Fracture response of alkali-activated slag mortars reinforced by carbon fibres

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Abstract. The paper reports a study of the effect of carbon fibres on the mechanical fracture parameters of alkali-activated slag mortars. The carbon fibres were added in the amount of 1, 2 and 3%, respectively with respect to the mass of the slag. The mechanical fracture parameters were determined using evaluation of fracture tests carried out on 40 × 40 × 160 mm beam specimens with an initial central edge notch. The monitored parameters were compressive strength, modulus of elasticity, effective fracture toughness and specific fracture energy. The specimen response during fracture tests was also monitored by means of acoustic emission. It was shown that as the addition of carbon fibres increased the value of compressive strength and modulus of elasticity of alkali-activated slag dropped to 50% in case of the highest amount of fibres. The effective fracture toughness is not significantly influenced by addition of carbon fibres. On the other hand, the fracture energy value gradually increases with addition of carbon fibres, up to more than twofold increase in case of the highest amount of fibres.

1. Introduction
Nowadays, the binders based on ordinary Portland cement (OPC) are the most commonly used in manufacturing of building materials like concrete, mortars and fine-grained composites. On the other hand, the great emphasis is placed on environmental protection through reductions of CO₂ emissions of which large amount is produced by cement industries. There are two feasible ways how to reduce the CO₂ emissions in production of above mentioned building materials. The first one is application of secondary raw materials as supplementary cementing materials [1]. The other way is use the alkali-activated binder, this type of material is even more effective in reducing CO₂ emissions and energy consumption [2, 3]. These binders are formed by mixing of some aluminosilicate based material, such as blast furnace slag or fly ash with an alkaline activator and water [4, 5]. The mechanical properties and application possibilities of materials based of alkali-activated binders are very similar to those based on OPC. Their major disadvantage is an increased shrinkage during hardening period, caused by both autogenous and drying shrinkage, which finally results in volume contraction, microcracking and deterioration of tensile and bending properties [6]. The addition of different types of fibres into alkali-activated matrix might lead to reduced cracking tendency and improvement tensile properties of these materials as in case of OPC based materials.

Only few papers concerning mechanical properties of alkali-activated slag composites with carbon fibres have been published so far [7–10]. They are focused mainly on determination of compressive and flexural strengths but unfortunately no information on the fracture properties of these composites is available in the literature. Considerable discrepancies in the influence of carbon fibres addition on...
the compressive strength were reported. While Vilaplana or Cui [9, 10] observed an improvement of mechanical properties with increasing amount of fibres, Alcaide et al. [8] reported strength deterioration of alkali-activated slag paste with 1 and 3% of carbon fibres.

Since no fracture tests on alkali-activated slag mortars with carbon fibres have been performed yet the main objective of this work was to determine the basic mechanical fracture parameters of these composites and analyse the effect of carbon fibres on fracture behaviour.

2. Material and specimens

The alkali-activated slag was composed of granulated blast furnace slag supplied by Kotouč, s.r.o. (CZ) finely ground to the specific surface of 383 m²/kg with the mean particle size of 15.5 μm and solid sodium silicate Susil MP 2.0 (Vodní sklo, CZ) as activator. The molar Na₂O/SiO₂ ratio of the silicate activator is 2.0 and the SiO₂ content is 52.4%. Quartz sand with maximum grain size of 2.5 mm was used as aggregate in order to prepare AAS mortar. The carbon fibres Tenax A HT C124 with the average length of 3 mm were added in the amount of 1, 2 and 3% respectively with respect to the mass of the slag. Since carbon fibres have nonpolar character 2% solution of Triton X-100 was used for easier dispersion of fibres in water. The designation of individual mixtures is based on carbon fibres content: CF0, CF1, CF2, and CF3. The mixture CF0 is reference without carbon fibres content.

For the preparation of the specimens, the following procedure was applied. At first, carbon fibres were dispersed in part of mixing water together with Triton X-100. Then, sodium silicate activator, slag and quartz aggregate was added and the mixture was stirred in a planetary mixer for about 7 min to prepare a fresh mixture. Finally, defoaming agent Lukosan S was added in order to detrain air bubbles from the fresh mixture. The amount of activator added was 20% of the mass of slag and an aggregate/slag ratio was 3.0 and water content varied with the amount of carbon fibres to achieve an accurate consistency. The composition of mixtures is presented in Table 1.

| Mixture component  | Unit | CF0 | CF1 | CF2 | CF3 |
|--------------------|------|-----|-----|-----|-----|
| Slag               | g    | 450 | 450 | 450 | 450 |
| Sodium silicate    | g    | 90  | 90  | 90  | 90  |
| Aggregate          | g    | 1350| 1350| 1350| 1350|
| Carbon fibres      | g    |    | 4.5 | 9.0 | 13.5|
| 2% Triton X-100    | g    |    | 5   | 10  | 15  |
| 1% Lukosan S       | g    |    | 5   | 10  | 15  |
| Water              | ml   | 180 | 210 | 235 | 270 |

The mixtures were cast into prismatic moulds of the size 40 × 40 × 160 mm. After 24 hrs the hardened specimens were immersed in a water bath at 20°C for another 27 days. Before the fracture tests were performed, all specimens were pulled out of the water and allowed to dry spontaneously under ambient conditions for 24 hrs.

3. Fracture tests

The three-point bending fracture tests were conducted on beam specimens with initial stress concentrator made by a diamond blade saw before testing. The depth of initial edge notch on the bottom side of specimen was approximately 1/3 of specimen depth. The span length was 120 mm. Fracture experiments were carried out on a very stiff LabTest 6-1000.1.10 multi-purpose mechanical testing machine with displacement control. If displacement increment loading of specimen is performed, it is possible record the load vs. displacement (F–d) diagrams using induction sensors connected in HBM SPIDER 8 device during the testing.
At the beginning of the specimen loading, there are quite often recorded small fluctuations in
the measured values. These are caused by the contact of specimen with the supports, pushing it into
the supports, and crushing of small protrusions on the specimen’s surface. These phenomena usually
soon stop, and the diagram continues with a linear part. Therefore, it is advisable to correct the beginning
part of the measured diagram, to obtain the proper estimation of the modulus of elasticity value. The first
step is approximation of the linear part of diagram with a straight line, follow by determination of
the intersection of this line with the horizontal axis, and then all points of the diagram are shifted
equidistantly, thus the intersection becomes the origin of the coordinate system. The processing of
measured diagrams including the above-mentioned phenomena was performed in GTDiPS software
[11], which uses advanced transformation methods to process extensive point sequences. The aim of
the corrections of the measured diagrams was primarily to shift the origin of the coordinate system,
the smoothing of the diagram and the reduction of the number of points. For this purpose, a chain of
transformation steps has been put together in the software. The selected $F–d$ diagrams, processed in
above mentioned way, with well recorded descending part are shown in Figure 1.

![Figure 1](image.png)

**Figure 1.** Selected $F–d$ diagrams of alkali-activated mortar specimens tested at three-
point bending.

After the above described corrections of measured $F–d$ diagrams, their initial parts were used to
estimate the modulus of elasticity values. Furthermore, the effective crack elongation and effective
fracture toughness values were determined using the Effective Crack Model [12], which combines
the linear elastic fracture mechanics and crack length approaches. The work of fracture and specific
fracture energy values were obtained from the whole $F–d$ diagrams according to the RILEM method
[13]. The informative compressive strength values were also determined on the fragments remaining
after the fracture experiments had been performed.

To describe acoustic emission signals formed during the three-point bending test, the attention was
focused on the number, duration, amplitude and energy of AE signals [14]. The AE signals were
recorded by measuring equipment DAKEL XEDO with four acoustic emission sensors IDK-09 with 35
dB preamplifier, which were attached to the specimen surface by beeswax. The number of AE events
responds to the material ability to withstand the fracture. The duration of an AE signal is the time
difference between the crossing of the first and last threshold. The amplitude is the highest measured
voltage in a waveform. This is an important parameter in AE inspection because it determines
the detectability of the signal. Signals with amplitudes below the operator-defined limit are not recorded.
Energy of AE signals is defined as an area under the envelope of the rectified linear voltage time signal from the transducer [15].

4. Results and discussion
The mean values (obtained from 3 independent measurements) and standard deviations (presented as error bars) of the informative compressive strength values of the tested alkali-activated slag mortars are depicted in Figure 2 (left). The compressive strength value decreased with addition of 1, 2 and 3% of carbon fibres by 5, 23 and 56%, respectively, which is in contradiction with some data reported in literature [9, 10]. It can be assumed that lower compressive strength value of alkali-activated slag mortars reinforced by carbon fibres is due to matrix higher porosity compared to denser matrix of non-reinforced mortar. However, the main difference is probably caused by the level of fibre dispersion, i.e. the way of pre-treatment, mixing and the type of dispersing agent. Ultrasonic treatment applied by Vilaplana [9] probably led to very well dispersed fibre mixture which resulted in the improvement of mechanical properties, Alcaide [8] used methylcellulose as dispersing agent which probably led to insufficient dispersion, thus lower strengths.

Figure 2. Compressive strength (left) and modulus of elasticity values of alkali-activated mortars with different amount of carbons fibres.

The mean values and standard deviations of selected mechanical fracture parameters obtained from recorded $F$–$d$ diagrams in above described way [12, 13], i.e. modulus of elasticity, effective fracture toughness and specific fracture energy, are summarized in Figure 2 (right) and Figure 3. Modulus of elasticity values increased with addition of 1% of carbon fibres by 10%. For higher amount of carbon fibres, the similar decrease of modulus of elasticity value was achieved as in case of compressive strength. In comparison to other parameters, the effective fracture toughness value is not significantly influenced by addition of carbon fibres, see Figure 3 (left). The difference with addition of carbon fibres is about 10% in comparison with reference mortar. On the other hand, the fracture energy value gradually increases with addition of carbon fibres, up to more than twofold increase in case of the highest amount of fibres. It follows that addition of carbon fibres into alkali-activated matrix does not have any significant effect on stable crack propagation. On the other hand, after the maximum load is reached more energy is needed to unstable crack propagation with addition of carbon fibres into the matrix, which can be connected with bridging of the crack by fibres.
Figure 3. Effective fracture toughness (left) and specific fracture energy values of alkali-activated mortars with different amount of carbons fibres.

Table 2. Mean values of selected AE parameters (coefficients of variation in %).

| Parameter                  | Unit        | CF0  | CF1  | CF2  | CF3  |
|----------------------------|-------------|------|------|------|------|
| Number of AE events        |             | 264  | 308  | 224  | 171  |
|                            |             | (0.6)| (0.4)| (1.2)| (0.8)|
| Duration of AE signals     | μs          | 1316 | 1177 | 1171 | 1171 |
|                            |             | (0.1)| (0.1)| (0.1)| (0.4)|
| Amplitude of AE signals    | mV          | 2861 | 2103 | 2183 | 2249 |
|                            |             | (0.2)| (0.3)| (0.6)| (0.2)|
| Energy of AE signals 10⁻⁵ | V·s         | 252.2| 221.8| 134.4| 94.6 |
|                            |             | (1.4)| (0.2)| (0.4)| (0.6)|

The mean values and coefficient of variations of selected parameters of AE signals obtained from AE measurements during the fracture tests are summarized in Table 2. The number of AE events values increased with the addition of carbon fibres in amount 1% by 17%. For the higher amount of carbon fibres, the decrease of the number of AE events value was recorded, the observed trend was similar as in case of modulus of elasticity values. The duration of AE signals values decreased with the addition of carbon fibres. The signal damping was 11% in comparison with reference specimens. The values of AE signal amplitude decreased with the addition of carbon fibres. Lower values of amplitude probably represent the formation of smaller microcracks. The energy of AE signal values gradually decreased with increasing amount of carbon fibres addition.

5. Conclusions
The aim of this study was to quantify the effect of carbon fibres as conductive filler on the fracture properties of alkali-activated slag mortar. Although carbon fibres were primarily added in order to enhance the electrical properties of the building material, it should not deteriorate its mechanical fracture properties. On the contrary, the addition of fibres into alkali-activated matrix should lead to a reduction in cracking tendency and improvement in tensile properties of these materials. The specimen response during fracture tests was also monitored by means of acoustic emission.

It was shown that as the addition of carbon fibres increased the value of compressive strength and modulus of elasticity of alkali-activated slag dropped to 50% in case of the highest amount of fibres.
This might have been caused by insufficient dispersion of carbon fibres in the alkali-activated slag matrix. The effective fracture toughness is not significantly influenced by addition of carbon fibres. On the other hand, the fracture energy value gradually increased with addition of carbon fibres, up to more than twofold increase in case of the highest amount of fibres.

The processed AE data showed that addition of carbon fibres affects mechanical properties; in a number of cases the decrease of these parameters was recorded. The work confirms that analysis of AE signals obtained during the fracture tests of alkali-activated composites provides additional information about composite damage and can be used to better understand the effects of carbon fibres addition to alkali-activated composites.

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References
[1] Keun-Hyeok Y, Yeon-Back J, Myung-Sug C and Sung-Ho T 2015 Effect of supplementary cementitious materials on reduction of CO2 emissions from concrete J Cleaner Prod 103 pp 774–783
[2] Keun-Hyeok Y, Jin-Kyu S and Keum-II S 2013 Assessment of CO2 reduction of alkali-activated concrete J Cleaner Prod 39 pp 265–272
[3] McLellan B C, Williams R P, Lay J, van Riessen A and Corder G D 2011 Costs and carbon emissions for geopolymer pastes in comparison to ordinary Portland cement J Cleaner Prod 19(10) pp 1080–1090
[4] Provis J L and van Deventer J S (eds) 2014 Alkali activated materials: state-of-the-art report, RILEM TC 224-AAM (Dordrecht: Springer)
[5] Shi C, Krivenko P V and Roy D 2006 Alkali-activated cements and concretes (London: Taylor & Francis)
[6] Cincotto M A, Melo A A and Repette W L 2003 Effect of different activators type and dosages and relation with autogenous shrinkage of activated blast furnace slag cement Proc. of the 11th International Congress on the Chemistry of Cement (Durban, South Africa) pp 1878–1888
[7] Natali A, Manzi S and Bignozzi M C 2011 Novel fiber-reinforced composite material based on sustainable geopolymer matrix Procedia Engineering 21 pp 1124–1131
[8] Alcaide J S, Alcocel E G, Puertas F, Lapuente R and Garces P 2007 Carbon fibre-reinforced, alkali-activated slag mortars Materiales de Construccion 57 pp 33–48
[9] Vilaplana J L, Bazea F J, Galao O., Alcocel E G, Zornoza Z and Garces P 2016 Con Build Mat 116 pp 63–71
[10] Cui H, Feng T, Yang H, Bao X, Tang W and Fi J 2018 Con Build Mat 161 pp 442–451
[11] Frantík P and Mašek J 2015 GTDiPS software http://gtdips.kitnarf.cz/
[12] Karihaloo B L 1995 Fracture Mechanics and Structural Concrete (New York: Longman Scientific & Technical)
[13] RILEM TC-50 FMC Recommendation 1985 Determination of the fracture energy of mortar and concrete by means of three-point bend test on notched beams Materials & Structures 18 pp 285–290
[14] Grosse C U and Ohtsu M 2008 Acoustic Emission Testing: Basics for Research – Applications in Civil Engineering; with contributions by numerous experts (Heidelberg: Springer)
[15] Sagar R V 2009 An experimental Study on Acoustic Emission Energy and Fracture Energy of Concrete Proc. of the National Seminar & Exhibition on Non-Destructive Evaluation (Tiruchirappalli, India) pp 225–228