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## UNCONVENTIONAL TREND IN ADOPTING THE NEW “ONE-PART” MIXTURE GEOPOLYMER FOR CONSTRUCTION APPLICATIONS

BY

LUCIAN PĂUNESCU<sup>1,\*</sup>, ADRIAN IOANA<sup>2</sup>, ENIKŐ VOLCEANOV<sup>3</sup> and  
BOGDAN VALENTIN PĂUNESCU<sup>4</sup>

<sup>1</sup>National University of Science and Technology “Politehnica”, Faculty of Applied Chemistry and Materials Science, Research Center for Environmental Protection and Eco-Friendly, Bucharest, Romania

<sup>2</sup>National University of Science and Technology “Politehnica”, Faculty of Materials Science and Engineering, Bucharest, Romania

<sup>3</sup>National University of Science and Technology “Politehnica”, Faculty of Engineering in Foreign Language, Bucharest, Romania

<sup>4</sup>Consitrans SA, Bucharest, Romania

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**Abstract.** The work aimed at producing “one-part” geopolymer composite material using coal fly ash and granulated blast furnace slag as alumina-silicate precursors, sodium carbonate as a solid alkali activator, and sodium lignosulphonate as a water-reducer superplasticizer, constituting a dry powder mixture, to which water was added later. This preparing way of the geopolymer is an alternative version to the traditional “two-part” geopolymer using liquid alkaline activation and is mainly suitable for in-situ construction applications. The results of the current experiment showed good thermal insulation properties, the density and heat conductivity values being quite low as well as quite high levels of compression and flexural strength, early strength values after only 7 days being high considering the final mechanical resistance values after 28 days.

**Keywords:** solid alkali activator, “one-part” geopolymer, fly ash, slag, sodium carbonate.

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\*Corresponding author; *e-mail*: lucianpaunescu16@gmail.com

## 1. Introduction

Recently, the manufacture of geopolymers as an alternative option to ordinary Portland cement, whose industrial production has caused huge CO<sub>2</sub> emissions into the atmosphere, being responsible for about 8% of entire world CO<sub>2</sub> emissions, has become a concern for researchers and producers of construction materials in the world (Andrew, 2017). The already conventional method of preparing geopolymers consists in developing the geopolymerization reaction between one or more alumina-silicate precursors with the aqueous solution of an alkaline activator. This method was claimed by the French researcher J. Davidovits at the end of the 20<sup>th</sup> century (Davidovits, 1991).

In their work (Duxson and Provis, 2008) have analysed the choice of suitable raw materials to be used as precursors in the production of geopolymers. In order to replace existing cement binders, as well as for the effective use of industrial waste, procedures for adapting the chemistry of geopolymer precursors and the behaviour of solid particles were investigated. The opportunity to produce geopolymers from a single part was identified as exceeding the potential of the already known design containing two parts (solid precursors and alkaline activator solution). Also, the potential value of class F-fly ash (with lower calcium content) in the synthesis of high-performance one-part geopolymers was highlighted.

In “one-part” mixtures, only one dry mix and water are required. The dry mix is made by mixing a solid alkaline activator with a solid alumina-silicate precursor, with or without a calcination stage. This mixture type for producing a geopolymer is considered by many specialists to be safer, easier to handle and adapted for in-situ works. The concentrated alkaline solutions used in the two-part mixture method have the disadvantage to be corrosive and dangerous. However, until now, the conventional “two-part” mixture method is the one adopted worldwide and used in various construction applications.

In the last two decades of the previous century, the first versions of “one-part” geopolymers were tried in the world. Some authors (Heitzmann *et al.*, 1987) proposed a dry mixture of metakaolin, blast furnace slag, amorphous silica, potassium hydroxide and potassium silicate, together with one of the materials fly ash, calcined shale, or calcined clay, mixed with Portland cement before the addition of water.

Also, J. Davidovits registered a patent on the production of a “one-part” mixture geopolymer including metakaolin, sodium or potassium disilicate, and metallurgical slag (Davidovits, 1994) and later, partially replaced the solid sodium or potassium hydroxide and silicate of the same chemical elements with a “synthetic lava” (reactive alumina-silicate glass) (Davidovits, 2015).

In another work (Ye *et al.*, 2016a), “one-part” geopolymer type was used to stabilize fly ash from municipal solid waste incineration mixed with red mud and NaOH, the water being added later.

A mechano-chemical activation method was chosen by (Mataalkah *et al.*, 2017), where fly ash was ball milled together with dry activators (such as CaO, MgO, and NaOH). It was found that sodium, calcium, and magnesium were incorporated into the ash structure due to the disruption of alumina-silicate bonds, leading to growing mechanical strength and moisture resistance as well as obtaining a finer microstructure compared to processing the raw materials by separate grinding and subsequent mixing.

Reactive amorphous silica and alumina are usually provided by alumina-silicate precursors (metakaolin, fly ash, or metallurgical slag) and are also supplemented by aqueous sodium (or potassium) silicate solution in conventional “two-part” geopolymers. “One-part” mixture geopolymers utilize various solid sources of amorphous silica. Regarding additional reactive alumina, solid sodium aluminate is the most commonly used material and has the ability to also act as an activator.

Rice husk ash, as a residual material from agricultural waste combustion, has a very high content of reactive silica and is mainly commercially supplied from China. Some researchers (Hajimohammadi and van Deventer, 2016) have used this residual material activated with solid sodium aluminate in “one-part” geopolymer mixtures. It was found that after only 24 hours of hardening the compression strength of this geopolymer type reached a high value (30 MPa) and that the ash was almost completely reacted. Also, it was discovered that poor quality-rice husk ash (containing large amount of unburned carbon) can be effectively used in the production of “one-part” geopolymer.

The ash resulting from burning corn stalks and cobs, with high pH, potassium oxide content of about 30 wt. %, and silica amount representing about 20 wt. % was used as a solid activator in “one-part” metakaolin-based geopolymers (Peys *et al.*, 2016).

Silica fume (finely dispersed amorphous SiO<sub>2</sub>), as a by-product of industrial manufacturing silicon and ferrosilicon alloys, was used in the “one-part” geopolymer mixture. According to (Ye *et al.*, 2016b), up to 25% silica fume added increased the compression strength of red mud-based geopolymer.

According to (Provis and van Deventer, 2014; Luukkonen *et al.*, 2018), the source of alkaline activation in a “one-part” geopolymer mixture can be any chemical compound that has the ability to provide alkaline cations, to increase the pH of the reactant mixture, and to favour the dissolution. The alkali activator in this case can be: solid NaOH, Na<sub>2</sub>SiO<sub>3</sub>, Na<sub>2</sub>SiO<sub>3</sub>·5H<sub>2</sub>O, Na<sub>2</sub>CO<sub>3</sub>, NaAlO<sub>2</sub>, CaSO<sub>4</sub>, Na<sub>2</sub>SO<sub>4</sub>, KOH, red mud as well as corn cob and stalk ash. Several of mentioned activators have some disadvantages. Thus, solid NaOH is corrosive, hygroscopic, and forms sodium carbonate in the presence of CO<sub>2</sub>. The manufacture of synthetic alkali silicates is disadvantageous due to high energy

consumption.  $\text{Na}_2\text{CO}_3$  as an alternative to synthetic alkali silicates has been shown to be at least as effective compared to  $\text{NaOH}$  as a thermal activator for kaolin, bentonite, or albite (Luukkonen *et al.*, 2018). However,  $\text{CaCO}_3$  as well as dolomite release  $\text{CO}_2$  if used at the high temperature required by alumina-silicate precursors for calcination. The work (Kovtun *et al.*, 2015) has shown appreciated results when using solid  $\text{Na}_2\text{CO}_3$  together with slaked lime  $[\text{Ca}(\text{OH})_2]$  as activators, without the calcination process, in the case of using blast furnace slag and silica fume as precursors, respectively. Very high values of compression strength were reached (50 and 85 MPa respectively, after 28 curing days at ambient temperature and  $85^\circ\text{C}$ , respectively). On the other hand,  $\text{NaAlO}_2$  represents an attractive source of alkaline activation because it also provides additional reactive aluminium.

Superplasticizers have a well-determined role regarding improving workability and rheology of concrete and mortar based on Portland cement. Generally, superplasticizer operation in geopolymer pastes is not satisfactory due to the high alkaline environment. However, naphthalene and modified polycarboxylates showed good performances in the case of “two-part” mixture geopolymers. In the work (Ye *et al.*, 2016b) sodium lignosulphonate (0.5 wt. %) in one-part mixture based on red mud was used obtaining the reduction of water/solid ratio from 0.55 to 0.45 and thus improving the compression strength.

The manufacturing technique of “one-part” mixture geopolymer composites involves a curing process both at ambient temperature and at relatively higher temperatures ( $50\text{--}85^\circ\text{C}$ ) depending on the nature of precursors. According to (Suwan and Fan, 2017), as a result of the dissolution of solid activators in “one-part” geopolymers, higher temperature can contribute to the improvement of hardening process. In general, thermal hardening accelerates the early strength growth of geopolymers (van Deventer *et al.*, 2010). According to the literature (van Deventer *et al.*, 2010) the curing “one-part” mixture geopolymer is preferable to take place under saturated conditions (100% relative humidity) and it is not necessary to add water during this process.

In accordance with (Mataalkah *et al.*, 2017), after the water addition to “one-part” geopolymer mixtures the geopolymerization process occurs in four distinct steps: ion exchange, hydrolysis, network decomposition, and release of silica and aluminum. Next, “one-part” geopolymer production follows a similar process to that of “two-part” geopolymer production (Duxson *et al.*, 2007). An important difference between the two types of geopolymer making is the release rate of silicon and aluminum. In the case of “one-part” mixtures, the solid silica sources react more slowly than in “two-part” mixtures. The paper’s authors (Hajimohammadi *et al.*, 2011) opted to mix solid geothermal silica with a solution of solid sodium silicate and sodium aluminate.

An interesting experiment conducted by (Zhang *et al.*, 2024) aimed at determining peculiarities of the preparation process of “one-part” alkali-activated geopolymer based on fly ash and granulated blast furnace slag. The “one-part”

alkali-activated mortar system included in the starting mixture: sand (below 4 mm) as a fine aggregate, class F-coal fly ash and granulated blast furnace slag as alumina-silicate binders, solid sodium silicate as an activator, water, and a superplasticizer. The ash/slag ratio, the binder content, and the solid activator/binder ratio were the variables in this experiment, while the water/binder ratio, superplasticizer/binder, and sodium silicate concentration were kept constant. The fly ash/slag ratio successively was 50/50, 30/70, and 10/90 and the binder quantity was 460, 650, and 1050 kg·m<sup>-3</sup>. The compression strength determining showed that mixtures with 0.15 activator/binder ratio and high binder content favoured obtaining the highest resistance values (58.3-76.3 MPa after 28 days). Flexural strength values increased with lower ash/slag ratio and higher activator/binder ratio, reaching between 1.64-5.86 MPa after 28 days of curing.

A comparison between “one-part” and “two-part” methods for the preparation of alkali-activated materials was presented in (Segura *et al.*, 2022). The comparative analysis showed that the preparation of materials by “one-part” technique is a safer and easier operation, being especially suitable for in-situ casting applications of geopolymer as a construction material. In this work, metakaolin mixed with granulated blast furnace slag were used as alumina-silicate precursors. Experimental results showed that the use of “one-part” system delays the setting time, increases the reaction heat, decreases the shrinkage and ensures a compression strength evaluated at 80-85% compared to corresponding strength of the conventional “two-part” mixture. In addition, the microstructural features of materials made in “one-part” mixture are almost similar to those of materials obtained through “two-part” technique.

According to (Luukkonen *et al.*, 2018), making “one-part” alkaline-activated geopolymers as alternative binders represents a suitable technical solution for construction applications involving in-situ casting. However, in the authors' opinion, further research in this area is needed to demonstrate the long-term durability of these materials.

Several works published in recent literature noted the above-mentioned advantages of “one-part” alkali-activated materials (Qin *et al.*, 2022; Elzeadani *et al.*, 2022; Yusslee and Beskhyroun, 2022; Finocchiaro *et al.*, 2024).

Although so far, the team of the current paper authors has gained a very good experience in the field of porous and high-strength conventional geopolymer production in the last years (Paunescu *et al.*, 2023a; Paunescu *et al.*, 2023b), the “one-part” mixture geopolymer method has not yet been tried. “One-part” mixture geopolymer versions designed and experimentally tested are presented below.

## 2. Methods and Materials

The binding phases of alkali-activated “one-part” geopolymers were identified as N-A-S-H and C-A-S-H gels similar to those in the “two-part” materials. N-A-S-H phase in geopolymer materials (sodium alumina silicate hydrate) is the main binding phase and is the first reaction product in geopolymerization. N-A-S-H gel forms an interconnected network of alumina-silicate tetrahedrons, contributing to the strength and durability of the geopolymer (Li *et al.*, 2020). C-A-S-H phases (calcium alumina silicate hydrate) play an important role in the strength and durability of the material. They are formed during the hydration process (Guo *et al.*, 2024).

It appears that the hardening mechanism of the alkali-activated “one-part” geopolymer consists of a very rapid dissolution reaction of the solid activators immediately after the addition of water, followed by slower dissolution reactions of the alumina-silicate components. Previous determinations have shown that the release rate of silicon from the solid silica source occurs much more slowly in the “one-part” geopolymer method compared to the conventional “two-part” procedure.

The new method of alkali-activated “one-part” mixture geopolymer requires a dry mixture consisting of solid alumina-silicate precursors, solid alkali sources and other possible solid additives, to which water is subsequently added, the preparation method being relatively similar to that of ordinary Portland cement. The component materials chosen for this dry mixture were: coal fly ash and granulated blast furnace slag as the alumina-silicate precursor mixture, sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) as the solid alkaline activator, and sodium lignosulphonate with the role of water-reducing superplasticizer.

The most well-known and frequently used industrial alumina-silicate by-product is coal fly ash. Recovered by electrostatic separation capture during the fine purification process of waste gases produced by coal burning in thermal power plants, fly ash has a particle size of less than 250  $\mu\text{m}$  and a content of 48.1%  $\text{SiO}_2$ , 26.4%  $\text{Al}_2\text{O}_3$ , 3.2%  $\text{MgO}$ , 3.6%  $\text{CaO}$ , 6.9%  $\text{Fe}_2\text{O}_3$ , 5.8%  $\text{Na}_2\text{O}$ , and 3.9%  $\text{K}_2\text{O}$ . The fly ash used in this experiment was acquired about 10 years ago from the Rovinari-thermal power plant (Romania) and the material was subjected to additional mechanical processing to reduce the grain size below 80  $\mu\text{m}$ .

Granulated blast furnace slag is also a frequently used by-product of the cast-iron industry as an alumina-silicate precursor for geopolymer making. The slag is industrially wet granulated to dimensions between 2-7 mm. Grinding the granulated product in ball mill allowed to decrease the slag granulation below 70  $\mu\text{m}$ . The slag was obtained by the authors more than 10 years ago from ArcelorMittal Galati (Romania) and stored for various subsequent experiments. The chemical composition of the slag includes 36.8%  $\text{SiO}_2$ , 12.0%  $\text{Al}_2\text{O}_3$ , 41.2%  $\text{CaO}$ , 5.6%  $\text{MgO}$ , 0.6%  $\text{MnO}$ , 0.8%  $\text{Fe}_2\text{O}_3$ , 0.3%  $\text{Na}_2\text{O}$ , and 0.4%  $\text{K}_2\text{O}$ .

Sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) in fine powder state originated in China was adopted as the solid alkaline activator. The purity of this water-soluble product was of 99.5%.

Sodium lignosulphonate as a water-reducer superplasticizer in a powder state has the ability to decrease the water/solid ratio up to 0.45, improving the compression strength. This additive chosen to complete the solid mixture is usually used in very low weight ratios (around 0.5 wt. %).

Investigating methods of the “one-part” mixture geopolymer specimens are presented below. The geopolymer density was determined by Archimedes’ method through the liquid intrusion technique (ASTM D792-20). The apparent porosity was determined using a vacuum saturation method (Kearsley and Wainright, 2002). Heat conductivity was investigated by the guarded-comparative-longitudinal heat flow method (ASTM E1225-04). Compression strength was measured with TA.XTplus Texture Analyzer and the flexural resistance was determined by carrying out the three-point bend test on the specimen (SR EN ISO 14125: 2000). Water uptake was measured through the immersion procedure of the specimen under water for 24 hours (ASTM C948) at the end of the curing process. The structural appearance of specimens was analysed with the Biological Microscope model MT5000, 1000 x magnification.

### 3. Results and Discussion

Four experimental versions of the one-part mixture geopolymer were adopted including the above-mentioned components. The composition of these versions is shown in Table 1.

**Table 1**  
*Composition of experimental versions*

Material	Version ( $\text{kg}\cdot\text{m}^{-3}$ )			
	1	2	3	4
Coal fly ash	200	250	300	350
Granulated blast furnace slag	250	200	150	100
Sodium carbonate	86	88	90	92
Sodium lignosulphonate	2	2	2	2
Water	241	242	243	244

According to the data presented in Table 1, the powder mixture including coal fly ash and granulated blast furnace slag was maintained at a constant weight value of  $450 \text{ kg}\cdot\text{m}^{-3}$ , the proportions of the two components being varied within the limits of 0.8:1 and 3.5:1. The solid alkaline activator  $\text{Na}_2\text{CO}_3$  increased from 86 to 92  $\text{kg}\cdot\text{m}^{-3}$ , while sodium lignosulphonate had a constant value in all tried experimental versions (2  $\text{kg}\cdot\text{m}^{-3}$ ). The amount of water subsequently added to the solid mixture slightly varied from 241 to 244  $\text{kg}\cdot\text{m}^{-3}$ .

Determining the physio-mechanical, heat, and morphological features of the “one-part” geopolymer samples was carried out at the end of 28 curing days at ambient temperature. Supplementary, determining the compression and flexural strength was also made after early 7 curing days. Results of these measures are presented in Table 2.

**Table 2**  
*Features of “one-part” geopolymer specimens*

Feature	Experimental version			
	1	2	3	4
Apparent density ( $\text{kg}\cdot\text{m}^{-3}$ )	645	592	544	479
Apparent porosity (%)	58.2	62.6	68.0	76.6
Heat conductivity ( $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ )	0.167	0.141	0.123	0.116
Compression strength (MPa)				
- after 7 days	25.6	20.3	15.0	8.9
- after 28 days	35.9	30.0	24.2	18.6
Flexural strength (MPa)				
- after 7 days	7.0	6.1	5.2	4.9
- after 28 days	9.8	8.9	8.0	7.3
Water uptake (vol. %)	3.5	3.1	2.9	2.8
Pore size ( $\mu\text{m}$ )	40-75	70-125	90-140	390-490

Examining the experimental results, it is observed that the one-part geopolymer tested in this experiment is a porous material with a tendency to have satisfactory thermal insulation properties, especially as the proportion of fly ash mixed with metallurgical slag sharply increases. The data in Table 2 show the significant decrease of the apparent density from 645 to 479  $\text{kg}\cdot\text{m}^{-3}$ , influenced by the increase of the fly ash/slag ratio from 0.8 to 3.5. Also, heat conductivity decreased from 0.167 to 0.116  $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , basically for the same reason.

The geopolymer strength made without expensive additions of nanomaterials or reinforcing fibres, which significantly increase its value, has reached moderate levels at the end of the 28 curing days process, but the high level of early compression and flexural strength (after only 7 days) was noted.

The early values of the two types of strength are already high (between 8.9-25.6 MPa for compression strength and between 4.9-7.0 MPa for flexural strength), considering the results of the final strength determining (between 18.6-35.9 MPa for compression strength and between 7.3-9.8 MPa for flexural strength).



The optimal versions in terms of mechanical strength were those in which the amount of  $\text{Na}_2\text{CO}_3$  as solid alkaline activator was lower (versions 1 and 2, but especially version 1) as well as in which the fly ash/slag ratio was low (0.8-1.25).

Images of the physical appearance of specimens made in the four experimental versions are shown in Fig. 1.

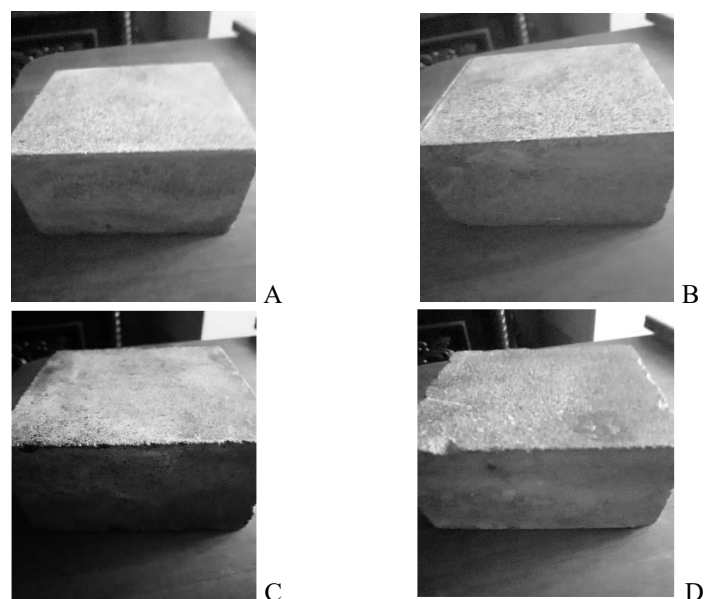


Fig. 1 – Physical appearance of specimens

A – version 1; B – version 2; C – version 3; D – version 4.

The microstructural aspect of the one-part geopolymer samples is exposed in Fig. 2.

If the difference between the physical appearance of the specimens shown in Fig. 1 is not very clearly noticeable, that between the microstructural peculiarities of the same samples presented in Fig. 2 is very evident by magnifying the images taken with the microscope

In general, the microstructure of the four samples is characterized by structural homogeneity and relative uniformity of dimensions of the component cells. Also, the microstructural network is closed without coalescence tendencies between neighbouring cells. The identified cell sizes of the samples are in the following ranges (see Table 2): 40-75  $\mu\text{m}$  (sample A), 70-125  $\mu\text{m}$  (sample B), 90-140  $\mu\text{m}$  (sample C), and 390-490  $\mu\text{m}$  (sample D).

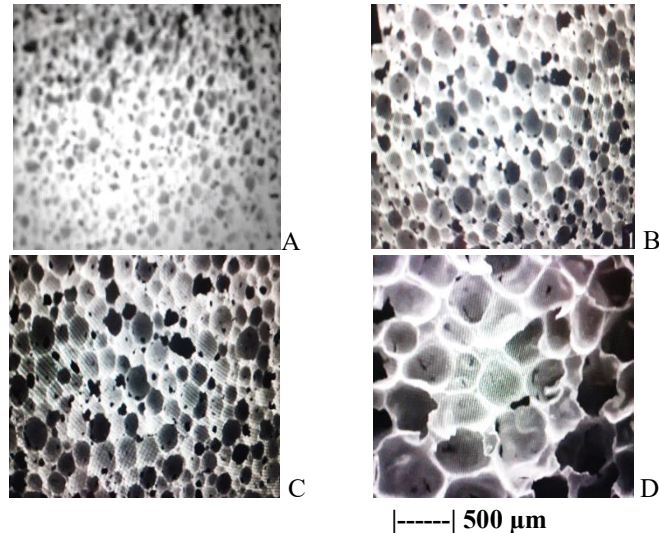


Fig. 2 – Microstructural aspect of geopolymer specimens  
A – version 1; B – version 2; C – version 3; D – version 4.

As a summary of the current work, the following aspects are appropriate for discussion. In the last 10-15 years, the new method of “one-part” mixture geopolymer has gained the attention and interest of construction specialists as an alternative version of the “two-part” geopolymer method. Unlike the currently applied traditional “two-part” method, the new method offers an advantageous technical solution for works that require in-situ construction operations.

Numerous researches conducted in this recent period have used a significant number of alumina-silicate precursors, additional sources of silica and alumina, solid alkaline activators, additives, reinforcing fibres, fresh geopolymer curing techniques, etc. Also, the research has indicated possible geopolymerization mechanisms and bonding phases and has evaluated the properties of “one-part” geopolymers.

According to the opinion of some of the research teams involved in the development of the “one-part” mixture geopolymer technique, the knowing process of this manufacturing method should not be considered complete, because other supplementary testing and documentations are still necessary to demonstrate the long-term durability of these materials prepared by the above-mentioned method.

#### 4. Conclusions

The “one-part” mixture geopolymers is an alternative variant to the currently used traditional “two-part” mixture, which has recently become an interesting solution, especially for in-situ geopolymer casting operations. The

work had as objective to produce and test a “one-part” geopolymer composite using coal fly ash and granulated blast furnace slag as alumina-silicate precursors, sodium carbonate as a solid alkali activator, and sodium lignosulphonate as a water-reducer superplasticizer, forming a dry powder mixture, to which water was added later. The fly ash/slag weight ratio was varied in the four variants between 0.8-3.5 and this influenced both the thermal insulation properties (density, heat conductivity, and porosity) and the compression and flexural strength of the geopolymer. Thus, the apparent density significantly decreased between versions 1 and 4 from  $645$  to  $479 \text{ kg}\cdot\text{m}^{-3}$  and heat conductivity also decreased from  $0.167$  to  $0.116 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , while the apparent porosity increased from  $58.2$  to  $76.6\%$ . The compression strength reached its maximum value ( $35.9$  MPa) in case of variant 1 after 28 curing days and significantly decreased to  $18.6$  MPa in case of variant 4 after 28 days. The early strength (after 7 days) had a high maximum value ( $25.6$  MPa) corresponding to variant 1 and decreased to  $8.9$  MPa in variant 4. A similar situation was also registered in case of flexural strength. Version 1 reached the highest values of the strength ( $7.0$  MPa) after 7 days and ( $9.8$  MPa) after 28 days.

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TENDINȚĂ NECONVENȚIONALĂ ÎN ADOPTAREA  
NOULUI AMESTEC GEOPOLIMERIC „MONOCOMPONENT” PENTRU  
APLICAȚII ÎN CONSTRUCȚII

(Rezumat)

Lucrarea a vizat producerea unui material compozit geopolimeric „monocomponent” utilizând cenușă zburătoare de cărbune și zgură granulată de furnal ca precursori aluminosilicatici, carbonat de sodiu ca activator alcalin solid și lignosulfonat de sodiu ca superplastifiant reducător de apă, constituind un amestec uscat de pulberi, la care s-a adăugat ulterior apă. Această metodă de preparare a geopolimerului este o versiune alternativă la geopolimerul tradițional „bicomponent”, care utilizează activarea alcalină lichidă și este potrivită, în principal, pentru aplicații de construcții in situ. Rezultatele experimentului actual au arătat proprietăți bune de izolare termică, valorile

densității și conductivității termice fiind destul de scăzute, precum și niveluri destul de ridicate de rezistență la compresiune și încovoiere, valorile timpurii ale rezistenței după numai 7 zile fiind ridicate, având în vedere valorile finale ale rezistenței mecanice după 28 de zile.