

## Conference Paper

# Mechanical Properties of Basalt Fiber Reinforced Fly Ash-Based Geopolymer Composites

Kinga Korniejenko<sup>1</sup>, Gábor Mucsi<sup>2</sup>, Nóra Papné Halyag<sup>2</sup>, Roland Szabó<sup>2</sup>, Dariusz Mierzwiński<sup>1</sup>, and Petr Louda<sup>3</sup>

<sup>1</sup>Cracow University of Technology, Faculty of Materials Engineering and Physics, Institute of Materials Engineering, Jana Pawła II 37, 31-864, Cracow, Poland

<sup>2</sup>University of Miskolc, Institute of Raw Material Preparation and Environmental Processing, 3515, Miskolc, Egyetemváros, Hungary

<sup>3</sup>Technical University of Liberec, Faculty of Mechanical Engineering, Department of Material Science, Studentska 2, Liberec, 461 17, Czech Republic

## Abstract

This article analyses the influence of a short basalt fibre admixture on the mechanical properties of geopolymers, especially compressive strength. This preliminary research is the first step towards the development of a composite for fire resistant applications in civil engineering. This study investigates the behaviour of a fly ash based geopolymer containing basalt fibres. Fly ash from the coal power plant 'Skawina' (located in: Skawina, Lesser Poland, Poland) was used as the raw material. The chemical composition of this fly ash is typical for class F. Three series of fly ash based geopolymers were cast. In the first, short basalt fibres were added as 1% by weight of fly ash, in the second short basalt fibres were added as 2% by weight of fly ash and the third functioned as a control series without any fibres. Each series of samples were tested on compressive strength after 28, 14 and 7 days, and specimen density was determined. Additionally, microstructural analysis was carried out after 28 days. The results show that the addition of basalt fibres can improve the mechanical properties of geopolymer composites.

**Keywords:** geopolymer, basalt fibre, composites, fire resistance

Corresponding Author:

Kinga Korniejenko

kinga.korniejenko@mech.pk.edu.pl

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## 1. Introduction

Geopolymer binder is a brittle material with a relatively low tensile strength and deformation capacity. To rectify these issues, reinforcement is required [1, 2]. In particular, the brittleness of these materials results in cracking under tensile and flexural loading [1, 2]. The challenge is to replace the conventional steel bars traditionally applied in construction industry, through the proper short fibres [3]. The one possibility is mineral fibres. They have reasonable mechanical properties and are a cost-effective alternative. Moreover, the mechanical properties of mineral fibres tend to be greater than those offered by the use of natural fibres for reinforcement [4, 5].

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Among the mineral additives, the widest research was conducted on basalt fibres. There were implemented to geopolymer matrix as fabrics [6–8], long fibres [9], as well as short fibres and microfibers. The short basalt fibres were investigated in different types of matrix, including: metakaolin, slag and fly ash. The microscopic investigations by using a scanning electron microscope, showed very good coherence of these fibres to the different matrix, in contrast to glass and carbon fibres [10].

Research based on a geopolymer matrix made of metakaolin with three mineral additives: short basalt fibres, wollastonite and thermolite particles were performed [11]. Optimal mechanical properties - compressive strength - were obtained for a composite with 5% wollastonite, 5% thermolite and 2% basalt fibre [11]. The compressive strength increased for the selected composition to 33.5 MPa, compared to a value of 13 MPa for the metakaolin matrix. Additionally, the selected composition was tested for strength after 3, 7, 28 and 90 days for resistance against aggressive environments, i.e. 15%  $\text{Na}_2\text{SO}_4$  solution and 15% NaCl solution. The fibre composites retained higher mechanical properties than the geopolymer matrix itself [11]. Other studies based on a geopolymer matrix made of metakaolin with the addition of 1, 3, 5, 7 and 10% by weight of short basalt fibres were conducted, among others in terms of flexural strength. The fibres had about 0.64 cm (0.25 inch) long and 13  $\mu\text{m}$  in diameter [12]. The strength properties increased with the addition of fibre. For a plain matrix, the flexural strength was 1.7 MPa and increased with the addition of basalt fibres - it was respectively: 1% - 3.6 MPa, 3% - 8.6 MPa, 5% - 13.5 MPa, 7% - 16.5 MPa and 10% - 19.5 MPa [12]

The investigations were also carried out on a matrix based on fly ash class F from Raichur Thermal Power Station, Karnataka, India (class F fly ash), with the addition of slag, basalt fibre, plasticizer (MYK Remicrete PC 5) and two types of aggregates (fine and coarse) [13]. The tests were carried out on composites with the addition of 0.5, 1, 1.5, 2, 2.5 fibres by weight and plain samples for comparison. The compressive strength was tested after 7 and 28 days. As expected, the strength values for all composites after 28 days were higher, compared to the values obtained after 7 days, for matrix material they were 30.8 MPa and 43.3 MPa, respectively. The composition with fibres had higher values than plain matrix. The best result was achieved for 2% fibre content - 40.1 MPa and 58.4 MPa [13]. Tensile tests were also tested. Strength values for the matrix material were: 2.2 MPa after 7 days and 2.4 MPa after 28 days. Maximum values were also obtained for composites with a 2% fibre addition: 3.2 MPa and 3.6 MPa [13].

Tests on the mechanical properties of composites with the addition of basalt fibres were also carried out on the basis of a matrix consisting of fly ash - class C, slag after processing of steel and sand [14]. The basalt fibres (diameter: 7-30  $\mu\text{m}$  and lengths:

12 mm), were added in following amount: 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5% by volume. Samples were tested after 3, 7 and 28 days [14]. The compressive strength increased with time for all composites, after 3 days it was about 15 MPa, after 7 days - about 22 MPa, and after 28 days - about 35 MPa. All composites achieved higher values than the matrix material. The highest values were obtained for 0.4% volume addition of basalt fibres, it was 40.3 MPa, compared to values around 35 MPa for matrix [14]. For bending strength, the value after 3 days was about 3 MPa, after 7 days - about 4 MPa, and after 28 days - about 6.5 MPa. All composites achieved higher values than the plain matrix. The highest values were obtained for 0.3% by volume of fibres, it was 7.3 MPa [14].

Compression, tensile and bending strength tests were carried out for samples with 0.5% and 1% basalt fibres. The geopolymer matrix was based on metakaolin. Additionally, the investigation were carried out on a matrix based on Portland cement for comparison [15]. The compressive strength decreased for both materials together with the addition of basalt fibres with a simultaneous increase in tensile and flexural strength [15]. The compressive strength for the geopolymer matrix were 39.5 MPa, and after addition of 0.5% and 1% basalt fibre 28.6 MPa and 36.9 MPa, respectively. For comparison, the value for concrete based on Portland cement, without the addition of fibres, was: 23.1 MPa [15]. The tensile strength values were: for the geopolymer matrix 3.2 MPa, for 0.5% fibre addition 4.3 MPa and for 1% basalt fibre addition 4.0 MPa. The tensile strength for concrete without fibre was 2.5 MPa. The values of bending strength are given in kN. They increased from 16 kN for the geopolymer matrix, through 17.2 kN for the composite with 0.5% fibre up to 21 kN for the composite with 1% fibre. For concrete based on Portland cement, without the addition of fibres, this value was: 9.6 kN [15].

Research with basalt fibres was also carried out on the basis of a geopolymer matrix composed of metakaolin, slag from the Bolu Cement Company in Turkey, sand and collemanite waste (borate mineral) from the Eti Mining Company mine in Turkey [16]. The tests were carried out with 0.4, 0.8 and 1.2% fibres addition by volume. The fibre dimension: diameter - 20  $\mu\text{m}$  and lengths - 12 mm. The specimens were investigated after 3, 7 and 28 days [16]. The mechanical properties increased in time. The best results for compressive strength were obtained for a sample containing 1.2% of basalt fibres - 64.8 MPa after 28 days, compared to the sample without reinforcement - 61.6 MPa. The best results for bending strength were also obtained for a composite with 1.2% fibre addition - 12.6 MPa, compared to the plain matrix - 8.8 MPa [16]. The behaviour of geopolymer materials at high temperatures were also be researched, i.e. 300 °C, 600 °C and 900 °C. Significant deterioration of mechanical properties was found at

temperatures of 600°C and 900°C, however samples with the addition of fibres still had higher values than the material of the geopolymer matrix [16].

Composites with a basalt fibre content of 0.5 and 1% by volume were investigated with using geopolymer matrix based on fly ash from the Eraring power plant in the region of New South Wales, Australia. At the same time, the tests were carried out for both - the traditional matrix and the geopolymer foamed with H<sub>2</sub>O<sub>2</sub> [17]. The fibres had the following dimensions: diameter - 16 µm and lengths - 5 mm. Samples were tested after 7 days [17]. In the case of compressive strength for non-foamed geopolymer, no significant differences were found between composites with different basalt fibre content and matrix material. The strength for all samples was about 20 MPa. In the case of foamed geopolymer, the addition of fibres resulted in an increasing of compressive strength by about 4.5 MPa [17]. Bending strength tests were carried out for the non-foamed matrix, it showed no significant value changes. It was about 6 MPa for both composites and plain matrix material. It should be noted that the introduction of PVA fibres in the same matrix in analogous amounts increased the flexural strength [17].

Tests with the addition of microfibers were conducted in the scope of compressive strength at ambient temperature - 30 °C, and at elevated temperatures: 200, 400 and 800 °C [18]. The tests were carried out on a metakaolin matrix reinforced with basalt microfibers up to 10 µm, 5, 10 and 15% of microfibers by weight were used [18]. At 30 °C, the composite with 15% microfibers showed the highest strength properties. Its compressive strength was 38.1 MPa, compared to 28.4 MPa for the plain matrix. Tests at elevated temperatures also showed an increasing of compressive strength of all tested samples at 200 °C, and decreasing of strength at higher temperatures. The composite with 15% addition of microfibers also showed the highest values at 200 °C - 43.8 MPa, compared to the plain matrix - 36.6 MPa. At 400 °C and 800 °C, the highest strength was achieved by a composite with a 10% of microfiber, it was 23.1 MPa and 16.1 MPa, respectively, and for comparison plain matrix had 14.8 MPa and 11.2 MPa, respectively [18]

Similar works with the addition of short basalt fibres were carried out on a geopolymer matrix based on fly ash from the Gladstone power plant in Queensland, Australia [19]. The fibres were added in amounts of 0, 0.5, 1 and 1.5% by weight. The tests were carried out at temperatures of 28 °C, 200 °C, 400 °C, 600 °C and 800 °C. The fibres had dimensions: diameter - 13 µm and length - 12.7 mm [19]. The results of the compressive strength at ambient temperature showed an increasing for samples with short fibres compared to the material without additives. The best results were achieved for 1% of fibres – ca. 36 MPa (plain matrix: ca. 29 MPa) [19]. At 200 °C, 400 °C and 600 °C, the

properties of composites increasing, only at 800 °C they decreased. The material had the highest strength properties at 400 °C for composites with 0.5 and 1% addition of basalt fibre – ca. 45 MPa, while for 1.5% and the control sample was over 35 MPa [19]. Studies confirmed the resistance of basalt fibre geopolymers to elevated temperatures [18, 19].

It is also worth paying attention to the research on the addition of basalt fibres to fly ash (class C) from the power plant in Thailand. In these studies, a very high weight proportion of basalt fibres was used: 0, 10, 15, 20, 25 and 30% [20]. The compressive strength tests were carried out after 7 and 28 days. All composites with the addition of basalt fibres obtained higher compressive strength than the basic matrix. The optimal addition was 10 - 15% of fibres [20]. At the same time, the composite with 15% fibres content achieved its maximum compressive strength - 44 MPa after 7 days and the same value was maintained after 28 days. The composite with 10% fibre content reached almost 40 MPa after 7 days, however, this value increased and after 28 days was about 45 MPa. For comparison, the results for the composite without fibre after 7 and 28 days were ca. 37 MPa [20].

As regards other mineral additives, work was carried out on fibres based on silica, aluminium oxide and zirconium [21]. Other mineral additives used in geopolymer composites include diatomaceous earth [22], however, this additive was not introduced in the form of fibres, but grains with a diameter of 1.2 µm and wollastonite, also introduced in the form of micro-additives [23, 24].

## 2. Materials and Methods

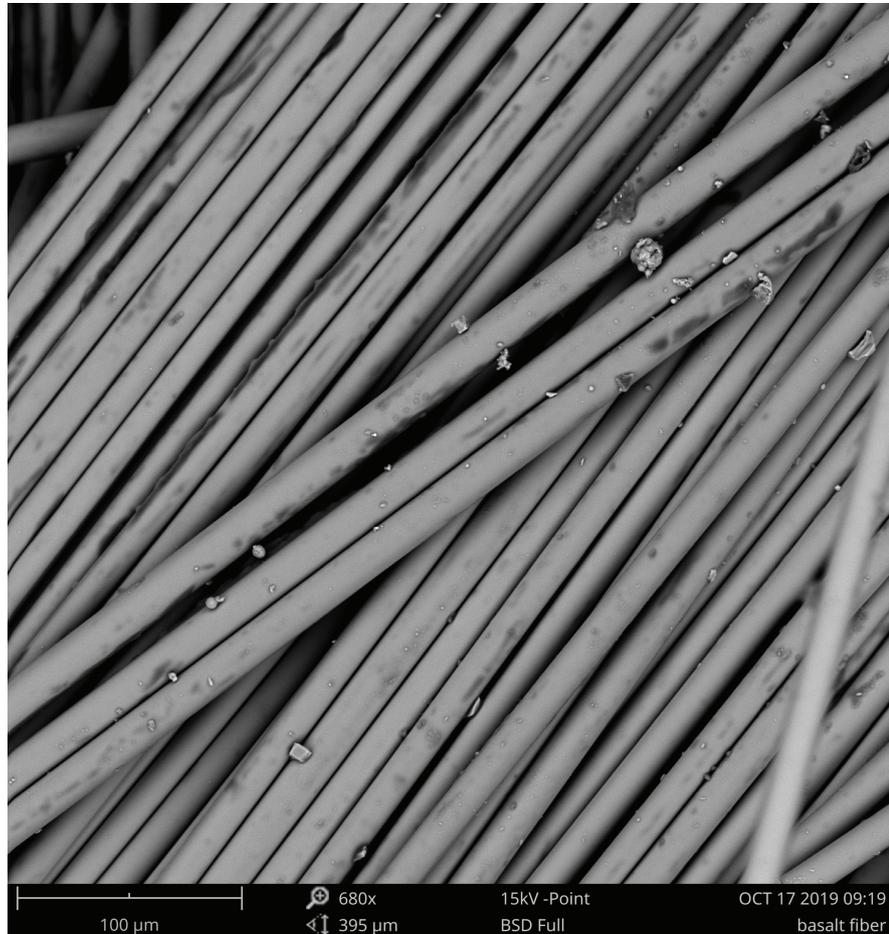
### 2.1. Materials

The matrix was made with using fly ash without any aggregate. The fly ash was delivered from the bituminous coal power plant 'Skawina' (located in: Skawina, Lesser Poland, Poland). It is obtained as combustion by-product through the electrostatic precipitation of fine particles from the exhaust gases from coal-fired furnaces.

The chemical composition of this fly ash is typically for class F [25]. It contains less than 5% of unburned material, less than 10% of iron compounds and low amount calcium compounds. The amount of the reactive silica in investigated material is reasonable – ca. 36%. It has good physical parameters - the content of particle under the size 45 µm is ca. 88% [25, 26]. The fly ash contains a lot of spherical particles and has good workability [26]. The mineralogical structure investigations show the large amount of

amorphous phase [26]. The fly ash has a features that predestine it as a raw material in geopolymerization process.

As the reinforcement the short basalt fibres has been applied (Figure 1).



**Figure 1:** SEM image – basalt fibres.

The fibres were about 5 mm length. They have been added in amount 1 and 2% by weight.

## 2.2. Samples preparation

Samples were prepared using sodium promoter, fly ash and basalt fibres. The synthesis of geopolymers was made with using 8M ratio sodium hydroxide solution. The three kind of samples were prepared:

1. plain samples (geopolymer mortar without fibres addition),
2. with addition of 1% short basalt fibres by weight,
3. with addition of 2% short basalt fibres by weight.

The fly ash, alkaline solution and fibres were mixed manually about 5 - 10 minutes to receive the homogeneous paste. Next, it was poured into sets of plastic moulds. The samples were hand-formed. They were left in laboratory condition for curing for 24 h. Next, they were heated in the laboratory drying cabinet for 8 h at 75 °C and left in the drying cabinet till they reach ambient temperature. Then, the samples were unmoulded. They were investigated after 7, 14 and 28 days.

### 2.3. Analytical procedure

The morphology was investigated by scanning electron microscope (SEM) on Phenom ProX. The research has been made for fibres and the samples previously broken during compressive strength tests. The investigations were made at various magnifications.

Compressive strength tests were performed according to the methodology described in the standard EN 12390-3. ('Testing hardened concrete. Compressive strength of test specimens'), because of the lack of separate standards for geopolymer materials. The tests involved at least 15 samples. Tests were provided on universal testing machine: Anyagvizsgáló Készülékek Gyára Budapest, type: SZF-1 with additional devices for measurement: Hottinger Baldwin Messtechnik GmbH. Samples used to the compressive strength test had cubic shape and dimensions (approx.): 20 mm × 20 mm × 20 mm.

## 3. Results and Discussion

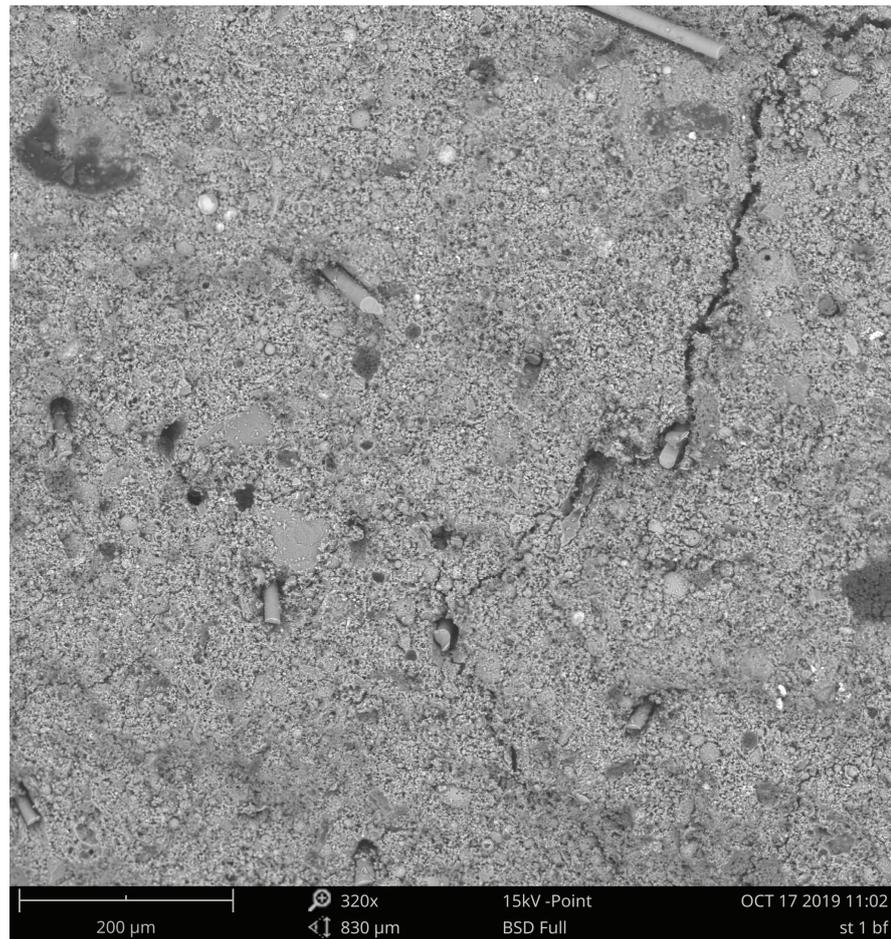
### 3.1. Microstructure investigations

The SEM observations were made for fibres (Figure 1) as well as for composition reinforced by fibres (Figures 2, 3 and 4). The images were made at various magnifications.

The observation of microstructure of composites gave a preliminary information about the distribution of fibres (Figure 2) and coherency of fibres and the geopolymer matrix (Figures 3 and 4).

The fibres are evenly distributed in all volume of the material (Figure 2). It is clearly visible that they are oriented in different directions. The material is probably anisotropic and the fibre distribution was regular. The fibre distribution in the matrix influences the properties of the specific composite. In case of the fibres aggregation appearance it could decrease its mechanical properties.

The microstructural observation also allows to notice that the structure is coherent and the fibres have good connection with matrix (Figure 3). Moreover, the some fibres



**Figure 2:** SEM image: composite with basalt fibres – distribution of fibres in composite.

probably have been partly dissolved (in the right upper corner in the Figure 3 we see shell after the basalt fibre).

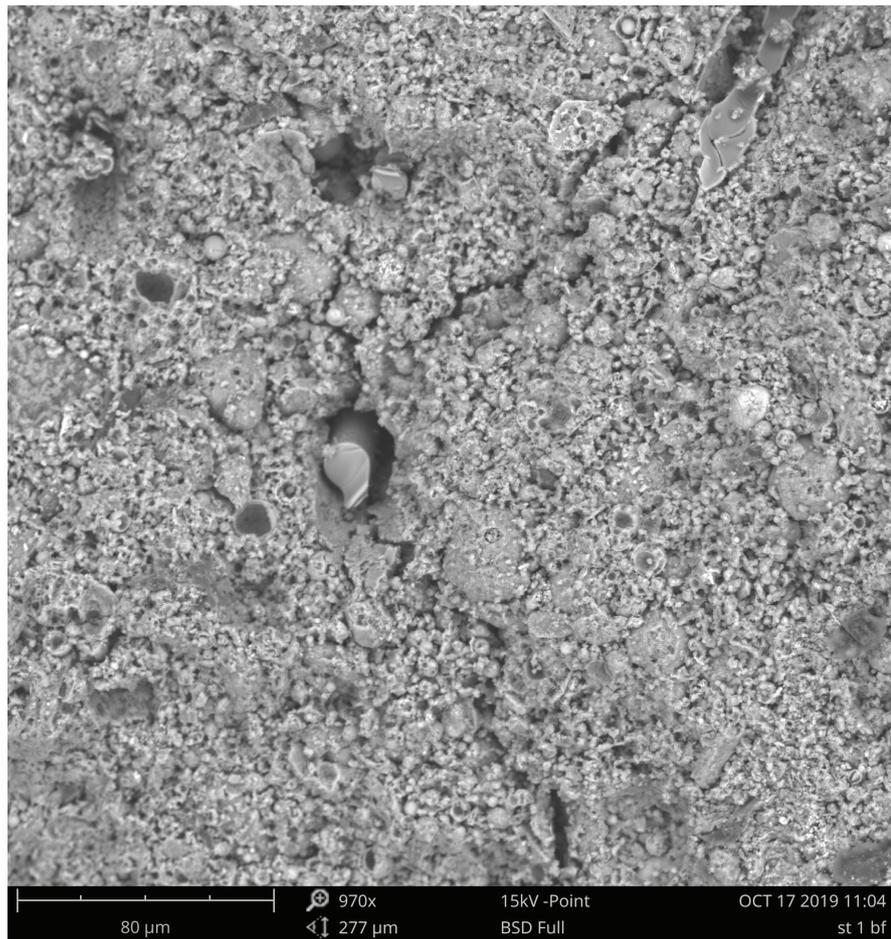
In the Figure 3 and 4 there is also visible the suppression the cracking by the fibre. The cracking is limited by the fibre presence (Figure 4). Additionally the fibre in the Figure 3 probably has been broken by the force during the compressive strength test. It shows that the fibres are actively involved in strength transmission.

The SEM observations allow to orient in overall structure of the composite and the role of fibres. The high coherence with matrix were noticed.

### 3.2. Mechanical properties - compressive strength

The results of the compressive strength tests are shown in Table 1.

The best results have been achieved for plain matrix after 28 days. It was more than 13 MPa. The achieved results are not coherent with predictions and literature sources



**Figure 3:** SEM image: composite with basalt fibres – basalt fibres and the cracking propagation.

TABLE 1: The results of compressive strength

	Basic sample	1% basalt fibres	2% basalt fibres
1 week	9.8 MPa (s.d.: 0.8)	8.7 MPa (s.d.: 1.0)	12.8 MPa (s.d.: 1.0)
2 weeks	10.0 MPa (s.d.: 2.7)	7.9 MPa (s.d.: 0.8)	8.7 MPa (s.d.: 0.7)
1 month	13.1 MPa (s.d.: 1.7)	10.7 MPa (s.d.: 1.1)	11.9 MPa (s.d.: 0.8)

s.d. = standard deviation

that show that compressive strength should increase according to the curing time [11–16]. This trend was only visible for the plain samples. For the samples with fibres this trend was not coherent. The achieved results show that the compressive strength for the samples with fibres was lower after two weeks in comparison with the samples after 1 week.

The compressive strength changes also depend on the fibre content. It is usually lower than for plain samples, the exception are the samples after 1 week with 2% of basalt fibres. The lower values for compressive strength for the samples with fibres have



**Figure 4:** SEM image: composite with basalt fibres – basalt fibres.

been previously reported in the literature [15]. However most of provided researches show rather increasing for this value [13, 16, 18] or lack of significant difference [17].

Next, the results of the compressive strength test have been compared with the samples' density (Table 2).

TABLE 2: Density of the samples

	Plain sample	1% basalt fibres	2% basalt fibres
1 week	1.49 g/cm <sup>3</sup>	1.48 g/cm <sup>3</sup>	1.52 g/cm <sup>3</sup>
2 weeks	1.60 g/cm <sup>3</sup>	1.46 g/cm <sup>3</sup>	1.47 g/cm <sup>3</sup>
1 month	1.47 g/cm <sup>3</sup>	1.44 g/cm <sup>3</sup>	1.43 g/cm <sup>3</sup>

Changes of density should be correlated with compressive strength. For the investigated samples this tendency did not appear. The lowest density is for samples with 2% of basalt fibres after 1 month and the compressive strength for this samples is 11.9 MPa. The highest density is for the plain samples after 2 weeks, but the compressive strength for this samples is lower – only 10.0 MPa. However differences in the density were not

significant. There is no correlation between the density and compressive strength for this series of samples.

The research also allows to observe indirectly the change in the samples behaviour. The plain samples were broken in brittle way (Figure 5), whereas for the samples with fibre the mechanism of crashing was more ductile (Figure 6).



**Figure 5:** Samples without fibres after compressive strength test.

The changing of the behaviour is coherent with the research reported in the literature.

It is worth to notice that the research were conducted for the geopolymer mortar without any aggregate, because of that the achieved values for compressive strength are relatively low in comparison with other reported in literature [11–19]. The sand addition could significantly improve this value.

## 4. Conclusions

Geopolymer composites based on fly ash reinforced with different amounts of basalt fibres have been manufactured and characterized. The composites were prepared using sodium promoter. The microstructure observation showed regular distribution and coherence fibres with the matrix. The results for this investigations are according to



**Figure 6:** Samples with 2% of basalt fibres after compressive strength test.

the prediction and literature sources. The results for compressive strength are slightly different than predicted and only partly coherent with other researches that have been published. The addition of 1% of basalt fibres decreased the compressive strength. The results for 2% of fibres addition are inconclusive. Firstly the compressive strength increased (after 7 days period), but next (after 14 and 28 days) it is lower than for basic matrix.

The fibre addition did not improve the compressive strength (after 28 days) of the composite significantly, but the presence of the fibres changes the character of failure mechanism from brittle to ductile. The changing the behaviour during the cracking from brittle to more ductile increases the safety of the material in potential application in construction industry. This study also presents that it is possible to produce the composites of reasonable properties from the fly ash and basalt fibres. This kind of composites could be applied in construction industry for the bricks, pavement elements and others.

## Founding

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