the blend contributes to better compressive strength of paste [11,12,34–38], mortar [39–41] and concrete [42,43]. On the other hand, increasing the FA content in the blend usually affects the prolongation of the setting time and reduction of the drying shrinkage [11,38,39]. Kumar et al [11] studied the setting time and strength development of alkali-activated FA–BFS blends depending on the FA–BFS mass ratio. The longest initial setting time as well as the lowest compressive strength was observed in pure alkali-activated FA. The addition of BFS positively affected the strength improvement but caused a sharp decrease in the setting

time. Jang et al [38] studied the influence of polycarboxylate- and naphthalene-based superplasticizers on workability, setting and compressive strength of Na-silicate activated FA–BFS pastes. It was found that the addition of 2–4% of polycarboxylate superplasticizer affected the strength increase up to 7 days, but after this period, the decrease in strength was observed comparing to the samples without the addition of the superplasticizer. Chi and Huang [39] reported high compressive strength of Na-silicate activated FA–BFS mortar with 50% of BFS in the blend at the concentration of 6% Na<sub>2</sub>O, but the drying shrinkage considerably exceeded the drying shrinkage of OPC mortar used as the benchmark material. Several authors studied the reaction products of alkali-activated FA–BFS blends and reported that the reaction products depend on the blend composition and alkali activation conditions. Coexistence of N–A–S–H gel and C–A–S–H gel [12,19,44] as well as the formation of one hybrid binding gel [44] were reported.

Based on available literature it is obvious that there are considerable data referring to alkali activation of individual components (FA and BFS) and their blends as well. However, given that FA and BFS are industrial byproducts highly heterogeneous in composition, it is clear that there is no universal alkali activation procedure applicable. There are no data regarding the synthesis of alkali-activated FA–BFS binder with optimal properties concerning mechanical strength, setting time and drying shrinkage. High compressive strength is achieved mainly at the expense of the setting time, i.e. high shrinkage. Inversely, optimal setting time often implies low compressive strength. The correction of undesirable properties by the utilization of various admixtures can cause the strength decrease. In addition, there are no data regarding the synthesis of FA–BFS mortar at elevated curing temperature. Synthesis at elevated curing temperature could be of great importance for precast concrete manufacturing, providing sufficiently high early strength [31,32].

Therefore, the objective of this paper was studying the influence of wide range of alkali activation conditions (blend composition, activator modulus and activator concentration) on physical–mechanical and microstructural characteristics of synthesized binders based on FA and BFS blends and determining the optimal synthesis conditions and binder characteristics.

## 2. Materials and methods

### 2.1. Materials

In this study, the following materials were used:

1. FA Svilajnac from thermal power plant “Morava” – Svilajnac, Serbia
2. BFS from “Zelezara Smederevo” d.o.o. – Smederevo, Serbia
3. Portland-composite cement (CEM II/B-M (S-L) 42.5N) from cement plant “Holcim”, d.o.o. – Novi Popovac, Serbia

Chemical composition of FA and BFS is given in Table 1. Based on the chemical analysis and according to ASTM C618, FA sample used in this study belongs to class F.

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