CELLULAR GEOPOLYMERIC MATERIAL MADE BY ALUMINA-SILICATE WASTES-BASED NONCONVENTIONAL TECHNIQUE

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ABSTRACT: Geopolymer froth with excellent physio-mechanical, heat, and morphological features was made from a mix including fly ash and metallurgical slag as alumina-silicate industrial by-products. Using the metallurgical slag and montmorillonite as a nanomaterial constituted the composition novelty of starting mixture as well as the use as surfactant of a rarely applied material (sunflower oil) in making process of geopolymer froth were originality elements. Results were appreciable, the compression resistance reaching 6.7 at the final curing process and 4.9 MPa at early age and flexure resistance registering 3.3 MPa and 3.0 MPa at the final curing process and early age, respectively. The new cellular product is adequate for applications in construction as a lightweight insulating material, having better features by comparing with the traditional lightweight porous concrete.
KEYWORDS: geopolymer froth, fly ash, slag, alumina-silicate, compression resistance.

1. INTRODUCTION

The last decade of the previous millennium highlighted a serious global ecological situation caused by partial affecting the ozone layer and the possible overheating of earth. The reason for this situation was the extremely high emission of CO_2 into the air. This accumulated great amounts intensively in few decades of the end of the millennium due to the excessively high consumption of fossil fuels whose residual gases have a strong carbon footprint [1].

Obviously, the reaction of the regional communities of the world states was prompt and immediately measures plans were established for the situation recovery, which reduced or eliminated the responsible industrial technologies [2]. Some economic sectors, especially the construction materials industry, were seriously affected. Portland cement, an essential material in the manufacture of construction concrete, had to be drastically limited, as it was necessary to find viable technological alternatives and at the same time, environmentally friendly.

Research of the French scientist Joseph Davidovits carried out in the 90s regarding activating alumina-silicate waste in alkaline environment through the geopolymerization reaction allowed obtaining so-called geopolymers, materials with excellent mechanical, chemical, and physical characteristics as well as pozzolanic properties [3] that ensure for these products the quality of cementitious materials similar to construction cement.

Geopolymers have the ability to offer a large range of quality requirements: from dense to porous materials. The category of porous geopolymers is the one to which the current paper refers.

Recently, porous geopolymers have attracted the attention of researchers and manufacturers of lightweight materials due to the exceptional combination of their mechanical properties and good structural stability at high temperature: high chemical protection, small heat growing coefficient, protection to heat shock, large surface area, low denseness, controlled permeability, and high mechanical resistance [4].

Numerous procedures are known to create pores in the geopolymer mass: impregnation of cellular polymer matrix using ceramic suspension, using template for porous materials, utilizing foam-creating additives forming pores, swelling ceramic mixtures, fillers addition with own porosity into ceramic mix, use of additives for producing ceramic suspensions, etc. [5].

Geopolymers are semi-crystalline amorphous materials generated by the polymerization reaction (known as geopolymerization) between an alumina-silicate precursor and an alkaline activating agent. Raw materials with the role of geopolymer precursor both in the natural state and as industrial by-products must be alumina-rich and silica-rich, playing an important role in hardening the geopolymer, because

together with other components they form the N-A-S-H gel, responsible for creating the typical resistance of geopolymer [6].

By dissolving the feedstock, aluminum and silicon are set free. Later, silicon into solution enters in reaction with this gel, generating the final silicon-rich gel. Following the polymerization action, the N-A-S-H gel (containing Na_2O, Al_2O_3, SiO_2, and H_2O) is formed [6].

The geopolymer solidification depends largely on aluminum. In mixtures characterized by high alkali concentrations, silicates are usually metastable because silica tetrahedra are invaded by H_2O, which facilitates the formation of silanol (Si-OH) until the equilibrium is made, when $Si(OH)_4$ is created. However, being in an alkaline environment, water invades continues until the silicate is entirely dissolved. In conclusion, only soluble silicon does not have the ability to create chemical material hardening. Si-O-Si connections and Si-O-Al connections are created due to aluminate and silicate tetrahedra bonding. Also, because they are not energetically stable, no Al-O-Al is formed. Thus, Si/Al ratio can obtain the lowest value (i.e. 1). The aluminum quantity must be enough so that silica dissolution does not occur [7].

Among the cellular geopolymer production procedures mentioned above, the main technique is that of direct expansion. This is based on the formation of gaseous products by dissolving chemical additives with the role of expanding agents in the geopolymer suspension made in an alkaline environment. Additives can be metal powders of aluminum or silicon as well as chemical compounds containing silicon.

Sanjayan et al. [8] used aluminum powder, fly ash being partial replaced by this. Outcomes indicated that the substitution of 5 % coal ash with aluminum powder in samples having alkali activator/ash ratio of 0.35 and Na_2SiO_3/NaOH mass ratio of 2.5 favours obtaining a product with the lowest density. The corresponding compression resistance dropped to 0.9 MPa.

The work [9] concerned the preparation of a geopolymer with high macroporosity (up to millimeter pore sizes) in alkali-bonded matrix, using the capacity of Si powder to generate hydrogen in an aqueous environment. The geopolymer characteristics were largely influenced by the quantity of silicon addition. Metakaolin was used as the basic raw material. K_2SiO_3 solution with a SiO_2/K_2O molar ratio of 2 and H_2O/K_2O between 13.5-23.0, together with KOH pellets dissolved in distilled water, constituted the alkaline activator.

The aim of the work [10] was to obtain supervised porousness as well as supervised heat features with geopolymeric products including SiC slurry. The free Si reaction in SiC slurry sets free hydrogen, which creates pores in the geopolymer mass at the end of the process.

Metakaolin-based geopolymer was produced by the direct expansion technique using hydrogen peroxide (H_2O_2) as an expanding product and surfactants to secure the porous structure [11]. H_2O_2 added in the alumina-silicate mix in alkaline environment does not disturb the geopolymerization reaction, which is only influenced by the alkaline solution concentration and the liquid/solid ratio. However, swelling degree of the material, denseness, and pore size have significant variations depending on the H_2O_2 content. Mechano-physical geopolymer properties can be also modified by some characteristics (the process heat regime, alkaline activator version, and the wetting agent concentration). The surface-active agent chemical composition and structure can influence the interaction of paste components changing its workability in liquid state and connectivity of macroporous network.

Geopolymer based on metakaolin applicable as membrane supports was made by gelcasting utilizing H_2O_2 and egg white dust as surface-active agent [12]. A suspension was obtained from metakaolin in alkaline environment at ambient temperature. The fairly high viscosity paste was foamed due to the breaking up of H_2O_2 in association with the protein acting as a wetting agent. The temperature required for the geopolymerization process was 75 °C. The geopolymer for membrane supports had the total porousness of 74 % (open porousness representing 65 %) and a compression resistance of 4.5 MPa.

H_2O_2 is frequently utilized as a blowing material in the production of cellular coal ash-geopolymer, leading to favourable effects on its denseness, heat conductivity, and porousness [13]. The metakaolin/coal ash weight ratio was 2:1 and the activator included Na_2SiO_3 and NaOH as an aqueous solution. The heat conductance of this cellular geopolymer type is under 0.107 $W \cdot m^{-1} \cdot K^{-1}$ and denseness does not exceed 0.56 $g \cdot cm^{-3}$ [14].

Joseph Davidovits, recognized as the inventor of geopolymer, proposed and experimentally used H_2O_2 and sodium perborate (NaH_2BO_4) as expanding agents to obtain products with denseness within the limits of 0.2-0.8 $g \cdot cm^{-3}$, heat conductance

of about 0.037 $W \cdot m^{-1} \cdot K^{-1}$, and the highest value of heat resistance at 1200 ℃ [15].

Making cellular geopolymer implies good stabilizing the newly formed porous structure, requiring a stabilizing agent. Jiang et al. [16] used sodium dodecyl sulfate, which exhibits this ability. Also, in previous studies, some salts such as NaCl, KCl, and $CaCl_2$ were mentioned that favourably influence the structural stability of pores.

Writers of the present work have tried experimental making of cellular geopolymer based on fly ash and metakaolin in alkaline environment composed of Na_2SiO_3 and NaOH utilizing sodium perborate as a more stable and easier to handle expanding agent, replacing the traditional H_2O_2 [17]. The best results were obtained under the conditions where the starting mixture included fly ash/metakaolin mass ratio at the maximum value (2.68), Na_2SiO_3/NaOH mass ratio of 2.48, alumina-silicate mix/alkaline activator ratio of 2.80, the nanomaterial additive (bentonite clay) had the maximum value, as well as sodium perborate and the stabilizing agent (sunflower oil). Optimal version characteristics were denseness of 0.47 $g \cdot cm^{-3}$, porousness of 76.4 %, thermal conductivity of 0.104 $W \cdot m^{-1} \cdot K^{-1}$, compressive strength of 7.5 MPa (at the final curing process), and flexure resistance of 3.4 (af final curing procedure).

Utilization of sodium perborate as an alternative foaming agent to the traditional H_2O_2 is also the solution adopted by Wattanarach et al. [18] for producing metakaolin-based cellular geopolymer. Sodium perborate (in the range of 0.5-2 wt. % of the metakaolin amount) was brewed with metakaolin powder and further, together with the liquid solution of activator until the paste was formed. 10M NaOH solution and Na_2SiO_3 solution in 2:3 weight ratio were activator components. Hardening the fresh mixture into silicon moulds was carried out at maximum 60 ℃ for 24 hours. The curing process of hardened specimens and removed from the moulds at 27 ℃ and 75 % relative humidity was scheduled for a duration of four weeks. The outcomes indicated that the denseness of the geopolymer was between 0.76-1.07 $g \cdot cm^{-3}$, thermal conductance had values in the range 0.218-0.325 $W \cdot m^{-1} \cdot K^{-1}$, and compressive resistance was around 5-6 MPa. The maximum values of the ranges mentioned above were reached under the conditions of using a weight proportion of 2 % of sodium perborate. The pore volume as well as the water absorption reached the highest values under the conditions of using the 2 % of foaming agent.

In another recent scientific article [19], geopolymer foams based on class F-coal ash and ground metallurgical slag were experimentally made utilizing sodium perborate as a blowing product, washing liquid as a surface-active product as well as 6M NaOH solution mixed with Na_2SiO_3 solution (in 1:2 ratio) as an alkaline activator. The surfactant dosage between 0.1-0.5 % favoured workability, fineness of foam pores, and the increase of compression resistance after 28 days up to 4.82 MPa. The heat conductivity values have reduced to 0.27-0.32 $W \cdot m^{-1} \cdot K^{-1}$. Additionally, the fire resistance has considerably improved, the temperature preservation into a 40 mm-geopolymer plate heated on one of his surfaces to 1000 ℃ was excellent, the temperature on the opposite surface being only 300 ℃ for 120 min. The geopolymer manufacturing method was that of direct foaming.

The current paper has chosen a preparation recipe based on coal ash and ground metallurgical slag as alumina-silicate secondary final materials of production and sodium perborate as a blowing agent substituting the traditional H_2O_2. A nanoclay (montmorillonite) is added to these basic components to increase the geopolymer resistance as well as a surfactant (sunflower oil) with the role of stabilizing agent for the porous structure. Compared to the recipe previously used by authors and presented in the work [17], granulated blast furnace slag and nanoclay were additionally added.

2. PROCEDURES AND MATERIALS

2.1 Procedures

The procedure of preparing the cellular geopolymer consisted in the first stage in making the mixture between 10M NaOH solution and Na_2SiO_3 solution in weight ratio of 0.67. Separately, the alumina-silicate waste (coal ash and metallurgical slag) in a fine dust state as well as nanoclay (montmorillonite) as a nanomaterial were mixed together with sodium perborate by stirring at a speed of 800 rpm for 2 min. The alkaline activator solution brewed in the first stage was poured at low speed over the solid mix. Their mixing for approximately 6-7 min was carried out until the paste was formed. The paste thus obtained was poured into silicon carbide-moulds of cubic and parallelepiped shape, respectively, (for the subsequent determination of flexural resistance) and was subjected to hardening in an electric laboratory oven at 70 ℃ for one day. Then, samples were set free and subjected to the curing process in ambient thermal conditions and low humidity for 28 days.

In chemical terms, by hydrolysis of the blowing agent (sodium perborate) in the presence of water, hydrogen peroxide and the anion $B(OH)_4^-$ are let out, according to Brotherton [20]. Then, H_2O_2 chemically decomposes into hydrogen and oxygen, both in a gaseous state, which, being blocked in the viscous mass, create bubbles that are later turned into pores.

Making process of geopolymer is based on ability transformation alumina-silicate materials through activating with aqueous alkaline solution into the new material type named geopolymer, with 3D-polymeric chain structure including Si-O-Al-O connections [21].

2.2 Materials

Alumina-silicate secondary final materials of industry (coal fly ash and metallurgical slag) and a nanomaterial (montmorillonite) were chosen as alumina and silica-rich materials for manufacturing process of cellular geopolymer.

Class F-fly ash (with low CaO content) was previously procured (about 7 years before) from Paroseni-Heat power station (Romania) with particle dimension under 250 μm, being required supplementary processed to reduce the maximum limit under 60 μm.

Metallurgical slag obtained 10 years ago from ArcelorMittal Galati (Romania) had grain size between 2-7 mm, supplementary mechanical processing being necessaire for obtaining grains under 50 μm.

Montmorillonite ($Al_2H_2O_{12}Si_4$) was commercially purchased. This nanomaterial as a nanoclay is found mainly in sedimentary bentonite rock (in proportion of 60-80 %). The dimensions of its particles are of the order of microns.

Oxide composition of ash, slag, and montmorillonite is shown in Table 1.

Table 1. Components of alumina-silicate feedstock

Component	Fly ash	Slag	Montmorillonite
SiO_2	54.4	36.4	43.0
Al_2O_3	26.5	11.6	16.8
Fe_2O_3	4.8	0.8	3.6
MgO	2.5	5.8	2.0
Na_2O	0.4	0.3	0.2
K_2O	0.6	0.4	0.02
CaO	3.5	41.8	0.11
TiO_2	1.5	-	-
Cl	-	-	0.19
SO_3	1.7	-	-
LOS	-	-	34.0

Sunflower oil proved effective as a surfactant, being previously tested by authors in the paper [17]. According to Phavangkham et al. [19], in general the amount of liquid surfactant must be introduced into the solid mixture in very small proportions (at most 0.5 % of the amount of solid).

Sodium silicate (Na_2SiO_3) or ”water glass” is soluble in water forming an alkaline solution. It has ability to improve the strength of ceramic materials and composites, according to Ayadi et al. [23]. Sodium silicate is commercially available as a liquid solution with concentration of 38-40 %.

Sodium hydroxide (NaOH) or caustic soda, contains 16.4 % Na_2O, 34.3 % SiO_2, and 49.3 % H_2O. There is in solid state being high soluble in water. NaOH exists on the market in solid state (pellets) with over 98 % purity. In association with sodium silicate, it constitutes an ideal alkaline activator in accordance with Prochon et al. [24], being chosen by authors for the current experiment.

2.3 Adopted methods to identify the specimen features

Methods used for determining cellular geopolymer characteristics were the following. Denseness was identified utilizing Archimedes’ method in conformity with the ASTM C373 standard. ISO 18754:2020 was adopted for determining porousness. The method chosen for verifying the specimen water absorption was that according to ASTM C373-18. The compression resistance of geopolymer samples was determined with a hydraulically operated compression testing machine with a pressing capacity of 1000 tons-force in accordance with the ASTM C133-97 (2015) standard, while three-point bending tests for determining the flexural resistance of specimens required a multi-operational apparatus for testing (named Instron) at a crosshead speed of about 1.0 mm/min at 23 °C, in accordance with ASTM D-790M. The heat conductance was measured by the guarded-comparative-longitudinal heat flow method (ASTM E1225-04). ASONA 100X Zoom Smartphone Digital Microscope was utilized for examining the structure at micro-scale of cellular geopolymer specimens.

3. OUTCOMES AND DISCUSSION

3.1 Outcomes

The choice of the experimental versions for the attempt to manufacture a high-performance cellular geopolymer was made considering the results reported in the literature as well as the own previous experience of authors in this field.

The dosage of alumina-silicate wastes (class F-coal ash and ground metallurgical slag) was set at 345 and 100 kg·m^{-3} respectively, being kept constant throughout the experiment. Also, the alkaline activator was prepared in a constant dosage of NaOH and Na_2SiO_3 solutions, i.e. 45 kg·m^{-3} for NaOH and 115 kg·m^{-3} for Na_2SiO_3, in the ratio 1/2.55.

The mixture composition in the four adopted variants is presented in Table 2.

Table 2. Composition of testing variants

Composition (kg·m^{-3})	**Variant**			
	1	2	3	4
Class F-coal ash	345	345	345	345
Ground metallurgical slag	100	100	100	100
Mont-morillonite	4.0	5.5	7.4	8.9
Sodium perborate	6.0	7.6	9.3	10.8
Sunflower oil	2.5	4.5	6.5	8.5
10M NaOH solution	45	45	45	45
Na_2SiO_3 solution	115	115	115	115
Water addition	80	80	80	80

Images of surface of cellular geopolymer specimens prepared according to recipes shown in Table 1, hardened at 70 °C for one day and further, subjected to the curing procedure in ambient thermal conditions for four weeks are exhibited in Figure 1.

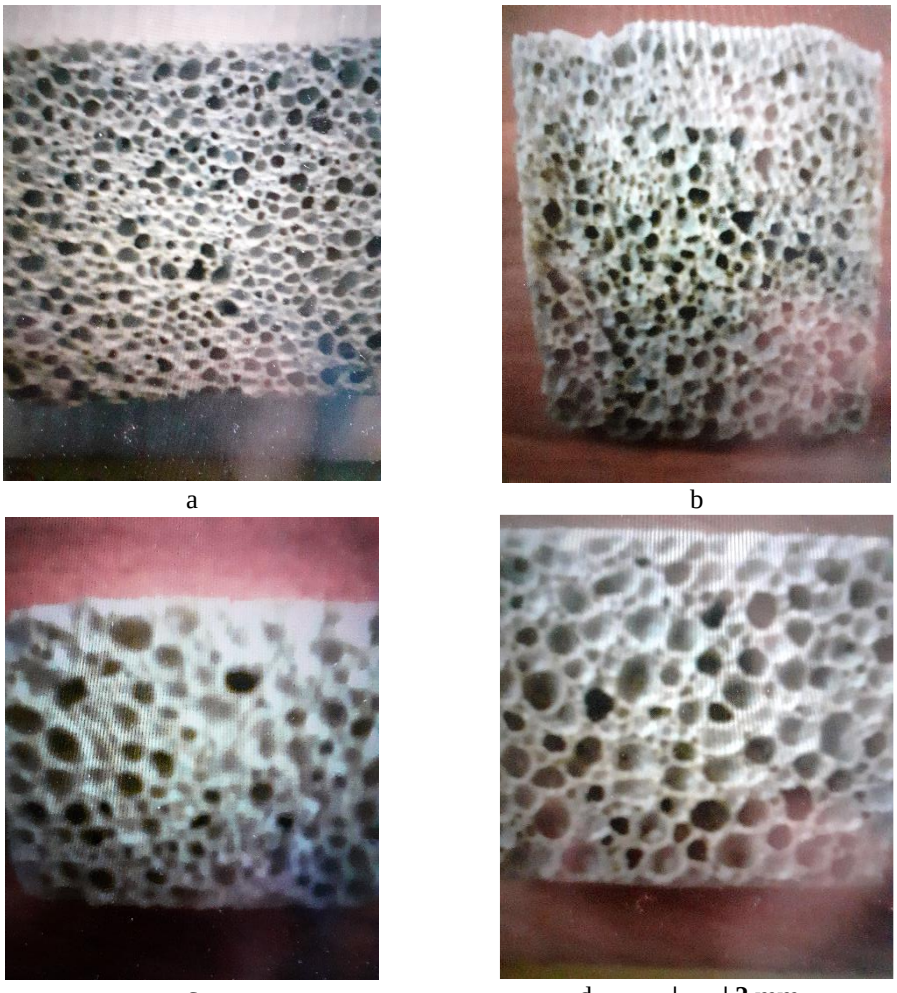

Figure 1. Surface of cell geopolymer samples after the curing process for 28 days
a – variant 1; b – variant 2; c – variant 3; d – variant 4.

Measuring the physio-mechanical, heat, and microstructural features of the geopolymer specimens was performed after 28 days, while the compression and flexural resistance was also determined after only 7 days to highlight the evolution of increasing the material strength at an early age. Results of these measurements are presented in Table 3.

Table 3. Particularities of cell geopolymer samples

Particularity	Variant 1	Variant 2	Variant 3	Variant 4
Denseness ($kg \cdot m^{-3}$)	910	780	690	610
Porousness (%)	43.8	52.7	61.0	68.5
Thermal conductance ($W \cdot m^{-1} \cdot K^{-1}$)	0.245	0.238	0.223	0.210
Compression resistance (MPa)				
- at early age	3.7	4.1	4.8	4.9
- at the end of curing process	5.0	5.6	6.5	6.7
Flexural resistance (MPa)				
- at early age	2.7	2.9	3.0	3.0
- at the end of curing process	3.0	3.2	3.3	3.3
Water uptake (vol %)	36.8	39.9	45.0	48.3
Pore dimensions (mm)	0.03-0.1	0.05-0.3	0.08-0.4	0.1-0.6

According to the data in Table 2, the variables in composition of the four making recipes were the blowing agent (sodium perborate), the nanomaterial (montmorillonite), and the surfactant (sunflower oil). All three variable components had increasing values from version 1 to version 4. Under these conditions, the above-mentioned abilities of these materials led to obtaining the maximum performances in the case of version 4. According to Table 3, denseness as well as heat conductivity had the lowest values (610 $kg \cdot m^{-3}$ and respectively, 0.210 $W \cdot m^{-1} \cdot K^{-1}$) and implicitly, porousness reached the highest value (68.5 %). The compression resistance had also an increasing slope of values, the highest being recorded after the end of the curing process (6.7 MPa). Also, flexural strength has grown in value up to 3.3 MPa, but similar or very close performances were also obtained in the case of versions 3 and 2. An interesting evolution of both types of mechanical resistance was noted following the determinations made at an early age (after 7 curing days). The values measured in all versions were very close to those recorded after 28 days. Regarding water uptake, this value has grown with growing the quantity of blowing product (sodium perborate), reaching a maximum of 48.3 vol. % in the case of version 4.

The variation of the structural aspect at a micro-scale of samples in accordance to the each preparing variant peculiarities is shown in Figure 2.

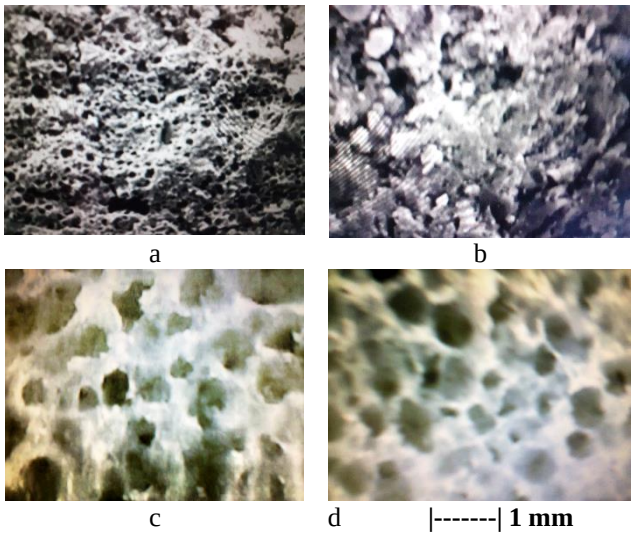

Figure 2. Structural appearance of geopolymer samples
a **–** variant 1; b – variant 2; c – variant 3; d – variant 4.

Obviously, the volume of cells in the porous structure considerably increased mainly due to increasing the amount of expanding agent (sodium perborate), also previously observed by Wattanarach et al. [18] and Paunescu et al. [17]. In version 4, the cell size range is within the limits of 0.1-0.6 mm, while in the first version, made with the lowest amount of sodium perborate, this size range is between 0.03-0.1 mm (i.e. 30-100 μm).

3.2 Discussion

The maximum amount of sodium borate (10.8 $kg \cdot m^{-3}$) represented 2.4 % of the used quantity of alumina-silicate waste. In general, the literature observations [17, 18] regarding the optimal proportion of sodium perborate addition showed that it would be around 2 % because above this limit the favourable effect of the blowing agent is radically attenuated. Under the concrete conditions of this

experiment, it was found that the proportion of 2.4 % can be applied with very good results regarding foaming the ash and slag-based cell geopolymer.

According to another work in which sodium borate was preferred over the traditional H_2O_2 [19], the ash-geopolymer was obtained with greater compression resistance than the geopolymer prepared with H_2O_2, under similar heat conductivity values. Table 3 confirms a high level of compression resistance of specimens made in versions 4 and 3, being obtained 6.7 and 6.5 MPa respectively, after 28 days, compared to 6 MPa of geopolymers expanded with H_2O_2.

To increase the strength of the cellular geopolymer, one of the possibilities is the use of a nanomaterial. In this experiment, a nanoclay called montmorillonite with micron particle sizes was chosen. This choice constituted one of the original solutions worldwide.

In order to growing the quality of cell product porousness, an original type of surfactant was adopted. Generally, surfactants have the role of reducing the tension on surface of the geopolymer froth in sludge form. They can improve the workability of the fresh material and especially ensure the distribution of finer pores after finishing the curing process [19]. The surfactant chosen for the experiment was sunflower oil, rarely used in the geopolymer foaming process, but known from the literature [25].

According to Wattaranach et al. [18], a comparison between the physio-mechanical and heat properties of a lightweight insulating concrete and a geopolymer froth based on metakaolin is clearly in favour of the geopolymer. Thus, the geopolymer denseness was determined between 760-1077 $kg \cdot m^{-3}$ compared to the maximum value of 1450 $kg \cdot m^{-3}$ of concrete and the compression resistance of the geopolymer was reported at 5-6 MPa compared to over 0.5 MPa corresponding to concrete. Thermal conductance values were in the range of 0.218-0.325 $W \cdot m^{-1} \cdot K^{-1}$, approximately similar to that of concrete (under 0.3 $W \cdot m^{-1} \cdot K^{-1}$).

4. CONCLUSION

Experimental manufacturing an environmentally friendly and economic geopolymer froth based on ash and blast furnace slag, alumina silicate secondary materials of energy and metallurgy industries was the research aim of this article. The current work showed that a mixture including the mentioned alumina-silicate waste, to which the addition of a nanomaterial (montmorillonite), a blowing agent (sodium perborate), and a surfactant (sunflower oil) as well as the traditional alkaline activator composed of aqueous solutions of NaOH and Na_2SiO_3, allowed to obtain a viscous slurry, which hardened at 70 °C for 24 hours and then subjected to the curing process for 28 days, generated a geopolymer froth with excellent characteristics. Optimal version of the final cell product had denseness of 610 $kg \cdot m^{-3}$, thermal conductance of 0.210 $W \cdot m^{-1} \cdot K^{-1}$, compression resistance of 6.7 MPa (at the end of process) and 4.9 MPa (at early age), flexure resistance of 3.3 MPa (at the end of curing) and 3.0 MPa (at early age), water uptake of 48.3 vol. %, and cell sizes between 0.1-0.6 mm. The new cellular product is adequate for applications in construction as a lightweight insulating material, having better features compared to the traditional lightweight insulating concrete.

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