Influence of curing conditions on mechanical and fracture properties of alkali activated slag concrete

H Šimonová, B Kucharczyková, V Bílek Jr., L Topolář and D Kocáb

1 Brno University of Technology, Faculty of Civil Engineering, Veveří 331/95, 602 00 Brno, Czech Republic
2 Brno University of Technology, Faculty of Chemistry, Purkyněova 464/118, 602 00 Brno, Czech Republic

E-mail: simonova.h@vutbr.cz

Abstract. The aim of this work is to determine the physical, mechanical and fracture characteristics of concrete specimens with alkali activated binder cured at different conditions. The concrete mixture with alkali activated ground granulated blast furnace slag was selected for the purpose of the experiment. The binder was activated by the liquid sodium silicate with the silicate modulus of 2.0. Natural sand and gravel were used as a fine and coarse aggregate, respectively. The maximum aggregate size was 16 mm. The binder : aggregate : water ratio was 1 : 3 : 0.46. During the specimens ageing, the dynamic modulus of elasticity was determined using a resonance method. The fracture characteristics were determined based on the results of the three-point bending test of specimens provided with an initial central edge notch. The fracture experiments were conducted at the age of 3 and 38 days. The loading force \( F \), the displacement measured in the middle of the span length \( d \), and the crack mouth opening displacement \( CMOD \) were continuously recorded during the test. The records of fracture tests were subsequently evaluated using the effective crack model, work-of-fracture method and double-\( K \) fracture model. The fracture tests were also accompanied by the monitoring of acoustic responses using the acoustic emission method.

1. Introduction
Alkali-activated materials are formed via dissolution of a suitable aluminosilicate precursor in presence of an alkaline activator, typically sodium hydroxide or sodium water glass. The dissolved species are polycondensates and they are thus rearranged to a binding phase. Their composition depends on the nature of activator and on the composition of the aluminosilicate precursor. Activation of low-calcium precursors like fly ashes and metakaolin result in the formation of the three-dimensional structure of sodium-aluminate-silicate-hydrates (NASH) while activation of high-calcium precursors, typically slag, leads to the formation of calcium-aluminium-silicate-hydrates (CASH) which are more similar to CSH phases formed during the Portland cement hydration. Differences in the structure and composition result also in the differences in their properties [1].

Alkali-activated slag (AAS) is popular because of its high early age strengths easily obtained at room temperatures but can suffer from the fast setting time and particularly from shrinkage and cracking. Despite many approaches to reduce shrinkage have been done more or less successfully, among others e.g. partial slag substitution by mineral admixtures [2], internal curing [3] or curing at elevated
temperatures [4] as well as the use of shrinkage-reducing admixtures [5], high cracking tendency of AAS is still an issue.

Ideally, cracking does not take place but if it does, there is an option to reduce its extent and consequences thanks to self-healing effect. For Portland cement-based materials improvement of self-healing ability various approaches including chemical admixtures, bacteria or microencapsulation has been used [6]. On the other hand, the self-healing phenomenon in AAS-based materials has been poorly investigated, despite it has certain potential. Moreover, AAS is very sensitive to curing conditions and therefore in the present study fracture parameters of AAS with different curing conditions and cracking development were determined.

2. Material and specimens

Ground granulated blast furnace slag with Blaine fineness of 400 m²/kg was used as a precursor for alkaline activation. According to X-ray diffraction, about 90% was amorphous phase and the rest were akermanite and calcite. For slag activation water glass having silicate modulus of 2.0 and solid content of 46.8 was used at the dose corresponding to 8% of Na₂O with respect to the slag weight. Three fractions, i.e. 0–4, 4–8 and 8–16 mm, of natural aggregate at the proportions given by Füller granulometric curve were used. Slag : aggregate : water ratio was 1 : 3 : 0.46 including water contained in water glass. Before the addition of solids, water glass with water were mixed together. Then the slag was added followed by an addition of aggregate. Total mixing time since the addition of slag to water was 6 minutes.

The AAS concrete mixture was cast into the polyethylene moulds with dimensions of 100 × 100 × 400 mm to prepare prismatic specimens intended for the fracture tests. The moulds were covered with a thin PE foil and stored in the laboratory with ambient temperature of 21 ± 2 °C and relative humidity of 50 ± 10 % for 24 hours. After that, all specimens were demoulded and left to dry freely at the same laboratory conditions. After another 24 hours when specimens were drying freely, the cracks caused by shrinkage appeared at specimen’s surfaces, see figure 1 (left). At this age (48 hours), the specimens were divided into two groups. One group of the test specimens was left to dry freely for the entire time of aging. The second group was immersed in the water-bath for the predetermined curing time, after which the specimens were again removed from the bath and left to dry freely until the testing time. This procedure was chosen to prevent the crack development and to start the self-healing process in AAS concrete specimens.

![Figure 1](image_url)

Figure 1. Specimen’s surface at the age of 2 days (left), 27 days stored at air (in the middle) and 27 days stored under water conditions.
The results of the fracture tests of the specimens stored at the air conditions performed at the age of 3 days were taken as the initial values of investigated parameters. To examine the influence of the curing conditions, the fracture tests were performed for both sets of specimens at the age of 38 days. The specimens cured in water were removed from the water bath at the age of 27 days and were left to dry freely until the testing time. Based on the curing conditions and the age of specimen when fracture tests were performed, the sets of specimens are designated as follows: 3_days_air, 38_days_air and 38_days_water. Each testing set consists of three specimens.

3. Methods

3.1. Resonance method
During the specimens ageing, the dynamic modulus of elasticity was monitored and determined using the resonance method according to the standard ČSN 73 1372 [7]. The natural frequency of longitudinal vibration was determined for each specimen. The vibration was generated by a mechanical impulse using the impact hammer and the natural frequency was measured by an oscilloscope Handyscope HS4 with a piezoelectric sensor. The dynamic modulus of elasticity $E_{dy}$ was then calculated from this natural frequency according to the standard [7].

3.2. Fracture test
The fracture characteristics were determined based on the results of the three-point bending test of specimens provided with an initial central edge notch. The notch was made with a diamond blade saw and its depth was approximately equal to 1/3 of the depth of the specimen. The span length was equal to 350 mm. The fracture experiments were performed at the age of 3 and 38 days. For this purpose, a very stiff multi-purpose mechanical testing machine LabTest 6.250 with the load range of 0–250 kN was used, see figure 2. The loading force ($F$), the displacement measured in the middle of the span length ($d$), and the crack mouth opening displacement (CMOD) were continuously recorded during the test with a frequency of 5 Hz. All performed fracture test were accompanied by the measurements using the acoustic emission technique.

Figure 2. The three-point bending fracture test configuration (left); overall view of the testing machine.

3.2.1. Processing of measured data. At the beginning of loading, small-sized fluctuations in the measured values of monitored parameters are often recorded. This effect is caused by the crushing of small protrusions on the specimen’s surface due to the pressure at the support and loading points. Therefore, it is advisable to correct the beginning part of the measured diagram in order to obtain the appropriate estimation of the modulus of elasticity value.
In the case of specimen made of composite with a brittle matrix, subjected to the displacement-controlled loading, stability loss can occur due to the insufficient stiffness of the test equipment in comparison to the specimen’s stiffness. This stability loss leads to an interruption in record of the $F$–$d$ and $F$–CMOD curves. Therefore, a procedure has been developed to identify and correct the data discontinuity in the case of fracture tests of specimens based on the brittle matrix [8]. The work-of-fracture and fracture energy values determined from measured diagrams without this correction could be overestimated.

The processing of measured diagrams including the above-mentioned phenomena was performed in GTDiPS software [9] which uses advanced transformation methods to process extensive point sequences. The correction of the measured diagrams in this case included primarily the shifting of the origin of the coordinate system, the smoothing of the diagram and the reduction of the number of points. For illustration, the figure 3 introduces the $F$–$d$ and $F$–CMOD diagrams after the advanced correction.

**Figure 3.** The corrected $F$–$d$ and $F$–CMOD diagrams.

3.2.2. **Evaluation of $F$–$d$ diagrams.** After the above described processing of recorded diagrams, at first the initial parts of $F$–$d$ diagrams were used to estimate the modulus of elasticity $E_c$ value according to [10]. The fracture toughness value $K_{ic}$ was determined using the effective crack model [10] which combines the linear elastic fracture mechanics and crack length approaches. To determine $K_{ic}$, the effective crack length $a_e$, corresponding to the maximum load $F_{max}$ and mid-span displacement $d$, had to be calculated first. The work-of-fracture $W_f$ value was obtained from the whole $F$–$d$ diagrams according to the RILEM method [11] and corresponds to area under the $F$–$d$ diagram. Subsequently, the specific fracture energy $G_f$ value was determined, that the $W_f$ values were divided by area of ligament (cross-section of specimens through which the crack grows).

3.2.3. **Evaluation of $F$–CMOD diagrams.** The $F$–CMOD diagrams (a part until the peak load) were subsequently evaluated using the double-$K$ fracture model [12]. The advantage of this model is that the double-$K$ fracture criterion can predict the crack initiation, stable crack propagation, and unstable fracture process during the crack propagation in quasi-brittle material. According to this criterion, the two size-independent parameters termed as initial cracking toughness $K_{ic}^{ini}$ and unstable fracture toughness $K_{ic}^{un}$ can predict different stages of the fracture process. This model is based on combination of the concept of cohesive forces acting on the faces of the fictitious (effective) crack increment with a criterion based on the stress intensity factor (details can be found in numerous publications – e.g. in Kumar and Barrai [12]). Finally, the value of the load $F_{ini}$ is determined according to [12]. This value can be defined as the load level at the beginning of stable crack propagation from the initial crack/notch.
3.3. *Acoustic emission method*

The acoustic emission (AE) method can be used for monitoring the fracture process of specimen during the three-point bending test. AE belongs to the non-destructive testing techniques [13]. Unlike other techniques, AE is a passive control method that can examine the entire volume structure of construction, structural member or – as in this case – of the specimen. The AE method detects only active damages, which begin to develop during the test. The AE activity arises from the rapid release of energy in a material because of stimulation by internal or external stress [14, 15]. To describe acoustic emission signals formed during the fracture test, the number of events $N_{AE}$, duration $D_{AE}$, amplitude $A_{AE}$ and energy $E_{AE}$ of AE signals were monitored and recorded.

3.4. *Basic mechanical parameters determination*

The informative compressive strength $f_c$ value was determined on the fragments remaining after the fracture experiments of AAS composites had been performed. The flexural strength $f_f$ value was estimated based on the maximum force recorded during the three-point bending fracture test. The tensile strength $f_t$ value was identified from $F$–$d$ diagrams using the artificial neural network (ANN) based on the inverse analysis method [16, 17].

4. *Results and discussion*

The average value (obtained usually from 3 independent measurements) and the coefficient of variations CoV of selected mechanical, fracture and acoustic emission parameters of AAS concretes cured at the different conditions are summarized in following tables.

Table 1 introduces the value of dynamic modulus of elasticity $E_{dyn}$ during the specimens ageing cured at the different conditions. All sets of test specimens exhibit the same $E_{dy}$ value at the age of 2 days before they are placed into different curing condition (see Table 1). In the case of air-stored specimens, the $E_{dyn}$ value slightly increases with specimens age (until the age of 7 days). Further desiccation and development of shrinkage cracks cause a gradual decrease in the value of this parameter (see Table 1). At the age of 34 days the value of $E_{dyn}$ was almost the same as at the age of 3 days. In the case of specimens cured in the water bath, the $E_{dyn}$ value gradually increases up to more than two times in comparison to specimens stored at air conditions. This increase is caused by presence of water and mainly by the self-healing process of AAS concrete. After the specimens are removed from the water bath at the age of 27 days and left to dry freely until the age 38 days, when the fracture tests are performed, the $E_{dyn}$ value decreases slightly. However, it is still about 75% higher than the value of the air-stored specimens.

| Specimen’s age | 2 days | 7 days | 16 days | 23 days | 28 days | 34 days |
|----------------|--------|--------|---------|---------|---------|---------|
| 3_days_air     | 11.6 (2.0) | –      | –       | –       | –       | –       |
| 38_days_air    | 11.6 (3.1) | 13.8 (1.2) | 12.6 (1.3) | 12.2 (1.5) | 12.2 (2.5) | 11.9 (1.4) |
| 38_days_water  | 11.8 (1.9) | 22.1 (1.5) | 26.3 (1.0) | 27.9 (0.7) | 24.6 (0.6) | 21.1 (0.4) |

Table 2. The average value of basic material properties (CoV in %) of AAS concrete.

| Parameter/Unit | 3_days_air | 38_days_air | 38_days_water |
|----------------|------------|-------------|---------------|
| $\gamma$ [kg/m$^3$] | 2280 (0.9) | 2270 (0.3) | 2270 (0.3) |
| $f_c$ [MPa] | 30.3 (0.3) | 40.2 (3.5) | 72.8 (1.4) |
| $f_t$ [MPa] | 1.98 (9.4) | 1.80 (7.6) | 3.72 (5.3) |
| $f_r$ [MPa] | 1.75 (9.6) | 1.69 (1.6) | 2.55 (2.2) |
Table 2 introduces the basic physical and mechanical parameters, namely the bulk density \( \gamma \), compressive \( f_c \), flexural \( f_f \) and tensile \( f_t \) strengths values. The bulk density was determined before the fracture tests were performed. The value of bulk density is not influenced by curing conditions and age of specimens. In the case of air-stored specimens, the compressive strength value increases with the specimen's age by about 10 MPa. On the contrary, the tensile and flexural strength values slightly decrease up to 10% because of further development of cracks caused by shrinkage. In the case of specimens cured in the water bath, the value of compressive, flexural and tensile strength increases by about 80, 110 and 50%, respectively in comparison with air-stored specimens.

Table 3 introduces the selected mechanical fracture parameters determined based on the above introduced non-linear fracture models. In the case of air-stored specimens, gradual development of cracks caused by shrinkage has a negative effect on the modulus of elasticity value \( E_c \) determined from the fracture test. The \( E_c \) value decreases by about 40% at the age of 38 days. On the other hand, the water curing has a positive effect and the \( E_c \) value is more than two times higher in comparison with the air-stored specimens. The fracture toughness value was determined using two different fracture models. Both models give the same results. In the case of air-stored specimens, the fracture toughness value decreases by about 15% with specimens age. Comparing the results at the age of 38 days, the fracture toughness and specific fracture energy values are by about 80% and 65% higher for specimens stored in the water-bath. The resistance to stable crack propagation, in this case expressed by \( K_{\text{ic}}\text{m} / K_{\text{ic}}\text{un} \) ratio, does not change with specimen's age in the case of air-cured specimens. In the case of the water-cured specimens, the resistance to stable crack propagation is more than two times higher in comparison with the air-cured specimens. A similar trend is observed in the case of the load ratio \( F_{\text{mu}} / F_{\text{max}} \), i.e. the ratio between the load level at the beginning of stable crack propagation and maximum load obtained during the test. The presence of cracks caused by shrinkage in air-stored AAS concrete leads to beginning of the stable crack propagation at earlier level. From obtained results, it is evident that water curing has a positive effect on both stable and unstable crack propagation and also on the post peak behaviour of AAS concrete. It is related to the self-healing process in the AAS concrete, when the cracks caused by early-age shrinkage are gradually healed up due to the presence of curing water.

It is well-known that alkali-activated materials need long time to achieve their final properties. However, they also need sufficient amount of water because water is a necessary medium for the dissolution and condensation reactions responsible for their hardening. Therefore, if they are left to dry, the mentioned reactions leading to the formation of the main binding phase (CASH) are suppressed and the properties of such material do not significantly change in time or even rather decrease as a consequence of ongoing shrinkage and other degradative processes, e.g. carbonation. On the other hand, if the specimens are returned to water, their hydration can continue which results in the further formation of CASH. It can grow in the pores but also inside the previously developed cracks and thus we expect that the origin of the observed self-healing effect lies in the formation of CASH. Hence, ongoing hydration itself but also the related self-healing are the main reasons for the increase in all fracture parameters of the investigated concretes immersed in water after the cracking.

**Table 3.** The average value of mechanical fracture parameters (CoV in %) of AAS concrete.

| Parameter/Unit | 3_days_air | 38_days_air | 38_days_water |
|----------------|------------|-------------|---------------|
| \( E_c \) [MPa] | 9.9 (11.6) | 6.1 (9.7) | 12.5 (7.8) |
| \( K_{\text{ic}}\text{m} \) [MPa-m\(^{1/2}\)] | 0.550 (15.5) | 0.471 (3.3) | 0.842 (3.3) |
| \( G_f \) [J/m\(^2\)] | 82.9 (6.2) | 90.6 (10.5) | 149.2 (9.9) |
| \( K_{\text{ic}}\text{un} \) [MPa-m\(^{1/2}\)] | 0.558 (11.1) | 0.462 (11.9) | 0.833 (4.1) |
| \( K_{\text{ic}}\text{m} / K_{\text{ic}}\text{un} \) [-] | 0.182 (30.8) | 0.191 (48.6) | 0.410 (8.2) |
| \( F_{\text{mu}} / F_{\text{max}} \) [-] | 0.322 (24.8) | 0.309 (40.3) | 0.595 (2.0) |
Table 4 introduces the selected parameters of AE signals obtained during the fracture tests. In the case of air-stored specimens, the number of AE events $N_{AE}$ decreases with specimens ageing. It is related to the nature of AE method, which detects only the active damages. The higher amount of cracks (damage of material) caused by shrinkage during specimens ageing leads to easier crack propagation through the specimens and therefore the detected number of AE event is lower. On the other hand, in the case of specimens stored in the water-bath, the number of AE events is higher because of the more compact structure of material and therefore a higher number of new cracks is initiated and propagated before the fracture. The maximum amplitude of AE signal $A_{AE}$ is connected with the intensity of AE event source. In general, the AE amplitude corresponds to the extent of fracture, whereas the energy emitted from the AE source depends on the crack propagation; small displacement emits waves of low energy, while large crack propagation generate higher amounts of energy [18]. This corresponds to obtained results, the lowest value of amplitude and energy of AE signals is detected in the case of air-stored specimens at the age of 38 days because of the material damage caused by shrinkage.

Table 4. The average value of acoustic emission parameters (CoV in %) of AAS concrete.

| Parameter/Unit | 3_days_air | 38_days_air | 38_days_water |
|----------------|------------|-------------|---------------|
| $N_{AE}$ [-]   | 38 (10.5)  | 8 (50.0)    | 64 (21.7)     |
| $D_{AE}$ [μs]  | 1355 (1.4) | 1648 (3.7)  | 1376 (4.8)    |
| $A_{AE}$ [mV]  | 1191 (0.8) | 861 (26.5)  | 1561 (15.9)   |
| $E_{AE}$ [$10^{-3}$V∙s] | 17.0 (22.9) | 4.7 (34.3)  | 41.4 (17.7)   |

5. Conclusions
The aim of this study was to quantify the effect of different curing conditions on the physical, mechanical and fracture characteristics of concrete specimens with alkali activated binder. One group of the test specimens was left to dry freely for the whole time of ageing. The second group was immersed in the water-bath for a predetermined curing time, after which the specimens were again removed from the bath and left to dry freely until the testing time. This procedure was chosen to prevent the crack development caused by shrinkage and to start the self-healing process in AAS concrete specimens.

From the presented experimental results, the positive effect of water curing on all monitored parameters is evident. Additional fracture tests will be performed after the dynamic modulus of elasticity value of the water-cured specimens will be stabilized.

Acknowledgments
This outcome has been achieved with the financial support of the Czech Science Foundation under project No 18-12289Y.

References
[1] Provis J L and van Deventer J S J (eds.) 2014 Alkali Activated Materials: State-of-the-Art Report, RILEM TC 224-AAM (Dordrecht: Springer Netherlands)
[2] Aydin S 2013 Construction and Building Materials 43 pp 131–138
[3] Sakulich A and Bentz D 2013 Materials and Structures 46 pp 1355–1367
[4] Aydin S and Baradan B 2012 Materials & design 35 pp 374–383
[5] Palacios M and Puertas F 2007 Cement and Concrete Research 37 pp 691–702
[6] Nguyen H H etal. 2018 Construction and Building Materials 165 pp 801–811
[7] ČSN 73 1372 2012 Non-destructive testing of concrete – Testing of concrete by resonance method (Prague: ÚNMZ)
[8] Frantík P, Průša J, Keršner Z and Macur J 2007 About stability loss during displacement-controlled loading Fibre Concrete (Prague) pp 99–102
[9] Frantík P and Mašek J 2015 GTDiPS software http://gtdips.kitnarf.cz/
[10] Karihaloo B L 1995 Fracture Mechanics and Structural Concrete (New York: Longman Scientific & Technical)
[11] RILEM TC-50 FMC Recommendation 1985 Materials & Structures 18 pp 285–290
[12] Kumar S and Barai S V 2011 Concrete Fracture Models and Applications (Berlin: Springer)
[13] Grosse C U and Ohtsu M 2008 Acoustic Emission Testing (Heidelberg: Springer)
[14] Ohtsu M 2015 Acoustic emission (AE) and related non-destructive evaluation (NDE) techniques in the fracture mechanics of concrete (Cambridge: Woodhead Publishing)
[15] Sagar R V 2009 Proc. of the National Seminar & Exhibition on Non-Destructive Evaluation (Tiruchirappalli, India) pp 225–228
[16] Novák D and Lehký D 2006 Engineering Application of Artificial Intelligence 19 pp 731–740
[17] Lehký D, Keršner Z and Novák D 2014 Advances in Engineering Software 72 pp 147–54
[18] Aggelis D E etal. 2011 Construction and Building Materials 25 pp 4126–4131