Research Article

Van Su Le*, Michal M. Szczypinski, Pavlína Hájková, Vladimir Kovacic, Totka Bakalova, Lukas Volesky, Le Chi Hiep, and Petr Louda

Mechanical properties of geopolymer foam at high temperature

https://doi.org/10.1515/secm-2020-0013
Received Sep 11, 2019; accepted Jan 24, 2020

Abstract: In this work, geopolymer foam composites containing waste basalt fibre (10, 30, and 50%wt) were exposed to elevated temperatures of 200, 400, 600, 800 and 1000°C. With an increase in high temperature, the geopolymer foams material exhibits a decrease in compressive strength and bending strength. When heated above 600°C, geopolymer foams materials exhibit a significant reduction in mechanical properties. It shows clearly with the naked eye that surface cracks in case of samples containing 10% of basalt filler. However, when increasing fillers with basalt fibres up to 30% and 50%, the cracking of the sample surface is no longer visible to the naked eye. Especially when the temperature increases, the mechanical properties also increase without decreasing in the sample of 50% by weighing to the binder. The results show that reinforcing the geopolymer foams with basalt ground fibre improves the mechanical properties at high temperatures.

Keywords: aluminium, compressive strength, flexural strength, agent powder, basalt fibre

1 Introduction

Cement and their variants are widely used in the construction industry. By 2030, about 4.83 billion tons of cement will be produced globally [1]. Cement is the primary aggregate to produce concrete. One of the weaknesses of concrete is its low fire-resistance compared to some other construction materials, and it causes global warming to affect the production of one tonne of cement generates one tonne of carbon dioxide [2].

Geopolymer is a break-through material. The production of one tonne of kaolinite-based geopolymeric cement generates only 0.18 tons of CO_2, compared with 1 tonne of CO_2 for Portland cement (six times less) [3]. Fly ash-based geopolymeric cement has attracted intensive research word-wide because it emits even less CO_2, up to nine times less than Portland cement.

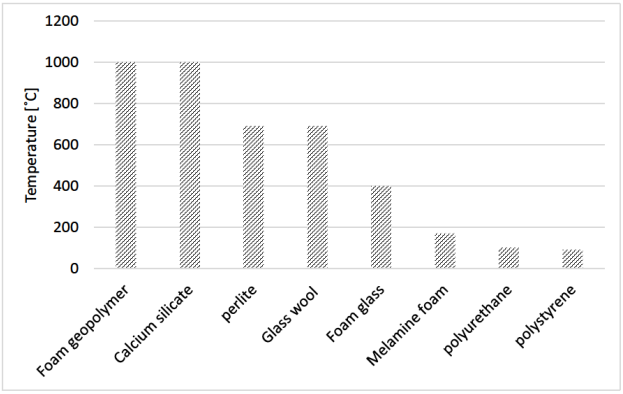


Figure 1: Maximum application temperature of some insulation materials [4].

Geopolymer materials have several advantages such as high durability, suitable fire-resistant and thermal stability, excellent mechanical properties, and resistance to acid attacks. Low-density geopolymers could be considered as potential materials for applications in many fields such as thermal insulation, fire resistance and other high-temperature applications (Figure 1). Due to its low thermal conductivity, geopolymer designed for fire resistance applications could be exposed to high temperature for an extended period.

The construction made of concrete usually has a large net mass [5]. The use of geopolymers of lower density is beneficial in term of reduced structural load-bearing with

***Corresponding Author: Van Su Le:** Faculty of Mechanical Engineering, Department of Material Science, Technical University of Liberec, Studentská 2, 461 17 Liberec, Czech Republic; Email: longsuvp90@gmail.com
Michal M. Szczypinski, Vladimir Kovacic, Totka Bakalova, Lukas Volesky, Le Chi Hiep, Petr Louda: Faculty of Mechanical Engineering, Department of Material Science, Technical University of Liberec, Studentská 2, 461 17 Liberec, Czech Republic
Pavlına Hájková: Faculty of Mechanical Engineering, Department of Material Science, Technical University of Liberec, Studentská 2, 461 17 Liberec, Czech Republic; Unipetrol Centre for Research and Education, Revoluční 84, 400 01 Ústí nad Labem, Czech Republic

further benefits of acoustic and thermal insulation [6, 7]. Different foaming agents can be used to synthesise low-density geopolymers. Metallic aluminium powder is commonly used and is very reactive in alkaline environments and react at room temperatures [7–9].

Fibre reinforcement has been used in various hardened binders to improve mechanical properties [10–15]. Reinforced geopolymer adds either steel fibres, glass fibres or carbon fibres or basalt fibres to carry mechanical properties and for high temperature resistant geopolymer composites [16–19].

This study was conducted to study the physical and mechanical properties of geopolymers with enhanced fillers. Evaluating these properties is essential for better material use purposes.

2 Materials and Methods

2.1 Used materials

During the experiments, the following raw materials were used: binder, grounded basalt fibre and foaming agent. The used binder material was *Baucis Lk*, supplied by *České Lupkové Závody* (Czech Republic). This is an inorganic two-component aluminosilicate material based on metakaolin and potassium alkaline silicate solution [20]. Grounded basalt fibre was used in the experimental work as reinforcement material. To obtain grounded fibres, the stone wool *Isover N* (manufactured by *Saint-Gobain Construction Product CZ a.s.*) was milled (see Figure 2).

The preparation of geopolymer coatings was carried out using aluminium powder (foaming agent). This powder produces bubbles in the material volume, which increases porosity and reduces the weight of the geopolymer coating. The chemical composition and particle size of the aluminium powder are shown in Table 1.

Table 1: The chemical composition and size of the aluminium powder.

Name	Diameter	Al	FeO	SiO	Cu
D50	65µm	98%	0.35%	0.4%	0.02%

Figure 2: Photograph of basalt fibre grounded using the mill.

2.2 Geopolymer synthesis

Geopolymers were synthesized using cement and the activator. The materials were homogenized with a stirrer for 5 minutes. After homogenization, the filler (basalt fibre) and aggregate (fine sand) were added to the mixture, and they were stirred for a further 5 minutes to full homogenization. Aluminium powder was added to the geopolymer slurry after 10 minutes of mixing period, and they were mixed for a further 30 s at high RPM. Immediately after mixing, samples were poured into test moulds. The synthesis of geopolymers was carried out according to Figure 3.

Afterwards, the polymer paste was poured into moulds of dimensions 40×40×160 mm (see Figure 4). In 2 to 8 minutes, geopolymer began to expand (pores formation) and finished after 20 to 30 minutes. Samples were cured at room temperature for 1 to 2 hours, and then the test specimens cut by hand-saw.

In this work, three samples of geopolymer foams with different weight percentages of basalt waste fibre (Table 2) were prepared and tested. All samples were made modules on 40×40×160 mm. They were cured after 28 days at room temperature. They were heated in a furnace at various temperatures of 200, 400, 600, 800 and 1000°C. The

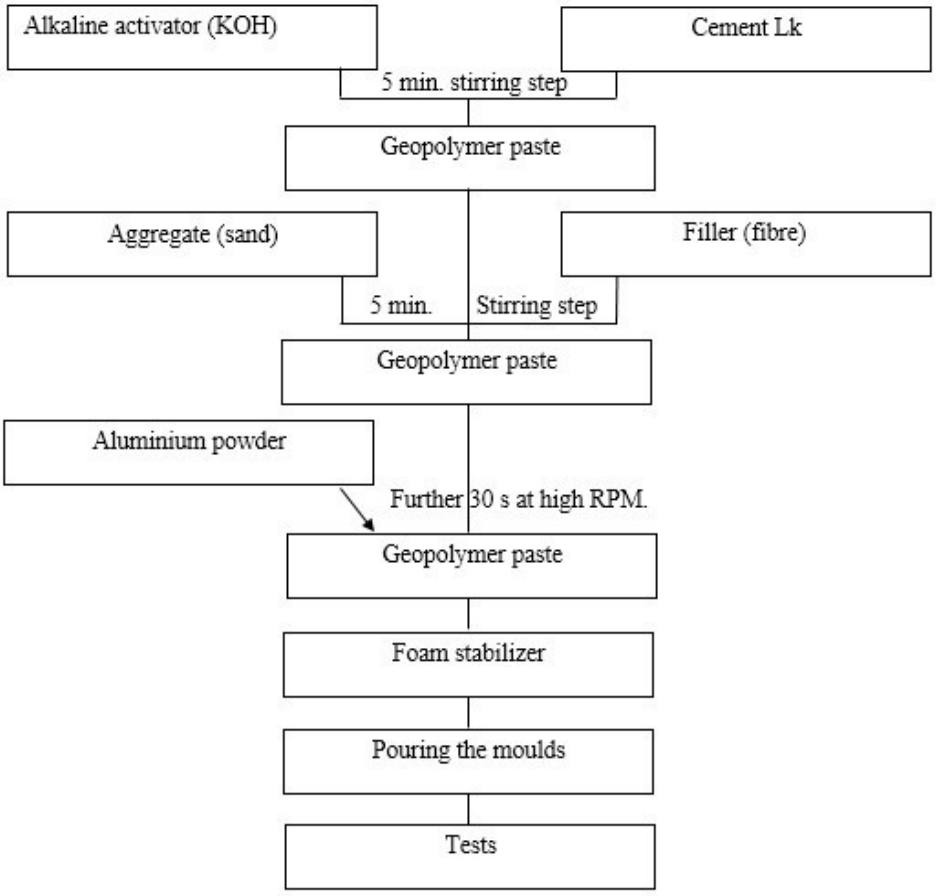


Figure 3: Schematic flowchart of geopolymer foams processing.

Table 2: Composition of geopolymer foams.

Mix ratio by weight			
Binder	Activator	Grounded basalt fibre	Aluminium powder
1	0.9	0.1	0.015
1	0.9	0.3	0.015
1	0.9	0.5	0.015

heating rate of the furnace was set to 5°C per minute until it reached the desired temperature, and the final temperature was kept for two hours. After they were holding them in the furnace until they lowered room temperature and then take to test.

2.3 Test procedures

The evaluation of the samples was carried out using mechanical tests, and subsequently, the structure of the tested samples was analysed. Drying of samples was done in a designated room at room temperature or an elevated temperature until the test requirements were met. The final result value was determined as the average of three measurements.

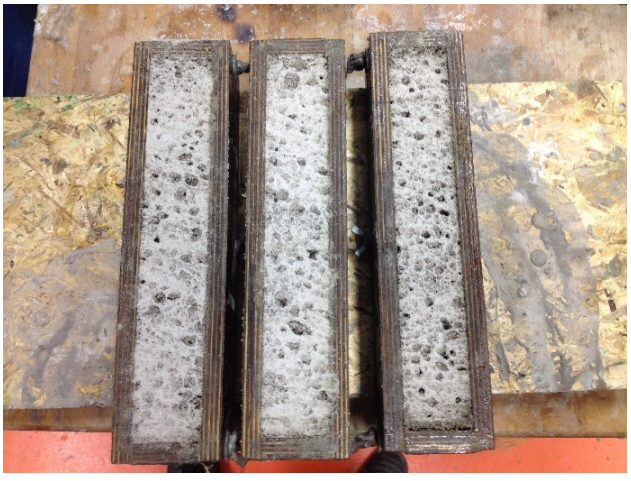
2.3.1 Apparent density

Apparent density was calculated with the following equation:

$$Apparent\ density = \frac{Mass}{Volume} \quad (1)$$

Where:
Apparent density (Kg/m^3);
Mass is the mass of the specimen (Kg);
Volume is the volume of the specimen (m^3);

(a)



(b)

(c)

Figure 4: Prepared samples: a) in moulds of size 40×40×160 mm, b) after cut by hand-saw c) samples removed from moulds.

2.3.2 Flexural strength and compressive strength

The strength was evaluated by a hydraulic press, universal Testing Mechanical INSTRON Model 4202 (Figure 5). Flexural strength was calculated from a three-point bending test on the samples of size 40×40×160 mm [21, 22]. Three cubes of 40 mm were cut from the test bar, and they were used for compressive strength testing. Continuously tested three samples were performed after 28 days.

The compressive strength of geopolymer foams (f_{cm}) was calculated by the equation:

$$f_{cm} = \frac{F_{\max}}{A_c} \tag{2}$$

Where:
f_{cm} is compressive strength (MPa);
$F_{\max}$ is the maximum applied load indicated by the testing machine (N);
A_c is the original cross-sectional area of a specimen in a compression test (mm^2);

The flexural strength (R_{mo}) was calculated by the equation:

$$R_{mo} = \frac{3F_{\max}L}{2bh^2} \tag{3}$$

Where:
R_{mo} is the flexural strength (MPa);
$F_{\max}$ is the maximum applied load indicated by the machine (N);
b is the average width of the specimen (mm);
h is the average depth of the specimen (mm);
L is span length (mm);

2.3.3 Weight loss

Weight loss was calculated using the equation:

$$W_L = \frac{W_0 - W}{W_0} * 100 \tag{4}$$

Where:
W_L is the weight loss (%);
W_0 is the initial mass (g);
W is the remaining mass at any given time(g);

2.3.4 Dry shrinkage

Dry shrinkage was calculated using the following equation:

$$S_L = \frac{L_0 - L}{L_0} \times 100 \tag{5}$$

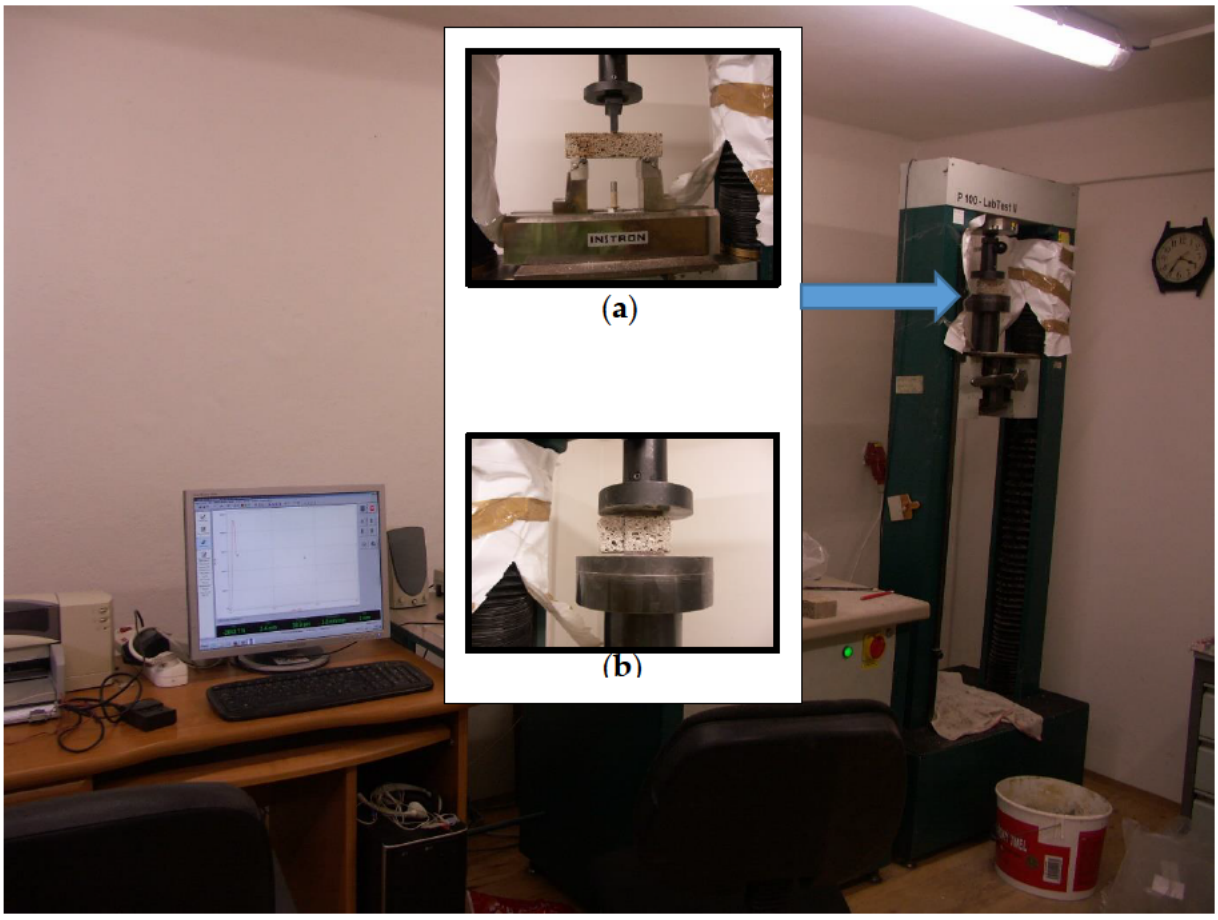


Figure 5: Universal testing machine INSTRON (Model 4202): a) test set-up for flexural strength b) test set-up for compressive strength.

Where:
S_L is the dry shrinkage (%);
L_0 is the length of the specimen (mm);
L is the remaining length of the specimen at any given time (mm);

2.3.5 Water Absorption

Water absorption is used to measure the permeability of geopolymer foams. The test made on the specimen (40×40×80) mm. All the samples were kept in room temperature for 28 days before they were tested. Each result was calculated from the average of three samples. According to ASTM C642 06 regulation, the samples were cured in an oven at a temperature of 100 to 110°C for not less than 24 h and determine the mass A. The samples were soaked in water for interval 24h. Surface-dry the sample by removing surface moisture with a towel, and determine the mass B.

Water absorption was calculated using the following equation:

$$\% = \left[\frac{B - A}{A}\right] \times 100 \tag{6}$$

Where:
A is the mass of the dry sample (g);
B is the mass of the wet sample (g);

3 Results and discussion

As part of this work, research was carried out that is relevant to building materials and those that have fireproofing properties. The physical characteristics of the material at room temperature and subjected to elevated temperatures were characterized. The density, water absorption, pore size, compressive and flexural strength were tested, and the weight loss and shrinkage of the materials after heating were examined. The building material that is to constitute a fire barrier must be characterized by the smallest

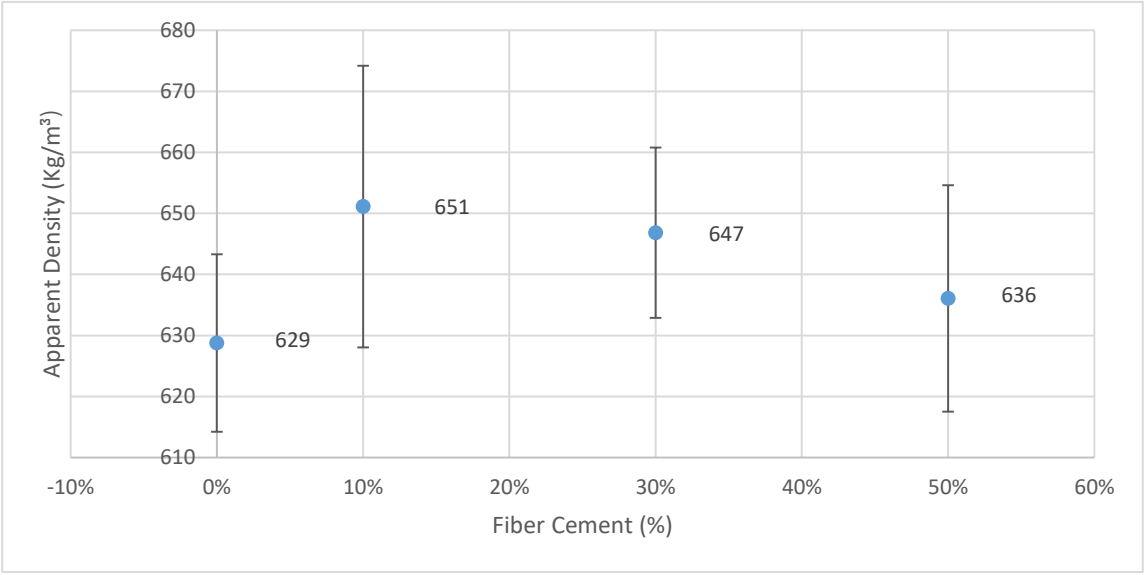


Figure 6: The density of geopolymer foams with increasing basalt waste fibre concentration at room temperature.

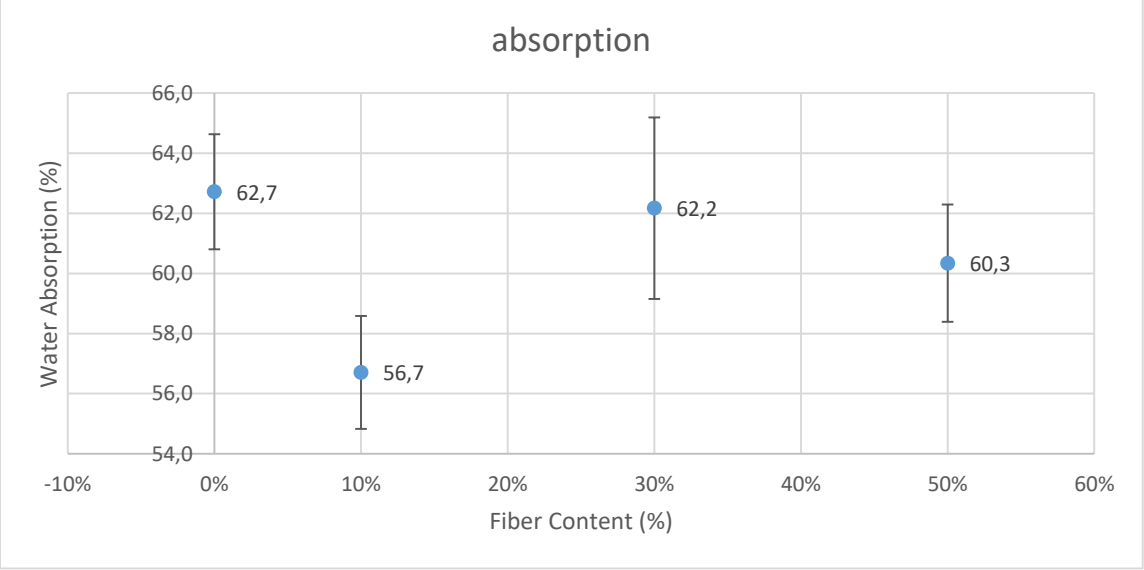


Figure 7: The absorption coefficient of geopolymer foams with increasing basalt waste fibre concentration.

possible change in physical parameters - otherwise a building disaster may occur during a fire. Compressive and flexural stregth testing will give an answer as to whether the material will continue to withstand when exposed to high temperatures.

3.1 Characterization of materials in room temperature

The addition of fibres contributes to the increase in the geopolymer foam density. However, it is not a significant increase (Figure 6). Furthermore, the addition of the fibre leads to reduced water absorption (Figure 7).

The fibre content also affects the pore size (Figure 8). The smallest pores occur in samples with a 50% fibre content (Figure 8c), the largest in samples with a content of 10% (Figure 8a).

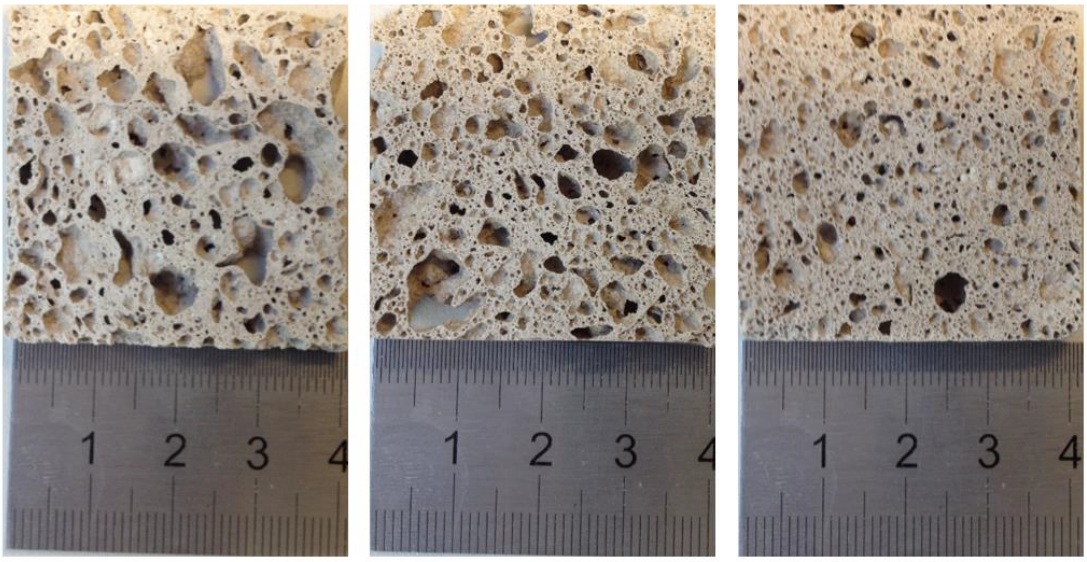


Figure 8: Pores size of geopolymer foams with various content of reinforcing fibre a) 10%, b) 30%, c) 50 % (total image width: 40 mm).

3.2 Characterization of material subjected to elevated temperature

All samples exhibit a colour change after heating from 200°C to 1000°C as follow: grey at 200°C, blackish-grey from 400°C to 800°C, yellowish-grey at 1000°C (Figure 9). There are visible cracks on samples containing 10% which were heated up to 600, 800 and 1000°C (Figure 9a). Samples reinforced with a larger amount of basalt fibre tend to crack less (Figure 9b and 9c). It is clearly seen that the fibers contribute to a change in the transfer of stress associated with sintering the material and thus inhibit the propagation of cracks from the places where these stresses arise.

The temperature of the material has a significant influence on the absorption coefficient. It decreases rapidly at 200°C, 400°C, and 600°C. However, at 600°C, 800°C, 1000°C, it doesn't decrease, and it's stable (Figure 10). This is particularly evident in the case of a sample with 50% fibre content, where coefficient doesn't decrease at range from 400°C to 1000°C. A similar situation occurs in the case of weight loss of tested samples at different temperatures (Figure 11). Weight loss increases in range from 200°C to 600°C. A further increase in temperature does not result in weight loss. The weight loss of unreinforced and the low fiber content samples is associated with the evaporation of water and gases in the pores, which are much larger than those of samples reinforced with more fibers. For the same reason, there is a significant change in the water absorption of samples with low (or no) fiber content.

Temperature also has a significant impact on the density (Figure 13) and shrinkage (Figure 12) of geopolymer composites – both increase with the temperature. The fastest growth is visible at high temperatures, above 400°C. In the case of density changes, a slight decrease was observed in all samples at a temperature of 400°C. The highest density changes at increasing temperature were observed in samples with less (10% and 30%) fibre content. Shrinkage is associated with the chemical nature of the material from which geopolymers are made. At high temperature there is a transformation from an amorphous to a crystalline phase. This happens following the crystallization of amorphous sodium aluminosilicates into nephelin.

Figures 14 and 15 show changes in strength (compression and flexural, respectively) depending on the temperature. The increasing dependence of strength on the temperature in the range from 400°C to 1000°C was observed. However, samples with a temperature of 200°C than 400°C were characterized by much higher strength. This is particularly evident in the case of samples with a lower fibre content (10% and 30%). Samples with 50% fibre content are characterized by the highest strength at temperatures above 400°C. At 1000°C, a sudden increase in strength in the sample with a 50% fibre content was observed (at 1000°C the compressive strength is 83.61% higher than compressive strength at 200°C). Samples reinforced with a higher fiber content are characterized by higher bending strength due to the smaller number of pores, which dramatically affect the strength of the material, increasing its fragility. In addition, the crystallization of aluminosilicate mentioned above occurs at a high temperature, which further promotes mechanical strength.

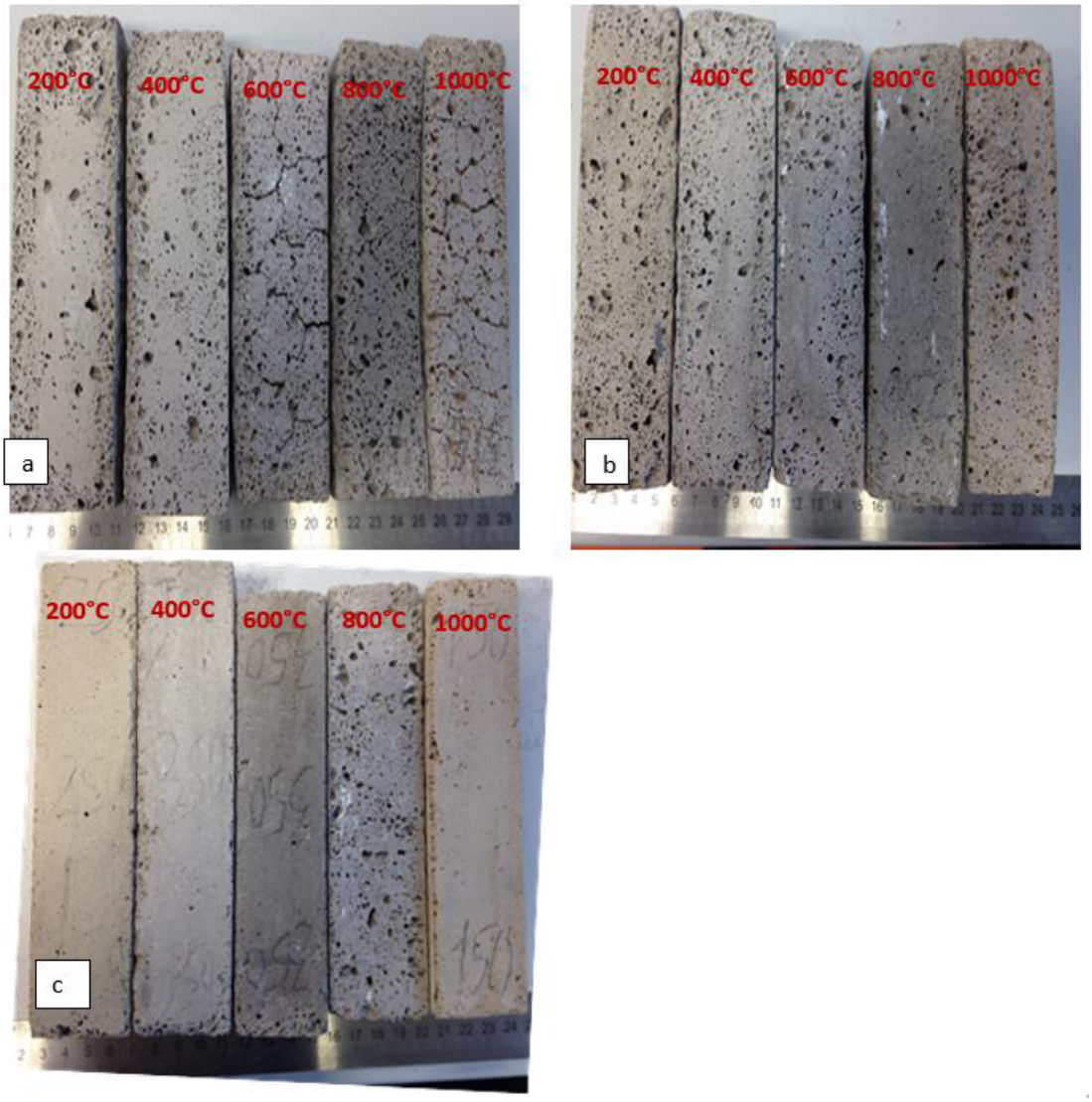


Figure 9: Colour change and cracks occurrence of geopolymer foams with different content of basalt waste fibre a) 10%, b) 30% and c) 50% after heating at various temperatures.

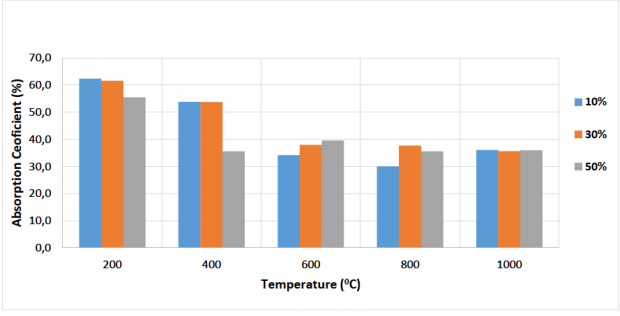


Figure 10: The absorption coefficient of geopolymer foams with different content of basalt waste fibre at high temperature.

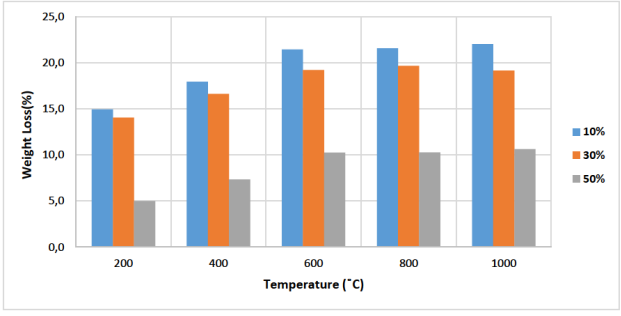


Figure 11: Weight loss of geopolymer foams with different content of basalt waste fibre at high temperature.

4 Conclusions

The results showed that the reinforcement of geopolymers with different content of basalt fibres influences the me-

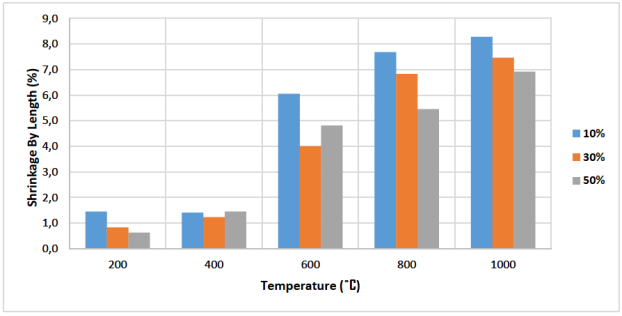


Figure 12: Shrinkage by the length of geopolymer foams with different content of basalt waste fibre at high temperature.

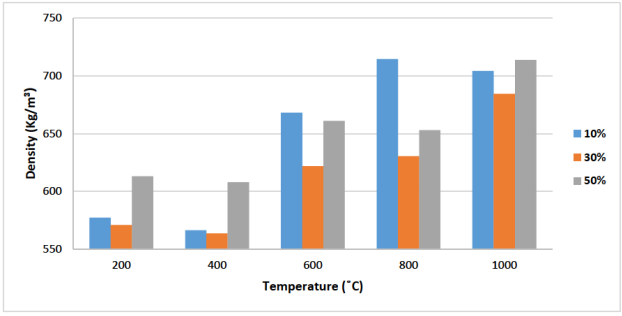


Figure 13: The density of geopolymer foams with different content of basalt waste fibre at high temperature.

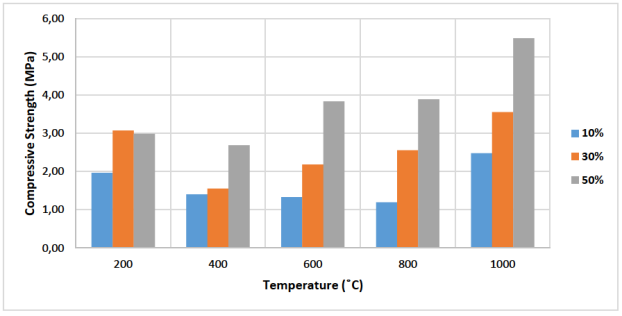


Figure 14: Change of compressive strength of geopolymer foams vs. heating temperatures.

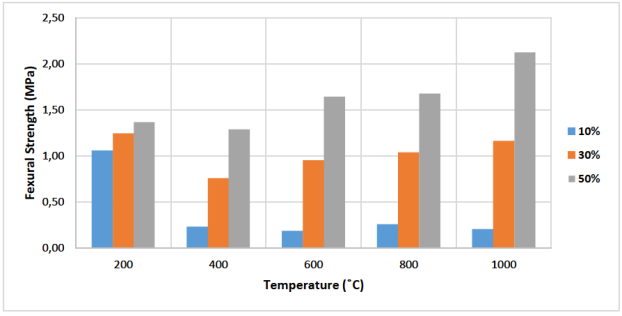


Figure 15: Evolution of Flexural strength of geopolymer foams vs. heating temperatures.

chanical properties of the obtained composites. The high fibre content improves practically all of the tested parameters, making the material also stable at very high temperatures. Through the reinforcement of the geopolymer composite with the right amount of basalt fibres, it's possible to obtain heat-resistant material. The research showed the influence of the content of introduced fibres in the formation of pores, crack propagation and in the formation of structural changes of the material, which ultimately result in its mechanical properties.

The obtained results are promising and lead us to further research towards the development of fireproof composite materials based on geopolymers.

Acknowledgement: The results of the project "Application of geopolymer composites as fire, AGK", registration number VI20172019055, were obtained through the financial support of the Ministry of the Interior in the programme "The Safety Research of the Czech Republic, 2015-2020 (BV III/1-VS)".

References

[1] (2018). *Cement: global production 1990-2030*. Available: https://www.statista.com/statistics/373845/global-cement-production-forecast/

[2] Davidovits J. Global warming impact on the cement and aggregates industries. World Resour Rev. 1994;6:263–78.

[3] Davidovits J. "Geopolymer chemistry and applications," in *Geopolymer Chemistry & Applications*, ed: Geopolymer Institute, 2008.

[4] Liefke E. Industrial applications of foamed inorganic polymers. Geopolymere. 1999;99:189–200.

[5] Just A, Middendorf B. Microstructure of high-strength foam concrete. Mater Charact. 2009;60(7):741–8.

[6] Feng J, Zhang R, Gong L, Li Y, Cao W, Cheng X. "Development of porous fly ash-based geopolymer with low thermal conductivity," *Materials & Design (1980-2015)*, vol. 65, pp. 529-533, 2015.

[7] Chiu YP, Lu YM, Shiau YC. "Applying inorganic geopolymers added with aluminium powder to fire-resistant fillers," *Materials Research Innovations*, vol. 19, pp. S5-642-S5-649, 2015.

[8] Caijun Shi B. "Composition of materials for use in cellular lightweight concrete and methods thereof," ed: advanced materials technologies, 2002.

[9] Škvára F. Ros tislav Šulc, Zdeněk Tišler, Petr Skřičík, Vít Šmilauer, and Z. Z. Cílová, "Preparation and properties of fly ash-based geopolymer foams," vol. 58, pp. 188-197, 2014.

[10] Almeida JH, Angrizani CC, Botelho EC, Amico SC. "Effect of fiber orientation on the shear behavior of glass fiber/epoxy composites," *Materials & Design (1980-2015)*, vol. 65, pp. 789-795, 2015.

[11] Alomayri T, Low IM. Synthesis and characterization of mechanical properties in cotton fiber-reinforced geopolymer composites. Journal of Asian Ceramic Societies. 2013;1(1):30–4.

[12] Alomayri T, Shaikh FU, Low IM. Mechanical and thermal properties of ambient cured cotton fabric-reinforced fly ash-based geopolymer composites. Ceram Int. 2014;40(9):14019–28.

[13] Alomayri T, Vickers L, Shaikh FU, Low IM. Mechanical properties of cotton fabric reinforced geopolymer composites at 200–1000°C. Journal of Advanced Ceramics. 2014;3(3):184–93.

[14] V. Sathish Kumar, Blessen Skariah Thomas, and Alex Christopher., "An Experimental Study on the Properties of Glass Fibre Reinforced Geopolymer Concrete," 2012.

[15] Hung TD, Louda P, Kroisová D, Bortnovsky O, Xiem NT. "New generation of geopolymer composite for fire-resistance," in *Advances in Composite Materials-Analysis of Natural and Man-Made Materials*, ed: InTech, 2011.

[16] Hung TD, et al. New generation of geopolymer composite for fire-resistance. Advances in Composite Materials-Analysis of Natural and Man-Made Materials. InTech; 2011.

[17] Masi G, Rickard WD, Bignozzi MC, van Riessen A. The effect of organic and inorganic fibres on the mechanical and thermal properties of aluminate activated geopolymers. Compos, Part B Eng. 2015;76:218–28.

[18] Timakul P, Rattanaprasit W, Aungkavattana P. Improving compressive strength of fly ash-based geopolymer composites by basalt fibers addition. Ceram Int. 2016;42(5):6288–95.

[19] D. Tran, P. Louda, O. Bortnovsky, and P. Bezucha, "Mechanical Properties of Silica-Based Geopolymer Composites Cured at Ambient Conditions in Accordance with Size-Independent Method," 2010.

[20] AS. České lupkové závody. (2019). *BAUCIS LK*. Available: http://www.cluz.cz/en/baucis-lk

[21] "ASTM C348 - 08, Standard Test Method for Flexural Strength of Hydraulic-Cement Mortars.," in *ASTM C348 - 08: Standard Test Method for Flexural Strength of Hydraulic-Cement Mortars.*, ed, 2008, pp. 1-6.

[22] "ASTM C78/C78M - 10, Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)." in *ASTM C78/C78M - 10: Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)*. ed, 2010, pp. 1-4.