EXPERIMENTAL STUDY OF ALKALI ACTIVATED FLY ASH CONCRETE WITH FLY ASH FROM ONE SERBIAN POWER PLANT

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Abstract

The use of alkali activated fly ash concrete (AAFAC) is an important step towards addressing two problems in the construction industry – environmental impact of cement production and large fly ash landfills.

In this study, AAFAC was prepared with class F fly ash from power plant 'Nikola Tesla B' in Serbia and a mixed alkali activator consisting of sodium silicate and sodium hydroxide solutions. The experimental programme consisted of three phases.

First, eight alkali activated fly ash pastes (AAFAP) with different activator combinations and a mass ratio of activator to fly ash of 0.6 were tested in order to investigate their effect on the compressive strength and workability.

In the second phase, four mixtures of AAFAC were made with one optimal activator solution and additional water was added to study its effect on workability and compressive strength.

Finally, eight mixtures of AAFAC were made to investigate the development of compressive strength through time. All samples were oven-cured at 80°C for 6 hours.

The results of compressive strength and workability of tested concretes showed that fly ash from power plant 'Nikola Tesla B' can be used as a binder in AAFAC made with sodium silicate and sodium hydroxide as alkali activators.

1. INTRODUCTION

The construction industry is facing great challenges in directing its activities towards sustainable development. Environmental protection, use of waste and recycled materials and reducing the use of non-renewable resources have become the focus of current research in the field of building materials. Unfortunately, the production of Portland cement, the active ingredient in concrete, generates a significant amount of carbon dioxide (CO_2) due to the

combustion of carbon-based fuels and the calcination of limestone [1]. For each ton of Portland cement clinker produced, approximately one ton of CO_2 is released [2]. With concrete production reaching nearly 25 billion t per year worldwide [3], the sustainability of concrete is a very real concern. A potential solution for these problems is the development of inorganic alumino-silicate polymer, called geopolymer, synthesized from materials of geological origin or by-product materials that are rich in silicon and aluminium.

Fly ash is most commonly used as a binder in geopolymer concrete. In Serbia for instance, about 200 million t of fly ash are being currently deposited at a surface area of 1.500 ha with a production of 6 million t of fly ash every year, mostly produced in 'Nikola Tesla A' and 'Nikola Tesla B' power plants located about 30 km away from Belgrade [4].

Alkali activated fly ash concrete (AAFAC) has attracted much interest in academic and commercial spheres over the past decade. Research shows a great potential for use of geopolymer concrete in the construction industry [5, 6]. AAFAC possess better durability properties compared to Portland cement concrete. Carbonation of alkali activated binders in service appears to be acceptably slow and alkali aggregate reaction seems to be restricted [7]. It has been shown that geopolymer binders possess excellent resistance to various acids [8].

Numerous research studies have shown that physical and mechanical characteristics of AAFAC are influenced by many factors: fly ash particle size and chemical composition, type and concentration of alkali activators (sodium oxide (Na_2O) concentration and silicon dioxide-to-sodium oxide (SiO_2/Na_2O) ratio), temperature and duration of curing [9, 10, 11]. Fly ash particle size distribution is a very important factor. In order to achieve optimal physical and mechanical characteristics of AAFAC, fly ash should have more than 80-90% particles of size smaller than 45 μm. Fernandez and Palomo [12] also reported that factors such as the percentage of calcium oxide (CaO) and the molar Si to Al ratio in the source material significantly influenced the compressive strength of geopolymers. The role and importance of CaO content in fly ash has been discussed by many researchers [13, 14], and it can be concluded that simply comparing the CaO content in fly ashes cannot give a clear prediction on the influence on strength of AAFAC.

The type and concentration of alkali activator solution is also an important factor influencing the mechanical properties of AAFAC. Xu and van Deventer [15] concluded that the addition of sodium silicate solution (Na_2SiO_3) to the sodium hydroxide (NaOH) solution as the activator enhanced the reaction between the source material and the solution. Palomo et al. [16] confirmed that activator solution that contained soluble silicates was proved to increase the rate of reaction compared to alkaline solutions that contained only hydroxides. The addition of Na_2SiO_3 to NaOH activator solution resulted in an increase in strength but a decrease in workability [17]. In addition to the type and ratio of activators, the molarity of the NaOH solution is also important. As the molarity of the sodium hydroxide solution increases there is an increase in strength and a decrease in porosity [18]. Hardjito [19] concluded that higher ratio of Na_2SiO_3 to NaOH by mass causes higher compressive strength of AAFAC. Research has demonstrated that curing time and temperature greatly affect the mechanical development of geopolymer binders. Hardjito and Rangan [20] demonstrated that with longer curing time at elevated temperature (60ºC), the compressive strength of AAFAC increased. Temperatures in the range of 50–90°C are widely accepted for successful geopolymerization.

2. EXPERIMENTAL PROGRAMME

In this study, the potential use of class F fly ash from power plant 'Nikola Tesla B' (Obrenovac, Serbia) as an alkali activated binder in concrete composites was studied. The experimental programme was designed to investigate the influence of various parameters on the concrete compressive strength and workability and it consisted of three phases. First, alkali activated fly ash pastes (AAFAP) were produced with different alkali activator combinations in order to investigate their effect on the compressive strength and workability. Based on the results of the first phase, in the second phase alkali activated fly ash concrete was made with one optimal activator solution and additional water was added to determine its effect on workability and compressive strength. Finally, in the third phase several different mixtures of AAFAC were made to study the development of compressive strength through time.

2.1 Materials

In the 2009/2010 repair of the 'Nikola Tesla B' power plant a new system for dry fly ash collection, transport and disposal was installed. During its pneumatic transport fly ash is separated into four fractions according to particle size and weight and the separate collection of each fraction is possible. In this study two samples of fly ash were taken from the two finest fractions. The samples were named FA-3 and FA-4.

The chemical composition of the fly ash samples, given in Table 1, was determined by X-ray fluorescence (XRF) analysis. As can be seen, both samples satisfy the ASTM C 618 [21] criteria for class F fly ash: SiO_2+ Al_2O_3+ Fe_2O_3 > 70% and LOI < 6% with FA-3 having a relatively high CaO content. Particle size distribution was tested using the Malvern Instruments Mastersizer 2000 and the results are presented in Figure 1. The average mean particle size is 16.775 μm for FA-3 and 8.533 μm for FA-4. Specific gravity was determined according to EN 450-1 [22] as 1960 kg/m^3 for FA-3 and 2070 kg/m^3 for FA-4.

Table 1: Chemical composition (%) of fly ash samples FA-3 and FA-4

Oxides	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3	Na_2O	K_2O	MgO	LOI (%)
FA-3	57.38	18.47	5.89	10.05	1.48	0.53	1.89	1.58	1.65
FA-4	58.24	20.23	5.33	7.62	2.21	0.52	1.51	2.01	1.64

For the activator, a combination of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) was chosen. Sodium silicate solution was obtained from Galenika-Magmasil, Serbia and its chemical composition was Na_2O=14.7%, SiO_2=28.08% and water 57.22% by mass. Sodium silicate solution $Na_2O{\cdot}n{\cdot}SiO_2$ had a module of n=1.91 and a specific gravity of 1514 kg/m^3. The NaOH used in the study was technical grade with 98% purity, in pellets, obtained from Superlab, Serbia. The activators were prepared by first dissolving the NaOH pellets in distilled water. After allowing the NaOH solution to cool down it was mixed with Na_2SiO_3. The solutions were left at room temperature (20±2ºC) in a capped plastic bottle for 24 h before paste and concrete mixing.

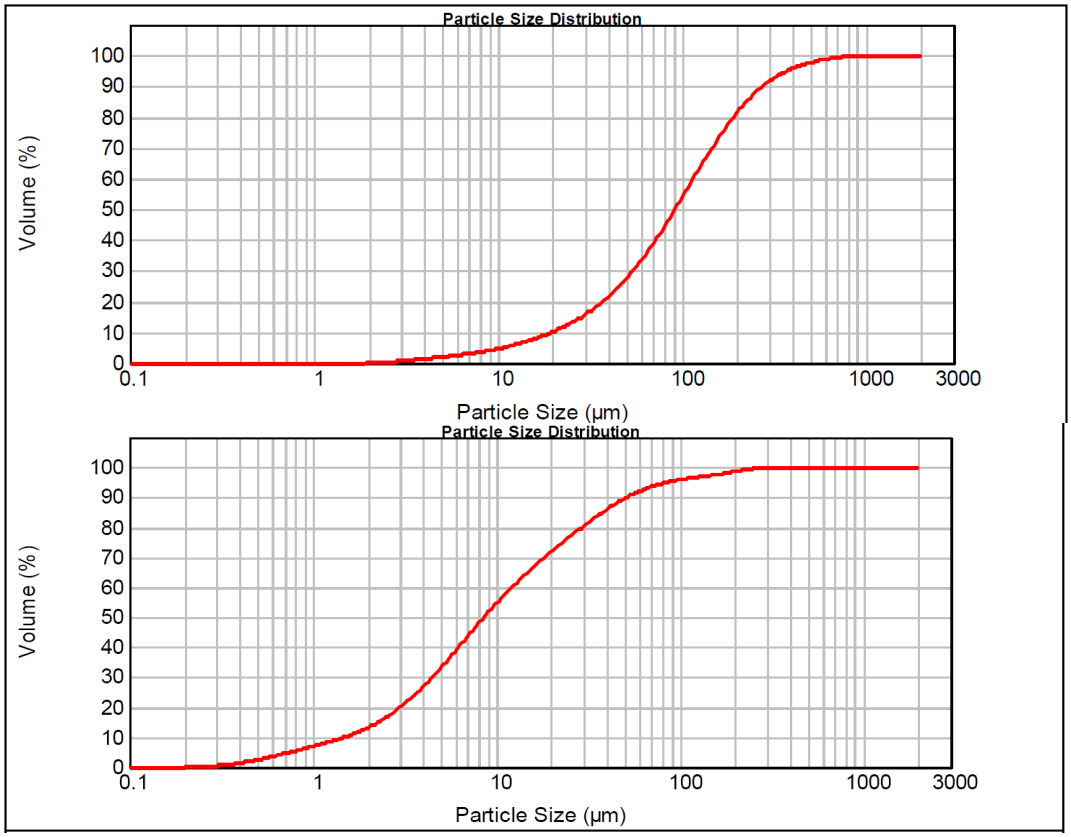

Figure 1: Cumulative particle size distribution of fly ash FA-3 (up) and FA-4 (down)

2.2 Phase 1 – Mix design and results

The aim of the first phase of the experimental programme was to find the best possible binder for later use in AAFAC. To achieve this, the effect of different activator combinations on the two fly ashes was studied. Since only the binder was of interest in this phase, alkali activated fly ash pastes were made instead of concrete, so as to remove any influence of aggregate on the geopolymerization process and mixing.

Activator solutions were prepared and mixed with fly ash. The properties of the activator solutions along with mixture proportions are presented in Table 2. Specimens A3.1-A3.8 were prepared with fly ash FA-3 and specimens A4.1-A4.8 with fly ash FA-4. The alkali activator-to-fly ash ratio (AA/FA) was 0.6 for all specimens. This value was chosen from trial mixtures because a workable paste couldn't be produced with a smaller AA/FA ratio. This stands in relative contrast to the findings of other researchers [19, 23].

In this phase two parameters were chosen as variables: the molarity of NaOH and the Na_2SiO_3/NaOH ratio. This choice was made based on the findings of numerous researchers [23, 24] as well as the values chosen: 10 M (mol/l) and 16 M concentrations for NaOH and 2, 3.5, 5 and 10 for the Na_2SiO_3/NaOH ratio. From these values another set of parameters was calculated: the sodium dioxide-to-fly ash ratio (Na_2O/FA) and the silicon dioxide-to-sodium dioxide ratio (SiO_2/Na_2O). The first two parameters may have a more tangible meaning and are easier to control in the mix design but the latter two are more significant in the geopolymerization process, as suggested by some researchers [25, 26]. As seen in Table 2,

these ratios range from 9.34 to 12.86 in the case of Na_2O/FA and from 1.04 to 1.64 in the case of SiO_2/Na_2O.

Another parameter cited by researchers as being important in the geopolymerization process is the water-to-geopolymer solids ratio (H_2O/SOL) [27]. It represents the mass ratio of water (contained in both the sodium silicate and sodium hydroxide solutions) to fly ash, Na_2O and SiO_2 (contained in Na_2SiO_3 and NaOH).

Table 2: Mixture proportions and compressive strength f_p of alkali activated fly ash pastes

Specimen	AA/FA (–)	NaOH (M)	Na_2SiO_3 /NaOH (–)	Na_2O/ FA (%)	SiO_2/ Na_2O (–)	H_2O/ SOL (–)	f_p /6 h (MPa)	f_p /24 h (MPa)
A3.1	0.6	10	2.0	10.72	1.04	0.312	40.78	44.53
A3.2	0.6	10	3.5	10.09	1.30	0.299	47.66	45.63
A3.3	0.6	10	5.0	9.77	1.44	0.292	45.00	48.00
A3.4	0.6	10	10.0	9.34	1.64	0.284	45.31	46.96
A3.5	0.6	16	2.0	12.86	0.87	0.290	42.00	48.00
A3.6	0.6	16	3.5	11.51	1.14	0.284	48.75	48.59
A3.7	0.6	16	5.0	10.84	1.30	0.282	45.94	48.13
A3.8	0.6	16	10.0	9.92	1.54	0.278	50.94	55.16
A4.1	0.6	10	2.0	10.72	1.04	0.312	57.11	54.30
A4.2	0.6	10	3.5	10.09	1.30	0.299	49.22	53.91
A4.3	0.6	10	5.0	9.77	1.44	0.292	55.31	56.88
A4.4	0.6	10	10.0	9.34	1.64	0.284	59.69	56.95
A4.5	0.6	16	2.0	12.86	0.87	0.290	–	–
A4.6	0.6	16	3.5	11.51	1.14	0.284	53.91	60.16
A4.7	0.6	16	5.0	10.84	1.30	0.282	58.83	59.92
A4.8	0.6	16	10.0	9.92	1.54	0.278	65.42	75.63

The preparation of paste samples was carried out according to EN 196-1 [28], with some necessary modifications. Fly ash was mixed with the activator solution in a RILEM-CEM mixer. Cubic specimens (40×40×40 mm) were cast for compressive strength testing. After placing in moulds and vibrating, the specimens were covered in a layer of plastic to reduce water evaporation. To accelerate the reaction process all the specimens were cured at 80ºC. This temperature was chosen as usual for accelerated curing of concrete and as suitable for the geopolymerization of fly ash. To test the speed of the reaction, specimens were cured for 6 and 24 h. Once the elevated heating was completed, all the specimens were stored at standard laboratory conditions (20±2ºC, relative humidity 60%) until testing at 48 h after mixing.

The first goal in interpreting the results of phase 1 was to find whether any significant difference exists between fly ashes FA-3 and FA-4. Firstly, the workability of pastes made with different fly ashes was very different as can be seen in Figure 2. The workability of all pastes with FA-3 was similar to that of modelling clay whereas all pastes with FA-4 had a honey-like viscosity.

Figure 2: Workability of paste with FA-3 (left) and FA-4 (right)

In Table 2 are also listed the compressive strengths f_p of all the specimens. The compressive strength in this work was determined as an average value from three samples. Mixture A4.5 had a very short setting time and hardened during the mixing so no samples were made. To further assess the possible difference between the fly ashes and curing times a two-factor analysis of variance (ANOVA) was carried out [29]. At a 95% level of significance the *F*-value for fly ash type was 33.61 and greater than $F_{critical} = 4.26$ whereas the *F*-value for curing time was 1.32 and smaller than $F_{critical} = 4.26$. This means that the influence of fly ash type is indeed statistically significant at this level while the influence of curing time is not. These findings are not unexpected since fly ash FA-4 has a finer particle size distribution than FA-3 and a more favourable chemical composition (lower SiO_2/Al_2O_3 ratio and a smaller CaO content). Because of this, for all further studies and analyses only FA-4 was considered and a curing time of 6 h was chosen.

The influence of the activator solution was harder to determine. Looking at Table 2 it can be seen that there is an increase in compressive strength both for rising molarity of NaOH and for the rising Na_2SiO_3/NaOH ratio. However if an attempt is made to translate these trends into Na_2O/FA and SiO_2/Na_2O ratios, no clear conclusion can be drawn. There also seems to exist a trend of increasing compressive strength with a decreasing H_2O/SOL ratio.

2.3 Phase 2 – Mix design and results

In phase 2 the aim was to produce AAFAC and to investigate another component material whose influence on the chemical reaction in geopolymerization remains unclear – water. To achieve this, four AAFAC were prepared with the same binder, chosen from phase 1, and additional water was added to the mix. When choosing an optimal binder from phase 1, aside from the compressive strength and workability, economical and ecological aspects needed to be analyzed as well.

Table 3 shows the price in EUR/m^3 of AAFAP A4.1-A4.8 on the Serbian market and the equivalent CO_2 of AAFAP, evaluated using life-cycle assessment explained in [30]. From Table 3 it can be seen that paste A4.4 (10 M NaOH and Na_2SiO_3/NaOH=10) has the lowest price, a reasonable amount of $CO_{2,eq}$ and the second highest compressive strength. For these reasons it was selected as the binder in phase 2 of the experimental programme.

AAFAC mixtures were prepared with 400 kg/m^3 of fly ash, aggregate with maximum size of 16 mm and in SSD condition. AA/FA ratio of 0.6 was used; however the amount of additional water was linearly increased so that the H_2O/SOL could be varied in a wider range. The mixture proportions of the AAFAC are given in Table 4.

Table 3: Price, $CO_{2,eq}$ and compressive strength of AAFAP from phase 1

Specimen	A4.1	A4.2	A4.3	A4.4	A4.5	A4.6	A4.7	A4.8
Price (EUR/m^3)	34.0	32.0	31.0	30.0	40.0	36.0	34.0	31.0
$CO_{2,eq}$ (kg/m^3)	238.4	250.2	256.0	264.0	263.2	266.7	268.4	270.8
f_p (MPa)	57.1	49.2	55.3	59.7	-	53.9	58.8	65.4

Table 4: Mixture proportions of alkali activated fly ash concretes in phase 2

Specimen	Fly ash (kg/m^3)	NaOH (10 M) (kg/m^3)	Na_2SiO_3 (kg/m^3)	Coarse aggr. (kg/m^3)	Fine aggr. (kg/m^3)	Additional water (kg/m^3)	Na_2SiO_3/ NaOH (–)	H_2O/ SOL (–)
B1	400.0	21.8	218.2	948.0	682.2	0.0	10.0	0.284
B2	400.0	21.8	218.2	968.7	671.5	10.0	10.0	0.304
B3	400.0	21.8	218.2	953.3	660.8	20.0	10.0	0.324
B4	400.0	21.8	218.2	965.9	669.6	28.0	10.0	0.340

The concrete mixtures were prepared by first adding all dry materials to the mixer and mixing them for 3 minutes. Subsequently the activator solution was slowly added and mixed for another 5 minutes. All samples were cast in 100×100 mm cubes, vibrated and sealed with a layer of plastic sheet. According to the results of phase 1, the specimens were cured for only 6 h at a temperature of 80ºC. After curing, all the specimens were stored at standard laboratory conditions until testing at 48 h after mixing.

Workability of AAFAC was measured with a standard cone slump test. The results in Figure 3 (left) show a linear increase in workability (slump) from 18.3 cm to 28 cm for additional water amounts of 0 to 28 kg/m^3. Compressive strength results shown in Figure 3 (right) show a decrease of compressive strength with the increase of water in the mixture but the correlation is poorer in this case. This effect could also be a result of a decreasing pH (i.e. molarity) of the solution with an increasing water amount.

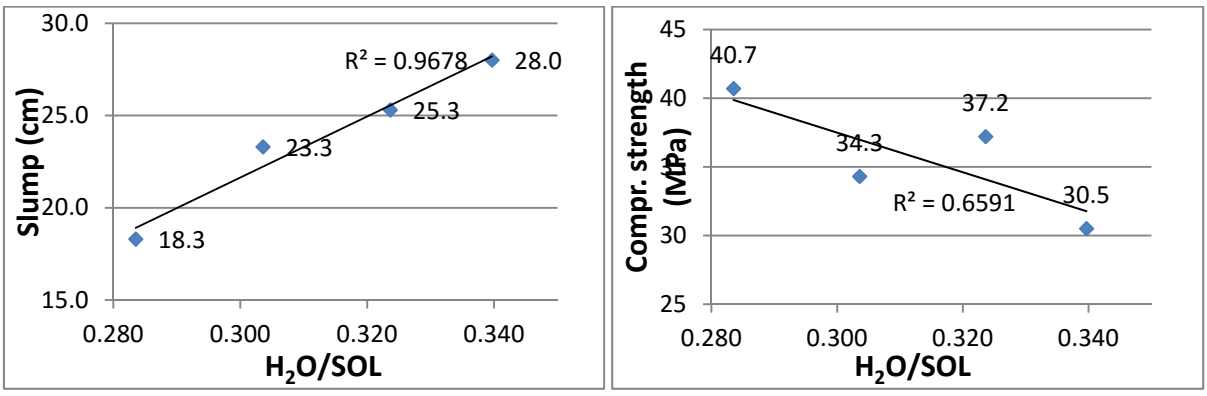

Figure 3: Slump (left) and compressive strength (right) of AAFAC specimens in relation to the H_2O/SOL ratio

2.4 Phase 3 – Mix design and results

Phase 3 was conducted in order to further investigate the influence of Na_2O/FA and SiO_2/Na_2O ratios on the geopolymerization process. All of the AAFAC mixtures were again designed with 400 kg/m^3 of fly ash and an AA/FA ratio of 0.6. Aggregate with maximum size of 16 mm was used in SSD condition. The specimens were prepared, cast and cured in the same way as in phase 2. After curing, all the specimens were stored at standard laboratory conditions until testing at 1, 3, 7 and 28 days after mixing.

As for the activator, it was decided not to vary specific molarities of NaOH or specific Na_2SiO_3/NaOH ratios but rather to design activator solutions for discrete values of Na_2O/FA and SiO_2/Na_2O ratios since the geopolymerization process in fact depends on them. Eight mixtures were designed for 2 different Na_2O/FA ratios and 5 different SiO_2/Na_2O ratios.

For Na_2O/FA ratio, values of 9.35 and 9.90 were chosen as being significantly different, based on the results of phase 1. In the first group of mixtures (Na_2O/FA=9.35), SiO_2/Na_2O ratios were 1.35, 1.45, 1.55 and 1.65 while in the second group (Na_2O/FA=9.90) they were 1.25, 1.35, 1.45 and 1.55. Not all combinations of Na_2O/FA and SiO_2/Na_2O ratios were made as they would have led to very high or low molarities of NaOH. As a result, the values of the H_2O/SOL ratio varied in a narrow range (0.277-0.312). The mixture proportions of the AAFAC are given in Table 5 and the values of the varied parameters are given in Table 6.

The compressive strength of the AAFAC specimens is shown in Figures 4-7. The mixtures are grouped according to the same SiO_2/Na_2O ratio except in the case of ratios 1.25 and 1.65. Two subjects can be analyzed from these figures: the development of compressive strength through time and the influence of activator combinations on the compressive strength.

The increase in compressive strength from day 1 to 28 ranges from 3% to 27% with an average value of 17%. For each SiO_2/Na_2O ratio, higher compressive strengths are achieved for the concretes with a higher Na_2O/FA ratio.

In Figure 8, concrete mixtures with different SiO_2/Na_2O ratios, but with a same Na_2O/FA ratio are compared. These results point to a fact that there is an optimal SiO_2/Na_2O ratio, not necessarily the highest one. For both values of Na_2O/FA ratio, the highest concrete compressive strengths at 28 days were obtained for SiO_2/Na_2O ratio equal to 1.45.

Table 5: Mixture proportions of alkali activated fly ash concretes in phase 3

Specimen	Fly ash (kg/m^3)	NaOH (kg/m^3)	Na_2SiO_3 (kg/m^3)	Coarse aggr. (kg/m^3)	Fine aggr. (kg/m^3)
C1	400.0	60.19	179.81	992.3	661.5
C2	400.0	46.87	193.13	993.2	662.1
C3	400.0	33.55	206.45	989.3	659.5
C4	400.0	20.24	219.76	989.9	659.9
C5	400.0	63.71	176.28	992.0	661.4
C6	400.0	49.62	190.38	993.0	662.0
C7	400.0	35.51	204.49	994.0	662.6
C8	400.0	21.41	218.59	986.3	657.5

Table 6: Values of varied parameters for alkali activated fly ash concretes in phase 3

Specimen	Na_2O/ FA (%)	SiO_2/ Na_2O (–)	NaOH (M)	Na_2SiO_3/ NaOH (–)	H_2O/ SOL (–)
C1	9.35	1.35	7.17	2.99	0.312
C2	9.35	1.45	7.64	4.12	0.302
C3	9.35	1.55	8.51	6.15	0.292
C4	9.35	1.65	10.47	10.86	0.282
C5	9.90	1.25	8.73	2.77	0.309
C6	9.90	1.35	9.62	3.84	0.298
C7	9.90	1.45	11.35	5.76	0.288
C8	9.90	1.55	15.98	10.21	0.277

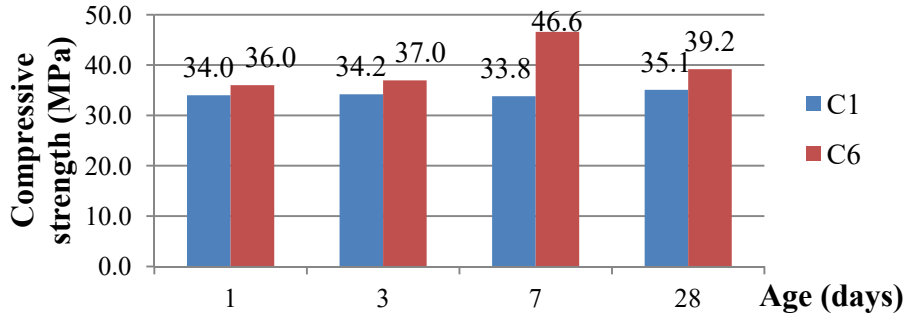

Figure 4: Development of compressive strength for mixtures with SiO_2/Na_2O=1.35

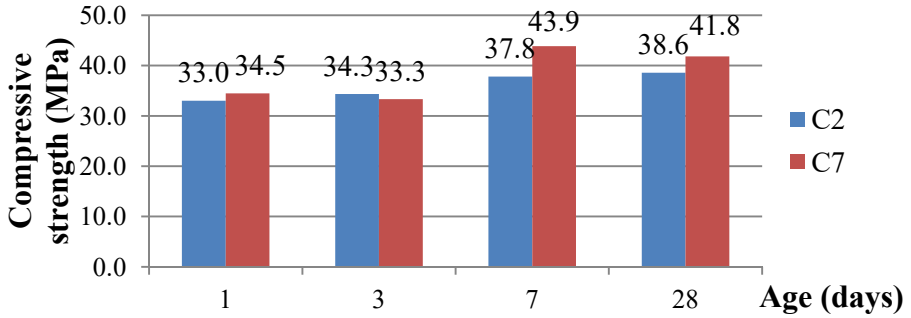

Figure 5: Development of compressive strength for mixtures with SiO_2/Na_2O=1.45

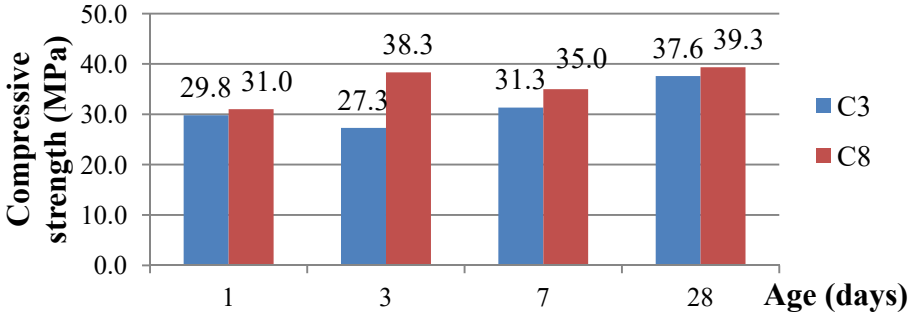

Figure 6: Development of compressive strength for mixtures with SiO_2/Na_2O=1.55

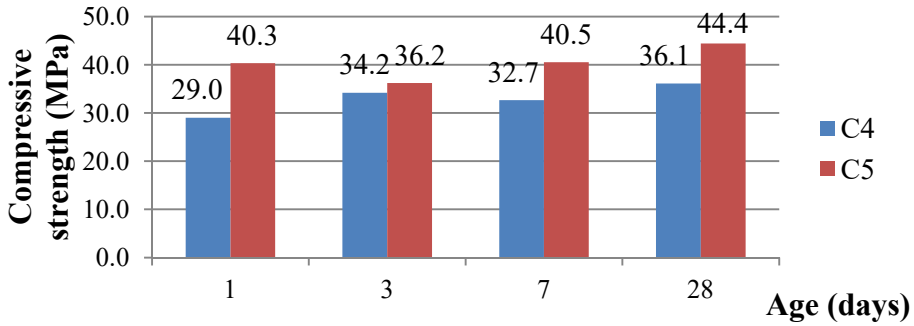

Figure 7: Development of compressive strength for mixtures with SiO_2/Na_2O=1.25 and 1.65

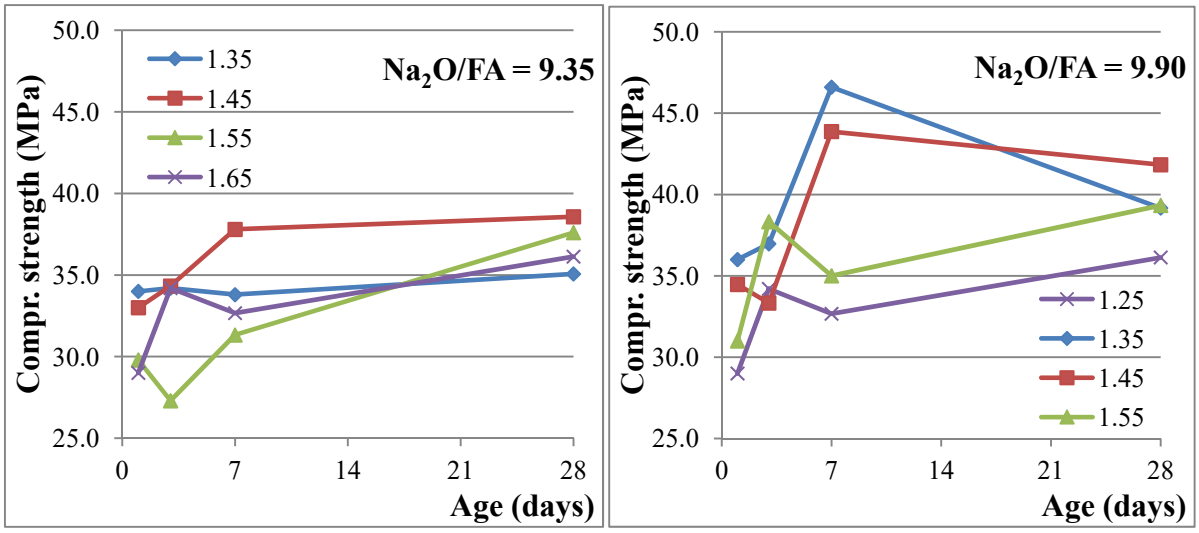

Figure 8: Development of compressive strength for mixtures with different SiO_2/Na_2O ratios

3. CONCLUSIONS

From the presented experimental research results the following conclusions can be drawn:

- Fly ash from 'Nikola Tesla B' power plant (Serbia) can be used as a base material for producing alkali activated pastes and concretes. The use of fly ash FA-4 with a smaller mean particle size and lower CaO content results in higher compressive strengths and better workability of AAFAP.
- There is no significant difference in compressive strength of AAFAP specimens cured at 80ºC for 6 h and for 24 h.
- When comparing AAFAC produced with the same SiO_2/Na_2O ratio but with different Na_2O/FA ratios, higher compressive strengths are obtained for the mixtures with a higher Na_2O/FA. For the Na_2O/FA ratio increase from 9.35 to 9.90, the average increase of concrete compressive strength is 15% while the values range from 2% to 40%.
- For both Na_2O/FA ratios, an optimal SiO_2/Na_2O ratio (regarding the concrete compressive strength) is equal to 1.45.

- The average increase of AAFAC compressive strength from day 1 to 28 is 17%, while the values range from 3% to 27%.
- Increasing the water-to-geopolymer solids ratio by adding additional water to the solution leads to increasing workability and decreasing compressive strength of AAFAC. However the decreasing compressive strength could also be a result of a lower pH value of the solution.
- Further research is necessary to better understand the influence of chemical composition and particle size distribution of fly ash and of Na_2O, SiO_2 and water contents on the physical and mechanical properties of fresh and hardened alkali activated fly ash concretes.

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