Evaluation of Mechanical Properties of Composite Geopolymer Blocks Reinforced with Basalt Fibres

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Geopolymers are modern, amorphous inorganic aluminosilicate polymers with specific composition and properties. However, to use a geopolymer as an engineering material, its mechanical properties need to be improved. As part of this work, a composite material was obtained - the matrix is a geopolymer, and the reinforcement is basalt fibers. Research on the reinforcement of geopolymer material with layers of basalt fabrics was carried out in order to verify the improvement of mechanical properties in relation to non-reinforced material. Static and dynamic mechanical tests for the evaluation of flexural, compressive, splitting tensile and impact strength were carried out. In case of flexural strength the increase was even 150 % and in the case of impact strength over 60 %. The developed basel fabric reinforcement had a significant impact on the mechanical properties of the tested geopolymer composite material.

Keywords: Geopolymer, Basalt fibers, Composite, Mechanical strength, Charpy impact test

1 Introduction

Geopolymers are modern, amorphous inorganic aluminosilicate polymers with specific composition and properties. Geopolymers are usually hard, mechanically resistant solids resembling a natural stone or concrete. Currently, geopolymeric materials, thanks to their unique properties, are increasingly used in the economy. It is anticipated that over the next few years geopolymers will completely replace traditional concretes based on Portland cement in advanced technologies [1, 2 and 3]. Geopolymers are used as fire insulation, carrier material, energy-saving ceramic tiles, self-hardening masses in foundries, fire protection composites. In addition, they are characterized by high compressive and bending strength, very high acid resistance and resistance to chlorides and sulphates. They are resistant to weather conditions at extreme temperatures (both high and low). Another very important advantage is the possibility of reinforcing geopolymers with steel. The geopolymer has a high degree of adhesion to steel and no corrosion occurs of the steel reinforcement. However, the most important is that the synthesis of geopolymers absorbs 2-3 times less energy than the synthesis of Portland cement and causes the emission of 4-8 times less carbon dioxide [2, 4 and 6].

Generally, the geopolymer synthesis method is reduced to one process: the disintegrated dried pozzolanic material (metakaolin or flyash) is mixed with an aqueous solution of the appropriate silicate (e.g. sodium or potassium silicate) with the addition of a strong base (concentrated sodium or potassium hydroxide). The resulting paste behaves in a similar way to cement slurry: it solidifies to a hard mass within a few hours. This process can be completely regulated, and the set time can vary from a few seconds to several dozen hours. Geopolymers consist of long chains - copolymers of oxides of silicon and aluminum and metal cations stabilizing them, most commonly sodium, potassium, lithium or calcium and bound water. The structure of geopolymers is similar to cage construction, as in the case of zeolites, and the main difference lies in the lack of long-range order. Depending on the short-range ordering, three basic structural units are distinguished: polysialate (Si-O-Al-O), poly(sialate-disiloxo) (Si-O-Al-O-Si-O) and poly(sialate-disiloxo) (Si-O-Al-O-Si-O). The amorphous geopolymer is described by the formula [2, 5, 7]:

\[ M_n\left(\text{SiO}_2\right)_z\text{AlO}_2^{-}wH_2O \]  \( (1) \)

where:

- \( n \) – degree of polycondensation;
- \( w \) – moles of bound water;
- \( z = 1, 2, 3 \);
- \( M = \text{Na}^+, \text{K}^+ \) cation.

However, to use a geopolymer as an engineering material, its mechanical properties need to be improved. As part of this work, a composite material was obtained - the matrix is a geopolymer, and the reinforcement is basalt fibers. The main purpose of using such reinforcement is to give the composite materials high strength parameters [8–12]. The increase of these parameters is approximately the higher the higher the fiber strength properties. In order to ensure proper functioning of the composite, good matrix adhesion to the reinforcement surface (adhesion) is necessary, which ensures proper transfer of the load to reinforcement in the form of fibers. However, the problem of matrix and reinforcement adhesion is very complex. Composites with brittle, e.g. ceramic, matrix should be characterized by moderate adhesion of individual com-

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ponents, because too strong their connection may increase the brittleness of the ceramic matrix, preventing the cracking of cracks propagating fibers. Strong mutual bonding of the components is desirable in composites with a discrete (metallic) matrix and polymeric, since microcracks forming on the surface of the brittle fibers are closed by the matrix. The reinforcement may be in the form of continuous or cut fibers and two- or three-dimensional fabrics and mats [13, 14, 15].

Basalt fiber is a product made of melted basalt aggregate. It is a magma rock with a fine-grained structure and black, gray or green color. It is very hard and resistant to acid and alkaline environments. The chemical composition of basalt is as follows: 45-55\% SiO₂, 14\%-20\% Al₂O₃, 10\%-14\% CaO, 5-14\% FeO, 5-12\% MgO, 0,5-2\% TiO₂, 2-6\% other basic compounds. Basalt fiber is obtained during the process of melting basalt rock at temperatures above 1400 °C in furnaces. The use of such a high temperature returns the material to the original form - lava. It flows through the head with holes with diameters from 6 to 22 μm, made of platinum. Leaking lava is subjected to cooling and drawing, and then wound on reels. Basalt fibers, depending on the purpose, can be supplied in the form of continuous or cut fibers and two- or three-dimensional fabrics and mats 

2.1 Materials

The basalt textile grid HTB 10/14-40 was obtained from Frisiverto s.r.o (Czech Republic) company. The textile had density 2.75 g/cm³, elastic modulus of 81 GPa, tensile strength of 1355 MPa, elongation at break 1.86%, the linear density 2400 tex. The mechanical parameters of the grid are shown in Table 1 [16].

Metakaolin KM60 was obtained from Keramost a.s. (Czech Republic). It is a powdered material containing predominantly aluminosilicate compounds including heat-treated floated kaolinite. A potassium-based alkaline activator is a combination of aqueous potassium glass marked as R36 and KOH potassium hydroxide with 98% purity. Table 2 shows the chemical composition of metakaolin KM60 and water glass R36. As fillers in geopolymer mortar matrix micro-silica (grain size <0.065 mm) and rough silica (grain size 0.6 – 1.25 mm) were used.

2.2 Preparation of geopolymer mortar matrix

The geopolymer paste (Table 3) was prepared as a two-component system using metakaolin powder and sodium silicate solution in a liquid-to-solid ratio (0.8:1) by mechanically stirring for 4 min. In the next step, the micro-silica sand was added to the geopolymer mixture and the mixture was stirred for an additional 2 min. Finally, rough silica were added to the mixture and the mixture was mixed for more 0.5 min. Specimens with dimensions of 30 mm x 30 mm x 150 mm were prepared for the three-point bending test and cylindrical specimens with dimensions of with the diameter of 45 mm and length of 90 mm for splitting tensile tests.

2.3 Preparation of geopolymer thin-plates

In order to evaluate the four-point bending strength for geopolymer composite thin-plates with embedded Basalt textile, specimens with a rectangular form with the dimensions of 400 mm x 100 mm x 15 mm (length, wide, height) were prepared. The label of textile reinforced geopolymer composite used in this experiment is defined as...
follows: GP represents the name of geopolymer composite thin-plates, L represents the number of textile layers, and B represents the name of Basalt textile. For example, label GP0L means that geopolymer composite plates reinforced without textile layer, label GP1LB means that geopolymer composite plates reinforced with the one layer of Basalt textile, respectively. For specimens reinforced with one textile layer, the thickness of geopolymer layers on bottom is 6 mm, then one textile layer is laid over geopolymer matrix followed by filling the rest of the molds. For specimens reinforced with the three textile layers, the thicknesses of matrix layers on the top and bottom are both 3 mm, the thickness of geopolymer layers in between other textile layer is 4.5 mm. For specimens reinforced with four textile layers, the thicknesses of geopolymer layer on the top and bottom are both 3 mm, the thicknesses of geopolymer layer in between different textile layers are 3 mm.

For Charpy impact test, six samples of 30 mm x 15 mm x 80 mm were cut from the full cured specimens that were formerly prepared to test four-point bending strength.

2.4 Methods of mechanical properties evaluation

Fig. 1 Four-point bending test procedure

The mechanical properties of geopolymer mortar matrix were obtained by the compressive and three-point bending strength. The three-point bending tests were conducted on 30x30x150 mm³ three prism specimens with test span 100 mm, and then the compressive strengths were measured on the far edge of both residual pieces obtained from the flexural test, which carried out according EN 196-1 CEN standard sand. The splitting tensile tests were conducted on cylinder specimens with the diameter of 45 mm and length of 90 mm. A total of 15 specimens for each test were prepared and tested at the age of 1, 14, and 28 days.

A four-point bending test with constant bending moment zone (Fig. 1) was used to determine the bending strength of the panels. Three samples from each of the examined mesh types and mesh size were tested.

The calculation of the measured data and the evaluation of the test results were made using the following equation:

\[
\sigma = \frac{Fl}{bh^2}
\]

where:
- \(\sigma\) – the flexural strength in MPa;
- \(F\) – load at a given point on the load deflection curve in N;
- \(b\) – the width of the tested sample in mm;
- \(h\) – the thickness of tested sample in mm;
- \(l\) – the support span in mm.

A Charpy impact tester with a 18kg pendulum hammer was employed to determine the impact strength. All six samples of 30 mm x 15 mm x 80 mm (wide, height, length) of each type were used. The impact strength \((\sigma)\) was calculated using the following equation:

\[
\sigma = \frac{E}{A}
\]

where:
- \(\sigma\) – impact strength;
- \(E\) – impact energy required to break a sample;
- \(A\) – cross-section area.

3 Results and discussion

3.1 Mechanical strength of geopolymer mortar matrix

Table 4 presents the results of strength test of geopolymer mortar after various number of days of mortar bonding. Flexural, compressive and splitting tensile strength were evaluated. An increase in the mechanical strength of the material is visible along with the subsequent binding days of the mortar. This gap is particularly noticeable when bending and squeezing after the first two weeks. In the case of flexural strength, the increase is more than double, while in the case of compressive strength, the increase was almost 50%.

Tab. 4 Result of mechanical strength of geopolymer mortar matrix

| Type                      | 1 day       | 14 days   | 28 days   |
|--------------------------|-------------|-----------|-----------|
| Flexural strength [MPa]   | 4.41 ± 0.38 | 8.9 ± 0.63| 11.08 ± 0.48|
| Compressive strength [MPa]| 27.66 ± 1.01| 40.67 ± 3.78| 64.23 ± 3.79|
| Splitting tensile strength [MPa] | 1.82 ± 0.33 | 4.27 ± 0.52 | 4.8 ± 0.25 |

3.2 Four-point bending strength test of reinforced composite geopolymer material

The results of the four-point bending strength test of geopolymer composite thin-plates reinforced with Basalt textile are presented in Table 5. A noticeable increase in bending strength occurs in the case of reinforcement with a triple layer of basalt fabric. The strength increased by 150% compared to unreinforced material. The addition of the next layer of fabric caused an increase in strength by over 16%. In the case of reinforcement with one layer of fabric, there was no significant change in the strength of
the composite in relation to the unreinforced material – the difference is in the margin of error. The results are visualized at graphs in Figures 2 and 3.

**Fig. 2** Flexural Loading – deflection curves of geopolymer thin-plates reinforced with Basalt grid corresponding to various textile layers

**Fig. 3** Ultimate flexural strength of all the specimens reinforced with Basalt textile

| Tab. 5 Result of four-point bending strength test of reinforced composite geopolymer material
| Series | Label | Ultimate Load [N] | Ultimate Stress [MPa] | Deflection [mm] |
|--------|-------|--------------------|-----------------------|-----------------|
| Without reinforcement | GP0L | 523.77(72.76) | 6.54(1.01) | 0.76(0.26) |
| Reinforced with basalt grid | GP1LB | 624.72(97.99) | 6.88(1.08) | 0.95(0.29) |
| | GP3LB | 1307.22(165.06) | 14.4(1.82) | 7.17(0.6) |
| | GP4LB | 1520.50(190.40) | 17.82(2.23) | 5.87(0.69) |

### 3.3 Charpy impact test of reinforced composite geopolymer material

The graph in Figure 4 presents the results of Charpy impact test of reinforced composite geopolymer material with various layer number of basalt grid. As in the case of bending strength test, a noticeable increase in bending strength occurs in the case of reinforcement with a triple layer of basalt fabric. The strength increased from 12.6 to 20.2 J/cm² in comparison non-reinforced material which means the increase of over 60%. The addition of fourth layer of fabric caused an increase in strength up to 22.0 J/cm². In the case of reinforcement with one layer of fabric, there was no significant change in the strength of the composite in relation to the unreinforced material – the slight decrease (12.3 J/cm²) is in the margin of error.

**Fig. 4** Impact strength of reinforced geopolymer

### 4 Conclusions

The reinforcement has a significant impact on the mechanical properties of the geopolymer material - both static and dynamic. The reinforcement makes it possible to reduce the mass of material in relation to a material without reinforcement characterized by similar parameters. This is particularly important in the case of engineering construction materials where lightness and high strength are required. Referring to the test results, it can be concluded that there is an optimal number of reinforcement layers, at which a high sudden increase in the strength of the reinforced material is noticeable. The research proved the legitimacy of using basalt fabrics as a reinforcement in geopolymer material and opens the door to more advanced construction applications.

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References

[1] DAVIDOVITS, J. (2008), Geopolymer chemistry and application, Institut Géopolymère, Saint-Quentin

[2] MIKUŁA, J., ŁACH. M. (2014). Geopolimery – nowa przyjazna środowisku alternatywa dla betonów na bazie cementu protlandzkiego. Wprowadzenie. In: Rozwiązania proekologiczne w zakresie produkcji. Nowoczesne materiały kompozytowe przyjazne środowisku. (J. Mikula, (Ed.)), pp. 13-32. Wydawnictwo PK, Cracow.

[3] BAKALOVA, T., M. KOLÍNOVÁ a P. LOUDA. (2014). Micro CT Analysis of Geopolymer Composites. Manufacturing technology, 14 (4). pp. 505 – 510.

[4] HIPS fireproof coatings can really take the heat, http://phys.org/news167306601.html (20.07.2009).

[5] DAVIDOVITS J., Mineral polymers and method of making them, US patent 434 9386, 1982.

[6] VAN JAARSVELD J. G. S., VAN DENVENTER J.S.J., LORENZEN L. (1997) The potential use of geopolymeric materials to immobilise toxic metals: Part I. Theory and applications, Materials Engineering, Vol. 10, No. 7, 659-669.

[7] BOCZKOWSKA A., (2016), Podstawowe informacje o kompozytach. In: Kompozyty i techniki ich wytwarzania (A. Boczkowska, G. Krzesiński (Ed.)) pp. 9-50. Oficyna Wydawnicza PW, Warszaw.

[8] BEHERA, P., BAHETI, V., MILITKY, J., & LOUDA, P. (2018). Elevated temperature properties of basalt microfibril filled geopolymer composites. Construction and Building Materials, 163, 850-860. Elsevier

[9] XIEM, N., LOUDA, P., KROISOVA, D., TRUNG, N., & THIEN, N. (2012). The influence of modified fly ash particles by heating on the compressive strength of geopolymer mortar. Chemické Listy. Vol: 106 SI, Supplement 3, pp. s557-s559.

[10] THANG, X. N., LOUDA, P., KROISOVÁ, D., KOVACIC, V., LE CHI, H., VU, N. L. (2012). Effects of commercial fibers reinforced on the mechanical properties of geopolymer mortar. Chemické Listy. Vol. 106. pp. 560-563.

[11] THANG, X., LOUDA, P., & KROISOVA, D. (2013). Thermophysical properties of woven fabrics reinforced geopolymer composites. World Journal of Engineering, 10(2), pp. 139-144. Emerald.

[12] HUNG, T. D., PERNICA, D., KROISOVÁ, D., BORTNOVSKY, O., LOUDA, P., & RYLICHÓVA, V. (2008). Composites base on geopolymer matrices: Preliminary fabrication, mechanical properties and future applications. In Advanced Materials Research (Vol. 55, pp. 477-480). Trans Tech Publications.

[13] BOCZKOWSKA A., KAPUŚCIŃSKI J., LINDEMANN Z., WITEMBERG-PERZYK D., WOJCIECHOWSKI S. (2003) Kompozyty. Wydanie II zmienione, Oficyna Wydawnicza PW, Warszaw.

[14] MATTHEWS F. L., RAWLINGS R. D. (1994) Composite Materials: Engineering and Science, Chapman & Hall, London.

[15] ŽMINDÁK, M., PELAGIĆ, Z., SOUKUP, J. (2015). Analysis of Fiber Orientation Influence to Dynamic Properties of Composite Structures, Manufacturing technology, 15 (3), pp. 490-494.

[16] FRISIVERTO PRODUCT CATALOGUE, https://www.frisiverto.cz/data/file/36.pdf.

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