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Influence of Curing and Water to the Mechanical Properties of Geopolymer Mortar

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***Abstract* - This paper presents experimental research investigating the effects of curing times, curing temperatures and water to the mechanical properties of geopolymer mortar incorporating different fly ash content. Geopolymer cements were synthesized from calcined kaolin and shale clay residues with Si/Al ratio of 2.0 with sodium and potassium silicates (Na_2SiO_3/K_2SiO_3) modulus ranging from 1.50 ÷ 1.95. Specimens were cured in two different environmental conditions: furnace and ambient. Tests were performed to establish the water to fly ash mass ratio increased the mechanical properties of geopolymer mortar decreased. Microstructural observations of fly ashes and pure geopolymer cement have been carried out by means of scanning electron microscopy (SEM) and energy-dispersive X-ray analysis (EDX).**

Keywords* - *Fly ash, compressive strength, geopolymer mortar, water – fly ash mass ratio.

I. INTRODUCTION

In 1979, the term "geopolymer" was first discovered to the chemical world by a French professor Joseph Davidovits [1], they are inorganic polymeric materials with a chemical composition similar to natural zeolite but containing an amorphous microstructure and possessing ceramic-like in their structures and properties [2-5]. Geopolymer are synthesized and hardened at ambient pressure and temperature, so the science can produce artificial stone at a temperature below 100 ^oC [6, 7]. This material (geopolymer cement) evolved into a mineral-based binder for use as a high strength industrial cement with significantly shorter cure times than OPC [2]. There are two main constituents of geopolymers, namely the source materials and the alkaline liquids. The source materials for geopolymers based on alumina-silicate should be rich in silicon (Si) and aluminium (Al) such as metakaolinite, slag, geological, blast furnace slag, fly ash, rice husk ash, etc. The choice of the source materials for making geopolymers depends on factors such as availability, cost, type of application, and specific demand of the end users. The most common alkaline liquid used in geopolymerization is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate (Na_2SiO_3) or potassium silicate (K_2SiO_3) [8, 9].

To discuss the chemical structure of geopolymers, the term 'sialate' is an abbreviation for silicon-oxo-aluminate and is used here to describe the bonding of silicon and aluminium by bridging oxygen. And the term poly(sialate) was suggested as a descriptor of silico-aluminate structure of the type of material [2, 4, 10]. The amorphous to semi-crystalline three dimensional of sialate network consists of SiO_4 and AlO_4 tetrahedral which are linked alternately by sharing all the oxygens to create basic polymeric Si-O-Al bonds (see in Fig. 1) [2, 4], so Prof. Davidovits called it geopolymer. To balance the negative charge of Al^{3+} in IV fold coordination, positive ions sodium (Na^+), potassium (K^+), lithium (Li^+), calcium (Ca^{2+}), barium (Ba^{2+}), ammonium (NH_4^+), hydronium (H_3O^+) must be present in the structural spaces [2].

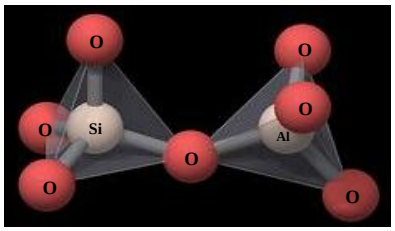

Fig. 1. Tetrahedral configuration of sialate Si-O-Al-O; Si, Al atoms in white and O atoms in pink [2, 11].

Geopolymerization involves a chemical reaction between various aluminosilicate oxides Al^{3+} in IV-V fold coordination with silicates, yielding polymeric Si-O-Al-O sialate bonds like the following:

$2(Si_2O_5,Al_2O_2) + K_2(H_3SiO_4)_2 + Ca(H_3SiO_4)_2 \rightarrow (K_2O,CaO)(8SiO_2, 2Al_2O_3, nH_2O)$

Poly(sialates) are described by the following empirical formula [1, 2, 4, 12] :

$M_n[-(SiO_2)_z - AlO_2]_n . wH_2O$, where M is a monovalent cation such as potassium (K^+) or sodium (Na^+) , n is the degree of polycondensation and z is either 1, 2, 3 or >> 3. Poly(sialate) are described as chain and ring polymers with Si^{4+} and Al^{3+} in IV-fold coordination with oxygen and range in from amorphous to semi-crystalline.

Davidovits has also distinguished four types of polysialates according to the ratio Si:Al they are of the types:

Poly(sialate): $M_n–(–Si–O–Al–O–)_n$ with Si:Al = 1:1

Poly(sialate-siloxo): $M_n–(–Si–O–Al–O–Si–O–)_n$ with Si:Al = 2:1

Poly(sialate-disiloxo): $M_n–(–Si–O–Al–O–Si–O–Si–O–)_n$ with Si:Al = 3:1

Poly(sialate-multisiloxo) with Si:Al >> 3:1, the polymeric structure results from the cross linking of poly(silicate) chains, sheets or networks with a sialate link (–Si–O–Al–O–) (2D or 3D cross-link).

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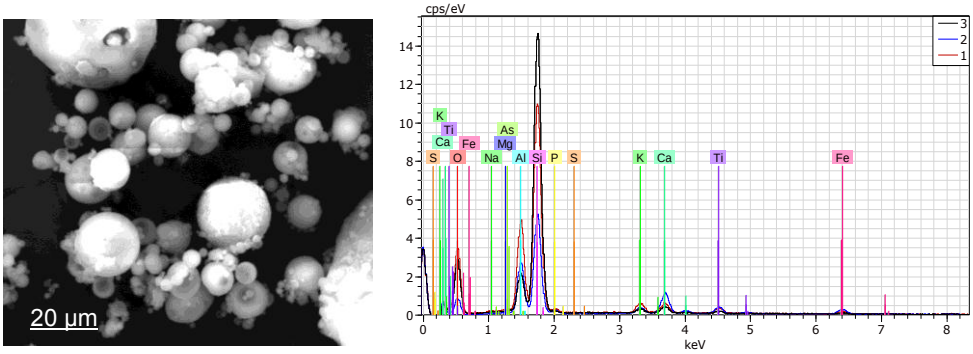

Fig. 2. SEM images the spherical microscopic structure and corresponding energy spectrum of fly ash FA1.

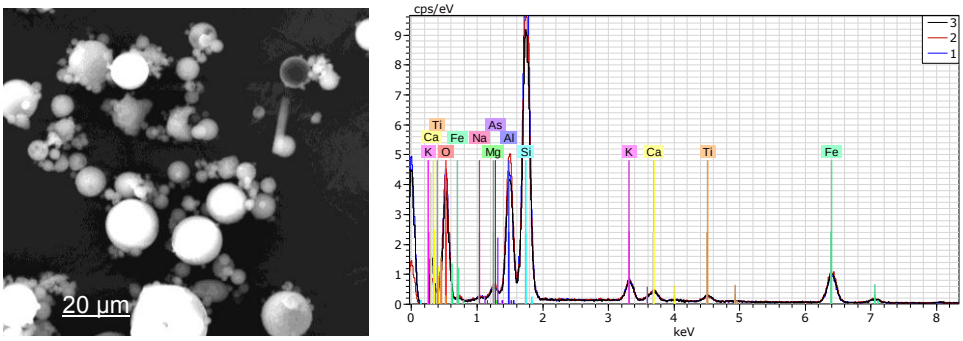

Fig. 3. SEM images the spherical microscopic structure and corresponding energy spectrum of fly ash FA2.

TABLE I: QUANTITATIVE ELEMENTAL ANALYSIS DATA OF FA1 AND FA2

Fly ash	O	Na	Mg	Al	Si	P	S	K	Ca	Ti	Fe	As
FA1 [%]	53.84	0.35	0.10	9.74	30.57	0.82	0.12	0.93	2.11	0.76	0.57	0.10
FA2 [%]	55.06	0.77	1.17	10.89	25.17	-	-	1.70	0.93	0.53	3.68	0.10

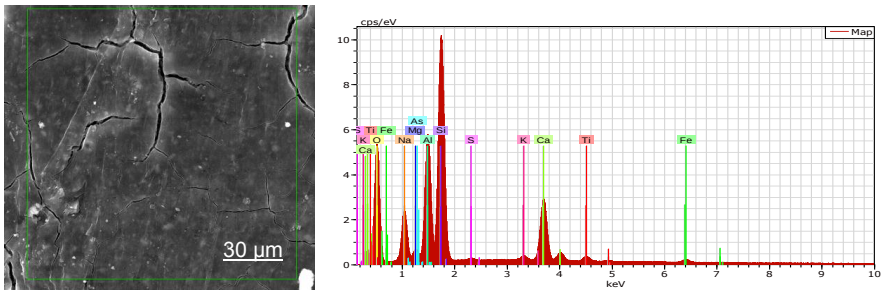

Fig. 4. SEM images and corresponding energy spectrum of an individual geopolymer matrix.

In this research, geopolymer resin was synthesized from shale fly dust burnt in rotary kiln (for 10 hours at 750 ^{o}C) with Si/Al molar ratio of 2.0 with sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). And the purpose of this research is observing the influence of adding fly ash and curing in order to obtain the mechanical properties of geopolymer mortar.

II. EXPERIMENTAL METHODS

Fly ashes used in this study came from different sources in Czech Republic. The fly ashes were already classified into 2 names of city and coded such as: FA1 (Kotel Plzeň), FA2 (Pražská Teplárenská).

They have color light grey due to its chemical compositions and contaminants as shown in Table I. Figs. 2, 3 indicates that fly ash particles are generally spherical in shape and range in size from 1 µm to 10 µm. All experiments were performed using the same batches of reagents and starting materials.

Geopolymer binders were prepared by mixing of alkali activator containing Na_2SiO_3/K_2SiO_3 and NaOH/KOH with modulus 1.50 ÷ 1.95. The microstructure of pure geopolymer matrix were analyzed by mean SEM and EDX in Fig. 4, corresponds to a Si/Al and Na/Al molar ratio of 2.0 and 0.8 respectively, that is poly(sialate-siloxo) [2].

Several series of samples were prepared to test the influential variables on the compressive strength of hardened geopolymer. The variables include modulus and content of the mixed alkali activator and the sample curing conditions. The technology of sample preparation is as follows. At first, the geopolymer resin was prepared by mixing the alkaline activator with the raw materials. The liquid and solid

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components were mixed about 10 minutes at room temperature until the solution homogenized. Next, the geopolymer resin mixture was mixed with different fly ash content. And if the mass content of this mixed fly ash increasing was also increased water from 2% to 20%. The mixing was done in an air conditioned room at approximately 20°C ± 3°C until the well homogenized mixture (about 5 minutes). Directly after mixing, the fresh mortar was poured in the moulds and vibrated for 2 minutes on the vibration table to remove air voids. Compressive strength testing was performed as per AS 1012.9 using (Ø50 x 100) mm diameter cylindrical moulds. Three cylinders of each sample were tested, with the experimental values being averaged.

There are two ways to curing these samples:

1. These samples were cured at room temperature for 3 days after casting. Next, the samples were removed from the moulds and left in laboratory ambient conditions until the day of test. The sample ages for the latter tests were 7, 14, and 28 days.

2. All the mixtures were cured in a furnace without delay time at the specific curing temperature for 24 hours and 48 hours ranging from 60°C ÷ 90°C. Samples were demoulded after the curing process in the oven until the testing age (see Fig. 5).

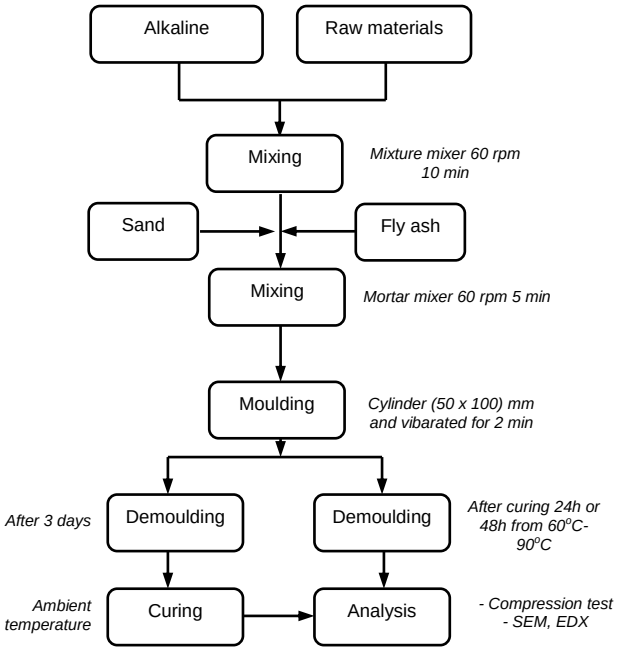

Fig. 5. Fabrication of the geopolymer mortar.

III. RESULTS AND DISCUSSION

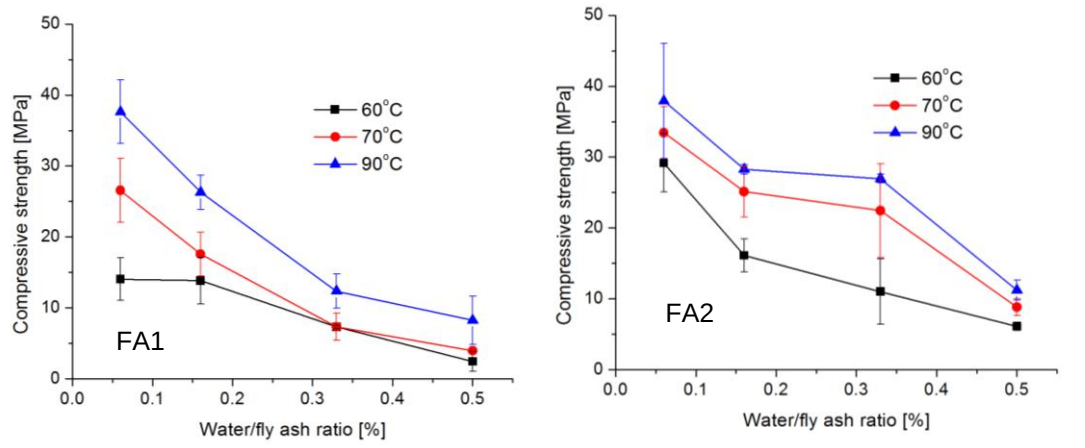

Fig. 6. Effect of water/fly ash ratio by mass for different curing temperatures for 24 hours.

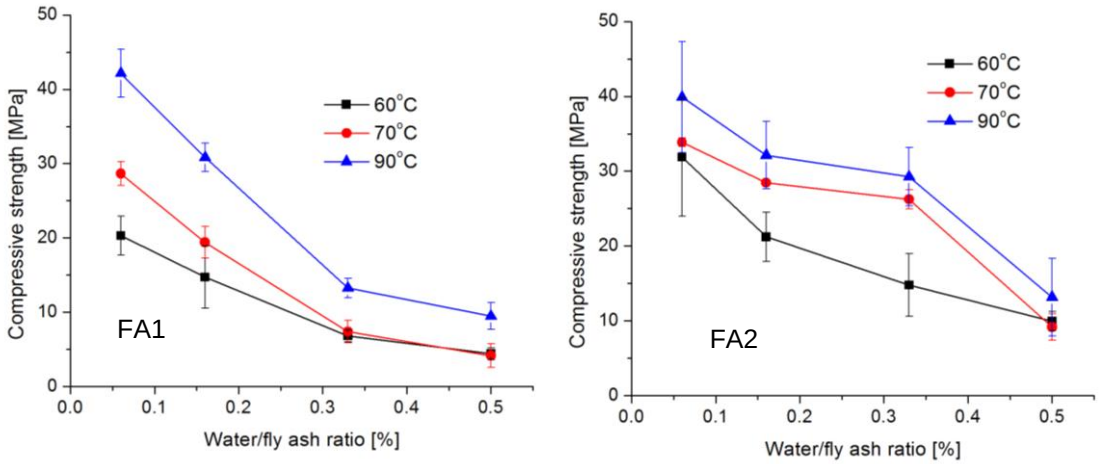

Fig. 7. Effect of water/fly ash ratio by mass for different curing temperatures for 48 hours.

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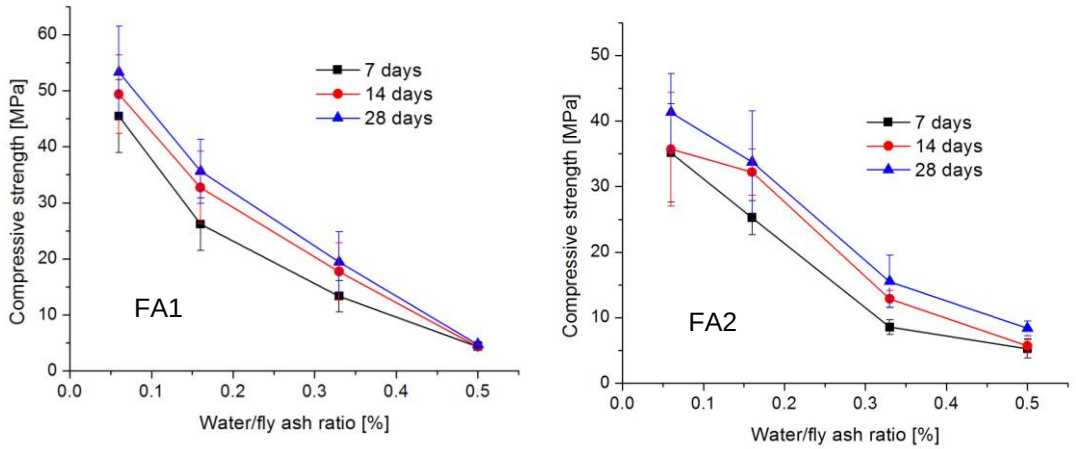

Fig. 8. Effect of water to fly ash ratio by mass for curing room temperature.

Tests were performed to establish the effect of water to fly ash ratio by mass on the compressive strength of geopolymer mortar. The test specimens were heat cured in a furnace at various temperatures for 24 hours presented in Fig. 6 and 48 hours in Fig. 7. Curing at higher temperatures for more than a couple of hours does seem to positively affect the development of compressive strength. However, we should note that curing for longer periods of time at elevated temperature appears to weaken the structure.

As we know that in Ordinary Portland cement mortar or concrete, water in the mixture chemically reacts with the cement to produce a paste that binds the aggregates. In contrast, the water in fly ash-based geopolymer mortar and concrete mixtures does not cause a chemical reaction. The results presented in Fig. 8 testify to the fact that water content in the geopolymer concrete mixture affected the properties of concrete in the fresh state as well as in the hardened state. Because the excess water in fresh mixtures which reduces the concentration of activator, the chemical reaction that occurs in geopolymers produces water that is eventually expelled from the binder, leads to a reduced geopolymerization reaction and thus lowers compressive strength. It can be seen that increasing the percentage of fly ash and adding water is significant reduce the compressive strength of samples, which exhibited nearly 24 % with FA2, 19 % with FA1 when curing in the oven at 70 ^{o}C for 48 hrs.

IV. CONCLUSIONS

The present study has shown that the final structure and physical properties of geopolymers mortar are dependent upon a variety of material parameters including water to fly ash ratio content, curing, particle size. Specifically, the calcium content of fly ash and the water/fly ash ratio seems to play an important that affect the final compressive strength of geopolymers mortar. Beside, rapid curing and/or curing at too high temperatures will also effect to physical properties of specimens because the samples have many crack inside. The current work has therefore shown that the manufacture of a geopolymer product for specific applications requires careful consideration of process conditions such as curing temperature, in addition to the initial mix design.

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