



# The influence of fly ash characteristics and reaction conditions on strength and structure of geopolymers



Violeta Nikolić<sup>a,\*</sup>, Miroslav Komljenović<sup>a</sup>, Zvezdana Baščarević<sup>a</sup>, Nataša Marjanović<sup>a</sup>, Zoran Miladinović<sup>b</sup>, Rada Petrović<sup>c</sup>

<sup>a</sup> Institute for Multidisciplinary Research, University of Belgrade, Serbia

<sup>b</sup> Institute of General and Physical Chemistry, Belgrade, Serbia

<sup>c</sup> Faculty of Technology and Metallurgy, University of Belgrade, Serbia

## HIGHLIGHTS

- Effects of FA characteristics and reaction conditions on geopolymer strength were studied.
- Development of FA-geopolymer structure was studied using SEM/EDS and NMR analyses.
- The correlation of FA characteristics, structure and strength of geopolymer was given.
- Strength of geopolymers was correlated with a fraction of Al-rich structural units.
- Rapid test for assessment of FA reactivity was evaluated.

## ARTICLE INFO

### Article history:

Received 17 March 2015

Received in revised form 27 May 2015

Accepted 8 July 2015

### Keywords:

Fly ash  
Reactivity  
Geopolymer  
Compressive strength

## ABSTRACT

Reactivity of Class F fly ash (FA), development of strength, and structure of FA-based geopolymers, depending on the reaction conditions, were examined in this paper. The results of SEM/EDS and NMR analyses revealed that the composition of aluminosilicate gel changed during reaction, i.e. Si/Al atomic ratio decreased with the reaction time. Higher strength of geopolymers was associated with a higher fraction of aluminum rich structural units, higher crosslinking, and more compact structure. A rapid test for the assessment of FA reactivity and thus the applicability of FA for geopolymer synthesis was proposed.

© 2015 Elsevier Ltd. All rights reserved.

## 1. Introduction

Geopolymers represent an alternative to Portland cement due to similar or even better binding properties [1,2]. Geopolymers are formed by alkali activation of solid aluminosilicate materials, usually fly ash (FA), or metakaolin, while alkali hydroxides and/or alkali silicates are generally used as alkali activators [3,4]. The main product of the reaction is a highly cross-linked network structure in the form of an amorphous aluminosilicate gel [5–7].

Since FA represents industrial waste that can be found all over the world, it is particularly attractive for the synthesis of geopolymers. Despite the fact that research in this area is intense and there are a large number of publications that suggest a wide range of

applications of these materials, FA-based geopolymers are still far from practical applications on a large scale. One of the main reasons is the variability of FA composition, which differs from source to source, and even within the same source. On the other hand, a large number of research results, which analyze different synthesis conditions of geopolymers, make it difficult to clearly identify the key factors that determine reactivity of FA, as well as the structure and characteristics of geopolymers.

There are many factors that influence FA reactivity, and thus characteristics of FA-based geopolymers [8–12]. The most important factors are characteristics of the initial material (particle size distribution, content of glassy phase, reactive silicon, and aluminum, presence of iron, calcium, and inert particles), nature and concentration of the activator, as well as the reaction conditions [1–4,7]. Compressive strength of geopolymers is commonly used as a quantitative indicator of FA reactivity and characteristics of FA-based geopolymers [13,14].

\* Corresponding author.

E-mail address: [violeta@imsi.bg.ac.rs](mailto:violeta@imsi.bg.ac.rs) (V. Nikolić).

Particle size distribution is the key physical factor in the process of geopolymer synthesis [15,16]. FA reactivity increases with increasing FA fineness regardless of the nature and concentration of alkali activator [17]. Classification (extraction of fine fraction), milling, or mechanical activation of FA significantly increases its reactivity, resulting in geopolymers with improved properties [18–20].

Content of glassy phase of the initial material is an important parameter, since only a glassy phase represents reactive material that is converted during geopolymerization into the compacted binder [5,21,22]. Accordingly, FA reactivity or FA solubility in an alkaline solution depends on the composition and content of FA glassy phase [23]. Generally, higher glass content is associated with lower Si/Al ratio in the glassy phase [24], which means that higher amount of glassy phase contains higher amount of soluble aluminum. The relative amount of soluble silicon and aluminum present in an initial FA defines the Si/Al ratio in aluminosilicate gel, and thus the mechanical properties of resulting geopolymers [21]. However, as the term “reactivity” of FA does not only refer to the glassy phase, but to the FA as a whole, it means that the content of FA glassy phase has limited significance. Therefore, an assessment of FA reactivity is usually based on the FA dissolution rate in an alkaline solution, by measuring FA mass change or concentration of dissolved FA components over time [25–27].

Nature and concentration of alkali activator have dominant influence on the structure and properties of geopolymers [17]. If activation of FA is performed by alkali hydroxide, a gel rich in aluminum is first formed, followed by a gel rich in silicon [28,29]. However, in systems where the activation of FA is performed by solution containing high-ordered silicate species, a gel rich in silicon is formed immediately, without the previous formation of an aluminum-rich gel [30].

Properties of geopolymers are also determined by reaction conditions (temperature, time, and relative humidity), whereby an elevated reaction temperature leads to faster development of geopolymer strength [29]. There are opposing views on the optimal temperature for FA-based geopolymers synthesis. Some authors consider that optimal temperature of synthesis is 75–80 °C, since a significant occurrence of microcracks [31] or even strength loss above this temperature was observed [32–34]. On the other hand, very high strength (over 100 MPa) of geopolymers is achieved by alkali activation of FA at 95 °C [35]. Longer reaction time is generally beneficial for geopolymer strength increase [29]; however, after 48 h of reaction at elevated temperature the increase of strength is usually not significant [36]. Some authors consider that in order to achieve optimal properties of geopolymers at elevated temperature (95 °C), a shorter reaction time (6 h compared to 24 h) is more beneficial [37]. At last, but certainly not least, high relative humidity (>90%) is imperative for achieving optimal composition and structure of reaction products, as well as mechanical characteristics of geopolymers [35,38,39].

Since the FA presents industrial waste with variables characteristics, one of the main obstacles toward practical application of FA-based geopolymers is a lack of standards and evaluation criteria for FA reactivity in the process of geopolymerization. Besides, the factors that control phase development and their influence on the geopolymers characteristics are not yet fully understood. Given that there are a large number of influencing factors, it is clear that there is no simple methodology for assessing FA reactivity in the process of geopolymerization. The main aim of the present work was to evaluate rapid tests for assessment of FA reactivity and suitability of FA for geopolymer synthesis, by studying the development of strength and structure of geopolymers as the function of FA characteristics and geopolymerization reaction conditions (temperature and time). Rapid tests might be useful for practical application.

## 2. Materials and methods

### 2.1. Materials

In this study, FA samples from three thermal power plants (TPP) from Serbia were used:

1. FA Morava, TPP Morava, Svilajnac.
2. FA Kolubara, TPP Kolubara, Veliki Crljani.
3. FA Kostolac B1, TPP Kostolac B1, Kostolac.

Sodium silicate solution was used as alkali activator (“Galenika-Magmasil”, 8.72% Na<sub>2</sub>O, 26.5% SiO<sub>2</sub>, 64.78% H<sub>2</sub>O). Starting sodium silicate modulus  $n = \text{SiO}_2/\text{Na}_2\text{O}$  (mass ratio) of 3.04 was further adjusted by adding NaOH (p.a. (min.99%), VWR).

### 2.2. Characterization of the initial FA samples

It should be noted that in this study FA was used in its original form, as received from electrostatic precipitators, without classification (extraction of fine fractions) or removal of large, less reactive particles (predominantly quartz and char).

#### 2.2.1. Chemical, physical, and mineralogical characterization of FA

Chemical composition of FA samples was determined by classic chemical analysis – alkali melting. FA fineness was determined by sieving through meshes of 63 μm and 43 μm, according to Serbian standard SRPS B.C1.018, which refers to pozzolanic materials – constituents for cement production – classification, technical conditions and test methods.

Mineral composition of FA samples was determined by means of X-ray diffraction (XRD) analysis. Powder diffractometer “Phillips PW 1710” (Cu Kα = 1.54178 Å) was used. The diffraction patterns were recorded within 5–50° 2θ range, with a step of 0.02° and holding time of 1 s per step. For the identification of the crystalline phases, the software PCPDFWIN was used.

#### 2.2.2. Glassy phase of FA

Glassy phase content was determined by dissolving FA in 1% HF acid – Arjuan’s method [40]. 1% HF dissolves the glassy phase of FA, while crystalline phases (usually quartz, mullite, hematite, and magnetite) remain intact [5,21,25]. This method involves the treatment 1 g of FA with 100 ml of 1% HF for six hours with constant stirring. After the treatment with HF acid, FA samples were dried at 105 °C to constant mass. The content of glassy phase was determined by subtracting the residual mass from the initial one.

#### 2.2.3. Solubility of FA in a highly alkaline medium

FA solubility in a highly alkaline medium was determined using concentrated sodium hydroxide (NaOH) solution, whereby two different methods were used:

1. FA dissolution at room temperature (20 °C, 5 h in 10 M NaOH) [27].
2. FA dissolution at elevated temperature (95 °C, 0.5 h in 7 M NaOH).

It is well known that the dissolution process accelerates as the temperature increases. The experiment at elevated temperature (95 °C) was chosen not only to accelerate endothermic reaction of dissolution, but also because the temperature of 95 °C represented the maximum temperature of reaction during this investigation.

Prior to dissolution the FA was dried at 105 °C for 1 h. The liquid/solid (L/S) mass ratio was 40. The dissolution process was performed with constant stirring. Given that the high dissolutions are necessary to avoid undesirable precipitation [41], the resulting solution of FA dissolution in NaOH solution was further diluted 50 times and acidified with 37% HCl to a pH value of ~7.

Optical emission spectrometry with inductively coupled plasma (ICP-OES, Spectro Analytical Instruments GmbH) was used to determine the concentration of dissolved elements.

### 2.3. Synthesis of geopolymers

The synthesis of geopolymers was performed according to previously optimized procedure [17]. Sodium silicate solution with modulus 1.5 was used as alkali activator, whereby modulus was adjusted by adding (10 M) NaOH solution to the starting sodium silicate solution. Concentration of the activator was 10% Na<sub>2</sub>O with respect to the FA mass. Geopolymer mortars were prepared by mixing FA with alkali activator and water, and then with standard sand (EN 196-1). Water was added in the amount required to obtain equal consistency (mortar flow measured on a flow table was 125 ± 5 mm). Water/binder ratios (water represents the total amount of water in the system, including water from the activator, while binder represents the total mass of FA and a solid part of activator) in the case of mortar based on FA Morava, Kolubara, and Kostolac B1 were 0.46, 0.61, and 0.67

Download English Version:

<https://daneshyari.com/en/article/6720293>

Download Persian Version:

<https://daneshyari.com/article/6720293>

[Daneshyari.com](https://daneshyari.com)