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## FLY ASH-GEOPOLYMER FOAM ACTIVATED IN ALKALINE ENVIRONMENT BASED ON POTASSIUM

BY

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**Abstract.** Fly ash-geopolymer foam was made under the conditions of choosing an alkaline activator solution composed of potassium hydroxide and potassium silicate compared to the sodium-based ones usually used. The adopted expanding agent was sodium perborate substituting the known hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), silica fume was chosen due to its ability for increasing the foam strength, and olive oil was the agreed surfactant replacing more effective but more expensive materials. The results showed porous products with excellent heat-insulating properties and simultaneously satisfactory compression and flexural resistance obtained at an early age (7 days) the quality level being almost similar to that of the best products worldwide.

**Keywords:** fly ash, geopolymer foam, sodium perborate, silica fume, heat-insulating properties.

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## 1. Introduction

Long-time durability, good thermal stability, high compressive strength also at early age, fast hardening, acid resistance are the main remarkable properties of new construction material types obtained by the geopolymerization reaction of alumina-silicate waste in alkaline environment. In addition, relatively low making costs complete the mentioned advantages. The notion of geopolymerization belongs to the French inventor J. Davidovits in the last decade of the 20<sup>th</sup> century (Davidovits, 1991). His innovative idea was published at a time when the effects of the global hydrocarbon crisis and the identification of the danger of overheating the earth's atmosphere due to excessive emissions of greenhouse gases (CO<sub>2</sub>) had become problems whose resolution was urgent at the global level.

Geopolymers have become extremely attractive and the main effect was the restriction of the industrial manufacturing activity of Portland cement considered largely responsible for the huge consumption of fossil fuel and CO<sub>2</sub> emissions in the atmosphere. Thus, the most frequently and intensively used construction material of the last two hundred years, with the role of binder for the production of concrete, also a material of great importance for humanity, must be replaced with a viable alternative version. Geopolymer concrete thus came to the attention of researchers and manufacturers from worldwide.

The geopolymer is an amorphous binder composed of alumina-silicate materials (such as fly ash, metakaolin, metallurgical slag, red mud, etc.) formed by polycondensation as a result of mixing them with an alkaline solution. According to (Kim *et al.*, 2022), the geopolymer structure is composed of disorderly connected silicon tetrahedra, with part of their cations replaced by aluminum. The imbalance caused by cation substitution is compensated by the materials contained in the alkaline activator.

An interesting application source is represented by geopolymer foams, porous materials with low shrinkage and high strength after moulding at ambient temperature due to their chemical and mechanical stability (Novais *et al.*, 2020). Geopolymer foam is more durable and more environmentally friendly compared to Portland cement-based foam due to the lower carbon footprint. The application fields of geopolymer foam are primarily focused on the construction sector due to its high resistance and ability for thermal and acoustic insulation, but also on photocatalytic degradation applications, catalyst supports, membrane supports, humidity control, sound absorption, and heavy metal adsorption.

The geopolymer foam determines specific properties of the porous material such as the distribution and size of pores, mechanical strength, thermal conductivity due to the pore interconnection (Seeber *et al.*, 2013).

The manufacture of geopolymer foam involves the use of an expanding agent in the mixture such as hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), sodium hypochlorite (NaOCl), silica fume or aluminum powder (Feng *et al.*, 2015; Ducman and Korat,

2016; Prud'homme *et al.*, 2010). Of these, hydrogen peroxide is industrially used on a large scale, having the option to decompose due to the instability of the H-O bond. The making process of fly ash-based geopolymer foam intensively uses  $H_2O_2$  as an expanding agent because it significantly favours the heat-insulating properties of the geopolymer (Pedziwiatr *et al.*, 2018; Novais *et al.*, 2016). The type of synthetic additive largely determines the thermal conductivity of the geopolymer foam. The inventor of geopolymer J. Davidovits used for the production of geopolymer foam  $H_2O_2$  and sodium perborate ( $NaH_2BO_4$ ) as foaming agents obtaining a porous material with density between  $0.2-0.8\text{ g}\cdot\text{cm}^{-3}$ , the highest thermal resistance at  $1200^\circ\text{C}$ , and heat conductivity of  $0.037\text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  (Davidovits, 2020).

Geopolymer foam was made according (Jiang *et al.*, 2020) using sodium dodecyl sulfate surfactant, which has the ability of surface activity, stability of air bubbles and implicitly, of pores. Previous studies have shown that some salts (such as  $CaCl_2$ ,  $NaCl$ ,  $KCl$ ), with which the foam inevitably comes into contact, have an important influence on the foaming process and on stability of the foam obtained using the mentioned surfactant.

The expansion method of the raw material by releasing and blocking inside the viscous mass of hydrogen bubbles resulting from the corrosion reaction of aluminum powder in an aqueous solution of hydrated lime- $Ca(OH)_2$  in order to produce a lightweight material based on gypsum (Paunescu *et al.*, 2022) was inspired by the manufacturing technique of autoclaved aerated concrete. Aluminum powder was prepared in the own experimental plant by unconventional microwave melting of aluminum as a recycled waste and atomization of this melt with strong jets of nitrogen gas. The product density was low ( $530-600\text{ kg}\cdot\text{m}^{-3}$ ), the compression resistance being satisfactory ( $1.2-2.2\text{ MPa}$ ).

The same method based on the own original making technique under energy-saving conditions of aluminum powder was applied to create a high-strength and heat-insulating cellular building concrete based on calcined gypsum. High proportions of calcined gypsum (74.7-82.3%) constituting the main binder, to which hydrated lime (5-7.8%), fly ash (3.6-11.1%), perlite (4.6-6.4%), and silica fume (0.3-1.2%) were added, allowed obtaining a building material structure with very small pores, low density (under  $641\text{ kg}\cdot\text{m}^{-3}$ ), and compression resistance up to  $4.1\text{ MPa}$  (Ioana *et al.*, 2022).

A geopolymer foam based on fly ash and metakaolin was designed and tested, applying the established method in the last decades regarding the alkaline activation of alumina-silicate waste in order to initiate and develop the geopolymerization reaction (Paunescu *et al.*, 2023a). The traditional expanding agent ( $H_2O_2$ ) has been substituted with sodium perborate ( $NaH_2BO_4$ ), a more stable and easier to use agent. The originality of the manufacturing recipe was the choice of a nanomaterial (bentonite clay), whose ability favours increasing the mechanical resistance of the porous material. The density and thermal

conductivity values ( $470 \text{ kg}\cdot\text{m}^{-3}$  and  $0.104 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , respectively) were low, but the compressive strength value increased, reaching 7.5 MPa.

Other geopolymer foam with heat-insulating properties was experimentally prepared by expanding the alumina-silicate mixture (fly ash and clay brick waste recycled from building demolition) activated by the contact with aqueous solution of sodium hydroxide and sodium silicate). Except for the two predominant alumina-silicate waste, they were also used in the starting mixture expanded perlite as a siliceous additive, olive oil as a surfactant as well as quartz sand as fine aggregate. Geopolymer foam features were: density between  $480\text{-}577 \text{ kg}\cdot\text{m}^{-3}$ , heat conductivity in the range of  $0.098\text{-}0.126 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , and compression resistance within the limits of 4.0-5.7 MPa (Paunescu *et al.*, 2023b).

Sodium perborate monohydrate ( $\text{NaH}_2\text{BO}_4$ ) with the role of expanding agent and sodium dodecyl sulfate ( $\text{NaC}_{12}\text{H}_{25}\text{SO}_4$ ) as surfactant with role of foam stabilizer, both in ratios between 1-3%, were used to produce in alkaline medium a fly ash-based geopolymer foam (Korat and Ducman, 2020). The curing process at  $70^\circ\text{C}$  for 1 day was applied, followed by storing the specimens for 3 days. The density geopolymer foam was between  $330\text{-}670 \text{ kg}\cdot\text{m}^{-3}$ , heat conductivity in the range of  $0.143\text{-}0.205 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , and compression resistance within the limits of 1.02-6.33 MPa.

In the current work, authors opted for the following composition of the mix used for preparing a fly ash-based geopolymer foam, in which the main change concerned replacing the alkaline activator composition, usually containing sodium silicate and sodium hydroxide in form of an aqueous solution, with silicate potassium and hydroxide potassium, respectively, in the same physical state. The expanding agent was chosen sodium perborate monohydrate, as a more stable and easier to handle agent compared to  $\text{H}_2\text{O}_2$  and an accepted vegetable oil (olive oil) (Bai *et al.*, 2018), more commercially available than the expensive surfactants mentioned in the literature.

## 2. Materials and Methods

Materials introduced into the solid mix for making the geopolymer foam were the following: class F fly ash with low CaO content (according to ASTM C 618-12), as the cementitious binder, sodium perborate monohydrate ( $\text{NaH}_2\text{BO}_4$ ) as an expanding agent, silica fume as a nano-material contributing to increase the foam strength, and olive oil as a vegetable surfactant having the role of foam stabilizer.

Fly ash used in this experiment had been received from Paroseni-Thermal Power Plant (Romania) and stored for different subsequent experiments 8 years ago, under the conditions that anthracite was the used fuel. The oxide composition of the ash recovered from the boiler electro-filter included: 54.4%  $\text{SiO}_2$ , 26.5%  $\text{Al}_2\text{O}_3$ , 4.3%  $\text{Fe}_2\text{O}_3$ , 3.5%  $\text{CaO}$ , 2.5%  $\text{MgO}$ , 1.5%  $\text{TiO}_2$ , 0.4%  $\text{Na}_2\text{O}$ , 0.6%  $\text{K}_2\text{O}$ , and 1.7%  $\text{SO}_3$ .

The common form of sodium perborate is monohydrate. It is a solid material soluble in water. Through its reaction with water, sodium perborate monohydrate releases hydrogen peroxide, which is a source of active hydrogen. The main application of sodium perborate is in the detergent domain (Burgess and Hubbard, 2013). It is commercially available (from China) in an almost pure state (96-98% purity). In the making process of metakaolin-based geopolymer foam using sodium perborate as an expanding agent (Wattanarach *et al.*, 2022), its weight proportion was within the limits of 0.5-2%.

Silica fume is a by-product of industrial making process of silicon metal or ferrosilicon alloys. Due to its chemical and physical features, it is a very reactive pozzolan. Applied in the case of concretes, silica fume as an extremely fine material (under 1  $\mu\text{m}$ ) significantly contributes to increasing the strength and durability. Silica fume mainly contains over 85% amorphous silicon dioxide ( $\text{SiO}_2$ ), according to ASTM C 1240 standard (What is silica fume, 2024).

Olive oil as a vegetable oil (Bai *et al.*, 2018) was used as an alternative to the expensive products on the market usable as surfactants (sodium lauryl sulfate, sodium dodecyl sulfate, etc.). Generally, the surfactant is a surface activating agent that changes the surface tension of a liquid.

The alkaline solution having the major role of activating the alumina-silicate by-product of the energy industry (coal fly ash) was chosen to be composed of potassium hydroxide (KOH) in form of solid pellets dissolved in deionized water (with 10 M molarity) and potassium silicate ( $\text{K}_2\text{SiO}_3$ ) in form of aqueous solution (38% concentration). The alkaline activator composition different from most of compositions used in previous geopolymer making processes, including sodium hydroxide and sodium silicate, still corresponds to the theoretical recommendation of the inventor J. Davidovits, who also mentioned these alkaline components in his patents.

Four experimental versions for the making recipe of fly ash-based geopolymer foam were chosen and they are shown in Table 1.

**Table 1**  
*Experimental version of making recipe*

Composition	Version ( $\text{kg}\cdot\text{m}^{-3}$ )			
	1	2	3	4
Class F fly ash	319.0	269.3	240.9	218.9
Sodium perborate	8.3	9.5	11.0	12.9
Silica fume	2.9	3.1	3.4	3.8
Olive oil	2.4	2.5	2.6	2.8
10 M KOH	47.6	47.6	47.6	47.6
$\text{K}_2\text{SiO}_3$	115.0	115.0	115.0	115.0
Deionized water	120.0	120.0	120.0	120.0

According to the data in Table 1, the alumina-silicate binder (fly ash) varied between 218.9-319.0 kg·m<sup>-3</sup>, decreasing from version 1 to version 4. Instead, sodium perborate, silica fume, and olive oil had increasing values within the limits of 8.3-12.9 kg·m<sup>-3</sup>, 2.9-3.8 kg·m<sup>-3</sup>, and 2.4-2.8 kg·m<sup>-3</sup>, respectively. The alkaline activator composition was kept constant in all versions, with K<sub>2</sub>SiO<sub>3</sub>/KOH ratio of 2.42. The fly ash/alkaline activator ratio had values starting from 1.96 in the case of version 1 and decreasing to 1.35 in version 4.

Sodium perborate monohydrate (NaH<sub>2</sub>BO<sub>4</sub>) was adopted in this experiment as an expanding agent. Its hydrolysis through contact with the aqueous solution releases hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), hydroperoxyl anion OOH<sup>-</sup>, and tetrahydroxyborate anion B(OH)<sub>4</sub><sup>-</sup> (Schubert, 2015). As a result of H<sub>2</sub>O<sub>2</sub> decomposition, hydrogen and oxygen both in molecular state are released and they remain trapped into the viscous mass of alumina-silicate material in the form of gas bubble contributing to the formation of the final product porous structure.

The manufacture of geopolymer foam involves the transformation of alumina-silicate waste into a geopolymer by its activating in the created alkaline environment, which allows the start and development of the geopolymerization reaction. This conversion process includes the formation of a three-dimensional polymer chain composed of Si-O-Al-O bonds. According to (Provis and Rees, 2009) (Provis and Rees, 2009), this structural transformation constitutes a highly complex process including three stages that can influence each other. Until now, the mechanism of the geopolymerization process has not been fully elucidated and needs to be further researched.

The geopolymer preparing method was adopted taking into account the general rules applied in the literature of the last years, but also in the own experiments carried out by the Romanian research team, which includes the authors of the current paper.

Thus, the preparation of the alkaline activator in liquid solution state was performed about 24 hours before the preparation of the solid mixture. The formation of the alkaline activator was obtained in a separate container, using KOH pellets dissolved in deionized water with 10 M molarity, mixed with K<sub>2</sub>SiO<sub>3</sub> aqueous solution. Their mixing was made by stirring with a rotary device (1000 rpm) for 4-5 min. In another container, the solid components of the material mixture composed of finely ground fly ash under 800 μm, sodium perborate monohydrate as an expanding agent, and silica fume as a nano-material were mixed. Their mixing was achieved by stirring with an electrically operated device (700 rpm) for 15 min. The contact between the alkaline activator and the solid mixture was achieved by slowly pouring the liquid mixture over the solid one and continuing to stir the new mixture with the same electrical device for another 5 min until the paste is formed. The liquid surfactant (olive oil) was added during the final mixing.

The curing process of the fresh geopolymer poured into metal moulds was adopted at room temperature in a thermally insulated oven at low humidity

for 2 days, according to (Kim *et al.*, 2022) which describes a fast-curing process for preparing metakaolin-based geopolymer foam. After hardening the fresh geopolymer, the specimens removed from the moulds were also stored at ambient temperature in a relatively dry atmosphere for 7 days.

The main methods applied in this paper for identifying the characteristics of geopolymer foam specimens are noted below. The apparent density of geopolymer foam specimens was measured using Archimedes' principle. The apparent porosity was determined by dividing the difference between wet and dry weight by the difference between wet weight and suspended weight of the sample (ASTM C642-97). Heat conductivity was identified at room temperature using the heat-flow method (SR EN 1946-3:2004). 1000 kN-compression fixture Wyoming Test Fixture was utilized for determining the compression resistance. The flexural strength was measured using the method indicated in SR EN ISO 1412:2000 (the three-point bending test). Sample immersion under water for 1 day (ASTM D570) allowed to determine the water-absorbing. Microstructural particularities of specimens were investigated with ASONA 100X Zoom Smartphone Microscope.

### 3. Results and Discussion

The investigation of physical, thermal, mechanical, and morphological features of geopolymer foam samples was carried out after the curing process of the fresh material for 2 days and the storage at room temperature for 7 days. The results of these determinations are presented in Table 2.

**Table 2**  
*Results of investigating the features of geopolymer foam samples*

Feature	Version 1	Version 2	Version 3	Version 4
Apparent density ( $\text{kg}\cdot\text{m}^{-3}$ )	495	447	421	401
Apparent porosity (%)	74.7	76.4	78.1	80.2
Heat conductivity ( $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ )	0.097	0.090	0.083	0.076
Compression resistance after 7 days (MPa)	4.3	4.5	5.1	5.4
Flexural resistance after 7 days (MPa)	2.8	2.9	3.1	3.6
Water-absorbing (vol. %)	3.9	4.5	5.7	9.5
Pore size (mm)	0.1-0.4	0.1-0.5	0.1-0.6	0.3-1.1

According to experimental results in Table 2, they were obtained geopolymer foam specimens with suitable heat-insulating properties and in the same time, with more than acceptable mechanical resistance at early age (7 days). Thus, the apparent density fell within the limits of low values ( $401\text{-}495\text{ kg}\cdot\text{m}^{-3}$ ), the specimens being slightly denser in the case of versions 1 and 2, in which the expanding agent (sodium perborate) was mixed in proportions below  $10\text{ kg}\cdot\text{m}^{-3}$ . Increasing this proportion up to  $12.9\text{ kg}\cdot\text{m}^{-3}$  created the conditions for developing pores with sizes that reached  $1.1\text{ mm}$  and the porosity of specimen 4 increased up to  $80.2\%$ .

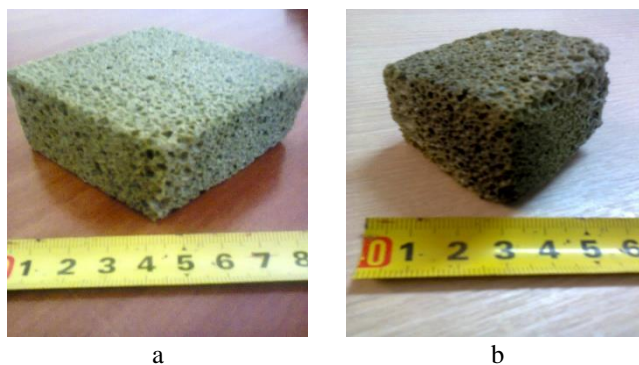
Heat conductivity of the four samples had values under  $0.1\text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , confirming their ability to offer very good thermal insulation.

Compression resistance of these specimens reached an acceptable level at an early age (after 7 days of hardening), strength values falling within the range of  $4.3\text{-}5.4\text{ MPa}$ . Increasing the consumption of silica fume up to  $3.8\text{ kg}\cdot\text{m}^{-3}$  decisively influenced the increase of the geopolymer compression strength.

Increasing the flexural strength was also noticed at an early age, the values corresponding to the four specimens between  $2.8\text{-}3.6\text{ MPa}$  being satisfactory and acceptable in the case of geopolymer foam.

Due to the relatively high volume of permeable voids that tend to absorb more water amount, determining the water-absorbing of geopolymer samples showed a fairly high absorption level within the limits of  $3.9\text{-}9.5\text{ vol. \%}$ , the highest absorption degree being reached in the case of the sample corresponding to version 4.

The physical appearance of geopolymer foam samples is shown in Fig. 1 and their microstructural peculiarities are presented in Fig. 2.





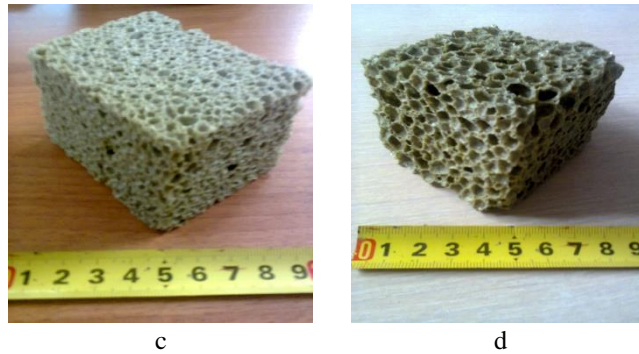


Fig. 1 – Physical appearance of geopolymer foam samples  
a – sample 1; b – sample 2; c – sample 3; d – sample 4.

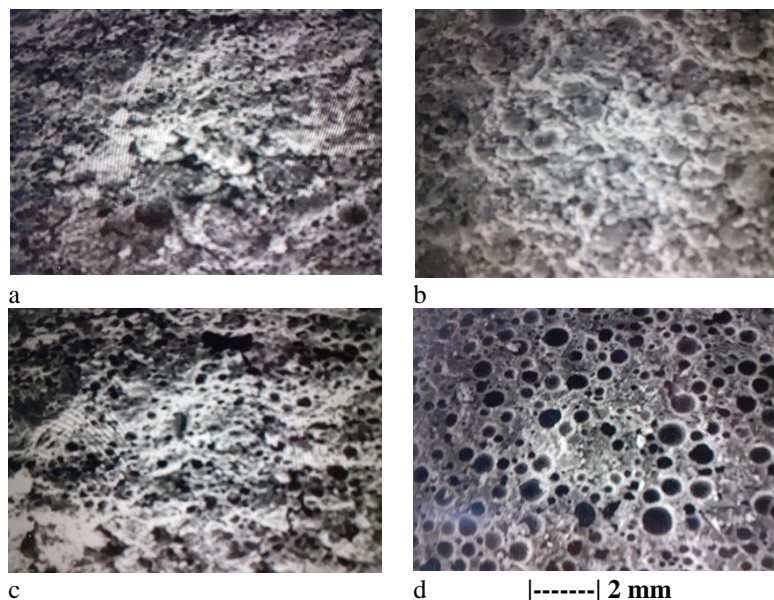


Fig. 2 – Microstructural peculiarities of geopolymer foam samples  
a – sample 1; b – sample 2; c – sample 3; d – sample 4.

The evolution of the outer surface appearance of specimens can be observed in Fig. 1. The size of the outer pores is continuously increasing, reaching the highest values in the case of the sample corresponding to version 4.

Figure 2 shows the appearance of the four specimens in cross section. The sample according to version 4 is characterized by a fairly homogeneous porous microstructure, in which most of the relatively large pores between 0.8-1.1 mm are together with significantly smaller pores up to 0.3 mm. The other sections corresponding to samples 1-3 have pores with considerably smaller

sizes, according to the data included in Table 2. Sample 3 contains pores with sizes between 0.1-0.6 mm, being attractive in terms of microstructure for thermal insulation making.

As mentioned above, the geopolymer foams can be employed as heat-insulating materials and ceramic precursor. Foams are also adequate for bulk-type adsorbents, pH buffering agents, and catalysts. Recently, possibility of tailoring their porosity, adsorbing, and leaching has increased the interest for applications in environment remediation (such as wastewater treatment), and renewable energy making (such as biogas) (Novais *et al.*, 2020).

In the current paper, the pursued goal was achieving a porous and strength material for thermal insulation. Therefore, due to the relatively large size pores, the sample corresponding to version 4 was not considered interesting by the authors, being probably suitable for some of the other applications mentioned above. Thus, the specimen obtained in experimental version 3 met the required assessment to be considered as the optimal sample of this test. Heat-insulating properties (apparent density of  $421 \text{ kg}\cdot\text{m}^{-3}$ , heat conductivity of  $0.083 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , and apparent porosity of 78.1%), mechanical strength values at an early age (compression resistance of 5.1 MPa, and flexural strength of 3.1 MPa), pore size between 100-600  $\mu\text{m}$  as well as water-absorbing of 5.7 vol. % (i.e. in acceptable limits) are features of the sample made in the optimal version.

Numerous studies carried out in the last 10-15 years aimed at the realization of geopolymer foam with insulating properties. According to (Lach, 2021), foams were obtained with densities whose values started from  $200 \text{ kg}\cdot\text{m}^{-3}$ , while thermal conductivities started from  $0.04 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ . Of course, in these experiments, the materials used as the gas-supplying agent for expanding varied greatly, including aluminum powder, silica fume, FeSi, SiC, sodium hypochlorite,  $\text{H}_2\text{O}_2$ , and sodium perborate. Alkaline environment was used in all cases for developing the geopolymerization process. Different surfactants (from expensive materials as sodium lauryl sulfate or sodium dodecyl sulfate, up to vegetable oils as alternative materials) were applied for stabilizing the foam. Under conditions of adopting some less used and less expensive materials, the current work obtained results quite close to those of the top.

#### 4. Conclusions

The work objective was achieving fly ash-based geopolymer foam intended for thermal insulation. Several changes made in the composition of the starting mixture aimed at replacing the usual expanding agent hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) with sodium perborate, a more stable and easier to handle agent. Also, the expensive surfactants usually used have been substituted with a vegetable oil (olive oil). The most important change in the geopolymer making recipe was adopting the composition of alkaline activator consisting of potassium hydroxide (KOH) and potassium silicate ( $\text{K}_2\text{SiO}_3$ ), abandoning the usual sodium-based

similar products. Of the four tested experimental versions, version 3 was adopted as the optimal solution. Its heat-insulating properties (apparent density of  $421 \text{ kg}\cdot\text{m}^{-3}$ , heat conductivity of  $0.083 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ , and apparent porosity of 78.1%), mechanical resistance values at an early age (compression strength of 5.1 MPa, and flexural strength of 3.1 MPa), and pore size within the limits of 100-600  $\mu\text{m}$  were the main characteristics of the optimal specimen.

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SPUMĂ GEOPOLIMERICĂ DIN CENUȘĂ ZBURĂTOARE ACTIVATĂ  
ÎN MEDIU ALCALIN PE BAZĂ DE POTASIU

(Rezumat)

Spuma geopolimerică din cenușă zburătoare a fost fabricată în condițiile alegerii unei soluții de activator alcalin compusă din hidroxid de potasiu și silicat de potasiu, față de acelea pe bază de sodiu utilizate în mod obișnuit. Agentul de expansiune adoptat a fost perboratul de sodiu substituind cunoscutul peroxid de hidrogen ( $H_2O_2$ ), praful de silice a fost ales datorită abilității lui de creștere a rezistenței spumei, iar uleiul de măsline a fost surfactantul agreat, înlocuind materiale mai eficiente, dar mai scumpe. Rezultatele au arătat produse poroase cu excelente proprietăți termoizolante și simultan, rezistențe satisfăcătoare la compresie și încovoiere obținute în termen scurt (7 zile), nivelul calitativ fiind aproape similar cu acela al celor mai bune produse din lume.