



# Geopolymer fly ash composites modified with cotton fibre

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## ABSTRACT

**Purpose:** The work's primary goal is to assess the influence of the cotton fibres addition and their proportion on the strength properties and thermal conductivity of foamed geopolymer composites based on fly ash.

**Design/methodology/approach:** Fly ash from a thermal power plant was used as the foundation material to create the geopolymer composites in this study. Volcanic silica was used as an additional source of silicon. As an additive, the recycled cotton flock was used in amounts of 0.5%, 1% and 2% by weight of dry ingredients. The density, compressive, and three-point bending strength of the created geopolymers were measured. Moreover, the thermal conductivity measurements for three temperature ranges: 0–20°C, 20–40°C, and 30–50°C for all investigated geopolymers were conducted. The structure of tested materials was observed using a scanning electron microscope (SEM).

**Findings:** It was demonstrated within the context of the study that the addition of cotton fibres to foamed fly ash-based geopolymers aids in slightly reducing their density. Cotton fibres can be used to boost the strength of the examined geopolymers; for samples with 1% cotton fibres added, compressive strength rose by around 22% and flexural strength by about 67%. Additionally, it is feasible to lower their thermal conductivity coefficient by incorporating cotton fibres into foamed fly ash-based geopolymers.

**Practical implications:** The results obtained highlight the potential of fly ash-based geopolymer composites with the addition of cotton flocks for application as insulating materials in the building industry.

**Originality/value:** The novelty of this work is the demonstration of the possibility of producing foamed geopolymers based on fly ash with the addition of recycled cotton fibres, with properties that make them suitable for use as building insulation materials.

**Keywords:** Amorphous materials, Inorganic foam, Fly ash, Cotton flock, Geopolymer composite

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## PROPERTIES



## 1. Introduction

Recently, geopolymers have received much attention as a green substitute for Portland cement (OPC). Globally, there is a tendency toward reducing how damaging construction development is to the environment [1-4]. In order to attain an environmentally neutral level of CO<sub>2</sub> emissions, numerous studies are being done to minimize energy consumption and emissions of greenhouse gases. Currently, 10% of the world's [5,6] energy consumption, including 40% in the European Union [7,8], is used to produce cement and consume significant resources. In addition, the process of calcining raw materials involves significant emissions of CO<sub>2</sub>, as well as other environmentally harmful gases [9].

Geopolymers, which are amorphous, synthetic, inorganic alkali aluminosilicates, are formed by reacting aluminium silicates with an alkali hydroxide/alkali silicate solution [9-12]. Since geopolymers are based on minimally processed materials of natural origin or made from industrial by-products, this substantially reduces the carbon impact of its manufacturing. [10, 13-17]. It is estimated that during the production process of geopolymers, 2-3 times less energy is used, and 4-8 times less CO<sub>2</sub> is emitted than in the production of Portland cement [18]. Therefore, geopolymers are considered ideal environmentally friendly building materials that can replace energy-intensive and high-emission cement, resulting in reduced environmental impact. However, the extent of geopolymers and their environmental impact is not yet well understood. Most studies focusing on life cycle assessment (LCA) descriptions emphasize how Portland or blended cement affects the environment [19-22]. Studies of this type on geopolymers are still relatively few.

Nevertheless, those published point to the environmental benefits of geopolymers as an alternative to ordinary cement. For example, it was estimated that, compared to OPC, for metakaolin-based geopolymer concretes and those produced from a mining source, CO<sub>2</sub> emissions during their production were 27% to 45% and 9% lower, respectively [23, 24]. On the other hand, according to Zhang et al. [25], the manufacture of geopolymers can result in CO<sub>2</sub> emissions up to 80% lower than those of OPCs. It is also worth noting that in recent years, geopolymers have been produced with strength properties, environmental resistance, or thermal properties at a level comparable to or even better than concrete [26].

Given the limitations of geopolymers as a replacement for concrete shortly, alternative directions for their use are being sought. Such an alternative could be geopolymer foams, which could find wide applications in civil engineering, such

as thermal insulation, due to their thermal properties. Because typically used insulation materials like polystyrene and cotton wool, for instance, have very high carbon footprint values, geopolymers have a high potency [27].

Geopolymer foams are produced using various activators, fillers, and precursors [28]. In general, direct foaming techniques are used to create the majority of foamed geopolymers. These techniques include adding a foaming agent to a paste or suspension of geopolymer to create the foam. The resulting foamed and often highly porous structure must maintain its form during curing, so it is often necessary to use structure-stabilizing agents. These agents play an important role during the foaming process, enabling the formation and maintenance of the porous structure [29]. As foaming agents used in the direct method, chemical and physical agents can be used, where the phenomenon of thermal decomposition or chemical reactions are used, during which gaseous products are released, forming a porous structure [8,30]. Metal powders [31-33], like aluminium powder or silicon-containing powder, as well as the extremely well-liked hydrogen peroxide [34-36], are among the most often employed foaming agents.

The primary goal of the study was to assess how cotton fibre addition and percentage affected the strength and thermal conductivity of foamed fly ash-based geopolymer composites. The study's scope included measurements of the raw materials' particle size distribution, fly ash's X-ray phase analysis, density, compressive strength, and flexural strength, as well as measurements of the composite geopolymer materials' thermal conductivity coefficient. In addition, the study employed scanning electron microscopy (SEM) to observe the microstructure of the tested materials.

Previous studies have primarily focused on the environmental benefits of geopolymers as an alternative to ordinary cement; however, very few have investigated their potential as insulating materials. The novelty of this research lies in the demonstration that it is possible to produce foamed geopolymers based on fly ash by adding recycled cotton fibres. This results in unique properties that make them suitable for building eco-friendly insulation materials.

## 2. Materials and methodology of research

### 2.1. Materials

Fly ash from a thermal power plant in Skawina, Poland, was utilized as the foundation material to create the geopolymer composites (Fig. 1). The ash utilized in the study is a fine-grained substance with pozzolanic

characteristics (Fig. 2, Tab. 1), generated by precipitation from waste gases from the burning of hard coal in power boilers. A particle size distribution analyser from Anton Paar GmbH was used to measure the distribution of raw material particle sizes (Graz, Austria).

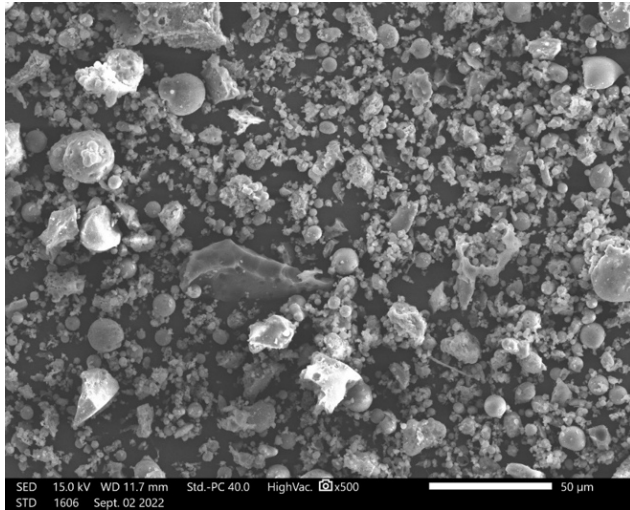


Fig. 1. Fly ash microphotographs taken using a SEM

Table 1. Particle size distribution of fly ash

	The mean value, $\mu\text{m}$
$D_{10}$	$2.011 \pm 0.065$
$D_{50}$	$10.722 \pm 0.216$
$D_{90}$	$24.474 \pm 0.786$
Mean size	$12.859 \pm 0.315$
Span	$2.085 \pm 0.029$

It is crucial to keep in mind that the fly ash particle size distribution might change based on a number of variables, including the coal’s composition, the combustion process, and the post-combustion treatment. Fly ash particles often come in a variety of sizes. Both fine and coarse particles are present, as shown by the bimodal or even multimodal distribution described in many investigations [37]. For instance, research by Sarkar et al. [38] that examined the particle size distribution of fly ash from several sources found that most of the particles were between 1 and 100  $\mu\text{m}$  in size. About 70% of the total mass comprised the fine fraction, which had particle sizes less than 10  $\mu\text{m}$ .

In comparison, the remaining 30% comprised the coarse fraction, which had particle diameters more than 10  $\mu\text{m}$ . These results show how the size distribution of fly ash particles is heterogeneous. The fly ash used in the current research had a spread of 2,085, which demonstrates that it has a very narrow particle size distribution. This feature draws attention to the uniformity of the particle sizes. Particle size study specifically shows that a significant majority of the particles, almost 90% ( $D_{90}$ ), had diameters less than 24.474  $\mu\text{m}$ . This result indicates that the bulk of the fly ash particles are rather small. A further measurement of 10.722  $\mu\text{m}$  for the median particle size ( $D_{50}$ ) shows that half of the particles have diameters lower than this value. Additionally, the research shows that a smaller portion of the particles, around 10% ( $D_{10}$ ), had diameters less than 2.011  $\mu\text{m}$ , representing the finer end of the particle size range. These findings demonstrate the regularity of the particle size distribution and provide important knowledge on the physical properties of the fly ash used in this investigation.

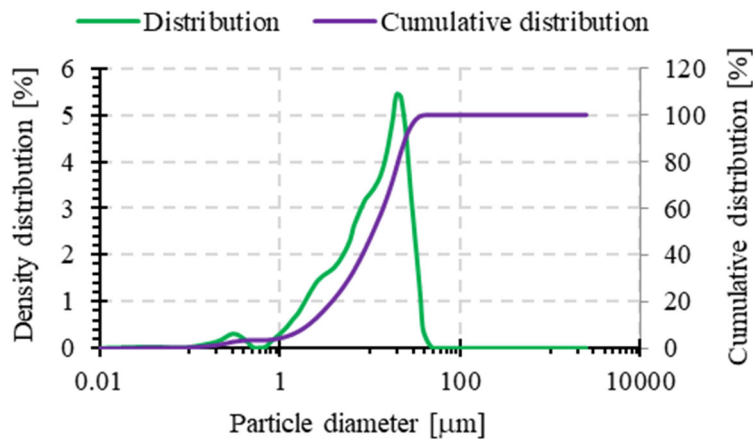


Fig. 2. The cumulative curve for fly ash and the particle size distribution

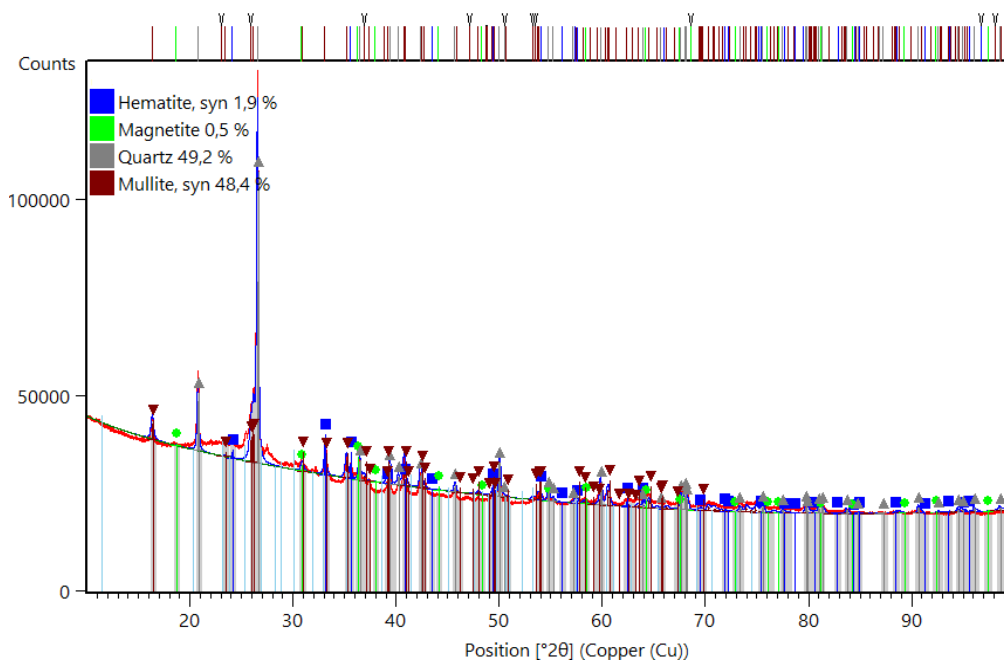


Fig. 3. Diffractogram for fly ash

Based on XRD phase analysis (Fig. 3), it was determined that the fly ash used for the geopolymer composites mainly consists of mullite ( $Al_6Si_2O_{13}$ ) and quartz ( $SiO_2$ ), with minor contributions from hematite ( $Fe_2O_3$ ) and magnetite ( $Fe_3O_4$ ). The study made use of a PANalytical Aeris X-ray diffractometer from Malvern PANalytical (Lelyweg 1, Almelo, The Netherlands), and Rietveld phase analysis was carried out using the PDF-4+ database from the International Center for Diffraction Data (ICDD).

During the production of geopolymers, T-180-type volcanic silica was used as an additional source of silicon (Fig. 4). The silica used, in the form of a light powder, was formed by mechanical and thermal processing at a temperature of about 950°C of natural volcanic rock. Silica is a chemically inert material, non-flammable, and harmless to humans. Figure 5 and Table 2 show the particle size distribution and cumulative curve for the silica used in the study.

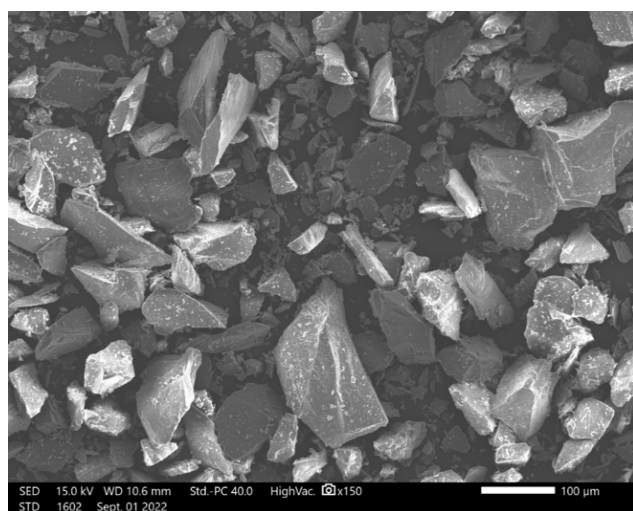


Fig. 4. SEM microphotography of volcanic silica particles

Table 2.

Particle size distribution of volcanic silica

	The mean value, μm
D <sub>10</sub>	7.137 ± 0.047
D <sub>50</sub>	20.828 ± 0.160
D <sub>90</sub>	44.253 ± 0.628
Mean size	24.814 ± 0.284
Span	1.782 ± 0.014

With a scattering ratio of 1.782, the particle size distribution of volcanic silica, which is analogous to fly ash, revealed a relatively restricted range of sizes. The fly ash, a common particulate material researched, displayed smaller particle sizes than the silica used in this study, which demonstrated greater particle sizes. The size distribution study showed that only 10% of the volcanic silica particles (D<sub>10</sub>) had a diameter lower than 7.137 μm, suggesting the



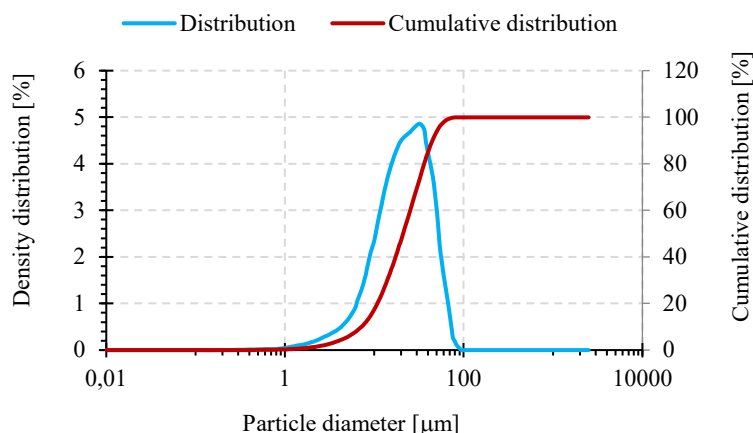


Fig. 5. The cumulative curve and particle size distribution for volcanic silica

existence of finer particles. This finding was based on the fact that there were other particles with diameters larger than 7.137  $\mu\text{m}$ . In contrast, the diameter of 50% of the particles (D50) was measured to be 20.828  $\mu\text{m}$ ; this value was used to reflect the sample's median particle size. In addition, a significant amount of 90% of the particles (D90) had a diameter smaller than 44.253  $\mu\text{m}$ , indicating the existence of coarser particles. The recycled cotton flock was used as an additive (Fig. 6), which was obtained by milling cotton fabric scraps and cotton jerseys. It is a biodegradable material with a low density of 0.22  $\text{g}/\text{cm}^3$ , an average fibre length of about 350  $\mu\text{m}$  (typically 150-500  $\mu\text{m}$ ), and a fibre width ranging from 10  $\mu\text{m}$  to 25  $\mu\text{m}$ . Cotton flock is used as a filler and reinforcing fibre in adhesives, epoxy resins, cosmetics, and other applications [39].

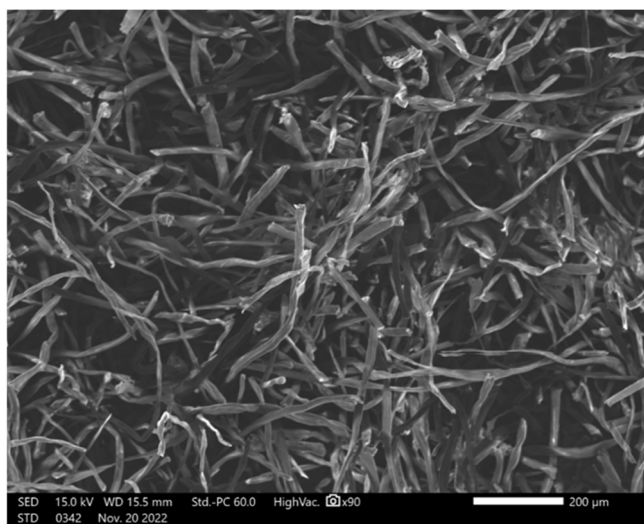


Fig. 6. SEM microphotography of cotton flock

## 2.2. Sample preparation

Properly selecting raw materials and activators significantly affects the properties of the produced geopolymers. The mechanism for forming geopolymers involves the formation of bonds through polycondensation, where the reaction occurs between a solid powder, which is the source of aluminosilicates, and an alkali hydroxide/silicate [13]. In the current investigation, dry component combinations were made in the first step per the chosen proportions depicted in Table 3 to create geopolymer composites.

First, an alkaline solution was prepared, which was later used to activate the material. The alkali utilized was a 10-mol solution of sodium hydroxide and a 1:2.5 mixture of an aqueous solution of sodium silicate R-145. The sodium hydroxide was in the form of flakes, which were dissolved in distilled water in appropriate, previously calculated proportions. To stabilize the temperature and concentration of the prepared solution, it was left for 24 hours under room conditions. The dry materials were then blended into a uniform mixture using a GEOLAB laboratory mixer (GEOLAB, Warsaw, Poland). After selecting the appropriate materials and activator needed for the geopolymer mortar, the next step was to combine them in the mixing process until a homogeneous paste was obtained. There were about 400 ml of activator per 1 kg of dry ingredients. To carry out the foaming process, after about 10 minutes of mixing, structure stabilizers in the form of gypsum and hydroxyethylcellulose (Glentham Life Sciences, UK) were first introduced into the geopolymer paste, followed by the addition of a foaming agent in the form of 35% hydrogen peroxide (Grupa Azoty, Pulawy, Poland) in the amount of 0.75%. To cure the samples, the

Table 3.

Percentage of components in the mixtures used to produce foamed geopolymers, along with designations

Sample ID	Fly ash	Silica	Gypsum	+	Hydroxyethyl cellulose	Cotton flock
	% by weight				% by weight (concerning dry ingredients)	
FA-0						0
FA-05						0.5
FA-1	80	10	10		0.5	1
FA-2						2

produced geopolymer paste was applied to the appropriate moulds and dried in an SLW 750 STD laboratory drier (POL-EKO-APARATURA, Wodzislaw Slaski, Poland). During 24 hours, the curing procedure was conducted at 75°C. After 28 days, the samples were evaluated.

### 2.3. Testing methods

A geometric approach based on measuring the mass and volume of the samples was used to analyse the density of the created geopolymers on a set of six samples. On specimens measuring 50 x 50 x 50 mm, all tested compositions were measured. Compressive and three-point bending strength measurements were performed per PN-EN 196-1:2016-07. For each of the compositions under test, six 50 x 50 x 50 mm compression specimens and four 50 x 50 x 200 mm bending specimens were created. A Matest 3000 kN testing device was used for the experiments (Matest, Treviolo, Italy). Standard deviations were calculated from the data and presented on graphs as error bars for both density measurements and compressive and flexural strengths. Plates with 20 x 200 x 200 mm measurements were produced to measure the heat conductivity coefficient. With a Netzsch HFM 446 plate apparatus (Netzsch, Wittelsbacherstrasse, Germany), thermal conductivity measurements were conducted for three temperature ranges: 0-20°C, 20-40°C, and 30-50°C. JEOL JSM-820 scanning electron microscope was used to observe the tested geopolymers (IXR Inc., Austin, TX, USA).

### 3. Results and discussion

Figure 7 displays the findings of the density measurements. The usage of cotton fibre addition affects the density of the tested foamed geopolymers, which may be inferred from the findings. The density values for the tested materials can be shown to decrease as the amount of cotton fibre in the combination increases. The reference sample had the highest density, 0.39 g/cm<sup>3</sup>, whereas the geopolymers with 2% cotton fibre added had the lowest density, 0.35 g/cm<sup>3</sup> (a drop of roughly 7.7% from the reference sample).

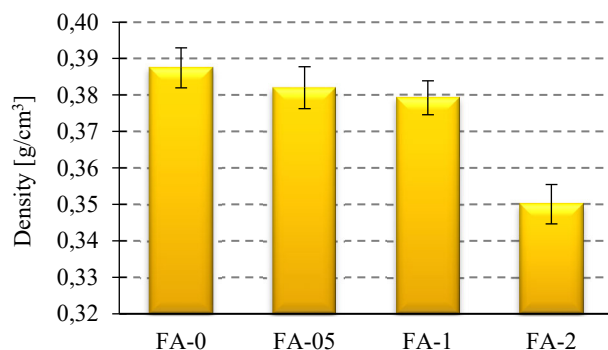


Fig. 7. The density of the tested geopolymer composites

The results of the compressive strength and flexural strength measurements are shown in Figures 8(a) and 8(b), respectively.

All the examined samples had compressive strength values of more than 1 MPa. In addition, it can be noted that the inclusion of cotton fibre caused an increase in the compressive strength of the tested geopolymers. The samples with 1% cotton fibre addition had the highest average compressive strength value, which was 1.31 MPa (approximately 22% higher than the reference sample). However, the obtained compressive strength value for the samples with 2% cotton fibre addition was nearly identical to that for the samples with 0.5% fibre addition and slightly lower compared to the samples with 1% fibre addition and amounted to 1.26 MPa. In comparison, this value was still higher compared to that obtained for the reference sample by about 17.6%. The introduction of cotton fibre enables the tested geopolymers' flexural strength to be increased; the highest flexural strength values were obtained for the samples with 1% cotton fibre addition and amounted to 0.75 MPa (about 67% more than the reference sample). This trend was also seen for compressive strength. The obtained average flexural strength value for the samples with 2% cotton fibre additive was 0.53 MPa, which was higher than the flexural strength of the reference sample by about 17.7%. It was similar to the value for the samples with 0.5% cotton additive and lower than the value for the samples with 1% fibre additive.

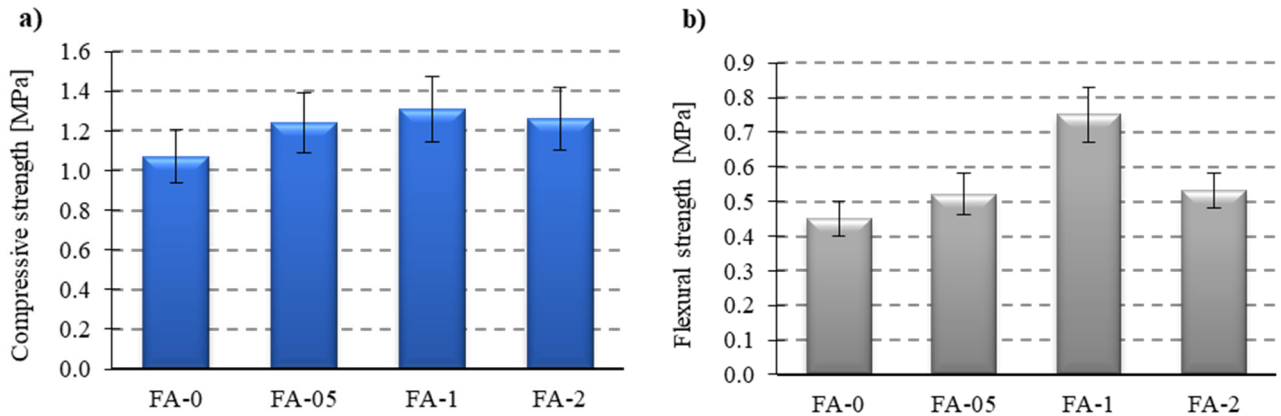


Fig. 8. Results for (a) compressive strength and (b) flexural strength of the geopolymer composites tested

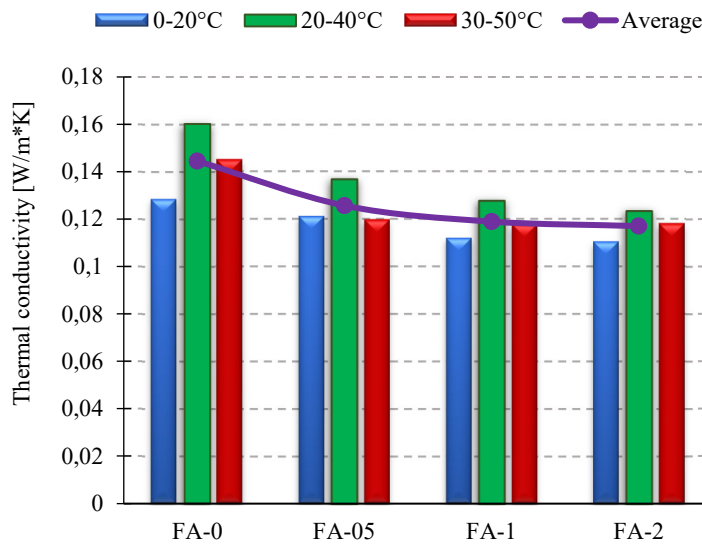


Fig. 9. Thermal conductivity in three ranges 0-20°C, 20-40°C, and 30-50°C for the tested geopolymer composites

Figure 9 displays the observed thermal conductivity values for the investigated geopolymers throughout three temperature ranges (0-20°C, 20-40°C, and 30-50°C).

The studied fly ash-based foamed geopolymers' thermal conductivity is partially reduced by the introduction of cotton fibres. When cotton fibres were added in amounts of 0.5%, 1%, and 2%, respectively, the average thermal conductivity coefficient was found to be 0.126 W/m²K, 0.119 W/m²K, and 0.117 W/m²K. These values represent decreases compared to the average thermal conductivity coefficient for the reference sample of about 13%, 17.6%, and 18.9%, respectively.

Figure 10 summarizes sample microphotographs showing the structure of the tested fly ash-based foamed geopolymers.

In the matrix of all samples, one can see tiny dissolved fly ash particles, unreacted spheroidal fly ash particles, and cotton fibres in the case of geopolymers with the flock. The investigated geopolymer composites' structures showed no discernible variations. Moreover, the porosity of every sample examined exhibited a consistent nature regardless of the quantity of cotton fibres used.

In summary, the results obtained in this paper are consistent with those reported in the literature. The thermal conductivity of foamed geopolymers is typically between 10% and 50% of that of ordinary concrete. Foamed geopolymers used for thermal applications typically have thermal conductivities that vary from 0.072 W/(m K) to 0.48 W/(m K), respectively, for densities of 300 kg/m³ to 1400 kg/m³ and

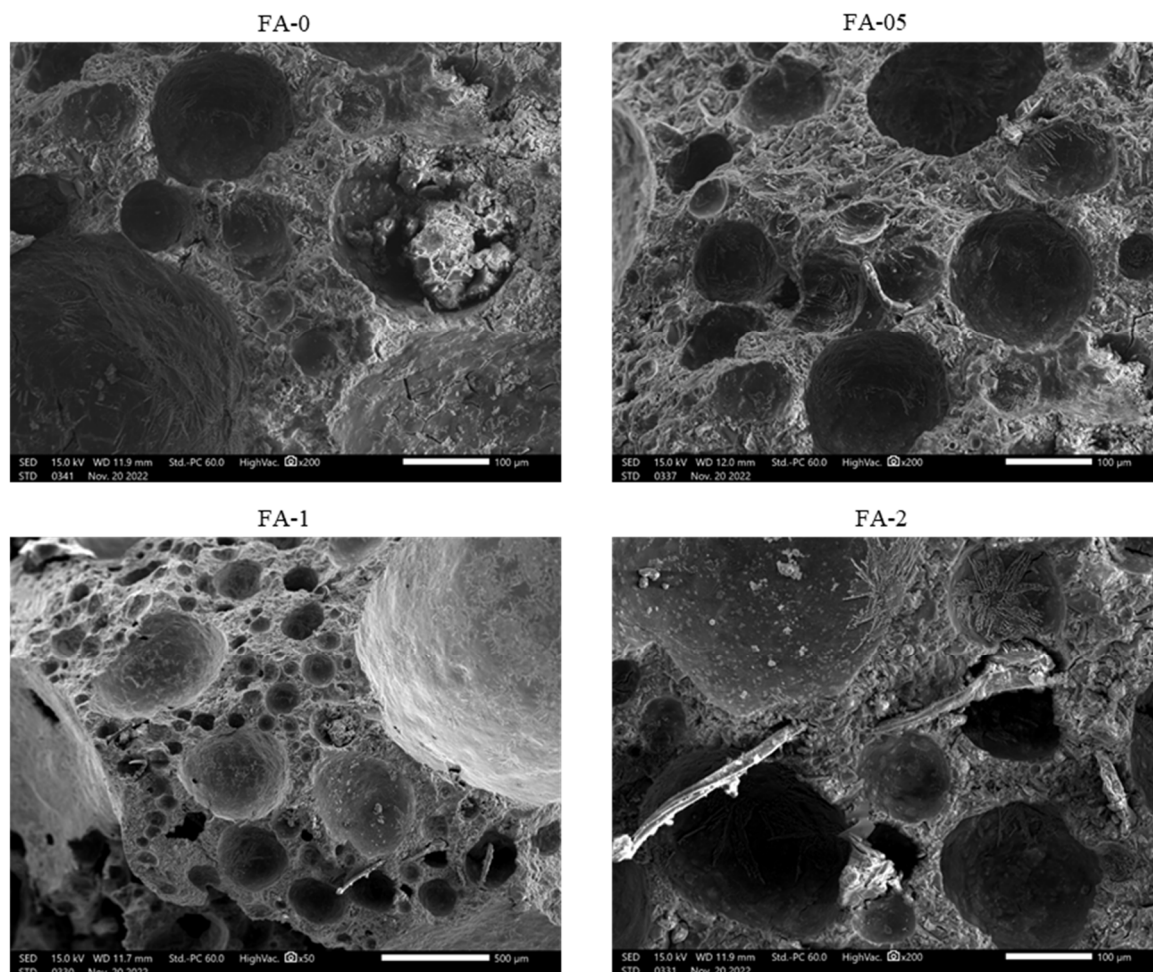


Fig. 10. SEM microstructure photographs of the tested geopolymer composites

compressive strengths of 0.7 MPa to 48 MPa [40-44]. Moreover, all of the produced foamed geopolymers had compressive strengths above 1 MPa, meaning they foam the JC/T2200-2013 standard that specifies the permissible compressive strengths of foam insulation boards made from ordinary Portland cement [40]. Also, the results reported in the literature for geopolymers reinforced with short fibres are consistent with the observed impact of the addition of cotton fibres on the strength of the tested geopolymers [45,46].

#### 4. Conclusions

The researchers focused on the density, strength, and thermal conductivity of foamed fly ash-based geopolymer composites, as well as the effect of adding cotton fibres as well as the amount of them. The following conclusions may be drawn from the results:

- I. The addition of cotton fibres to foamed fly ash-based geopolymers helps to lower their density.
- II. The evaluated geopolymers' strength qualities can be improved by including cotton fibres; for samples with 1% cotton fibres added, the compressive strength increased by around 22% and flexural strength by about 67%.
- III. Moreover, the addition of cotton fibres introduced into the foamed fly ash-based geopolymers makes it possible to reduce their thermal conductivity coefficient, which demonstrates their potential for use in the building sector as insulating materials.

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