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Mechanical properties of alkali activated geopolymer paste using different Romanian fly ash sources – experimental results

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Abstract. As concrete demand is constantly increasing in recent years and also considering that cement production is both a consumer of natural resources and a source of carbon dioxide release into the atmosphere, there have been worldwide investigations into green alternatives for making concrete environmentally friendlier and simultaneously to satisfy the development of infrastructure facilities. The use of fly ash as a component of cementitious binders is not new but when considering the specific case of alkaline activation and fly ash representing the only source for the binder formation, it necessitates a more complete understanding of its specific reactions during the alkaline activation process. Since the fly ash varies dramatically, not only from one source to another, but also from one batch to another even when provided by the same power plant, its chemistry in obtaining alkali-activated materials during the geopolymerisation process and the final mechanical properties are considered crucial for the performance of geopolymer concrete. This paper will provide a review of the experimental results concerning the physical and mechanical evaluation of the alkali-activated fly ash-based geopolymer materials, developed with different types of fly ash, for a better understanding of geopolymer concrete production control.

1 Introduction

Concrete is the second most widely used material in the world, being considered the most versatile, durable and reliable building material - therefore the need to produce large quantities of Portland cement (PC) as binder for concrete are constantly increasing. The cement industry is considered to be the second highest carbon dioxide generator, being preceded only by the automotive industry. Each tonne of produced cement requires between 60 and 130 kg of liquid fuel or its equivalent, depending on the type of cement and the manufacturing process used, and approximately 110 KWh of electricity [1]. One tonne of produced cement releases between 0.8 and 1.1 tonnes of CO_2 into the atmosphere, as a subsequent consequence of combustion and calcareous calcination [2]. Only in 2014, the world cement production represented approximately 4.2 billion tonnes [3], indicating an obvious increase related to the previous years.

Environmental problems associated with ordinary Portland cement production are extremely well known and they represent a problem which is carefully monitored, in terms of the amount of carbon dioxide released into the atmosphere during its production. Constant efforts are therefore made in order to counteract this effect in future years. As the industry is constantly growing, the involvement of modern building materials in the economic world is more than necessary.
Alkaline activation of the fly ash represents a special procedure, able to generate the solidification of fly ash powder when mixing it with a certain type of alkaline activator and creating a new binding material. Consequently, it has opened the perspective of obtaining a new building material when incorporating aggregates, as an alternative to traditional concrete and cement-based composites [4-7]. Developing these types of composites, with similar or even superior, properties to cementitious composites, without using cement in the composition, creates an extraordinary opportunity for the environment and for the construction industry, constituting itself as an alternative to traditional technology, both due to mechanical properties and high resistance in aggressive environments [8].

The large amount of fly ash resulting from the energy industry in Romania can create new opportunities to use this waste as a substitute for Portland cement in the production of new materials. In 2017 only, one power plant in Romania produced nearly 650,000 tonnes of fly ash, 50,000 tonnes of slag and 50,000 tonnes of gypsum. Despite the slag recovery by recycling means its reuse into the coal supply of boilers (43,000 t/year) and the fly ash delivery to the construction industry (cement, concrete, mortar for stabilizing soils, roads etc.), representing approximately 162,000 t/year, the unused

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quantity of fly ash from the power plant is still an important environmental problem, with almost 565,000 t/year stored in waste dumps [9].
When fly ash is used as a main binding material in the production of alkali activated materials, a careful consideration is needed when choosing the primary materials used for the mix. Chemical properties of the components have to be taken into consideration so that the geopolymerisation reaction can properly occur [10]. The solid part (fly ash) and the liquid part (alkaline activator) in the system should not be considered as additions, as they represent the main constituents of the binder [11].

The physical, chemical and mechanical properties of the alkali-activated geopolymer materials are strongly influenced by the properties of raw material such as fly ash [12, 13]. Since the properties of fly ash vary, depending on several parameters (the quality of coal, temperature and process of combustion etc.) [12-15], understanding the mechanisms that generate the geopolymerisation process have to be well known.

The aim of this paper is to present the preliminary results obtained on alkali-activated fly ash-based geopolymer paste using different Romanian power-plant source materials for producing this type of material, based on efficient source materials, available at national level and also presenting the effect of their physical and chemical properties on the compressive strength of the binder.

2 Experimental method – materials and methods

This chapter summarizes both the state-of-the art information about the raw components used for the development of alkali-activated fly ash-based geopolymer pastes (FAGP) and also about the applied particular experimental methodology (mix design, moulding methods, curing and also testing methods).

2.1 Materials

2.1.1 Fly ash

Fly ash (FA) is a by-product obtained by electrostatic or mechanical precipitation of the pulverized particles resulting from the coal-fired combustion gases of the furnaces in power plants. It is a fine powder, consisting mainly of spherical shaped glass particles [16, 17]. Although some certain characteristics proved to be common for all fly ash types, due to factors like coal type as raw material for FA production and the burning process as well, fly ash powders show significant differences when coming to their chemical composition. The raised variation of the chemical composition, leading to different performance of FA as binding material, when used as primary raw material for producing alkali-activated fly ash-based binding paste (FAGPP), represents an essential aspect for the general development of the concept [18].

For the current research, Class F fly ash was used for preparing the FAGPP specimens and was characterized as follows [19]: $SiO_2 + Al_2O_3 + Fe_2O_3 > 70\%$, $SO_3 < 5\%$ and L.O.I. <6%.

Two different Romanian power plant, low-calcium, fly ashes (FA.S1 and FA.S2) were used in this study (Fig. 1a and Fig. 1b), focused on the development and implicitly, the comparative analyses of FAGPP mechanical properties [20]. The chemical composition of the FA samples was established by X-ray fluorescence analysis (XRF analysis), presented in Table 1. Also their relevant physical properties are presented in Table 2.

a)  b) 

Fig. 1. a) FA.S1 sample b) FA.S2 sample

Table 1. XRF analysis data for the fly ash samples.

Oxides	FA.S1 %	FA.S2 %
SiO_2	53.61	54.32
Al_2O_3	26.16	22.04
Fe_2O_3	7.58	9.02
CaO	2.42	5.85
MgO	1,49	2,48
SO_3	0.26	0.20
Na_2O	0.59	0.54
P_2O_5	0,12	0,16
TiO_2	1.04	0.86
Mn_2O_3	0,08	0.06
L.O.I.	3.57	3.05

Table 2. Physical properties of fly ash samples

Properties	FA.S1	FA.S2
Colour	Light grey	Greyish white
Residue retained on 45 μm, %	37.6	59.4

2.1.2 Alkaline Activator

When in contact, the reactive solids from the fly ash and the alkaline solution react and a hard, water-resistant material is generated due to the formation of an alumino-silicate network [21] and geopolymerisation occurs, resulting from the dissolution of the Si and Al compounds of the fly ash [22].

By using an alkaline hydroxide activating solution and a silicate solution, the SiO_2 / Al_2O_3 and Na_2O / SiO_2 ratios in the mixture are optimized to obtain higher mechanical performances [23, 24].

The most commonly used combination for the production of the alkaline activator is sodium hydroxide (NaOH) or potassium hydroxide (KOH), together with a sodium silicate solution ($nSiO_2Na_2O$) or a potassium silicate solution ($nSiO_2K_2O$) [25]. Waterglass adds silicon atoms and mixed liquid, both of which play an important role in producing the geopolymerisation process.

It is recommended to use sodium hydroxide of purity ranging between 94 and 99%. Regarding the sodium hydroxide solution, it should be prepared 24 hours before use, but not earlier than 36 hours because it loses its properties as an activator [26]. Sodium hydroxide concentration has an important influence on the final mechanical properties of the alkali-activated fly ash-based geopolymer material [27, 28].

The alkaline activator used for this experimental study was a combination of sodium hydroxide solution (NaOH) and sodium silicate solution (Na_2SiO_3), as shown in Fig. 2a and Fig. 2b. The sodium silicate solution was purchased from the local market and its chemical composition was SiO_2=30%, Na_2O=14% and H_2O=56%. The sodium hydroxide solution was prepared by dissolving the NaOH flakes into water until the desired concentration of the solution was achieved. Two distinct NaOH solutions, of 10M and 12M respectively, were prepared for FAGPP mixes. The 10M NaOH solution was prepared by dissolving 400g of NaOH flakes into water for 1 litre solution (40g x 10 = 400g NaOH/litre, where 40g is the molecular weight of NaOH) [20].

Fig. 2. a) NaOH flakes b) Na_2SiO_3 solution

2.2 Mix design of FAGPP

Producing FAGPP does not imply a different technology than in the case of conventional mortars, the use of a pallet mixer is convenient for a proper mix. Preliminary results obtained on FAGPP, when using Romanian local raw materials, indicated that an alkaline activator/fly ash ratio (AA/FA) of 0.5 for FA.S1 and 1.0 for FA.S2 of fly ash provided good workability [29]. The Na_2SiO_3/NaOH solution ratios ranged between 0.5 and 2.5, for a comparative analysis regarding the variation effect on the material behaviour. Table 3 summarizes the fly ash-based geopolymer paste mixes developed in the current experimental work.

The FA and AA were mixed together for three minutes, until a homogeneous paste was obtained. The mixtures were then placed in 40 mm x 40 mm x 160 mm moulds and heat cured at 70°C for 24 hours. A glass film was placed on top of every mould in order to prevent excessive water release from the mixtures. After demoulding, the FAGPP specimens were stored in the climatic chamber at the temperature T (20±1)°C and relative humidity RH (60±5)% until the age of 7 days, when their mechanical performance was tested. Fig. 3 shows aspects related to the preparation of the FAGPP samples.

Table 3. Mix design of FAGPP.

FA type	AA / FA ratio	Na_2SiO_3 / NaOH ratio	NaOH molarity
FA.S1	0.5	0.5	10M and 12M
		1.0	
		1.5	
FA.S2	1.0	2.0	
		2.5	

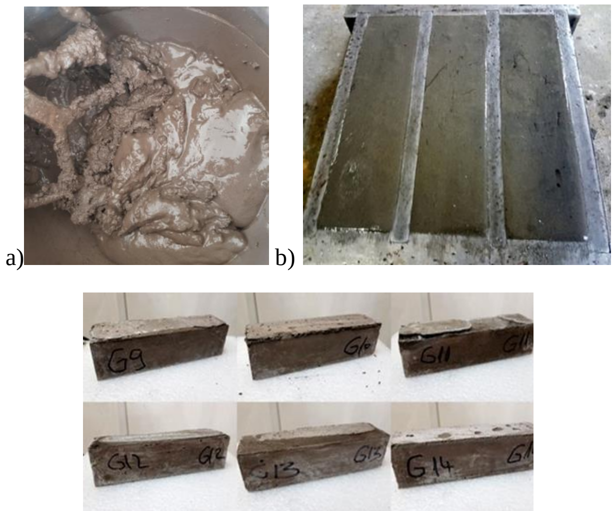

Fig. 3. a) Final mixing sequence b) Moulding c) FAGPP specimens after de-moulding

2.3 Testing method of FAGPP samples

The FAGPP compressive strength testing (as shown in Fig. 4) was performed at the age of 7 days, using three prismatic specimens for each type of alkali-activated fly ash-based geopolymer paste; the mean value of the results was considered relevant for the data interpretation. The testing method was in accordance to EN 196-1:2006, namely the standard testing methodology for ordinary Cement mortars. Early age testing at 7 days was considered relevant for the comparative evaluation, as previously experiments proved that generally the FAGPP reach most of their compressive potential by this age [20].

Fig. 4. FAGPP samples after compressive test

3 Results and discussions

Alkali-activated fly ash-based geopolymer materials properties can be notably influenced by several important factors such as the source and quality of the fly ash used as the precursor material, the NaOH solution concentration and the Na_2SiO_3/NaOH solution ratio. The physical and chemical properties of the fly ash samples, as well as the different ratios of alkaline liquid ratios, at the age of 7 days, and FAGPP compressive strength were consider relevant for the comparative evaluation of the above nominated parameters.

3.1 Fly ash parameters

The oxide composition of the two samples of fly ash were analysed by XRF analysis and are listed in Table 1. Based on literature [30], both samples of fly ash are categorized as Class F fly ash, because the amount of SiO_2, Al_2O_3 and Fe_2O_3 is more than 70% (Table 4). A high difference occurs in CaO content, where FA.S2 has a higher percentage than FA.S1. The literature suggests [31] that a low content of CaO in the chemical composition of the fly ash leads to better compressive strength of the material.

Table 4. Class F compliance of the FA samples.

FA	$SiO_2 + Al_2O_3 + Fe_2O_3$ %
FA.S1	87.35
FA.S2	85.38

The reactivity of fly ash samples highly depends on the LOI, which represents the unburned material [31, 32]. Both samples of fly ash have LOI less than 6%, as recommended by standards [30] and also less than 5%, as recommended by literature [31]. The fineness of the fly ash is defined as the percentage of particles retained on the 45µm mesh sieve. As shown in Table 2, FA.S1 has a lower percentage of retained particles on the mesh sieve (37.6%), therefore it has a higher fineness.

Although the chemical composition and the L.O.I. parameters of the fly ash samples used (FA.S1 and FA.S2) are only slightly different, as shown in Table 1, it can be easily noticed (Table 3) that the FAGPP mix design for each FA sample is significantly different. In order to achieve the desired workability and to obtain a proper geopolymerisation process, the alkaline activator/fly ash ratio for FA.S1 was 0.5 and for FA.S2 was 1.0. The FAGPP samples prepared with FA.S2 showed lower compressive strength values for all the Na_2SiO_3/NaOH ratios and for both, 10M and 12M NaOH solution concentration. the fineness of the two fly ash samples being different (Table 2), shows that this parameter plays an important role in the workability of the mix and further on, in the compressive strength development of the alkali activated material (Fig. 5 and Fig. 6): – the finer the fly ash, the better the workability of the mix, therefore, a lower alkaline activator/fly ash ratio.

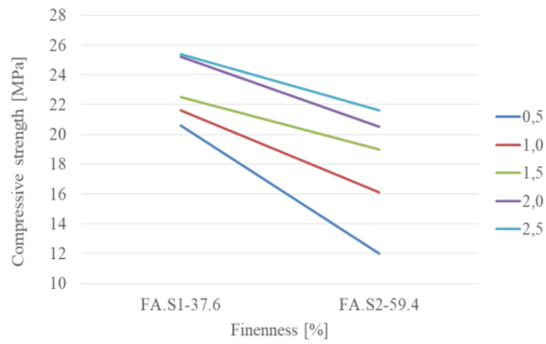

Fig. 5. Effect of fineness to compressive strength (10M NaOH solution – 0.5 to 2,5 alkaline activator ratio)

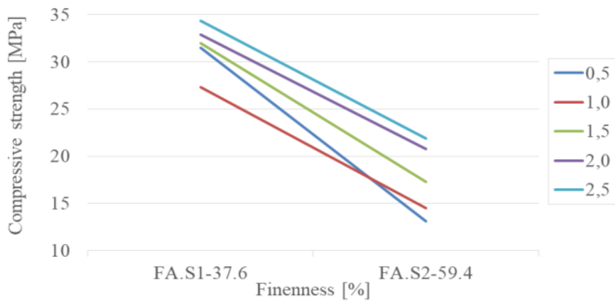

Fig. 6. Effect of fineness to compressive strength (12M NaOH solution – 0.5 to 2.5 alkaline activator ratio)

The influence of fly ash fineness (in terms of particles retained on the 45µm sieve) on the compressive strength is graphically emphasised in the figures above. The graphs indicate that the compressive strength decreases as the percentage of particles retained on the 45 µm sieve increases, therefore the fly ash sample with a lower fineness generates lower compressive strength values. A low percentage of particle fly ash retained on the sieve shows higher specific surface area, therefore a higher reactivity of the fly ash and subsequently, an increase in the compressive strength of the FAGPP.

The decrease in the amount of fly ash retained on the 45 µm sieve causes a decrease of the final pore size of the binder and also of the total porosity, with an increase of the density of the material and, therefore, a higher final compressive strength. This happens because fly ash is more reactive as smaller particles, compared to bigger ones, in its physical composition [33,34,35].

3.2 Influence of the Na_2SiO_3/NaOH ratio and the NaOH solution concentration on the compressive strength

The effect of sodium silicate solution to sodium hydroxide solution (Na_2SiO_3/NaOH ratio), by mass, on the compressive strength of the FAGPPs specimens has been observed by analysing it for each of the molar concentrations of sodium hydroxide solution and for each fly ash sample, FA.S1 and FA.S2. The results are graphically presented in Fig. 7 and Fig. 8, both proving that the best compressive values were obtained for the Na_2SiO_3/NaOH ratio of 2.5 (for both samples of fly ash). For the mixtures prepared using FA.S1, the ratios that induced lower compressive strength were generally generated by a lower geopolymerisation reaction during the mixing of the components. The compressive strength evolution strongly depends the complexity of the geopolymerisation reaction and the concentration of the alkaline activator constituents.

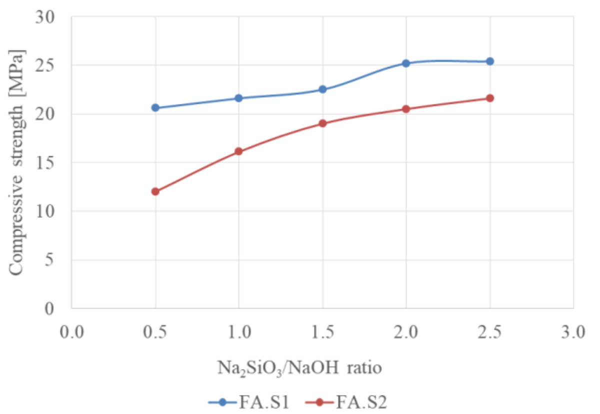

Fig. 7. Effect of Na_2SiO_3/NaOH ratio on the compressive strength (10M NaOH solution)

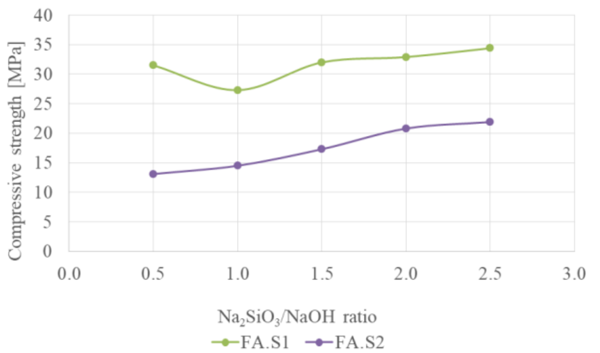

Fig. 8. Effect of Na_2SiO_3/NaOH ratio on the compressive strength (12M NaOH solution)

Fig. 7 and Fig. 8 also illustrate the effect of NaOH solution concentration on the compressive strength of the FAGPP specimens. The compressive strength of the geopolymer binder increases with the increase in the concentration of the NaOH solution and is also significantly affecting the microstructure of the alkali-activated geopolymer binder in terms of geopolymer synthesis [20]. A higher concentration of sodium hydroxide solution leads to a greater dissolution of precursor (fly ash), hence a higher compressive strength is achieved.

4 Summary and conclusions

The experimental procedures for obtaining FAGPP, using Romanian local raw materials, were performed in controlled laboratory conditions, regarding the temperature, the relative humidity, the pressure and the air flow velocity, in order to collect proper data that could be used for future studies in the topic. Careful investigation of specific ratio variations within the paste when using fly ash from different sources provided important conclusions regarding further approaches of the mix design of the geopolymer material. Optimal dosage of the geopolymer paste for superior compressive performance will be used in geopolymer mortar and concrete design.

The best results were provided by the alkali-activated FAGPP when using FA.S1, on mixes developed by using the 2.5 Na_2SiO_3/NaOH solution ratio and 10M NaOH concentration: 25.4 MPa, respectively 12M NaOH concentration: 34.4 MPa.

Fly ash FA.S1 provided both proper workability of the mixes and better compressive strength of the material. The compressive strength of the FAGPP increased with the decrease of the percentage of particles of the fly ash retained on the 45 μm sieve. The compressive strength of the geopolymer paste was influenced by both the physical and chemical characteristics of fly ash, however, the compressive strength was more affected by the fineness of the fly ash. Also, the compressive strength of the FAGPP samples increased with the increase of the molar concentration of the NaOH solution molarity.

The results obtained on FAGPP specimens contribute to the necessity of ongoing research on the identification and exploitation of the various Romanian sources of by-products, resulting both from secondary materials and wastes, from technological processes in various industries. Besides the economic advantages, an important aspect is the ecological one, namely the re-use of industrial wastes and by-products (sources of environmental pollution) into the economic circuit. Likewise, total replacement of cement in innovative, smart materials strongly contributes to the reduction of carbon dioxide emissions.

Further similar studies will be performed using different types of fly ash, from different Romanian power-plants, for a stronger confirmation of the present conclusion. The influence of fly ash chemical composition on geopolymer paste matrix, with respect to the evaluated NaOH concentration and the Na_2SiO_3/NaOH solution ratio and the optimization of the mix design, represent the near future challenges of the ongoing research.

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