

## The effect of alkali activator on the development of mechanical properties of fly ash based geopolymer

*The alkali activation of fly ash is a physical-chemical process of mixing fly ash with alkaline activators in order to produce materials with high mechanical properties - geopolymers.*

*Three types of geopolymers were synthesized. The same fly ash was used in all the samples, but the alkali activator has been changed: water glass (sample A), sodium hydroxide (sample B) and combination of water glass and sodium hydroxide (sample C). After preparation, all samples were thermally cured in the laboratory oven at the temperature of 85 °C for 24 hours. Mechanical strengths were measured after 3, 7 and 28 days, and during that period of 28 days all the samples were hermetically sealed and stored at room temperature.*

*The results obtained indicate that, geopolymer, synthesized by mixing fly ash with the combination of water glass and sodium hydroxide as alkaline activator, developed the highest compressive strengths during all period of interest, about 21.23 MPa at the age of 3 days and 21.28 MPa at the age of 7 days, which then reached 22.52 MPa after 28 days of curing at room temperature.*

**Key words:** geopolymers, alkali activator, compressive strengths

### INTRODUCTION

The alkali activation of fly ash (FA) is a physical-chemical process of mixing glassy component of FA with certain alkaline activators. The mixture is cured under certain temperature to make solid materials. The alkaline solution most often used is a sodium silicate solution, although potassium silicate solutions and other alkaline solutions have also been used (Palomo et al., 1999). The final structure and physical properties of FA based geopolymers depends heavily on the chemical composition of the starting material, nature and concentration of alkali activator. Palomo et al. also found that different fly ashes activated with NaOH cured at 85 °C for 24 h produced material with compressive strength between 35-40 MPa, and about 90 MPa if some water glass was added to the NaOH solution ( $\text{SiO}_2/\text{Na}_2\text{O} = 1.23$ ).

### MATERIALS AND METHODS

#### Materials

A type F (as defined in ASTM C618) fly ash from the Croatian power plant Plomin 2 (Plomin) was used in the present study. The chemical analysis of fly ash is given elsewhere (Mužek et al., 2012). Technical - grade sodium silicate solution (a type S, water glass,  $3\text{Na}_2\text{O} \times 3\text{SiO}_2$ ) and sodium hydroxide, p. a., (Kemika) were used as alkaline activator.

#### Instrumentation

The FTIR spectra of geopolymers were made on KBr pastille on a Perkin Elmer Spectrum One in the range from 4000 to 450  $\text{cm}^{-1}$ .

Toni Technik hydraulic press modular system was used for the compressive strength measurements.

#### Synthesis of geopolymers

Three types of geopolymers were synthesized. Certain amounts of fly ash were mixed with alkali activators. The same fly ash was used in all the samples, but the alkali activator has been changed: water glass (sample A), 16 M NaOH solution (sample B) and 16 M NaOH solution and sodium silicate solution (sample C). In samples B and C solution/ash ratio was 0.40. To provide good workability extra water was added. After adding all the components, geopolymer paste started to form. The mixture was mixed for 10-15 minutes and poured into polypropylene cylindrical containers along with constant stirring. Containers were hermetically sealed to prevent moisture evaporation. The fresh pastes were heat cured at 85 °C for 24 h in an oven. After 24 h, the geopolymer samples were removed from the oven and kept at room temperature for 28 days.

In the sample A, the fly ash was activated with a water glass solution to prepare paste specimens. In order to provide good workability, the solution/ash ratio was 0.72. Total water in system was only water from sodium silicate which itself contained  $w = 67.75$  % of water. No extra water was added. After mixing,

Author's address: University of Split, Faculty of Chemistry and Technology, Teslina 10/V, 21000 Split, \*muky@ktf-split.hr

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the fresh pastes were cast immediately into metallic moulds (4 x 4 x 16 cm) that were later kept in a hermetically sealed tin mould to prevent moisture evaporation. They were exposed to heat curing in a laboratory convection oven at 85 °C for 24 h. After removing from the oven, the alkali-activated fly ash (AAFA) specimens were covered with plastic to protect the samples from excessive water loss, and kept at room temperature for 28 days, respectively.

The compressive strength of all paste specimens prepared, was determined after 3, 7 and 28 days in accordance to the Croatian standard HRN EN 196-1:2005.

## RESULTS AND DISCUSSION

### FTIR analysis of geopolymer samples

The FTIR spectra for geopolymer system (sample C), as well as the spectrum for the original FA, are plotted in Fig. 1. These spectra reveal the differences between the original FA and geopolymeric materials formed. The main broad band at 1087.07 cm<sup>-1</sup> in the original FA, corresponding to asymmetric stretching vibrations of Si–O–Si and Al–O–Si (Vempati et al., 1994; Mollah et al., 1994) becomes sharper and shifts towards lower frequencies (~ 1023 cm<sup>-1</sup>) as a result of the formation of new reaction products associated with ongoing alkali activation.

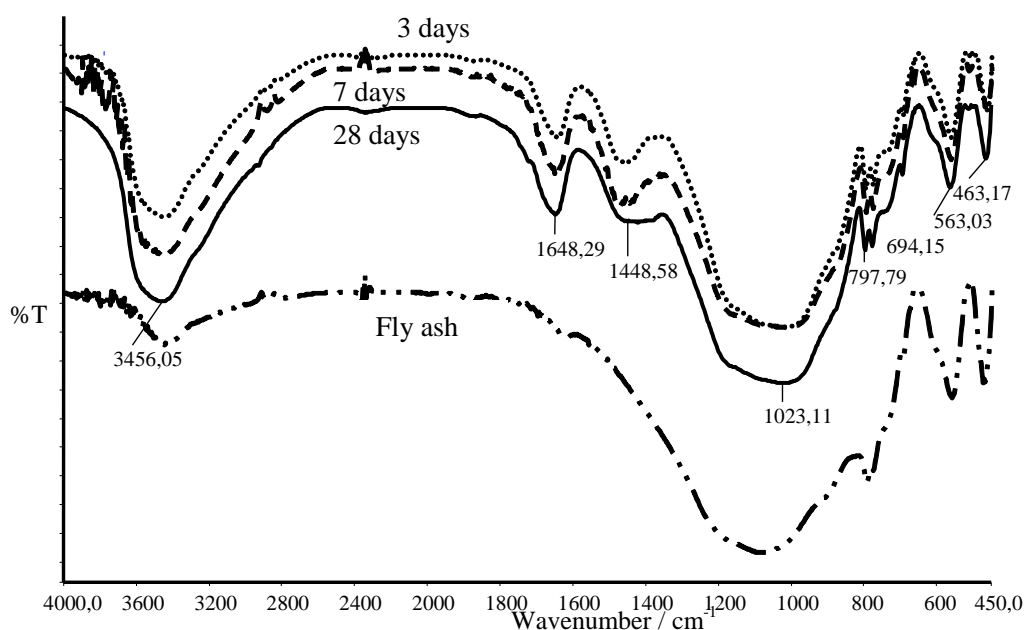


Figure 1 - FTIR spectra for the original FA and sample C after 3, 7 and 28 days of curing in the air at room temperature.

The bands located at ~ 795 cm<sup>-1</sup> and ~ 463 cm<sup>-1</sup> are ascribed to bending vibrations of Si–O–Si and O–Si–O bonds implying to the presence of quartz which is hardly affected by alkaline activation of FA (Bakharev, 2005; Lee & van Deventer, 2002). The bands located at ~ 694 cm<sup>-1</sup> and ~ 563 cm<sup>-1</sup> are specifying the presence of mullite.

In all geopolymeric materials, new bands appeared in the regions of ~1650 cm<sup>-1</sup> and ~ 3456 cm<sup>-1</sup> that were attributed to bending vibrations (H–O–H) and stretching vibrations (–OH, H–O–H). Water is needful for process of geopolymerisation as it implicates the destruction of solid particles and the hydrolysis of dissolved Al<sup>3+</sup> and Si<sup>4+</sup> ions. Bond at ~ 1450 cm<sup>-1</sup> assigned to the stretching vibrations of O–

C–O bond occurred in all AAFA samples implying to the presence of the sodium bicarbonate.

This observation of changes in the FTIR spectra of the AAFA materials indicate that the geopolymerisation reaction occurred leading to the formation of the main reaction product, an amorphous aluminosilicate gel in all samples examined.

### Effect of alkali activator on development of mechanical properties

The compressive strength, of all geopolymer pastes formed, was determined after 3, 7 and 28 days of curing in the air at room temperature. The values of compressive strengths measurements increased over the whole period of interest, as it can be seen from the Fig. 2. The sample C showed maximum

compressive strength during all period of interest, about 21.23 MPa at the age of 3 days and 21.28 MPa at the age of 7 days, which then reached 22.52 MPa after 28 days of curing at room temperature.

The increase in the 28-days compressive strength measurements was also found in the samples A and B, although, their values are significantly lower than those obtained for sample C.

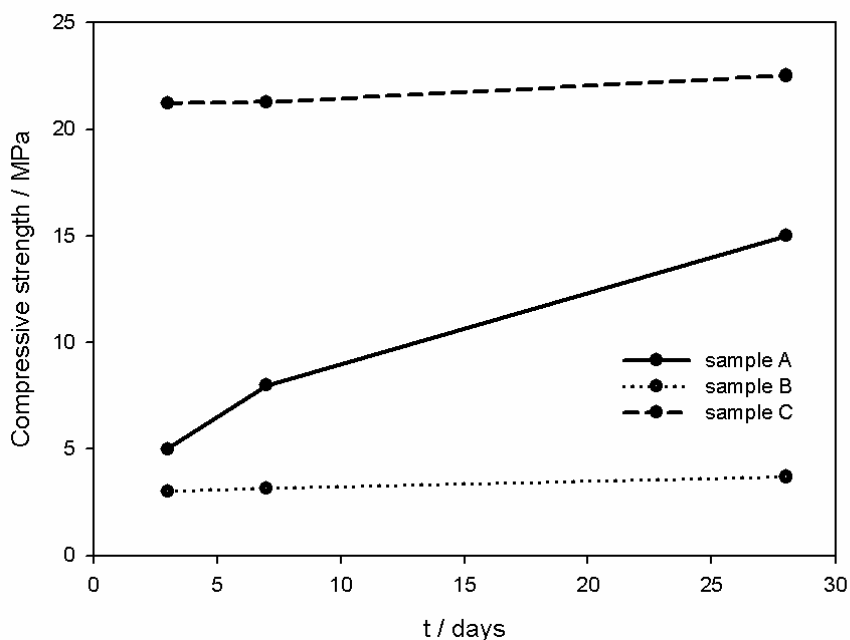


Figure 2 - Development of the compressive strengths for all the samples during the period of 28 days.

The mixture of sodium hydroxide with sodium silicate solution was found to affect significantly the compressive strength of geopolymers. Similar effect was found by Panias et al. (2007). According to the authors, sodium hydroxide acts on the dissolution process, as well as on the bonding of the solid particles in the final structure. Sodium silicate solution controls the soluble silicate concentration and the predominant silicate species in the geopolymeric system, improving the mechanical strength of the produced materials.

## CONCLUSIONS

The geopolymerisation reaction occurs in all alkali activated samples prepared. The usage of alkali activator reflects on the values of compressive strength measured during all period of interest.

The sample C showed maximum compressive strength, 22.52 MPa, after 28 days of curing at room temperature. It is evident that the mixture of sodium hydroxide with water glass as alkaline activator is needful to gain geopolymeric materials with higher compressive strength that can find their applications as construction materials.

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## IZVOD

### UTICAJ ALKALNOG AKTIVATORA NA RAZVOJ MEHANIČKIH KARAKTERISTIKA LETEĆEG PEPELA NA GEOPOLIMER

*Alkalna aktivacija letećeg pepela predstavlja fizikalno-kemijski proces miješanja letećeg pepela sa alkalnim aktivatorima kako bi se proizveo materijal visokih mehaničkih čvrstoća - geopolimer.*

*Sintetizirane su tri vrste geopolimera. Kod svih sinteza koristio se isti leteći pepeo, dok se mijenjala samo vrsta alkalnog aktivatora: vodenog stakla (uzorak A), natrijev hidroksid (uzorak B) i kombinacija vodenog stakla i natrijevog hidroksida (uzorak C). Nakon sinteze, svi uzorci su podvrgnuti toplinskoj obradi u trajanju od 24 sata u laboratorijskoj peći na temperaturi od 85 °C. Mehaničke čvrstoće su testirane nakon 3, 7 i 28 dana njegovanja. U tom periodu od 28 dana svi uzorci su hermetički njegovani pri sobnoj temperaturi.*

*Geopolimer sintetiziran miješanjem letećeg pepela sa kombinacijom vodenog stakla i natrijevog hidroksida, kao alkalnog aktivatora, je razvio najviše mehaničke čvrstoće tijekom cijelog perioda ispitivanja: 21.23 MPa nakon 3 dana, 21.28 MPa nakon 7 dana i, konačno, 22.52 MPa nakon 28 dana njegovanja pri sobnoj temperaturi.*

***Ključne riječi:*** geopolimeri, alkalni aktivator, mehaničke čvrstoće

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