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## FOAMED GEOPOLYMER WITH CUSTOMIZED PORE STRUCTURE

### Article Highlights

- By varying the concentration of H<sub>2</sub>O<sub>2</sub> and surfactant, the porosity can be designed optionally
- It is possible to produce closed-pore thermal insulating materials or open-pore catalyst supports
- Foams with thermal conductivity in the range of 0.130–0.054 W/mK were produced using only H<sub>2</sub>O<sub>2</sub>
- The use of sodium oleate as a surfactant increased the chance of open-cell formation
- For catalyst support, foams with 75–80 vol% total porosity and >1 MPa strength were achieved

### Abstract

*Due to their favorable production conditions and promising properties (e.g., low shrinkage after foaming, mechanical and chemical stability, high-temperature resistance), geopolymer foams are suitable for heat- and sound insulating refractory building materials. Another promising field of application may be their use as catalyst supports in water treatment. Metakaolin-based foams were prepared by a direct foaming process with high total porosity (> 75 vol%), low bulk density (< 500 kg/m<sup>3</sup>), approximately 1 MPa compressive strength, and low thermal conductivity (0.095 W/mK). By varying the concentration of foaming agent (H<sub>2</sub>O<sub>2</sub> solution) and stabilizing agent (sodium oleate), it is possible to produce foams with designed porosity and pore size distribution. Foams with mainly closed pores are suitable for thermal insulation, while those with significantly open pores can use as catalyst supports. The computed tomography images showed that the concentration of stabilizing agent is a key parameter in forming a homogeneous pore structure and open pores; up to 24 vol% open porosity can be achieved without significantly affecting other properties. The physical properties of the foams are equally influenced by the thickness of cell walls and the size of the cells themselves.*

*Keywords: catalyst support, geopolymer, heat insulator, mechanical property, porous material, thermal conductivity.*

Cellular materials, especially foamed inorganic solids, have rapidly growing importance in the field of filters, thermal insulators, and catalyst supports. Proper thermal insulation is crucial to avoid wasting energy, whereas inorganic insulators have a great advantage over organic ones: require less hazardous

substances for production and have better recycling potential. On the other hand, wastewater treatment is a global environmental problem, where photocatalysis can provide a clean solution, and durable catalyst supports are required. Porosity plays an important role in both cases, and the cell structure must be carefully controlled to achieve the expected properties. Three factors considerably define the cellular material: the properties of the raw material, the topology and shape of the cells, and their bulk density [1].

Foamed inorganic solids can be prepared easily by mixing a cementitious binder with a foaming agent. Although foams made of ordinary Portland cement (OPC) have good thermal insulation properties, the en-

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environmental effects of cement production (e.g., high energy demand, CO<sub>2</sub> emissions, landscape destruction, etc.) give serious cause for concern [2].

Binders with lower environmental impact, such as alkali-activated types of cement (and geopolymers), can be good alternatives, proven to have superior physical properties in versatile applications [3,4]. Geopolymers are a significant section of alkali-activated cements and are obtained by mixing a low-calcium natural material (e.g., metakaolin or clay) or an industrial by-product (e.g., fly ash) with a strongly alkaline solution. The resulting cement paste sets at room temperature and has excellent physical properties even at an early age (high compressive strength, good thermal resistance, low shrinkage, etc.) [4].

Production of foams is one of the fastest-growing research fields in recent years of geopolymer applications [5]. Three main methods can achieve the desired porosity (or by their combination):

method 1 mixing gas-releasing agents such as hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) [6–12] or fine aluminum powder [7, 12–14] with the geopolymer paste/mortar,

method 2 mechanically introducing a substantial volume fraction of air bubbles into the paste [15,16], usually through the use of an organic surfactant which plays a stabilizing agent role,

method 3 mixing a highly porous aggregate with the matrix (for example perlite [16], EPS [17,18], microspheres [18], aggregate from recycled lightweight blocks [19,20]).

In some cases, due to deteriorating mechanical strength, fiber reinforcement is also applied in the geopolymer foams [13,20].

H<sub>2</sub>O<sub>2</sub> solution and aluminum powder are used in the largest quantities as chemical foaming agents, which decompose to O<sub>2</sub> and generate H<sub>2</sub> gas, respectively, by the following equations [7,21]:



The foaming process can serve a dual purpose: making thermally insulating materials [6–14,17–19,21–25] or foamed inorganic solids for catalyst support applications [15,16,26–28]. In recent studies, the investigated parameters were the chemical composition of the alkali activator solution (for example sodium silicate/sodium hydroxide mass ratio) [6,9,14], the activator/binder mass ratio [6,11,14,23], and the foaming agent concentration [6,7,11,14,21,24,28]. In some references, the foaming agent is also combined

with a stabilizing component (surfactant) [21,24], but only Korat and Ducman [21] have investigated the effect of the surfactant concentration. In some cases, surfactants were formed through in-situ saponification of vegetable oils, resulting in a high volume of open porosity [26–28].

Generally, foams derived from fly ash [6,7,14,22,24,25] or metakaolin [9,10,15,26,27] have good thermal insulation properties, with lower than 1000 kg/m<sup>3</sup> bulk density and < 0.1 W/mK heat conductivity and a compressive strength between 1–4 MPa. By foaming, ground granulated blast furnace slag also provides porous structures with slightly higher bulk density and heat conductivity [11]. Still, these foams are mainly made using highly porous materials according to the third method described above [20].

According to the literature, the activator/binder mass ratio needs to be around 0.8 and the sodium silicate/sodium hydroxide mass ratio around 2.5 to achieve the lowest thermal conductivity (< 0.1 W/mK) [6,9,14]. Likewise, a high concentration of sodium hydroxide in the activating solution leads to an unstable H<sub>2</sub>O<sub>2</sub> decomposition, while the presence of more sodium silicate in the activating solution results in a more controlled H<sub>2</sub>O<sub>2</sub> decomposition rate during foaming [9]. Using a low, 0.55 solid/liquid mass ratio (high activating solution content) and Al powder results in relatively low density (430 kg/m<sup>3</sup>) and low thermal conductivity (0.079 W/mK) [23].

Researchers have also shown that the concentration of the foaming agent has a strong effect on bulk density, compressive strength, and heat conductivity. Al powder was applied between 0.01 and 0.2 wt% [10,23,24], while the H<sub>2</sub>O<sub>2</sub> content was varied between 0.1 and 20 wt% [6,7,9,24,26,27], depending on the concentration of the solution. Ducman and Korat [7] have shown that using Al powder in the range of 0.07–0.2 wt% resulted in bulk densities between 0.64 and 0.74 g/cm<sup>3</sup> with compressive strengths within the range of 3.3 and 4.3 MPa, while samples prepared from 0.2–2 wt% H<sub>2</sub>O<sub>2</sub> had densities between 0.61 and 1.00 g/cm<sup>3</sup> and compressive strengths in the range of 2.9 and 9.3 MPa. Hajimohammadi *et al.* [9] have found that producing foams with homogeneously small pore sizes leads to low bulk density and helps gain more strength while providing better thermal insulation capability. Korat and Ducman [21] have established that the stabilizing agent performs best when its amount is adjusted to the amount of the foaming agent. It is difficult to compare the results because of the lack of a consistent definition of foaming agent concentration in the literature.

The current study presents the link between the

composition of the foam (concentration of the foaming agent and surfactant) and the cell structure of the foamed specimens. In addition, the most important physical properties such as bulk density, total and open porosity, water absorption, compressive strength, and thermal conductivity were also studied. By clarifying the relationship between cell structure and physical properties, the aim was to produce geopolymer foams with controlled properties that could act as thermal insulators or catalyst supports.

## MATERIAL AND METHODS

### Materials

Metakaolin, obtained from New-Zealand kaolin by calcination at 750 °C for 8 h, was used for geopolymer production. The original clay contained mainly kaolinite and halloysite phases and quartz and cristobalite as minor constituents (identified using a Philips PW 3710 X-Ray powder diffractometer - reported in a previous article [29]). The chemical composition of the metakaolin was determined by wet chemical analysis, resulting in SiO<sub>2</sub> 56.3, Al<sub>2</sub>O<sub>3</sub> 38.0, TiO<sub>2</sub> 0.2, Fe<sub>2</sub>O<sub>3</sub> 0.9, CaO 0.5, MgO 0.5, Na<sub>2</sub>O 0.4, K<sub>2</sub>O 1.0, LOI 2.2 wt%. The mean particle size was 15.8 μm (determined by a Fritsch Laser Particle Sizer).

The alkali activator solution was produced using commercially available sodium silicate solution (SiO<sub>2</sub> 28.6, Na<sub>2</sub>O 6.8, H<sub>2</sub>O 64.5 wt%, ANDA) and laboratory-grade sodium hydroxide pellet (Scharlab). In addition, H<sub>2</sub>O<sub>2</sub> solution with 30 wt% (Reanal) was applied as a chemical foaming agent and sodium oleate powder (Sigma) as a stabilizing agent.

### Sample preparation

Metakaolin-based foamed geopolymers (FG) were made. An alkali activator solution was first prepared during the experiments by dissolving the required amounts of sodium hydroxide pellets in sodium silicate solution (and in distilled water as needed). Then the specified amount of H<sub>2</sub>O<sub>2</sub> solution and sodium oleate powder was added to the activating solution (Table 1). After this mixture cooled down to room temperature, it was mixed with the metakaolin powder for 1 min. Next, the slurry was cast into cylindrical molds of dimensions Ø36 mm × 36 mm. One hour after the final setting (195-240 min depending on composition), the "cup" (the excess amount of foamed sample) was removed with a sharp knife to prevent cracking, which may occur due to the shrinkage of the sample. The specimens were demolded at 1 day of age and stored at ambient conditions (21-23 °C and RH = 50 ± 10%) until 7 days. Large-size samples were prepared in a wooden mold with 200 mm × 200 mm × 15 mm.

The composition of the geopolymer paste applied in the experiments was as follows: SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> = 3.4, Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> = 1.0, the sodium silicate modulus of the alkali activator solution = 1.4, the amount of Na<sub>2</sub>O in relation to the dry metakaolin was 23.1 wt%, the mass ratio of the sodium silicate and sodium hydroxide pellets was 5.4, while that of the activator solution and metakaolin powder was 1.3.

In stage I, the concentration of the chemical foaming agent was varied as 0.0, 0.8, 1.6, 2.3, 3.1, 4.5, 6.0, 11.3 wt% H<sub>2</sub>O<sub>2</sub> solution relative to the mass of metakaolin. In stage II, compositions with the most favorable physical properties were compared with the mixtures supplemented with a fixed amount of sodium oleate surfactant (1.4 wt% relative to the mass of metakaolin). Finally, in stage III, the surfactant concentration was applied as 0.1, 0.3, 0.5, 1.0, 1.4, 2.1, and 2.8 wt% relative to the mass of metakaolin with a fixed amount of chemical foaming agent. The investigated mixtures are presented in Table 1.

Table 1. Foaming agent contents of the specimens

Series	Foaming agent concentration, wt%	
	H <sub>2</sub> O <sub>2</sub> solution	Sodium oleate
I	0.0, 0.8, 1.6, 2.3, 3.1, 4.5, 6.0, 11.3	0.0
II	3.1, 4.5, 6.0	0.0
	3.1, 4.5, 6.0	1.4
III	4.5	0.1, 0.3, 0.5, 1.0, 1.4, 2.1, 2.8

According to the following system, Samples were named FG (foamed geopolymer) H<sub>2</sub>O<sub>2</sub> solution concentration/sodium oleate concentration; "Δ" indicates the varied parameter. For example, FG Δ/0.0 means a foamed geopolymer sample with various H<sub>2</sub>O<sub>2</sub> solutions without sodium oleate content.

### Characterization

A Controls 5 universal testing machine performed compressive strength tests with a loading rate of 2400±1 N/s according to EN 196-1. However, the sample sizes differed from the standard (Ø 36 × 36 mm cylindrical samples were tested). Three samples of each formulation were tested, and the average data were reported at the age of 7 days. The sample surfaces were polished flat and parallel before testing if it was necessary.

The bulk density of geopolymer samples was obtained by measuring the mass to volume ratios in foamed samples (0.0 wt% < H<sub>2</sub>O<sub>2</sub> content ( $\rho_b$ )).

The porosities and water absorption were determined by Archimedes' method.

The samples were placed in distilled water for 2 hours and weighed on a hydrostatic balance. Water absorption was calculated by the following equation (Eq. (3)):

$$w_A(\text{wt}\%) = 100 \cdot (m_2 - m_1) / m_1 \quad (3)$$

where  $m_1$  is the weight of the dry sample,  $m_2$  is the weight of the sample after 2 hours of water absorption. The apparent porosity was obtained by the Eq. (4):

$$P_A(\text{vol}\%) = \rho_b \cdot w_A \quad (4)$$

where  $\rho_b$  is the bulk density. Total porosity was calculated using the true density and bulk density of samples with the following equation (Eq. (5)):

$$P_T(\text{vol}\%) = 100 \cdot (1 - \rho_b / \rho_T) \quad (5)$$

where  $\rho_b$  is the apparent density of the foamed samples, while  $\rho_T$  is the true density of the geopolymer matrix (1961 kg/m<sup>3</sup>), determined by the pycnometer method.

Heat conductivity ( $\lambda$ ) was measured using the MTPS (Modified Transient Plane Source) method with a C-Therm TCi equipment. Before characterization, the bottom of the samples was polished and the powder removed from the exposed pores; no contact agent was used for measurement. The larger (200 mm × 200 mm × 15 mm) samples were examined using the heat flow meter method with NETZSCH HFM 436/3/1 Lambda equipment. Before measurement, the surface of samples was polished parallel.

The MTPS technique is a fast thermal conductivity measuring method. It does not require large-sized specimens, but the thermal conductivity value obtained is lower than the one measured with a heat flow meter, which is generally used to determine the thermal resistance of insulators. For this reason, the MTPS method was used to compare the thermal conductivity values of the differently foamed specimens and investigate the tendency in thermal conductivity during the series of experiments. Based on the series II and III results, two compositions were chosen; a: using only H<sub>2</sub>O<sub>2</sub> (FG3.4/4.5/0.0) and b: combination of H<sub>2</sub>O<sub>2</sub> and surfactant (FG3.4/4.5/1.4). Large-sized samples (200 × 200 × 15 mm) were prepared from these compositions as described at the beginning of the Sample preparation section. Their thermal conductivity was measured by the heat flow meter method.

Samples were prepared separately for cell structure investigation, where the top of the specimens was not removed. The cell structure was characterized by a NIKON XT H 225 ST X-ray tomography, which was

also used to assess the distribution of different pores (type, size) in the samples. It is important to notice that this method is non-destructive, which is essential in the case of such fine structures. The pore size distribution and pore wall thickness values were measured by a KEYENCE VHX 2000 digital microscope. Before the examination, the cross-section of the samples was polished flat and coated with gold/palladium films to a thickness of 2–3 nm (using a BALZERS SCD020 type unit) for better contrast. Pore size distribution and pore wall thickness values were obtained by taking the average of 100 pore diameters and 50 pore wall thicknesses.

## RESULTS AND DISCUSSION

### Effect of foaming agent concentration

One must note that the water content of the chemical foaming agent, H<sub>2</sub>O<sub>2</sub> solution, was not compensated. Consequently, with the increase in H<sub>2</sub>O<sub>2</sub>, the water content of the geopolymer paste also raised from 28.9 wt% (0.0 wt% H<sub>2</sub>O<sub>2</sub>) to 31.1 wt% (11.3 wt% H<sub>2</sub>O<sub>2</sub>).

The compressive strength and bulk density results of the stage I tests can be seen in Figure 1a. The values of the dense samples (0.0 wt% H<sub>2</sub>O<sub>2</sub>) were outstandingly high (61.3 MPa and 1631 kg/m<sup>3</sup>, respectively) and therefore are not depicted in this figure. Compressive strength rapidly falls from ~60 MPa below 10 MPa by applying 1.6 wt% of H<sub>2</sub>O<sub>2</sub>, but further addition of foaming agent causes a slower decline in strength. A similar trend can be observed for bulk density results. For instance, a higher foaming agent concentration does not lead to a proportionately lower bulk density, while the smallest attainable bulk density was around 500 kg/m<sup>3</sup>. In addition, Ducman and Korat [7] report similar results with 2.9 MPa and a bulk density of 0.61 g/cm<sup>3</sup>. Their higher strength result can result from a different loading rate, applying 5 N/s 480 times lower than the loading rate used in the present study.

Porosity and water absorption results (Figure 1b) indicate a change in structure when the foaming agent concentration exceeded 3.1 wt%. Total porosity did not change considerably even if the H<sub>2</sub>O<sub>2</sub> concentration was raised from 3.1 to 11.3 wt% (the highest total porosity was ~75 vol%). In contrast, water absorption rapidly rose from 6–10 wt% to 36 wt%, and a similar but less remarkable tendency can be seen in the case of open porosity (from ~10 vol% to 18 vol%). These two properties are closely related: more water can enter the sample due to the higher number of open pores.

The thermal conductivity of the dense sample was 1.280 W/mK, determined by the MTPS method.

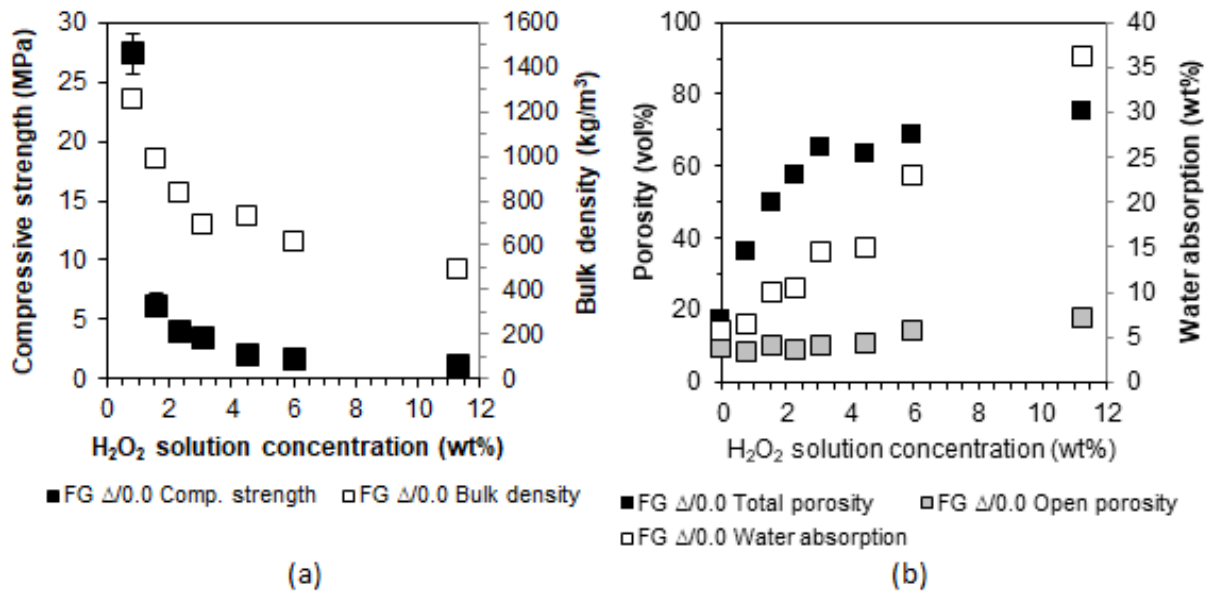


Figure 1. Compressive strength, bulk density (a), total porosity, open porosity, and water absorption (b) results in stage I testing at 7 days of age.

On the other hand, thermal conductivity for foamed samples was 0.130–0.060 W/mK (Table 2). The lowest result was achieved with 4.5 wt% of H<sub>2</sub>O<sub>2</sub> and got slightly higher (0.070 W/mK) with the increase of foaming agent.

Table 2. Thermal conductivity results in stage I testing at 7 days of age

Sample name	Thermal conductivity (W/mK)
FG 0.0/0.0	1.280 ± 0.009
FG 0.8/0.0	0.130 ± 0.019
FG 1.6/0.0	0.090 ± 0.001
FG 2.3/0.0	0.066 ± 0.003
FG 3.1/0.0	0.080 ± 0.001
FG 4.5/0.0	0.067 ± 0.002
FG 6.0/0.0	0.070 ± 0.001
FG 11.3/0.0	0.070 ± 0.006

According to CT images (Figure 2) and pore characteristics (Table 3), the concentration of H<sub>2</sub>O<sub>2</sub> solution has a subtle effect on cell size, cell wall thickness, and pore size distribution. For example, using only H<sub>2</sub>O<sub>2</sub> as a foaming agent leads to a heterogeneous pore structure, which, based on light microscopic images (Figure 3), consists of macrocells larger than 250–350 μm diameter, and the space between them being populated with much smaller ones, causing increased cell wall thicknesses (Table 3). Bai *et al.* [27] experienced similar phenomena when making foams using a mixture of H<sub>2</sub>O<sub>2</sub> and oil as

surfactants.

Table 3. Cell diameters and cell-wall thicknesses of the foam samples using H<sub>2</sub>O<sub>2</sub> as a foaming agent

Sample name	Average cell diameter (μm)	Average cell wall thickness (μm)
FG 1.6/0.0	516 ± 158	215 ± 135
FG 4.5/0.0	703 ± 661	709 ± 476
FG 6.0/0.0	1546 ± 532	592 ± 382
FG 3.1/1.4	140 ± 31	47 ± 36
FG 6.0/1.4	427 ± 84	38 ± 26
FG 4.5/0.5	392 ± 60	35 ± 32

Increasing the amount of the H<sub>2</sub>O<sub>2</sub> solution leads to larger pore sizes (1.6 wt%, 4.5 wt%, and 6.0 wt% to around 520 μm, 700 μm, and 1550 μm, respectively). It can be explained by the release of a larger amount of O<sub>2</sub>, as well as the larger water contents. A higher O<sub>2</sub> concentration means that cell walls need to withstand increased oxygen pressure. A higher water content (due to the higher H<sub>2</sub>O<sub>2</sub> concentration) and thus lower viscosity of the geopolymer paste may also weaken the cell walls. Increasing the foaming agent content causes the cell size distribution to become heterogeneous in any given volume and the cross-section of the sample, meaning that the bottom of the sample is populated with much smaller cells than the top. Due to their buoyancy, the larger pores are likely to rise in the plastic sample (before the setting). The most significant cells are found directly under the sample cap, which may be trapped

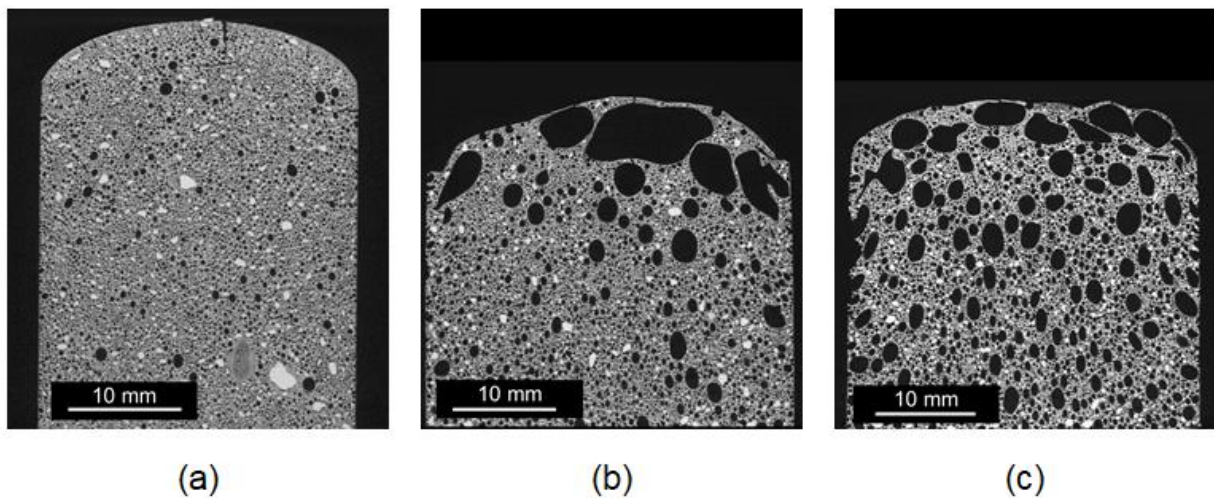


Figure 2. The pore structure of the foam samples in stage I testing using  $H_2O_2$  as a foaming agent (selected samples were the following:  $a = FG\ 1.6/0.0$ ,  $b = FG\ 4.5/0.0$ , and  $c = 6.0/0.0$ ).

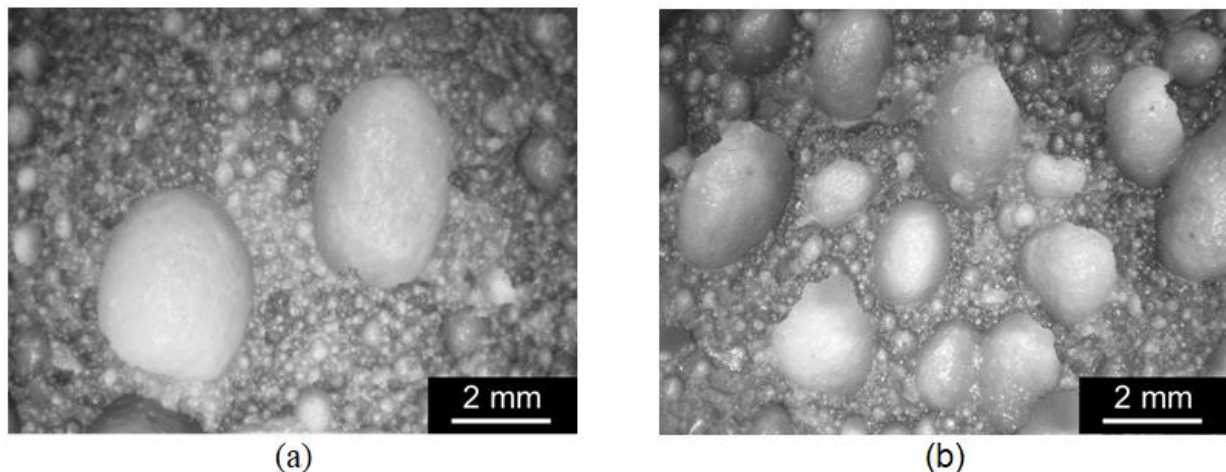


Figure 3. The cell wall structure of foamed samples in stage I testing using  $H_2O_2$  (selected samples were the following:  $a = FG\ 4.5/0.0$  and  $b = FG\ 6.0/0.0$ ).

because geopolymer samples tend to have a thin, rapidly setting layer on their top. It is due to the reaction between  $CO_2$  in the air and water glass in the activator solution. The white particles on the CT images, indicating a higher X-ray absorbance in the rendering mode, are undissolved metakaolin particles. Their amount also decreases with increasing  $H_2O_2$  contents.

The microstructure development explains many phenomena in changing physical properties (Figure 2). Higher than 1.6 wt%  $H_2O_2$  concentration leads to heterogeneous cell size and thicker cell walls. The paste cannot hold the higher concentration of released oxygen, and the foaming gas "escapes" from it, which causes the collapse of the foam. Therefore, a higher concentration of  $H_2O_2$  results in unpredictable properties instead of the expected lower bulk density or higher total porosity. The rupturing pores also cause

more open cells, increasing water absorption (Figure 1). The thicker cell walls explain the moderate compressive strength and the slight increase in thermal conductivity (Tables 2 and 3).

#### Effect of surfactant concentration

A foam stabilizer was used to avoid the pore coalescence and collapse of the pore structure in stages II and III. Figure 4a shows the compressive strength and bulk density results of geopolymer foams where sodium oleate was applied as a commonly used surfactant (1.4 wt%), the results are compared to those of samples prepared with  $H_2O_2$  only. The surfactant used has a strong effect on physical properties. A much lower bulk density (with the minimum value of  $\sim 360\ kg/m^3$ ) can be achieved with considerable strength loss. In addition, a surfactant concentration

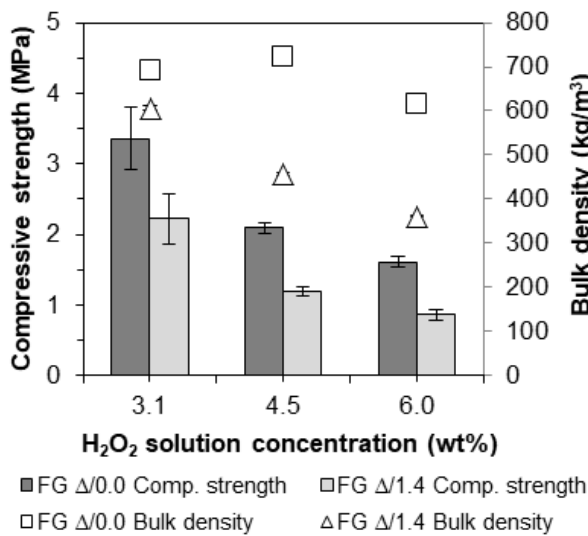
even as low as 0.1 wt% remarkably reduces bulk density (from 724 kg/m<sup>3</sup> to 512 kg/m<sup>3</sup>), with a further increase in sodium oleate level impairing compressive strength, causing it to drop from ~2.0 MPa (0.1 wt% sodium oleate) to ~1.2 MPa (≥ 1.0 wt% sodium oleate), (Figure 4b).

It was already noticeable from the bulk density results but even more apparent when considering porosity and water absorption data that in the case of 3.1 wt% H<sub>2</sub>O<sub>2</sub>, there is no significant difference between the properties of samples made with or without

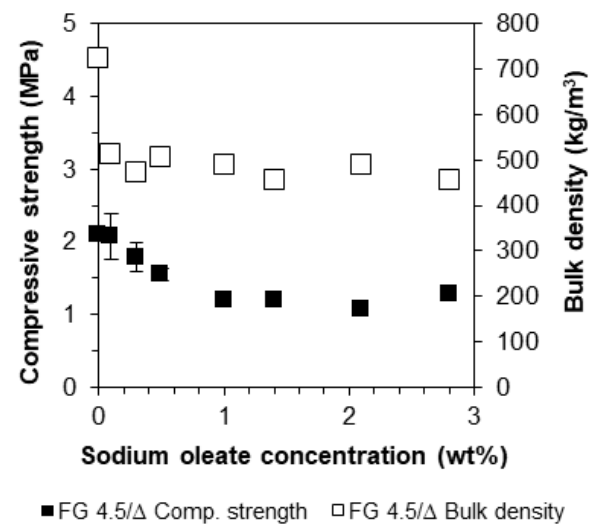
surfactant (Figure 5a). However, with the rise of H<sub>2</sub>O<sub>2</sub> concentration, bulk density, and compressive strength drop remarkably, but there is also a sharp rise in porosities and water absorption.

Varying the amount of surfactant showed that (Figure 5b), similarly to bulk density results, 0.1 wt% is sufficient for the highest achievable total porosity (~75 vol%). Further increase in the sodium oleate content leads to a higher proportion of open porosity (from ~10 vol% to ~20 vol%), with greater water absorption.

The lower bulk density and greater porosity

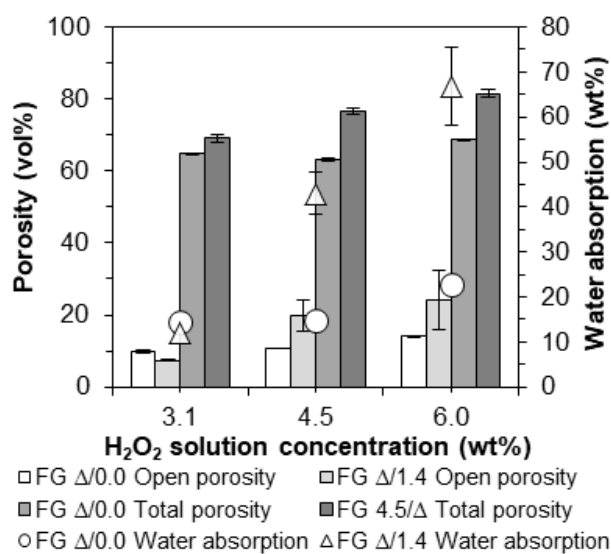


(a)

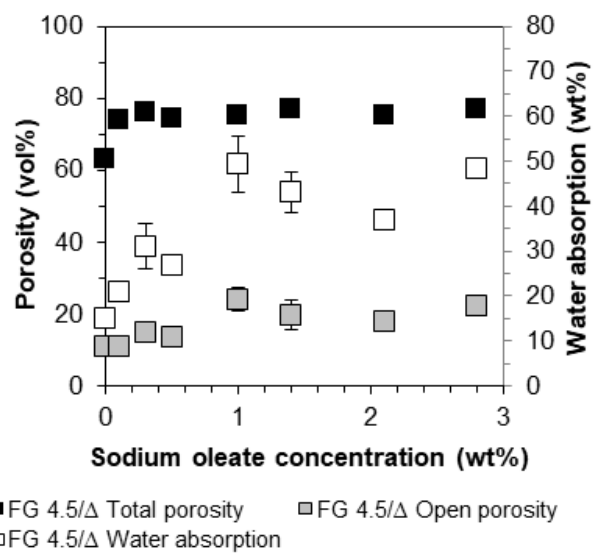


(b)

Figure 4. Compressive strength and bulk density results in stage II (a) and stage III (b) testing at 7 days of age.



(a)



(b)

Figure 5. Total, apparent porosity and water absorption results in stage II (a) and stage III (b) testing at 7 days of age.

caused lower thermal conductivity when applied surfactant (Figure 6a). Nevertheless, the MTPS method is moderately accurate in such a small thermal conductivity data range. It may be why there is no significant difference between samples prepared with varying amounts of  $H_2O_2$ . It can also be stated that a surfactant content higher than 0.5 wt% does not affect the insulating properties significantly (Figure 6b).

Large-size samples with the composition of FG 4.5/0.0 and FG 4.5/1.4 were also prepared, and their thermal conductivities were determined by the heat flow meter method at 7 days of age:

- FG3.4/4.5/0.0:  $\lambda = 0.159$  W/mK
- FG3.4/4.5/1.4:  $\lambda = 0.095$  W/mK

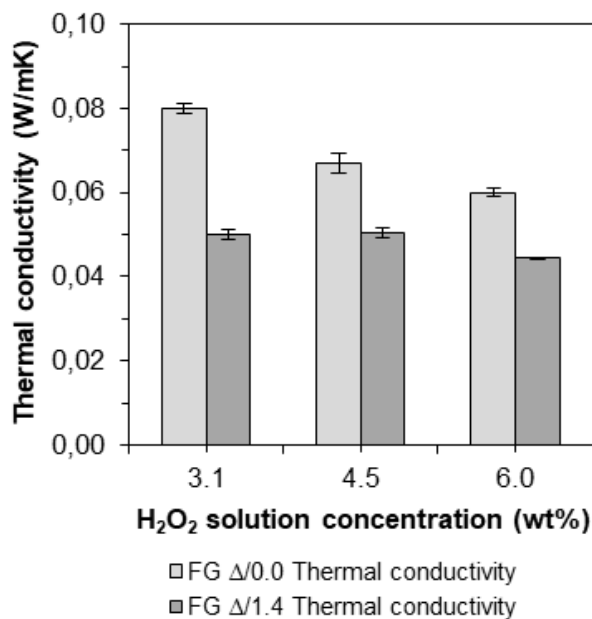
It can be established that the surfactant notably influenced the thermal conductivity of the foams, and samples can be prepared with lower than 0.1 W/mK thermal conductivity.

The application of a surfactant makes significant changes in the pore structure, even with the smallest amount of surfactant (0.1 wt% sodium oleate). It influences pore distribution, size, and thickness of pore walls. Namely, pore sizes decrease from the range of 500–1500  $\mu\text{m}$  to 150–450  $\mu\text{m}$ , the pore structure becomes more homogeneous (the standard deviation of pore size is around 40–60  $\mu\text{m}$ ), and the thickness of the pore walls also decreases to 25–40  $\mu\text{m}$  (Table 3). The small voids and homogeneous pore structure

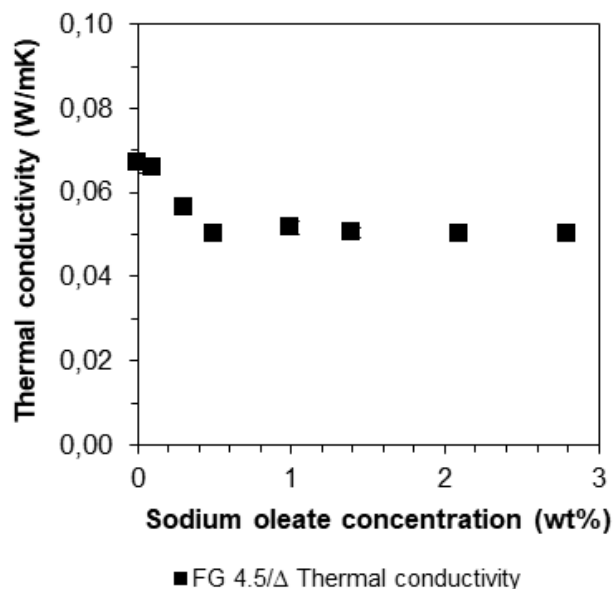
explain the lower bulk density, higher porosity (Figures 4a and 5a), and better insulating abilities (lower thermal conductivity, Figure 6a). Still, the thin pore walls weaken the structure, leading to lower compressive strength (Figure 4a) than using a foaming agent alone with the same concentration. Wide cracks can also be seen because of the increased shrinkage of the samples, but they can be avoided if the tops of the samples are removed shortly after the final set as described in the Sample preparation.

It is also an important observation that if the pore structure is homogeneous, then compressive strength and thermal conductivity are more dependent on the thickness of pore walls than on the diameter of cells (Figure 7).

Sodium oleate concentration has a limited impact on physical properties (compressive strength, thermal conductivity), which implies that the amount of foaming agent has a greater influence on cell size and cell wall thickness than has the surfactant content. Surfactant affects only the water absorption and thus the open porosity. Higher amounts of surfactant increase the chance of the formation of open cells. Therefore, the concentration of the surfactant needs to be carefully selected, depending on the application. For insulation, a sufficient quantity is required (in the case of this research,  $\sim 0.5$  wt%); for catalyst support applications, however, an even higher surfactant concentration is needed.



(a)



(b)

Figure 6. Thermal conductivity results in stage II (a) and stage III (b) testing at 7 days of age.



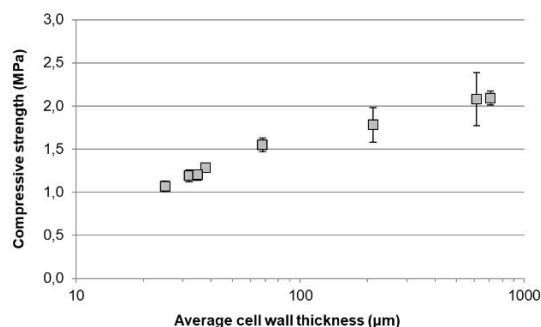


Figure 7. The relationship between compressive strength and average cell wall thickness for samples with a homogeneous pore size distribution.

## CONCLUSION

This paper aimed to investigate the effect of the foaming agent ( $\text{H}_2\text{O}_2$ ) and surfactant (sodium oleate) concentrations on the physical properties of foamed geopolymers. As a result, it can be concluded that foams with a customized pore structure can be produced.

Favorable insulating properties can be achieved by combining  $\text{H}_2\text{O}_2$  and sodium oleate: 75–80 vol% total porosity,  $< 500 \text{ kg/m}^3$  bulk density,  $> 1 \text{ MPa}$  compressive strength,  $0.095 \text{ W/mK}$  thermal conductivity (heat flow meter method).

Homogeneous cell structure can be achieved by using only  $\text{H}_2\text{O}_2$  up to the amount of 3 wt%. Above this threshold, the cell walls start to collapse due to the increased pressure by  $\text{O}_2$  formation, leading to a heterogeneous cell structure with a higher proportion of open cells.

A sodium oleate concentration of 0.5 wt% is sufficient for forming fine, homogeneous pores. Higher surfactant levels lead to a higher proportion of open cells. The surfactant plays a key role in producing satisfactory catalyst support with a high specific surface area.

Based on the experiments performed, it can be said that in the case of a homogeneous pore structure, cell wall thickness has a greater impact on the physical properties of geopolymers than the pore size itself.

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## Nomenclature

$\varnothing$	Sample diameter, mm
EN 196-1	Methods of testing cement - Part 1: Determination of strength
$m_1$	Weight of the dry sample, g
$m_2$	Weight of the sample after 2 hours of water absorption, g
min.	Minute
$P_A$	Apparent porosity, vol%
$P_T$	Total porosity, vol%
vol%	Volume %
$W_A$	Water absorption, wt%
wt%	Weight %
Greek	
$\Delta$	Varied parameter during the foaming process
$\lambda$	Thermal conductivity, W/mK
$\rho_b$	Bulk density, $\text{kg/m}^3$
$\rho_t$	True density, $\text{kg/m}^3$
Subscripts	
aq.	Aqueous solution
g	Gas
l	Liquid
s	Solid
Abbreviations	
CT	Computed tomography
EPS	Expanded polystyrene
FG	Foamed geopolymer
LOI	Loss on ignition
MTPS	Modified Transient Plane Source

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NAUČNI RAD

## PENASTI GEOPOLIMER SA PODEŠENOM STRUKTUROM PORA

*Zbog svojih povoljnih uslova proizvodnje i svojstava (npr. nisko skupljanje nakon formiranja pene, mehanička i hemijska stabilnost, otpornost na visoke temperature), geopolimerne pene su pogodne za toplotnu i zvučno izolacione vatrostalne građevinske materijale. Još jedno obećavajuće polje primene može biti njihova upotreba kao nosača katalizatora u obradi vode. Pene na bazi metakaolina pripremljene su postupkom direktnog formiranja pene sa visokom ukupnom poroznošću (> 75% v/v), malom zapreminskom gustinom (< 500 kg/m<sup>3</sup>), kompresivnom čvrstoćom od približno 1 MPa i niskom toplotnom provodljivošću (0,095 W/mK). Promenom koncentracije sredstva za formiranje pene (rastvor H<sub>2</sub>O<sub>2</sub>) i stabilizatora (natrijum oleat), moguće je proizvesti pene sa projektovanom poroznošću i distribucijom veličine pora. Pene sa uglavnom zatvorenim porama su pogodne za toplotnu izolaciju, dok one sa znatno otvorenim porama mogu koristiti kao nosači katalizatora. Snimci kompjuterizovane tomografije su pokazali da je koncentracija stabilizacionog agensa ključni parametar u formiranju homogene strukture pora i otvorenih pora; do 24% v/v otvorene poroznosti može se postići bez značajnog uticaja na druga svojstva. Na fizička svojstva pena podjednako utiču i debljina ćelijskih zidova i veličina samih ćelija.*

*Ključne reči: nosač katalizatora, geopolimer, toplotni izolator, mehanička svojstva, porozni materijal, toplotna provodljivost.*