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Thermal insulation and thermally resistant materials made of geopolymer foams

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Abstract

The foamed geopolymers are promising alternative to other foamed materials. The article presents the method of its production and the results of the research on such materials produced from fly ash from CHP plant. It provides and analyses the strength and thermal conductivity of the expanded geopolymers. It also determines the relationship between the thermal conductivity and density of foamed geopolymers. The density of the obtained materials ranged from approx. 400 kg/m^3 to 650 kg/m^3. The research results confirm that the foamed geopolymers produced from fly ash from CHP plant have reasonable mechanical properties and performance of a construction product.

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Keywords: Fly ash; geopolymers foam; thermal insulation

1. Introduction

Geopolymers are relatively new materials whose specific properties find wider and wider application in various industries. Nowadays they are applied mainly in construction and in the future may replace conventional concrete based on Portland cement. In fact, geopolymers may find application wherever the thermal insulation or fireproof properties are desirable e.g. in the interiors of airplanes and cars, in heat shields for space shuttles, in fire-resistant composites, or in foamed panels used as thermal insulation in the construction industry [1,2]. Geopolymers have

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been used since the early 1970s. At that time the fireproof particle boards were invented which consisted of the wooden core covered with two geopolymer layers [3].

Foamed inorganic polymers (geopolymers) are unique in terms of mechanical properties and fire safety. Additionally, they are light materials. Therefore, they may be applied in various branches of the industry, especially in construction [4,5].

Among such materials perhaps the most known is TROLIT™ [5]. TROLIT™ is an inorganic geopolymer foam manufactured from: 1) solid ingredients being the source of silicon and aluminum (they are most often by–products of various high-temperature technological processes); 2) liquid activator consisting of water solutions of alkaline metals; 3). Expanding agents (foaming agents), such as peroxides, perborates etc.

In order to manufacture TROLIT™ the solid and liquid ingredients are mixed in the dedicated appliance. Then, the obtained paste is poured into the molds where the polymerization takes place and the material is freely shaped (virtually any shape may be obtained with regard to dimensions and size, and the exact amount of the poured material depends on the volume of the mold and on the desired density of the final product). Then, the mold is closed and the foaming agent decomposes causing the material to expand. After the polymerization the material is unmolded and put into dryer to be hardened. Then, the machining takes place in order to meet the customer's particular needs for e.g., drilling, milling, cutting [5].

Table 1 presents the characteristics of TROLIT™ geopolymer foams which are nonflammable and whose max. application temperature is up to 1000 °C [5].

Table 1. Physical characteristics of TROLIT™ foams [1,5].

Bulk density (kg/m^3)	200–800
Max. temperature of application (^oC)	1000
Max. thermostability (^oC)	1200
Thermal conductivity ($W/(m \cdot K)$)	≥0.037 (depending on density)
Pore parameter (mm)	0.5–3.0
Fire protection [DIN 4102]	Inflammable, classification A1
Compressive strength (N/mm^2)	0.5–2.0
Tensile strength (N/mm^2)	≈250
Shrinkage (800 °C) (%)	< 1.5
Specific heat ($kJ/(kg \cdot K)$)	ca. 1.2
Linear heat expansion ((920–60)°C) (K^{-1})	9×10^{-6}

Fig. 1 presents the comparison of the max. application temperatures of some typical insulation materials.

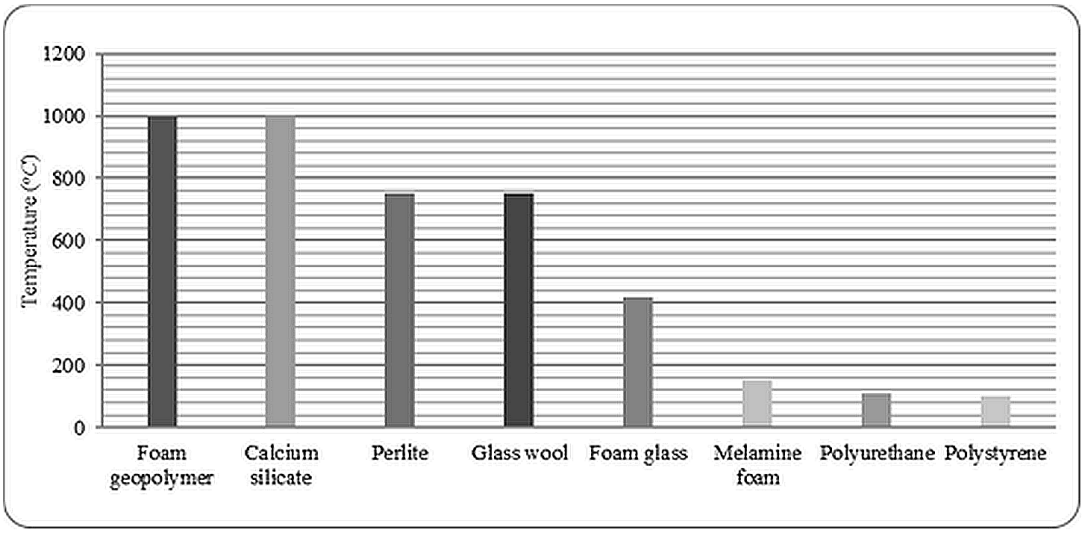

Fig. 1. Max. application temperature for some insulation materials (according to [5]).

In order to produce the foamed polymer one must take into account and optimally select two key parameters [1]: i.e.

- kinetics of peroxide decomposition (oxygen production)
- the increase of viscosity of the pre-condensate geopolymer

As there are no standard procedures and parameters pertaining to the process of production of geopolymer foams, such parameters must be selected individually i.e. dedicated to the locally available raw materials [1].

The activators in the production of geopolymers are mainly sodium hydroxide and sodium silicate, but also potassium hydroxide and potassium silicate are used [6,7]. In the production of foamed geopolymers one primarily uses fly ash from coal combustion [8,9], metakaolin [6], but also other materials e.g., palm oil fuel ash [10,11]. The typical gas-producing agents are aluminum powder or materials containing elemental silicon (silica powder, FeSi or SiC). In the alkaline environment the reaction occurs involving Al or Si and the gas hydrogen is released (generated) [4]. During the process of expanding geopolymers very often various surfactants are added (e.g., Sika Lightcrete 02) containing 40 wt% solution of fatty acid, amide and sodium salt of C14–C16 sulphonic acid in water. In order to decrease viscosity other additives are used e.g., polyacrylic acid (Dolapix CE-64 by Zschimmer & Schwarz) [6]. Apart from that, sodium hypochlorite (NaOCl) [13] as well as 1, 2 or 3 % of hydrogen peroxide and sodium perborate [12] are used as a foaming agent. The increase of porosity may be achieved by means of in situ formation of surfactants by virtue of the saponification of oil in the geopolymer alkaline environment [14].

Geopolymer foams are often unstable in the production process and have a tendency to collapse which may be prevented with the addition of foam stabilizers e.g., Portland cement or lime [4]. To stabilize fresh geopolymer foam one adds non-ionic surfactants e.g., Tween80 by VWR BDH Prolabo (Polyoxyethylene (20) sorbitan monooleate – $C_{64}H_{124}O_{26}$), and TritonX – 100 by Sigma-Aldrich (polyethylene glycol tert-octylphenyl ether – $C_{14}H_{22}O$ $(C_2H_4O)_n$, n= 9–10) [6]. The compressive strength of geopolymer foams made from fly ash varies depending on the particular material and shaping method e.g., amounting to 0.9–4.35 MPa [15], >3.7 MPa [16] and approx. 3.3–3.7 MPa [17] and amounting to 0.78 MPa for geopolymer foams made from perlite [18].

In this research we focused on the possibility of production of the geopolymer foams from the fly ash provided by the local CHP plant, and on determining such foaming methods that allow for the use of most available raw materials and for the repetitive industrial-scale applications.

2. Experimental

2.1. Materials

In order to obtain the desired geopolymer, flakes of technical sodium hydroxide and an aqueous solution of sodium silicate of R-145 module 2.5 and molar density of about 1.45g/cm^3 were used. Tap water was used instead of the distilled one. To prepare the alkaline solution, the aqueous solution of sodium silicate and water was poured over solid sodium hydroxide. The resulting solution was thoroughly mixed and allowed to equilibrate to constant concentration and temperature.

In the research, we used fly ash from the CHP plant in Skawina. Its oxide content and the physical and chemical properties are presented in Tables 2–4. In the production of geopolymer foams we used microspheres (by Micromex) and 35% hydrogen peroxide by Chempur. The size of the grains of microspheres amounted to 100–500 μm.

Table 2. Oxide content of the fly ash from the Skawina CHP plant.

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	TiO_2	P_2O_5	BaO
Content (%)	55.89	23.49	5.92	2.72	2.61	0.59	3.55	0.16	1.09	0.82	0.20

Table. 3. Chemical properties of the fly ash from the Skawina CHP plant.

Parameter	SiO_2 reactive	CaO free	Na_2O_{eq}	Chlorides (Cl-)	Soluble phosphates as P_2O_5	Loss on ignition
Result (%)	35.86	0.02	4.76	0.034	0.0008 (8 mg/kg)	2.84

Table. 4. Physical properties of the fly ash from the Skawina CHP plant.

Parameter	Fineness (%)	Density (*kg/m^3*)	Pozzolanic activity index after 28 days (%)	Beginning of the binding (*min*)	
				Cement (for comparison)	75% cement (for comparison) + 25% fly ash
Result	34.2 ± 0.3	2180 ± 20	75 ± 3	175 ± 10	240 ± 15

2.2. Examination methods

The compressive strength tests were conducted with a testing machine MATEST 3000 kN on the samples whose dimensions were 100 × 100 × 100 mm. The thermal conductivity coefficient λ was calculated in accordance with the PN-EN 12667:2002. The measurements were carried out under the steady-state conditions of heat transfer, at the average sample temperature amounting to 10 °C, for the samples whose dimensions were 300 × 300 × 100 mm. The applied temperature difference was 20 °C for the thickness of the sample (T1=0 °C, T2=20 °C, Tmean=10 °C). The tests were conducted in the closed test chamber (having thermally-insulated walls) in the one-sample plate apparatus HFM 436/3/0, NETZSCH provided with the heat-flow density sensor. The results were compared with the certified reference values IRMM-440 no 30. The bulk density of the product was calculated as a ratio of the mass to the volume of the sample, without referring to the certified specimen.

3. Results and discussion

In order to obtain geopolymer foams from the fly ash from the Skawina CHP plant the solid ingredients such as fly ash and microspheres were mixed to achieve homogeneity and then applied with the alkaline solution (14M aqueous solution of NaOH + liquid glass at a ratio of 1:2.5). During the mixing the solution was gradually added until the desired consistence of the mixture was achieved. Then, it was mixed for 7–10 min. Then, the proper

amount of the hydrogen peroxide was added (seven various recipes were prepared – see Table 5). After the mixing the obtained material was poured into plastic molds and put in the lab dryer for 24 h at 75 °C. After drying the unmolded samples were stored in the laboratory conditions for 28 days. Finally, the properties of the produced foams were determined.

Fig. 2. The exemplary surface of the obtained geopolymer foams (sample F4).

Table 5. Composition and physical properties of geopolymer foams.

Sample labels	Composition, additives (*% vol.*)	Density (kg/m^3)	Heat conductivity ($W/(m \cdot K)$)	Compressive strength after 28 days (*MPa*)
F1	30% microspheres; 7% H_2O_2	401.47	0.0947	1.9
F2	30% microspheres; 5% H_2O_2	414.07	0.0980	1.9
F3	30% microspheres; 4% H_2O_2	421.49	0.0826	2.1
F4	20% microspheres; 5% H_2O_2	458.09	0.1103	2.3
F5	20% microspheres; 4% H_2O_2	474.65	0.1261	2.3
F6	10% microspheres; 5% H_2O_2	520.53	0.1019	2.9
F7	10% microspheres; 2% H_2O_2	640.46	0.1273	3.4

Table 5 presents the composition of the examined materials and their characteristics such as density, heat conductivity coefficient and compressive strength (which, dependent on the sample, amounted to 1.9–3.4 MPa). The compressive strength and the value of the heat conductivity coefficient were dependent on the density of the obtained geopolymer foams.

Fig. 2 presents the structure (physical appearance) of the obtained geopolymer foams (sample F4). The visual analysis shows that the structure of materials is homogeneous, that means the pores are evenly distributed and have almost the same size in the sample. However this parameters are various between different samples. Fig. 3 presents the relationship between the heat conductivity coefficient and the density of the geopolymer foams. The relationship is linear: the lower the density the lower the coefficient.

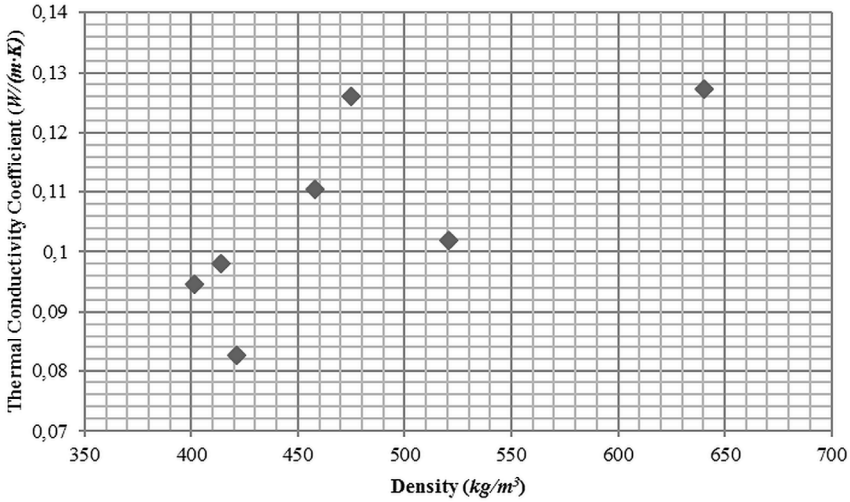

Fig. 3. The relationship between the heat conductivity coefficient and the geopolymer density.

According to the conducted research, it is possible to obtain geopolymer foams whose density is approx. 400 kg/m^3 and heat conductivity is less than 1 W/(m·K) using raw materials similar to fly ash from the Skawina CHP plant. The foams were produced by means of the foaming agent i.e. 35% hydrogen peroxide. In order to ensure a good ratio of open to closed pores and to improve the insulating properties of the product the microspheres were added (10–30% vol.). The research confirmed the positive relationship between the heat conductivity coefficient and the density of the obtained materials [4,5]. Comparing of prepared foam geopolymer with Trolit and other foam materials available on the market there has a similar density, a little bit worse thermal properties such as thermal conductivity, but better compressive strength than other material. It may be useful especially for construction purposes. The authors of the research currently focus on the method of production of similar materials whose density is approx. 200 kg/m^3 and heat conductivity coefficient <0.06 W/(m·K).

4. Conclusions

The foamed geopolymers can be a promising alternative to other foamed materials. The research results confirm that the foamed geopolymers produced from fly ash from CHP plant have reasonable mechanical properties and performance of a construction product. It is possible to obtain foamed geopolymers whose density amounts to 200–400 kg/m^3 by means of the proper mixing (homogenization) method and adding the more foaming agent. The research shows that the hydrogen peroxide (H_2O_2) is a good foaming agent. It allows obtaining a homogeneous material structure (pores are evenly distributed and have almost the same size in the sample). In the article, there are presented the composition with the additive of form 2% to 7% of the hydrogen peroxide (H_2O_2). The amount of it has an influence on density and mechanical properties. The higher amount of the hydrogen peroxide (H_2O_2) caused lower material density and also lower compressive strength. The use of microspheres from fly ash allows reaching appropriate proportions of cells. In the material, there are open as well as close cells. The presence of close cells gives the material better insulating properties comparing with material that contains only open cells.

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