Porous geopolymeric material for insulation applications

Material geopolimeric poros pentru aplicații în izolații

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Abstract. *Cellular fly ash-based geopolymer with suitable physico-thermal and mechanical strength characteristics was prepared using sodium perborate as an expanding agent, palm oil as a surfactant for stabilization of the froth, montmorillonite as a nanoclay for growing the strength of geopolymer , and an alkaline activator composed of potassium hydroxide and silicate solutions. Results showed excellent thermal insulation properties (density between 438-502 kg·m^{-3} and heat conductivity in the range of 0.077-0.103 W·m^{-1}·K^{-1}) as well as adequate compression and flexural strength up to 5.4 and 4.1 MPa respectively, being interesting the excellent strength values obtained at early age only 7 days.*

Key words: *geopolymer, porosity, insulation, expanding agent, foam.*

Rezumat. *Geopolimer celular pe bază de cenușă zburătoare cu caracteristici adecvate fizico-termice și de rezistență mecanică a fost preparat utilizând perborat de sodiu ca agent de expansiune, ulei de palmier ca surfactant pentru stabilizarea spumei,*

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montmorillonit ca o nanoargilă pentru creşterea rezistenţei geopolimerului şi un activator alcalin compus din soluţii de hidroxid şi silicat de potasiu. Rezultatele au arătat excelente proprietăţi termoizolante (densitate între 438-502 kg·m^{-3} şi conductivitate termică în intervalul 0,077-0,103 W·m^{-1}·K^{-1}), precum şi rezistenţă la compresiune şi încovoiere până la 5,4 şi respectiv, 4,1 MPa, fiind interesante excelentele valori ale rezistenţelor obţinute timpuriu, după numai 7 zile.

Cuvinte cheie: geopolimer, porozitate, izolaţie, agent de expansiune, spumă.

1. Introduction

The new type of inorganic cementitious material, named geopolymer, has offered a huge interest for world specialists since the last decade of 20th century and in present. The amorphous alkali alumina-silicate gel, as the major binding phase generated into geopolymer, clearly distinguishes this material for the traditional Portland cement [1]. The geopolymer is mainly composed of calcium silicate hydrate (C-S-H) gel phase [2]. Main abilities of geopolymers are rapid reaching of early strength [3], high bonding resistance [4], fire-resistance [5], high durability [6], and very low carbon footprint [7]. The strength of geopolymers used as thermal or acoustic insulating board coming in direct contact with external factors is exhibired by its high resistance to bacteria, insects or rodents [8].

The pore structure represents an important key in reaching the qualitative performances of geopolymer. Higher porosity can contribute to improving some of its properties such as strength, heat conductivity, permeability depending on the porous structure [9]. Pores have a major role in sorption, capillary condensation, and determine the heat insulation level of the geopolymer [10, 11].

Several pore-generating methods are recently known. Thus, impregnating the polymer matrix with a ceramic suspension, introducing froth-forming additives, direct expanding, the use of templates for porous materials, adding lightweight fillers into a ceramic mix, utilizing additives for suspension generation [12] are possible methods.

Among the methods for producing porous geopolymer, the main procedure is that of direct expanding, which is based on the release of gaseous products by chemically dissolving additives acting as expanding agents in the suspension generated in the alkaline environment.

More expanding agent variants (aluminum powder, zinc powder, and hydrogen peroxide-H_2O_2) were tested in [13]. The use of aluminum powder aimed to determine its effect on the geopolymerization reaction at early age, while the other agents were involved in evaluating the effect of geopolymerization for longer times up to 28 days. The research results showed that the expanding agent type strongly influences the generation and composition of N-A-S-H gel. Aluminum powder proved to be the most suitable for gel formation. The duration of the geopolymerization reaction influences the proportion of fly ash content reduction in the geopolymer compared to the initial one. The mentioned experiment [13] used an extremely low CaO content (0.82 %). 12M NaOH and sodium silicate composed the alkaline activator. Aluminum powder

(0.1 wt. %) contributed to obtaining a porous and interconnected void material. Zinc powder (0.1 wt. %) showed an unfavourable influence on the geopolymerization reaction. H_2O_2 (1 wt. %) showed the best abilities for an excellent control of froth density and pore size.

In the work [14], aluminum powder was used as an expanding agent for preparing porous fly ash-geopolymer. Results indicated that substituting 5 % fly ash with aluminum powder into a mixture characterized by alkali activator/fly ash ratio of 0.35 and Na_2SiO_3/NaOH weight ratio of 2.5 favours making a lowest density-product. Corresponding compression strength dropped to 0.9 MPa.

The production of cellular geopolymer requires proper stabilization of the created porous structure. For this purpose, some authors have used sodium dodecyl sulfate, which has experimentally proven this capacity. Also, sodium, potassium or calcium chlorides (NaCL, KCl, $CaCl_2$) have been used due to the increase in surface activity and their beneficial influence on the structural stability of the porous matrix, which, however, can decrease the foaming ability of sodium dodecyl sulfate [15].

Metakaolin-based geopolymer was manufactured through the direct expansion procedure and the use of surfactants to protect the formed porous structure [16]. The chosen expanding agent was hydrogen peroxide added to the alumina-silicate mix and the favourable effect of the alkaline environment led to developing the geopolymerization reaction and turning the mixture into a porous geopolymer material.

Porous fly ash-based geopolymer was produced using also hydrogen peroxide [17]. Results revealed that the H_2O_2 content has the ability to control the main properties of the geopolymer (porosity, heat conductivity, and mechanical strength). The porous geopolymer has adequate properties for thermal insulation applications. Heat conductivity lower than 0.107 $W \cdot m^{-1} \cdot K^{-1}$ and density of 0.56 $g \cdot cm^{-3}$ were obtained. The study promoted the valorization of alumina-silicate waste, fly ash being such an example.

The French geopolymer inventor J. Davidovits designed and experimentally tested a porous geopolymer using H_2O_2 and sodium perborate (NaH_2BO_4) as expanding agents [18]. The products had density in the range of 0.2-0.8 $g \cdot cm^{-3}$, heat conductivity of around 0.037 $W \cdot m^{-1} \cdot K^{-1}$, and high heat resistance at 1200 °C.

Cellular fly ash-based geopolymer using sodium borate monohydrate as a foaming agent was made in [19]. Sodium dodecyl sulfate as a foam-stabilizing agent was also introduced in the mix. The product was examined both at ambient temperature and after keeping at high temperature (1000 °C). The density decreased from 1.2 to 0.3 $g \cdot cm^{-3}$ depending on the fly ash content. The compression strength increased between 1-6 MPa. The use of sodium borate allowed to obtain the heat resistance similar to the case of using H_2O_2.

In another work from the literature [20], results of making cellular geopolymer based on fly ash using sodium perborate as a foaming agent and washing liquid as a surfactant with role in foam stabilizing are presented. The paper was mainly focused on the content effect of the surfactant on heat and mechanical features of the geopolymer samples. By using this surfactant type within the limits of 0.1-0.5 wt. %,

porosity became finer, compression strength after 28 days of curing had values in the range of 4.2-4.8 MPa, increasing depending on the surfactant ratio, and heat conductivity was between 0.27-0.32 $W \cdot m^{-1} \cdot K^{-1}$. Also, the fire-resistant of the cellular geopolymer proved to be excellent.

Different variants of surfactants usable for stabilizing foams formed through cellular geopolymer manufacturing processes are highlighted in the work [21]. It is known that vegetable oils extracted from many plants (olive oil, palm oil, rapeseed oil, sunflower oil, cottonseed oil, corn oil, etc.) can be used in industrial applications such as polymer synthesis. According to [21], palm oil is extracted from the oil palm tree and is used in several applications such as oleochemicals, ingredients of several foods, health products, household products, biodiesel, etc. The fractionated products of palm oil and fatty acids (palm olein and palm stearin) are used as renewable raw materials for polymer making.

The current work brought as main novelties in the making recipe of cellular geopolymer the use of palm oil as a surfactant of vegetable origin [22] with the role of foam stabilizer as well as the combination KOH/K_2SiO_3 representing the composition of the alkaline activator chosen by the authors, unlike the frequently used combination $NaOH/Na_2SiO_3$. Both sodium and potassium were recommended and claimed by the inventor of the alkaline-geopolymerization process (J. Davidovits), but most researchers in this domain prefer sodium hydroxide and silicate.

2. Methods and materials

Production mechanism of cellular geopolymer requires forming the gas bubbles and then stabilizing the cellular structure. In this experiment, the direct expanding procedure was chosen that is based on the chemical reaction of releasing gaseous products inside the softened mass of raw materials. The experiment has promoted the use of sodium perborate (NaH_2BO_4) as an blowing agent considered more stable and easy handling compared to hydrogen peroxide (H_2O_2) large-scale used by researchers that investigate the cellular geopolymer producing [19, 20]. Avoiding the bubbles coalescence becomes a required operation and is performed through stabilizing the foamed mass. This is possible by diminishing the surface tension of gas bubbles additing the surfactant. In this case, the surfactant was palm oil (of vegetable origin). On the other hand, the use of a nanomaterial from the nanoclay category (montmorillonite) had the role of improving the cellular geopolymer strength. An important stage of the making method flux of cellular fly ash-based geopolymer was mixing in an electrically operated device (with 750 rpm for 12 min) until the paste is formed. Then, the paste was poured into SiC-moulds (made in China) and introduced into a laboratory electrical oven. The thermal process temperature was 65 °C for 24 hours. During the maintaining process at the mentioned temperature, alumina-silicate precursor materials polycondense forming a three-dimensional network of bonded molecules. Gas bubbles remain trapped into this network generating the cellular structure. The curing process for 7 and 28 days respectively, in which specimens were

removed from the moulds, allowed hardening the geopolymer samples before their determining physical, heat, mechanical, and microstructural features.

The fabrication of cellular fly ash-geopolymer required the following materials: class F-fly ash as the basic alumina-silicate raw material, montmorillonite as a nanoclay, sodium perborate as an expanding agent, palm oil as a surfactant, potassium hydroxide solution, and potassium silicate solution.

Class F-fly ash was purchased almost ten years ago from the Romanian Paroseni-Thermal power plant. The material had a particle size below 250 μm and required additional mechanical processing to lower the maximum granulation level below 70 μm. The oxide composition of fly ash included: 54.4 % SiO_2, 26.5 % Al_2O_3, 4,3 % Fe_2O_3, 3.5 % CaO, 2.5 % MgO, 1.5 % TiO_2, 0.4 % Na_2O, 0.6 % K_2O, and 1.7 % SO_3.

Montmorillonite ($Al_2H_2O_{12}Si_4$) a versatile mineral nanomaterial is found in natural bentonite deposits of volcanic origine. Its use advantages in making polymer composite are: high surface area, high adsorption capacity, high contribution as a reinforcing agent growing the mechanical strength of composites. The particle size of this powder is on the order of microns. Its chemical composition contains 43.0 % SiO_2, 16.8 % Al_2O_3, 3.6 % Fe_2O_3, 2.0 % MgO, 0.2 % Na_2O, 0.11 % CaO, 0.19 % Cl, and 34.0 % LOS.

Water-soluble sodium perborate (NaH_2BO_4) was chosen as an expanding agent. Through decomposition, this releases hydrogen perborate (H_2O_2), that decomposes in turn into water vapour and oxygen. Sodium perborate is available in high purity (up to 99 %) on the market, the product being made in China. Its particle size is very fine (under 24 μm). According to the date in the literature [23], the optimal proportion of sodium perborate into the mix is between 0.5-2.0 % of the total alumina-silicate precursor quantity.

Palm oil has been used in its commercially available form as a cooking product. It contains 50 % saturated, 40 % monounsaturated, and 10 % polyunsaturated fatty acids. The level of palmitic acid is high representing about 44 % of fatty acids [24].

Potassium hydroxide solution as a component of the alkaline activator used in this experiment was prepared using commercially available KOH pellets dissolved in distilled water to form an aqueous solution with 10 M molarity.

Potassium silicate solution as the other component of the alkaline activator adopted in this experiment was purchased from the market as an aqueous solution with 38 % concentration.

Methods of investigating physical, thermal, mechanical, and microstructural features of cellular geopolymer samples were generally those known and applied in cases of this kind. Density was measured using Archimedes' principle in conformity with the ASTM C373 standard and ISO 18754:2020 was applied for identifying the porosity. The compression strength of geopolymer specimens was determined with a hydraulically operated compression testing machine with a pressing capacity of 1000 tons-force (about 107 MPa) according to the ASTM C133-97 (2015) standard. Three-point bending tests to measure the flexural strength of samples required a multi-operational apparatus for testing (Instron type) at a crosshead speed of 1.2 $mm \cdot min^{-1}$ at

23 ℃, in accordance with ASTM D790-17. The water uptake of specimens was determined in accordance with ASTM C373-18 standard by their immersion under water. Microstructural aspect of samples was examined with ASONa 100X Zoom Smartphone Microscope.

In conformity with the above-mentioned materials used in this experiment, four making recipe variants were adopted including the quantities shown in Table 1.

Table 1

Composition of experimental variants

Composition	Variant 1 ($kg \cdot m^{-3}$)	Variant 2 ($kg \cdot m^{-3}$)	Variant 3 ($kg \cdot m^{-3}$)	Variant 4 ($kg \cdot m^{-3}$)
Class F-fly ash	290	290	290	290
Montmorillonite	2.7	3.4	4.0	4.7
Palm oil	0.4	0.4	0.3	0.2
Sodium perborate	6.1	6.5	6.9	7.3
10M KOH solution	48	48	48	48
K_2SiO_3 solution	114	114	114	114
Distilled water addition	80	80	80	80

According to the data in Table 1, it was exhibited the tendency to increase the porosity of samples and to decrease its fineness by growing the sodium perborate content between 6.1-7.3 $kg \cdot m^{-3}$ and decreasing the amount of palm oil from 0.4 to 0.2 $kg \cdot m^{-3}$. Also, by increasing the amount of montmorillonite from 2.7 to 4.7 $kg \cdot m^{-3}$, it was desired to improve the mechanical strength of the final cellular products. Using the constant K_2SiO_3/KOH ratio of 2.38, it has ensured an optimal ratio between the two components of the alkaline activator capable to initiate and to develop the geopolymerization reaction, thus leading to obtaining the designed geopolymer.

3. Results and discussion

As stated above, determining the compressive and flexural strength was performed both at early age after 7 days and at the end of the curing process after 28 days. Measuring results regarding features of cellular geopolymer specimens are presented in Table 2.

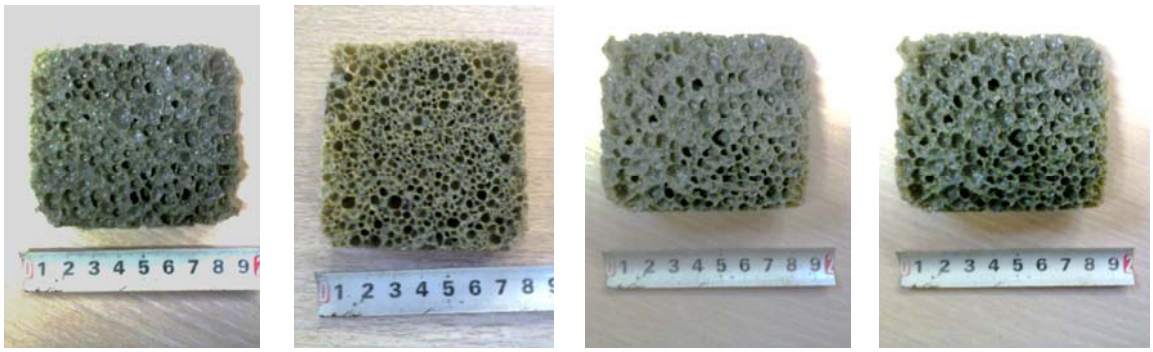
Table 2

Features of cellular geopolymer specimens

Feature	Variant 1	Variant 2	Variant 3	Variant 4
Apparent density ($kg \cdot m^{-3}$)	502	480	459	438
Porosity (%)	73.5	75.0	77.4	80.9
Heat conductivity ($W \cdot m^{-1} \cdot K^{-1}$)	0.103	0.094	0.086	0.077
Compression strength				

Feature	Variant 1	Variant 2	Variant 3	Variant 4
- at early age (7 days)	4.3	4.7	5.0	5.3
- at 28 days	4.6	4.9	5.2	5.4
Flexural strength				
- at early age (7 days)	3.0	3.3	3.6	3.9
- at 28 days	3.1	3.5	3.9	4.1
Water uptake (vol. %)	5.1	6.3	7.4	8.4
Pore size (mm)	0.1-0.3	0.1-0.4	0.2-0.5	0.4-1.0

In general, physico-thermal characteristics of fly ash-based cellular geopolymer were influenced not very much by the increase of the amount of expanding agent (sodium perborate) and by the slight decrease of the already low amount of palm oil as a froth stabilizer. The apparent density decreased in value from 502 $kg \cdot m^{-3}$ (variant 1) to 438 $kg \cdot m^{-3}$ (variant 4). By default, the porosity increased slightly between 73.5-80.9 %, while the heat conductivity decreased from 0.103 $W \cdot m^{-1} \cdot K^{-1}$ to values very suitable for thermal insulation in construction (0.077 $W \cdot m^{-1} \cdot K^{-1}$ in the case of variant 4 and also 0.086 $W \cdot m^{-1} \cdot K^{-1}$ in the case of variant 3). In terms of mechanical strength, rapid increase in compression and flexural strength values during the curing process was observed, so that at early age (after only 7 days), values close to the level obtained after 28 days of curing were reached. Thus, the compression strength of the specimen produced in variant 4 was 5.3 MPa after 7 days and 5.4 MPa after 28 days. Also, the flexural strength reached 3.9 MPa after 7 days and 4.1 MPa at the end of the curing process. Water uptake measured after immersion of cellular geopolymer samples under water showed an increase between 5.1-8.4 vol. % between specimens corresponding to variants 1-4, but the level is considered normal for this type of material. Appearances of specimen surfaces corresponding to the four preparing variants are shown in Fig. 1.



a b c d

Fig.1. Appearance of specimen surfaces corresponding to the four preparing variants
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

According to Fig. 1, the macrostructural dimension of specimens is increasing from the preparing variant 1 to variant 4. Images of the microstructural configuration of cellular geopolymer are shown in Fig. 2.

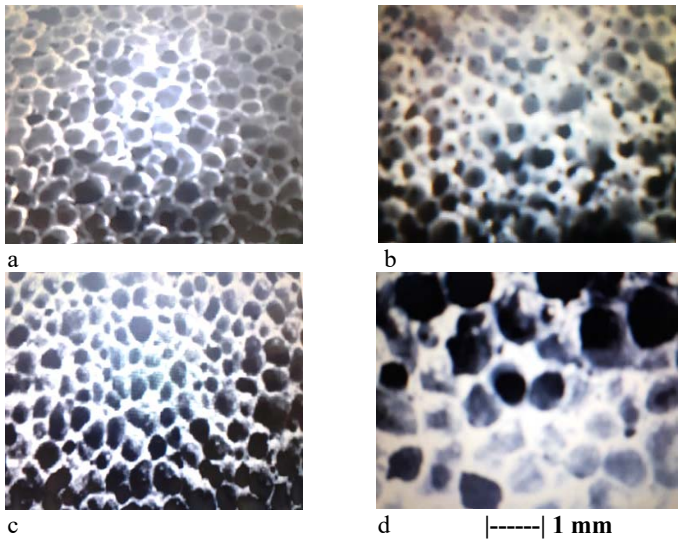


Fig. 2. Microstructural configuration of cellular geopolymer samples
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

According to pictures in Fig. 2, the microstructural homogeneity of specimens is remarkable. Images (a)-(c) are characterized by very close dimensions, the dimensional ranges of the cells being shown in Table 2. These ranges are 0.1-0.3 mm (variant 1), 0.1-0.4 mm (variant 2), and 0.2-0.5 mm (variant 3). In the case of variant 4, the cell dimensions are slightly larger, being within the limits of 0.4-1.0 mm.

The experimental results provided some observations regarding the particularities of the geopolymer foaming process using sodium perborate. The maximum amount of sodium perborate in variant 4 of 7.3 $kg \cdot m^{-3}$ represented 2.5 % of the amount of alumina-silicate waste (fly ash). According to the literature [23], the recommended weight proportion of sodium perborate is around 2 %, because higher proportions can affect the foaming effect of this agent. The results obtained in this experiment proved that a proportion of 2.5 % did not affect the expanding process in any way.

The strength increasing of the cellular fly ash-geopolymer was possible through the use of a nanomaterial from nanoclay category (montmorillonite). This procedure constituted one of the original solutions. Also, the choice of palm oil as a less demanding surfactant from the group of vegetable oils is an original decision. Unlike most technical solutions adopted in the world for creating the alkaline activator including sodium hydroxide and silicate, in this experiment the combination of potassium hydroxide and potassium silicate was adopted. The combination of all these material choices, which are usually little used in cellular geopolymer manufacturing processes, allowed to obtain a set of specimens with excellent properties at the level of the most appreciated materials of this type.

4. Conclusions

The work aimed at the production of a cellular fly ash-based geopolymer using sodium perborate as an expanding agent. The experiment involved applying additive material types less frequently used in similar processes such as: montmorillonite as a nanomaterial, palm oil as a surfactant as well as potassium hydroxide and potassium silicate as components of the alkaline activator. Cellular geopolymer specimens had excellent heat insulation properties as well as compression and flexure strength, being applicable as insulation materials in building construction.

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