Utilization of basalt fabrics as reinforcement for alkali-activated blast furnace slag systems

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Abstract. Alkali-activated materials based on the alkali-activated blast furnace slag (AABFS) seem to be a suitable alternative construction material due to the environmental and economic aspects. More widespread application potential could be supported by the utilization of fabric reinforcement into the matrix to form AABFS composite with improved mechanical properties. The basalt fibres or fabrics (BFs) represent a reinforcing material suitable for the AABFS due to its favourable price/properties ratio. However, the question of fibres stability under the alkaline conditions should be taken into account. The effect of BFs implementation in one or more layers into various types of matrices to the mechanical properties was studied with the regard to the compressive and flexural strengths. The used matrices were based on the alkali-activation of the BFS with various activators. The fabric/matrix adhesion same as the transition zone properties were studied using the SEM analysis and pull-out tests. Indication of appreciable improvement of mechanical properties utilizing one layer of BFs for some of the mixtures activated using sodium water glass and sodium carbonate was found out.

Keywords Alkali-activated materials, blast furnace slag, basalt fabric, pull-out test

1. Introduction

Alkali-activation of secondary raw materials like blast furnace slag (BFS) using the convenient alkaline activator allows to produce alternative construction materials e. g. AABFS with many favourable properties in contrary to the traditional ordinary Portland cement (OPC). [1, 2] Starting with the much-discussed environmental benefits of carbon dioxide emissions lowering along with the ability to achieve lower cost when the composition of the alkali-activated materials is optimised [3] and continued by many favourable properties like lower hydration heat in contrary to the OPC [4], high resistance to the aggressive environments [5] or good high temperature performances [6]. Still, there are some disadvantages like high shrinkage accompanied with formation of micro and macro cracks or formation of efflorescences as described in publication [7].

AABFSs represents suitable matrix for basalt fabric implementation to form inorganic composite with improved mechanical and thermal properties as well as durability. The main purposes of fibre/fabric reinforcement of the matrix are to provide a control of the cracking and to increase the fracture toughness of the brittle matrix through the stress transfer during both micro and macro-cracking. The ability of the stress transfer depends on the volumetric fibre fraction, fibres orientation and distribution. However, in order to achieve the desired properties, the existence of quality adhesion
between the fibres and the matrix (F/M) same as formation of transition zone at their interface is necessary as discussed in studies [8–10]. Since the AABFS matrix forms highly alkaline environment for the reinforcing material and due to the price/parameter ratio basalt fibres have been chosen instead of the alkali resistant glass fibres in this paper.

There were no publications where the same type of matrix and basalt fabric were used, otherwise that is the reason why this paper has originated. Many authors used chopped basalt fibres instead of the fabric, hence possibilities of utilization of basalt fibres as a reinforcing material were tested. Timukal et al. [11] reported that reinforcement of Na$_2$SiO$_3$ and NaOH activated fly ash matrix utilizing 10 wt. % of basalt fibres led to the increase of compressive strength about 37% after 28 days but utilization of the dosage higher than 15 wt. % resulted in decrease of compressive strength in contrary to the reference because of lower precursor dissolution leading to the less binder content. Similar results were presented in Rill et al.’s paper [12] where the addition of 10 wt. % of the BFs to the matrix based on the metakaolin activated using the KOH resulted in the increase of flexural strength from 1.9 to 19.5 MPa. Masi et al. [13] noted that just a minimal chemical interaction between the AAM (based on fly ash activated with sodium aluminate solution) and the BFs had been found. They also reported improvement of the flexural strength between 600 and 1000°C due to the better F/M interaction after the sintering. These materials have the potential for applications at elevated temperatures up to about 800°C because they represent replacement of ceramic based composites for the midrange temperature applications. This issue was discussed more in study of Welter et al. [14].

This paper focuses on the preliminary studies with a goal to determine the influence of the basalt fabric utilization to the AABFS matrix on the mechanical properties and possibilities of characterization of the F/M interaction.

2. Experimental procedure

2.1. Raw materials

2.1.1. Blast furnace slag (BFS). Blast furnace slag was obtained from Kotouč Štramberk, Ltd. having a Blaine specific surface area of 400 m$^2$·kg$^{-1}$. Its chemical composition is given in Table 1. The BFS was 84% amorphous, while the rest was crystalline (akermanite – 9.5%, calcite – 3.7%, merwinite – 2.3% and quartz – 0.5%) according to the X-ray diffraction (XRD) using Rietveld method.

| Chemical composition in wt. %       |
|-------------------------------------|
| CaO          | SiO$_2$ | MgO | Al$_2$O$_3$ | SO$_3$ | TiO$_2$ | K$_2$O | MnO | Na$_2$O | Fe$_2$O$_3$ |
| 41.1         | 34.7    | 10.5 | 9.1         | 1.4    | 1.0     | 0.9    | 0.6 | 0.4     | 0.3        |

2.1.2. Alkaline activators. Liquid sodium water glass (WG) obtained from Vodní sklo, Inc. – Silicate modulus (M_s) = 2.24; Sodium hydroxide (SH) – PENTA Ltd. (pure); Sodium carbonate (SC) – INCHEMA Ltd. (pure).

2.1.3. Basalt fabric (BF). Basalt fabric (BF) in form of filament yarns connected into the square net was provided by the Institute for textile technique at RWTH Aachen University. BFs have been used in form of biaxial warp knitted textile but also as unique filaments yarns in same cases (pull-out test, SEM). Filament yarns have different tex in longitudinal (2.6) and transverse directions (1.3). Table 2 gives the information about the chemical composition of basalt fibres from Kammeney Vek determined by X-ray fluorescence (XRF). Amorphous structure of BFs was confirmed using the XRD.
Table 2. Chemical composition of the BFs from Kammeney Vek determined by XRF analysis.

| Chemical composition in wt. % | SiO₂ | Al₂O₃ | Fe₂O₃ | CaO | MgO | Na₂O | K₂O | TiO₂ | Other |
|-----------------------------|------|-------|-------|-----|-----|------|-----|------|-------|
|                             | 55.7 | 15.4  | 10.8  | 7.4 | 4.1 | 2.4  | 1.5 | 1.2  | 1.4   |

2.1.4. Other materials. Standard siliceous sand according to ČSN EN 196-1 was used for the mortar preparation. STACHEMENT AC 600 produced by Stachema Kolín Ltd. was used as a shrinkage reducing admixture (SRA).

2.2. Tests and methods

2.2.1. Mechanical properties testing. Testing samples were prepared according to ČSN EN 196-1. Specimens with dimension 40 × 40 × 160 mm were used for compressive and flexural strengths (three-point bend arrangement) determination using the Desttest 4310 Compact A (Beton System, Ltd.). Water to binder (w/b) ratio (0.49) was kept the same along with the SRA dosage (0.75 wt. % by the BFS weight) and sand to binder ratio (3:1) for all mortars. Various alkaline activator mixtures were used for the alkali-activation of the BFS meanwhile. Starting with the WG/SC and WG/SH mixtures with silicate modulus (Mₛ) equal to 0.5 using activator dosage ranging from 6 to 12 wt. % by the BFS weight. Demoulded specimens were kept in wet chamber until further testing was done. These tests were performed on mortars (reference) and mortars reinforced with the basalt fabric(s). One layer of basalt fabric was added to the middle of the specimen commonly, if there were more layers the layers were distributed equally, so the same amount of mortar was between the layers always.

2.2.2. Single fibre yarn pull-out test. This method was used for a direct estimation of fibre/matrix (F/M) interfacial bond properties based on pulling out of a single fibre yarn of its surrounding matrix (mortars). Testing samples had shape of cubes (40 × 40 × 40 mm) where the fibre yarn was placed into the middle with embedment length (EL) of 5 mm. Pull-out tests were performed for the same EL after 72 hours of curing at wet conditions based on previous research. Fifteen samples were tested and evaluated for each matrix type. The measurement was done using the Universal mechanical testing machine Instron 5985 (testing speed 2 mm/min; distance between jaws 100 mm) with a maximal force (Fₘ) before the fibre broke down (fibre breakage about Fₘ = 333 N) or was pull out determination. Interfacial shear bond strength was then calculated based on the Choi’s study [15].

2.2.3. SEM analysis. The Zeiss Evo LS 10 microscope with EDX analysis was used for microstructure of interfacial zone properties study. Accelerating voltage was 10 kV. Samples were AABFS pastes (w/b = 0.36) reinforced with one yarn of basalt fibres. They were broken down after defined period (7/28 days of hydration) and excessive fibres were removed. These fragments were placed on the carbon tape, sputter coated with gold and underwent the SEM analysis.

3. Results and discussion

3.1. WG/SC and WG/SH activated BFS matrices

Figure 1 and Figure 2 show results of flexural and compressive strengths development as a function of alkaline activator dosage and BF reinforcement.

The highest compressive strength (83.8 ±4.2) MPa was obtained for 10% Na₂O WG/SC activated matrix without utilization of BFs reinforcement after 112 days. It should be noticed that increase of compressive strength for this mixture have continued up to 112 days while the other activator dosages did not show further gain of compressive strength after 56 days of curing. The highest flexural strength was found out for 8% Na₂O dosage. Differences in flexural strengths for 8 and 10% Na₂O dosages are less crucial than the ones for the compressive strengths, thus the 10% Na₂O seems to be preferable.
WG/SC activated systems showed lower initial mechanical strength when compared to the WG/SH activated ones due to the lower pH of initial solution. The most suitable activator dosages for WG/SH mixtures were 6% Na$_2$O (56.3 ±4.3 MPa) or 8% Na$_2$O (55 ±5.4 MPa) based on the results obtained after 112 days. Further increase of activator dosage up to 10 or 12 % led to the compressive and flexural strengths decrease. Comparison of the best mixtures of each activator type should be done as well. The difference in compressive strengths obtained after 112 days was 36.8% in favour of the WG/SC.

Addition of one layer of basalt fabric to the 10% Na$_2$O WG/SC led to the compressive strength improvement in most cases when compared to the reference. More precisely, it resulted in the compressive strengths increase about 24.7; 0.5; 17.5 and 14.5% after 7; 28; 56 and 84 days of curing. No beneficial effect of BFs addition in one layer on the mechanical properties was found out for other tested WG/SC and WG/SH mixtures in general observation of the entire interval.

Quality F/M adhesion same as the formation of transition zone is necessary for the effective stress transfer realisation, hence for the composite to be effective. No signs of quality F/M adhesion was found out for WG/SC. There were some indications of improvement in case of WG/SH after 7 days of hydration but no longer as will be described later. The deterioration of compressive and flexural strengths can be related to many factors beside the poor F/M adhesion. Starting with the presence of local fabric disorientations (leading to ineffective stress transfer, moreover local stress concentration and crack propagation) as described in Arnon’s publication [6]. The influence of poor constitution of the BFs used same as deterioration of fibres mechanical properties along with the decrease of their stress transfer capability due to the degradation under the highly alkaline environments should be considered as well.

![Figure 1. Flexural and compressive strengths development as a function of alkaline activator dosage (6–12% Na$_2$O) and basalt fabric reinforcement for WG/SC activated BFS systems.](image)
3.2. Influence of number of fabric layers on the mechanical properties

Implementation of more fabric layers should be associated with improvement of mechanical properties as also mentioned in study of Du et al. [7]. Hence, the most suitable matrix (BFS activated using the 10% \( \text{Na}_2\text{O} \) WG/SC activator mixture) underwent further testing with increased number of basalt fabrics to two and three. The flexural and compressive strengths development is showed in Figure 3.

The results for samples reinforced with zero or one layer of BFs usually overlap each other considering the standard deviations. Still, one layer of the basalt fabric seems to be the most applicable when the reinforcement is required. Utilization of two or three layers caused lowering of compressive strength during the entire testing period. There was no significant difference between the results for reinforcement with two or three layers. Addition of basalt fabric to the matrix can be accompanied with the formation of lower amount of hydration products. It could explain the compressive strength drop against the non-reinforced reference sample same as the more significant drop with increasing number of fabric layers.

These results partially correlate with the ones presented in Du et al.´s paper [7]. Authors stated that in case of cementitious matrix reinforced with basalt fabric either one or two fabric layers had not exhibit significant reinforcement efficiency. Which is in a good correlation with the results obtained in this paper. On the other hand, Du et al. reported the strain-hardening behaviour when the cementitious matrix was reinforced with three or more layers of basalt fabrics. These results do not match the ones presented here. This difference should not be caused just by other matrix type. It is related to the deterioration of the fibres stress transfer capability due to their degradation. Existence of local stress concentrations caused by local bending of the fabric cannot neither be excluded.
3.3. Fibre/matrix transition zone characterization

Nominal values of the interfacial shear bond strength ($F_i$) should be taken with reserve due to the limited instrumental equipment. Still, it brings interesting option for comparison of different matrices when various samples were tested under the same conditions. Embedment length about 5 mm and curing time of three days were selected as the most suitable to secure the fibre pulling out instead of its breakage while maintaining at least some time for the fibre/matrix interface formation.

The $F_i$ results for various matrices (6–10% Na$_2$O WG/SC; WG/SH AABFS) reinforced with a single yarn of basalt fibres obtained using the pull-out tests are given in Figure 5. $F_i$ increases up to 10% Na$_2$O, but beyond this dosage significant drop was noticed for WG/SC activator mixture, which is in a good correlation with the compressive strengths results. This was expected, because increase of the $F_i$ is closely related to the increase of the compressive strength, respectively mechanical properties development at all. It indicates that fibre resistance against the pull-out is realized mainly due to the frictional bonds whose strength is related to the strength of the matrix itself and do not indicate such a significant contribution of chemical bonds formed between the F/M. The $F_i$ increases with increasing activator dosage from 6 to 12% Na$_2$O for WG/SH activator mixture. It seems that chemical interactions beside the frictional forces are contributing to the total $F_i$ in case of WG/SH AABFS, because the $F_i$ should be lower for the 12% Na$_2$O dosage based on the compressive strength results.

The SEM images (Figure 4) showed that some sort of chemical F/M interaction could be realised for the 10% Na$_2$O WG/SH after 7 days of hydration since the etched surface of the fibres was visible along with the fact that matrix fragments extracted from surrounding matrix were adhered to the fibres surface. However, this was no longer visible after 28 days of curing. So, it cannot be excluded that the presence of protrusions on the fibres could be caused just by lower degree of hydration around the fibres. Still, the 10% Na$_2$O WG/SC AABFS did not show any signs of quality adhesion or F/M interaction at all after 7 or 28 days of curing (there was not observed presence of adhered paste on the surface of the fibres). The surface of the fibres sustained smooth without signs of their rapid degradation.
Figure 4. SEM images showing the detail of F/M interaction for 10% Na$_2$O WG/SC (left) and WG/SH (right) activated BFS pastes reinforced with the BFs after 7 days (top line) and 28 days (bottom line) of curing.

Figure 5. Interfacial shear bond strength development as a function of the activator dosage for WG/SC and WG/SH AABFS systems with fibre EL 5 mm after 72 hours of hydration.
4. Conclusion
There was not observed any significant improvement of tested mechanical properties when the various matrices were reinforced with one or more layers of basalt fabric. This is related to low F/M interaction associated with poor adhesion and ineffective stress transfer realisation or deterioration of fibres stress transfer capability due to their degradation. The presence of local fabrics disorientations cannot be excluded either. Some sort of noticeable improvement was noticed in case of $M_s = 0.5; 10\% \text{Na}_2\text{O} \text{WG/SC AABFS}$ matrix only. Despite these results utilization of basalt reinforcement in suitable form in the AABFS represents a promising possibility to increase the application potential of this material type however further and thorough research must be done to accomplish this goal.

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