

THE EFFECT OF FLY ASH/ALKALINE ACTIVATOR RATIO IN CLASS F FLY ASH BASED GEOPOLYMERS

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Abstract

This study focused on the effect of fly ash/alkaline activator ratio and Na₂SiO₃/NaOH ratio in class F based geopolymers. The objective of this study is to observe the variation of the compressive strength of the final product depending the solid/liquid ratio. In order to form the geopolymer, various parameters were changed during the geopolymerisation process. The fly ash/alkaline activator ratio studied was: 1.5; 2.0; 2.5. The geopolymeric pasta was heated up to 400 °C, 600 °C and 800 °C. After heating the compressive value of geopolymer, with the fly ash and alkaline activator ratio equal to 2 and Na₂SiO₃/NaOH ratio of 2.5, decreased with approximative 40 MPa comparative with the value of the sample obtained from the same composition dried for 7 days at normal temperature.

Keywords: geopolymers, fly ash, fly ash/alkaline activator ratio, Na₂SiO₃/NaOH ratio.

Introduction

Geopolymers are a class of ceramic materials total inorganic [1], based on alumina and silica, which are chemical balanced by alkaline ions from group I (Li⁺, Na⁺, K⁺) [2]. Thus materials are rigid gels, obtained in normal boundary condition or cured at high temperature [3] and it can be converted in crystalline materials or glass [4]. Any geopolymer can be splitted into two main constituents, the base material and the activator (an alkaline liquid). The major constituent is the base material, it must be rich in silicon and aluminum, it can be a natural mineral, such as clays [5-7], kaolin [8-10], or waste, such as ash [11], slags [12] etc.

Ash is a secondary product derived from coal combustion in power plants. From burning of the coal sprayed in the combustion chambers, carbon and volatile materials are produced. However, some impurities of clay, quartz, etc. merge in exhaust gases and when discharged from the burners they are captured in the gas filters. With the cooling of the exhaust gases, the fused materials solidify in glass spherical particles called fly ash [13]. Due to the fusion-in-suspension, fly ash particles are generally solid spheres and tubular ecosystems with some globe-shaped particles that contain smaller spheres.

The chemical and mineralogical composition depends to a large extent on the composition of coal, given that there are many types of coal (anthracite, bituminous lignite or

sub-bituminous lignite) burnt in many stations, the properties of this ashes can be very different depending on source and collection method [14].

According to the standard of natural calcined or non-calcined pozzolanic materials (ASTM C618), there are two categories of fly ash. [15] (Table 1).

In the furnace firing zone, coal burning produces heat up to 1500 °C. At this temperature, inorganic minerals (quartz, calcite, gypsum, pyrite, feldspar, etc.) are combined in small liquid drops. These drops are evacuated from the furnace's combustion chamber together with exhaust gases. Once leave out the firing zone, these drops get a spherical shape of glass particle. Fly ash is collected from the gases evacuated by mechanical and electrostatic precipitators. Bottom ash can also be used to make geopolymers [16].

Table 1. Types of ashes according to ASTM C618 standard

Class	Description according ASTM C618	Chemical requires
F	Fly ash produced by burning bituminous coal or anthracite that meets the chemical condition. This ash class has pozzolonic properties.	$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \geq 70\%$
C	Fly ash produced by the combustion of sub-bituminous coal or lignite that meets the chemical condition. This class has, besides pozzolonic properties, cementitious properties. *)	$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \geq 50\%$

*) Some class C ashes may contain more than 10% calcium oxide.

Experimental

The fly ash used in the experiment belongs to the ASTM C618 class F class because the sum of the major oxides percent exceeds 70%, according to equation (1) established from the data in table 2:

$$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 = 83.09\% \quad (1)$$

Table 2. Fly Ash Composition (weight percents)

Oxide	Percentage (%)	Oxide	Percentage (%)
SiO ₂	52.11	Na ₂ O	0.42
Al ₂ O ₃	23.59	K ₂ O	0.80
Fe ₂ O ₃	7.39	P ₂ O ₅	1.31
TiO ₂	0.88	SO ₃	0.49
CaO	2.61	MnO	0.03
MgO	0.78	LOI	9.59

LOI - loss on ignition

For a better description of the raw material used, a XRD analyze was performed, the results are presented in the picture below (fig. 1).

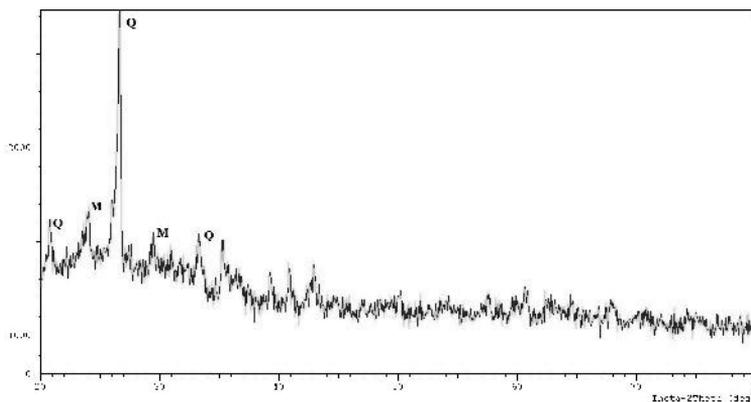


Fig. 1. X Ray Pattern (XRD) for raw Fly Ash, Q = quartz, M = mullite

Table 3. Mix Design Details for Various Ratios of Fly Ash/Alkaline Activator and Na₂SiO₃/NaOH

Fly ash/ Alkaline Activator Ratio	Na ₂ SiO ₃ /NaOH Ratio	Fly Ash (g)	Na ₂ SiO ₃ (g)	NaOH (g)
1.5	0.5	505	115	225
	1.0		170	170
	1.5		205	135
	2.0		225	115
	2.5		240	95
	3.0		255	85
2.0	0.5	565	95	190
	1.0		140	140
	1.5		170	115
	2.0		190	95
	2.5		200	80
	3.0		210	70
2.5	0.5	605	80	160
	1.0		120	120
	1.5		145	95
	2.0		160	80
	2.5		170	70
	3.0		180	60

Results and Discussion

At normal temperatures, the reaction rate of the pozzolonic material is lower than the hydration rate of the cement, so fly ash-based concrete must be **cured** under "special" conditions in order to benefit of all the properties of this compound. When using a very large amount of ash in a concrete, it is recommended that the curing time to be at least 7 days. If it is desired to decrease the curing temperature, other additions may be added to the material.

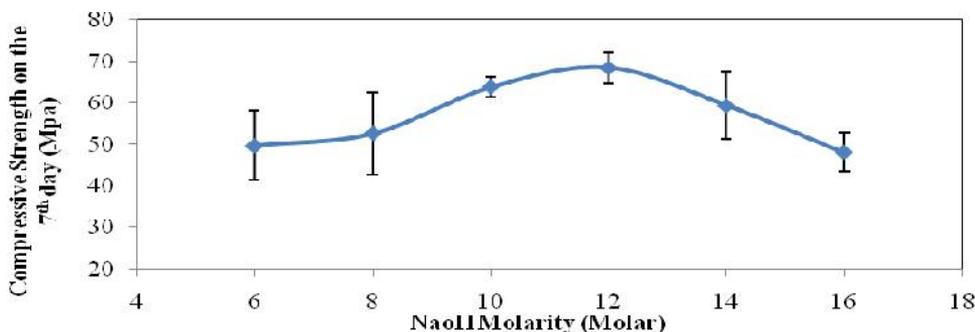


Fig. 2. Compressive Strength of Various NaOH Molarities

The compressive strength (Fig. 3) of the geopolymeric composites is closely related to the amount of activator used as well as its concentration. For the activated geopolymer with a ratio of 2.5 $\text{Na}_2\text{SiO}_3/\text{NaOH}$ of 1.5%, the compressive strength obtained after 7 days was about 40 MPa, for 2% of about 70 MPa, and for 2.5% of about 55 MPa.

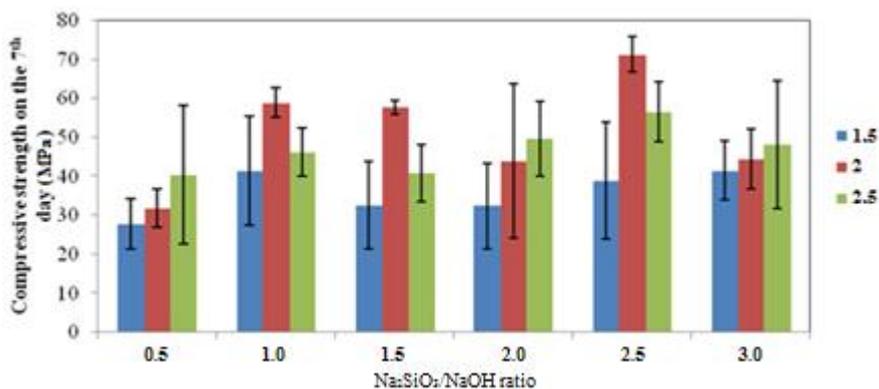


Fig. 3. Compressive Strength of Various Proportions of Reactants

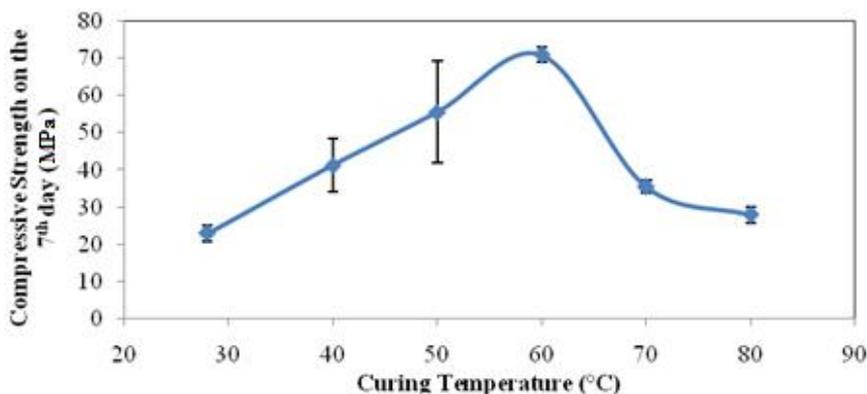


Fig. 4. Compressive Strength for Various Curing Temperatures (°C)



Fig. 5. The optimum Fly ash-based geopolymers with optimum compressive strength of 8.61 MPa, Fly Ash/Activator ratio= 2.0 and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio = 2.5, after heat treatment at 400°C, 600°C, and 800°C

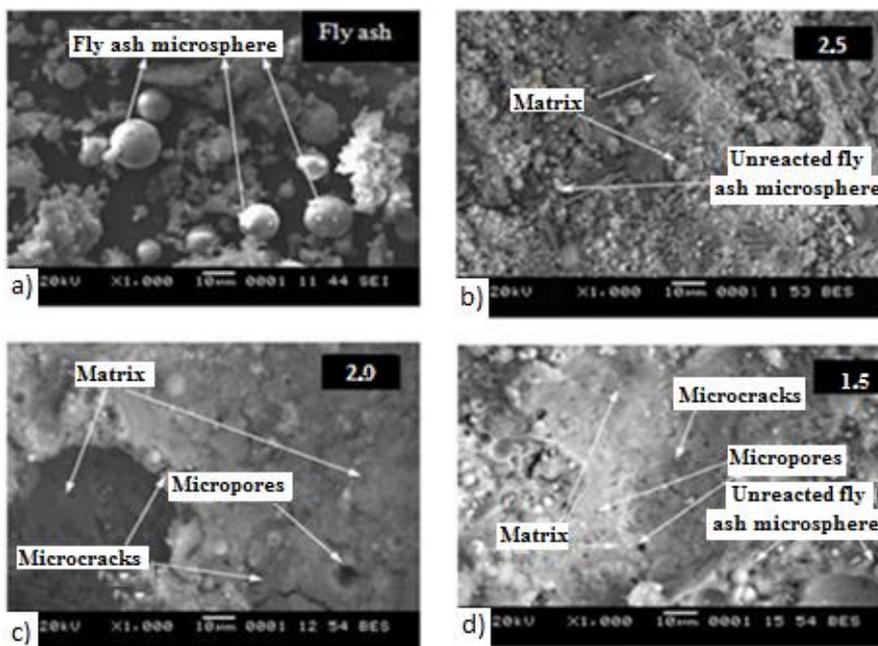


Fig. 6. SEM micrographs of the original a) fly ash and the synthesized geopolymers at activator/fly ash ratios of b) 2.5, c) 2.0, and d) 1.5 and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ solution ratio of 2.5

Heating of the geopolymeric paste at high temperature has a negative effect on compressive strength, the optimal value being only 8,61 MPa for the geopolymer with the ratio of fly ash to activator 2.0 and the ratio of $\text{Na}_2\text{SiO}_3/\text{NaOH}$ of 2.5, heated at 400 °C, 600 °C and 800 °C (Fig. 7).

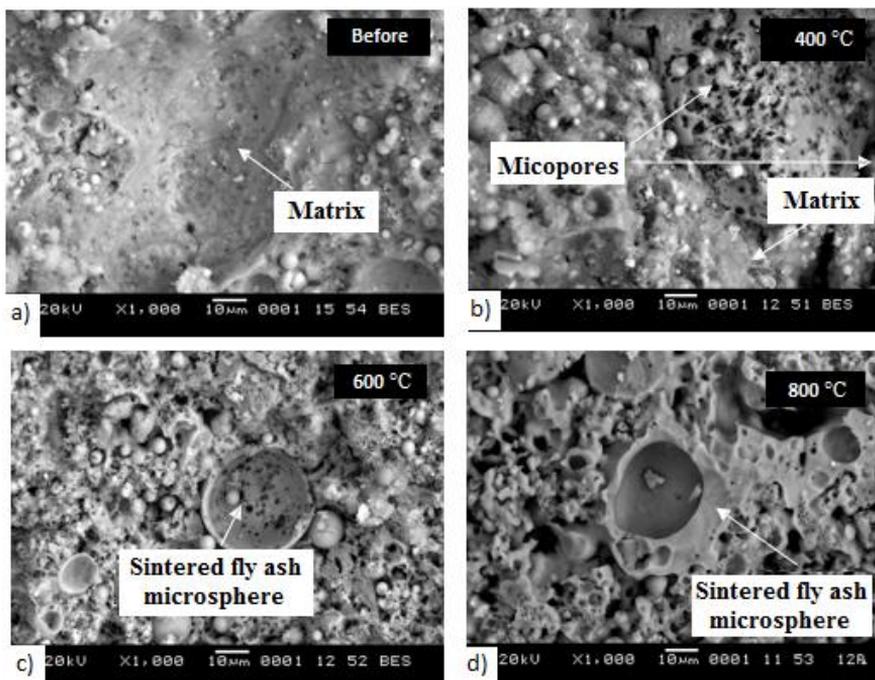


Fig. 7. SEM micrographs of fly ash-based geopolymers,
a) before heat treatment and after heat treatment at b) 400 °C, c) 600 °C and d) 800 °C.

Conclusion

The properties of the final geopolymer are directly depending of: Si/Al ratio, base material, particle dimensions, alkaline activator concentration, calcium quantity, time of synthesizing/hardening and geopolymerization temperatures.

According to our study, after heating, the compressive strength value of geopolymer, with the fly ash and alkaline activator ratio equal to 2 and $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio of 2.5, is decreasing with approximate 40 MPa, comparative with the value of the sample obtained from the same composition cured for 7 days at normal temperature.

The value of compressive strength after 7 days for the geopolymer with fly ash/alkaline activator ratio 2 was (i) 60 MPa for 1.5 $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio, (ii) 40 MPa for 2.0 $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratio and (iii) 70 MPa for 2.5 $\text{Na}_2\text{SiO}_3/\text{NaOH}$.

Curing at high temperature has a negative effect on compressive strength, the optimal value obtained was 8.61 MPa.

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