

BULETINUL INSTITUTULUI POLITEHNIC DIN IAȘI

Publicat de

Universitatea Tehnică „Gheorghe Asachi” din Iași

Volumul 69 (73), Numărul 1, 2023

Secția

CHIMIE și INGINERIE CHIMICĂ

DOI: 10.5281/zenodo.7767038

## ENVIRONMENTAL FRIENDLY MANUFACTURING THE GEOPOLYMER FOAM FROM ALUMINOSILICATE WASTES COMPLETELY EXCLUDING THE CEMENT

BY

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Received: October 4, 2022

Accepted for publication: February 10, 2023

**Abstract.** Geopolymer foam was produced by foaming with hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) of aluminosilicate mixture composed of fly ash and clay brick waste activated with an alkaline activator (water glass and sodium hydroxide in aqueous solution). Other mixture components were expanded perlite as a siliceous additive, a usual fine aggregate (sand), and a less frequently used surfactant (olive oil). The characteristics of the geopolymer foam were: density in the range of 420 - 560 kg/m<sup>3</sup>, porosity between 71.5 - 76.9%, thermal conductivity within the limits 0.080 - 0.122 W/m · K, and compressive strength between 4.1-5.6 MPa. The low values of density and thermal conductivity indicated good thermal insulation properties of the geopolymer foam, similar to those of this type product presented in the literature. The residual materials contributed to the low level of costs and the complete replacement of ordinary cement in the concrete composition led to the significant reduction of CO<sub>2</sub> emissions.

**Keywords:** geopolymer foam, aluminosilicate binder, coal fly ash, hydrogen peroxide, alkaline activator.

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## 1. Introduction

In the last decades, the world economy is facing a profound crisis due to the high energy consumption on the one hand and, on the other hand, as a consequence of this excessive consumption, with major ecological problems caused by greenhouse gas emissions with serious consequences on the planet climate (Simmons and Coyle, 2021). Under these conditions, the world community took several decisions regarding the limitation of production activities with excess energy consumption and established limit levels of polluting emissions into the atmosphere. The construction materials industry and especially the cement industry were strongly affected. Therefore, the concern of researchers for the complete replacement or drastic reduction of this important binder in the concrete making process is justified. Several types of natural aluminosilicate materials (kaolinite, calcined kaolinite, and clay) as well as wastes or industrial by-products (coal fly ash, rice husk, granulated blast furnace slag, red mud) are theoretically considered adequate for this purpose (Davidovits, 2008) and the literature has already presented numerous experimental results. Certainly, the production of new types of concrete mainly aimed at obtaining high mechanical strength. The objective of the current work is to create porous, lightweight concretes with low thermal conductivity using aluminosilicate residual materials. Both techniques for making the geopolymer concrete (high-strength or porous) lead to very low CO<sub>2</sub> emissions during processing raw materials, much lower compared to the manufacture of Portland cement.

According to the literature (Pereira *et al.*, 2019; Castillo *et al.*, 2022), the geopolymer is a hydrated aluminosilicate inorganic material obtained by activation of silicon and aluminum in medium or high alkaline solutions through the polymerization reaction.

Further, the results obtained in the field of making the geopolymer foam shown in international literature will be presented. In principle, the main difference between the production techniques of high-strength and respectively, porous geopolymer concretes is the use of a method of foaming the mixture (by mechanical methods or with a supplying agent). According to the literature (Movais *et al.*, 2016), the most used foaming agent for producing geopolymer foam based on coal fly ash is hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), which favourable influences thermal insulation properties (porosity, thermal conductivity) of the product. The mixture composition adopted in this paper included metakaolin (natural aluminosilicate) and coal fly ash (industrial by-product) in 2:1 weight ratio, activated with hydrated water glass (Na<sub>2</sub>SiO<sub>3</sub>) and sodium hydroxide (NaOH) aqueous solution (Chithambar Ganesh and Muthukannan, 2018) as alkaline activators. The thermal conductivity of geopolymer foam had values below 0.107 W/m·K and its density dropped below 560 kg/m<sup>3</sup>.

The manufacture of geopolymer with metakaolin as an aluminosilicate binder substituting the cement was controlled in the paper (Jaya *et al.*, 2020) by

the values of some parameters considered decisive: the alkaline concentration at 10M, the proportion of alkaline activator at 1%, and the metakaolin/activator ratio at 0.8. The experiment showed that the activator proportion influences the mechanical strength and the metakaolin/activator ratio determines the thermal conductivity values. The use of H<sub>2</sub>O<sub>2</sub> and the surfactant Tween 80 (polyethylene glycosorbitan monooleate) allowed obtaining a geopolymer foam with density between 471-1212 kg/m<sup>3</sup>, porosity between 36-80%, thermal conductivity between 0.11-0.30 W/m·K, and a compressive strength within acceptable limits (0.4-6 MPa).

Geopolymer foam was made by alkaline activation of the mixture composed of metakaolin and calcium phosphate biomass ash in 1:1 weight ratio and its expansion with H<sub>2</sub>O<sub>2</sub> (5 wt. %) (Muri *et al.*, 2017). Foam characteristics were: density of 310 kg/m<sup>3</sup>, thermal conductivity of 0.073 W/m·K, and compressive strength of 0.6 MPa. Self-bearing thermal insulation partitions and lightweight cores for thermal-structural sandwich panels are the recommended application fields.

According to (Agostini *et al.*, 2021), polypropylene fibers were used in the making process of geopolymer foam based on coal fly ash. Water glass and NaOH in aqueous solution were used as alkaline activators. The mechanical foaming method was adopted being performed by mixing the foaming agent with distilled water at high pressure. The foam thus prepared was added to the geopolymer mixture in proportions of 40 and 60 wt. %. Polypropylene fibers were added in very small amounts (below 0.5% of the fly ash amount). By increasing the fiber content, the mechanical strength of geopolymer foam increased, but the thermal conductivity was affected. However, the conductivity value was approximately similar to that of conventional lightweight concrete.

Metakaolin, as a natural aluminosilicate binder, activated with 10M NaOH solution and water glass in the 2:3 weight ratio were used for preparing the geopolymer foam (Wattanarach *et al.*, 2022). Sodium perborate (NaH<sub>2</sub>BO<sub>4</sub>) (between 0.5-2 wt. %) was the foaming agent firstly mixed with metakaolin and then with the alkaline activator solution in order to form a paste, which was poured into silicon molds. Curing the geopolymer foam samples was carried out below 60°C for 24 hours followed by their aging at room temperature for 28 days. Increasing the NaH<sub>2</sub>BO<sub>4</sub> weight proportion between 0.5-2%, porosity increased from 54.7 to 67.6%, density decreased from 1077 to 750 kg/m<sup>3</sup>, thermal conductivity decreased from 0.325 to 0.218 W/m·K, and compressive strength decreased from 6.7 to 5.2 MPa.

According to (Kurek *et al.*, 2022; Zhang *et al.*, 2021), the geopolymer foam manufacturing technique can involve numerous types of additives (polypropylene fiber, carbon nano-tubes, nano-silica, rosin, sodium dodecyl sulfate, sodium lauryl ether sulfate, proteins, etc.) and foaming agents (H<sub>2</sub>O<sub>2</sub>, NaH<sub>2</sub>BO<sub>4</sub>, sodium hypochlorite, etc.). Organic materials such as expanded perlite or cellulose fibers are also suitable in terms of ecology for this purpose. The paper (Kurek *et al.*, 2022) used a mixture of metakaolin (90 wt. %) and sand (10 wt. %) as aluminosilicate binder, activated with aqueous solution of water glass and 8M NaOH (2.5:1 weight ratio). H<sub>2</sub>O<sub>2</sub> (3 wt. %) and aluminium powder (very low

ratio) were chosen as foaming agents. Expanded perlite as a siliceous additive significantly decreased the thermal conductivity and density of geopolymer foam. Compressive strength did not been practically influenced.

Other making recipe of geopolymer foam based on coal fly ash used water glass as a foaming agent and detergent (between 0.1-0.5 wt. %) as surfactant influencing the geopolymer macrostructure fineness (Phavongkham *et al.*, 2020). The pore size decreased with the surfactant proportion increasing. The thermal conductivity was lowered from 0.32 to 0.27 W/m·K, while the compressive strength increased from 4.21 to 4.82 MPa due to the curing process (28 days). Also, the increase of surfactant ratio up to 0.5 wt. % contributed to the fire resistance increasing.

Making the geopolymer foam with low thermal conductivity and low density (*i.e.* thermal insulation properties) using only recovered aluminosilicate residues (coal fly ash and clay brick waste) was the objective of the current paper. Coal fly ash constitutes a by-product of energy industry resulted from the coal burning and the residual clay brick comes from the demolition of buildings. The complete replacement of ordinary Portland cement with recycled materials whose processing (grinding) requires a low consumption of electricity without polluting gas emissions (mainly CO<sub>2</sub>) into the atmosphere is the main element of this work originality. Activating the aluminosilicate binder was obtained by the traditional method involving the alkaline activator (water glass and NaOH aqueous solution), which facilitates developing the polymerization reaction and the adopted foaming agent was H<sub>2</sub>O<sub>2</sub> frequently used in similar processes according to the literature.

## 2. Methods and Materials

Under the conditions of using an aluminosilicate mixture as a binder, the adopted method implies the choice of an alkaline activator for the polymerization reaction to take place. Taking into account the need to foam the mixture for obtaining a porous structure, adopting a foaming agent is the appropriate solution. The alkaline activator was chosen as a mixture composed of NaOH dissolved in aqueous solution and water glass (Na<sub>2</sub>SiO<sub>3</sub>) available in liquid state. The foaming agent chosen was H<sub>2</sub>O<sub>2</sub> whose decomposition occurs by exposure to light (Pedziwiatr *et al.*, 2020). The reaction of releasing water and oxygen (1) inside the mass of the aluminosilicate mixture offers the possibility of forming numerous bubbles, which are then turned into pores.



A siliceous additive (expanded perlite) was also introduced into the aluminosilicate mixture, having favourable influence on the thermal conductivity decreasing.

Olive oil was adopted in experiment as a surfactant which added in very low weight proportions (0.1-0.5%) into the geopolymer paste has ability to

improve the pore and structure fineness of foam (Zhao *et al.*, 2015). According to (Bai *et al.*, 2018), geopolymer foam was made using H<sub>2</sub>O<sub>2</sub> solution as a pore-forming agent and vegetable oil as a stabilization agent. The foamed product had remarkable characteristics: density between 370-740 kg/m<sup>3</sup>, thermal conductivity between 0.11-0.17 W/m·K, porosity between 66-83%, and compressive strength between 0.3-11.6 MPa. It was experimentally found that the proportions of the stabilizing agent and the foaming agent have a significant influence on the performance of the foam. The highest proportions of the two agents contribute to the accentuated improvement of the thermal insulation properties (density, thermal conductivity, and porosity) and to the strong decrease of the compressive strength.

The method of making the geopolymer foam was divided into several distinct stages. Firstly, the dry solid materials that make up the mixture (fly ash, clay, and expanded perlite) were separately ground and together with the dry fine sand as aggregate were homogeneously mixed, then, the alkaline activator solution was added. The geopolymer paste was obtained by mixing the solid and liquid component materials in a laboratory mixer for 15 min. Finally, the liquid foaming agent H<sub>2</sub>O<sub>2</sub> (30% concentration) and the surfactant (olive oil) were added to the paste and mixed. The fresh geopolymer was poured into stainless steel molds and cured at room temperature for 24 hours. After this time, the specimens were removed from the molds and left for aging up to 28 days.

The list of materials chosen for the manufacture of geopolymer foam as well as their role was shown above. Coal fly ash was provided by Paroseni thermal power plant. Its initial grain size (under 200 μm) was reduced by grinding to under 100 μm. Clay brick residues were selected from the demolition of buildings and ground to a grain size below 120 μm. Expanded perlite (Celik *et al.*, 2013) was purchased from the market and was mechanical processed in a ball mill for decreasing the grain dimension under 130 μm. River sand (Bala and Khan, 2003) purchased from the market had fine size of grain below 200 μm. The chemical composition of fly ash, clay, perlite, and sand is indicated in Table 1.

**Table 1**  
*Oxide composition of raw materials*

Composition	Coal fly ash (wt. %)	Clay brick (wt. %)	Perlite (wt. %)	Sand (wt. %)
SiO <sub>2</sub>	49.8	59.0	70.7	97.9
Al <sub>2</sub> O <sub>3</sub>	23.5	28.9	13.0	1.1
Fe <sub>2</sub> O <sub>3</sub>	6.1	1.7	1.0	0.5
K <sub>2</sub> O	4.0	2.3	4.3	0.3
Na <sub>2</sub> O	1.6	1.2	3.5	-
CaO	3.6	1.0	2.0	-
MgO	3.1	0.9	1.8	-
TiO <sub>2</sub>	1.1	1.3	-	-

The H<sub>2</sub>O<sub>2</sub> solution was purchased from the market. Its concentration chosen by the authors was 30%.

The tests for characterizing the geopolymer specimens were carried out after the 29 days of curing process. The density was measured using the gravimetric method (Metrology, 2015) and the test of water absorption (ASTM C1585) was performed by the specimen immersion in distilled water for 1 hour. Porosity was calculated as volumetric proportion between the true and apparent density, where the true density was measured with multivolume pycnometer 1305 (Papa *et al.*, 2022). Measuring the thermal conductivity (at 30°C) was made with HFM 446 Lambda apparatus based on the heat-flow method (SR EN 1946-3:2004). The compressive strength was determined using 100 kN-hydraulic axial press machine (EN 826:2013). The microstructural characteristics of specimens was investigated with ASONA 100X Zoom Smartphone Digital Microscope. Identifying the crystalline phases of the geopolymer foam was achieved using X-ray diffractometer Bruker-AXS D8 Advance with CuK $\alpha$  radiation (EN 13925-2:2003).

### 3. Results and Discussion

In order to experimentally making the geopolymer foam according to the objective stated above, three variants of recipes were tested. Coal fly ash and clay waste as aluminosilicate binder, expanded perlite as a siliceous additive as well as fine sand were the solids of the mixture. The liquid components were the alkaline activator, H<sub>2</sub>O<sub>2</sub> as a foaming agent, and olive oil as a surfactant. The fly ash/clay waste ratio was kept constant at 3.65. Also, the weight ratio was kept constant between sand, perlite, and activator at the values of 1.95: 0.55: 1, while the proportions of the other components varied in the three variants according to the data in Table 2.

**Table 2**  
*Experimental variants composition*

Component	Experimental variant (wt. %)		
	1	2	3
Coal fly ash	25.04	24.57	24.20
Clay	6.86	6.73	6.63
Sand	36.80	36.69	36.58
Perlite	10.38	10.35	10.32
H <sub>2</sub> O <sub>2</sub>	1.99	2.72	3.32
Olive oil	0.06	0.12	0.19
Alkaline activator	18.87	18.82	18.76

Images of the three geopolymer foams produced in this experiment are exposed in Fig. 1. The different appearance of the three specimens is due to the increase of the H<sub>2</sub>O<sub>2</sub> and surfactant contents.

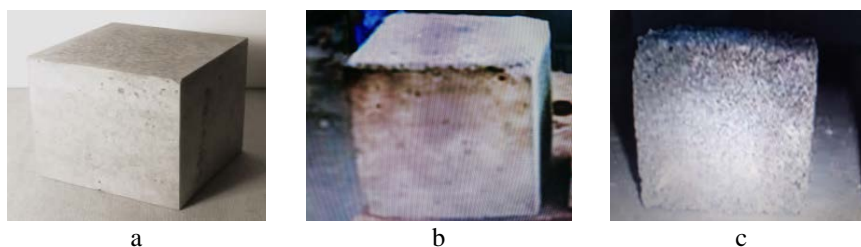


Fig. 1 – Appearance of the geopolymer foam specimens  
a – variant 1; b – variant 2; c – variant 3.

The results of applying the characterization methods mentioned above are presented in Table 3.

**Table 3**  
*Physical, thermal, and mechanical features of geopolymer foam specimens*

Feature	Variant		
	1	2	3
Apparent density (kg/m <sup>3</sup> )	560	495	420
Thermal conductivity (W/m·K)	0.122	0.098	0.080
Compressive strength (MPa)	5.6	4.9	4.1
Water absorption (vol. %)	3.8	3.5	3.1
Porosity (%)	71.5	73.8	76.9
Pore size (mm)	0.2-0.8	0.5-1.0	1.0-1.8

The features shown in Table 3 correspond in general to the geopolymer foam type peculiarities produced by other making methods presented in the literature. The apparent density falls within the acceptable limits (420-560 kg/m<sup>3</sup>) with the mention that variant 3 which used the highest weight proportions of foaming agent (3.32 wt. %) and surfactant (0.19 wt. %) led to the lowest density value (420 kg/m<sup>3</sup>). As a consequence of the low density, the thermal conductivity of this specimen had the lowest value (0.080 W/m·K) and the porosity reached it the highest (76.9%). These characteristics indicate good thermal insulation properties of the porous material, which, by the way, was followed in this experiment. The compressive strength recorded acceptable values for the made geopolymer type, the highest value being obtained in the case of variant 1 in which H<sub>2</sub>O<sub>2</sub> and olive oil had the lowest contents. Variant 3 reached a lower value of mechanical strength (4.1 MPa), but sufficiently high by comparison with other values of this geopolymer foam characteristic previously made in the world.

The microstructural appearance of the geopolymer foam specimens is shown in Fig. 2.



Fig. 2 – Appearance of the geopolymer foam specimens  
a – variant 1; b – variant 2; c – variant 3.

Figure 2 confirmed that the three specimens microstructure is characterized by closed pores without the tendency to intercommunicate with neighbouring pores.

The analysis of the crystalline phases in geopolymer foam identified mainly quartz and to a small extent mullite. The two crystalline phases which do not easily dissolve in the alkaline activator reduce the effectiveness of the polymerization process.

The geopolymer foam whose achievement is presented in the current work was prepared using wastes as aluminosilicate materials: coal fly ash as a by-product of energy industry and clay brick waste recycled from the building demolition. By comparison with other making processes of geopolymer foam, the use in exclusivity of wastes, which contributed to reducing the costs, was the original element of the paper.

The geopolymer foam specimens made in the three experimental variants corresponded to the requirements of products with good thermal insulation properties for building construction (density between 420-560 kg/m<sup>3</sup>, thermal conductivity between 0.080-0.122 W/m·K, and porosity between 71.5-76.9%). Improved by the curing process, the compressive strength had enough high values (4.1-5.6 MPa) similar to those of other geopolymer foams known from the literature. Also, the water absorption (between 3.1-3.8 vol. %) was within acceptable limits for a porous product of this type.

#### 4. Conclusions

Taking into account that the making of concrete for construction includes materials whose processing involves high consumption of fossil fuel and implicitly, high emissions of greenhouse gases (mainly CO<sub>2</sub>), the production of geopolymers has become a requirement. Manufacturing the geopolymer is based on aluminosilicate materials (rich in silica and alumina) in the category including both natural materials and residual materials as industrial by-products or wastes. This paper aimed at the production of a porous geopolymer with suitable heat insulation properties using as an aluminosilicate binder a mixture of fly ash from energy industry and clay brick waste from building demolition activated with an



alkaline activator composed of water glass and NaOH. An additive such as expanded perlite with an important role in foaming the geopolymer and increasing the thermal conductivity was also used. The mixture for the production of geopolymer foam was completed with a commonly used foaming agent (hydrogen peroxide H<sub>2</sub>O<sub>2</sub>), a common fine aggregate (river sand), and a surfactant known in the literature, but less often used (olive oil). The main features of the geopolymer foam samples were: density between 420-560 kg/m<sup>3</sup>, thermal conductivity between 0.080-0.122 W/m·K, porosity between 71.5-76.9%, and compressive strength in the range of 4.1-5.6 MPa.

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## FABRICARE ÎN CONDIȚII ECOLOGICE A SPUMEI GEOPOLIMERICE DIN DEȘEURI ALUMINOSILICATICE EXCLUZÂND COMPLET CIMENTUL

(Rezumat)

Spuma geopolimerică a fost produsă prin spumarea cu peroxid de hidrogen ( $H_2O_2$ ) a amestecului aluminosilicatic compus din cenușă zburătoare și deșeu de cărămidă argiloasă activat cu un activator alcalin (apă de sticlă și hidroxid de sodiu în soluție apoasă). Alți componenți ai amestecului au fost perlită expandată ca un aditiv silicios, un agregat fin uzual (nisip) și un aditiv tensioactiv mai puțin utilizat (ulei de măsline). Caracteristicile spumei geopolimerice au fost: densitatea în intervalul 420-560 kg/m<sup>3</sup>, porozitatea între 71,5-76,9%, conductivitatea termică în limitele 0,080-0,122 W/m·K și rezistența la compresiune între 4,1-5,6 MPa. Valorile mici ale densității și conductivității termice au indicat bune proprietăți termoizolante ale spumei geopolimerice, similare cu acelea ale acestui tip de produs prezentate în literatura de specialitate. Materialele reziduale au contribuit la nivelul redus al costurilor, iar completa înlocuire a cimentului obișnuit în compoziția betonului a condus la reducerea semnificativă a emisiilor de CO<sub>2</sub>.