Acta Polytechnica CTU Proceedings **47**:71–74, 2024
https://doi.org/10.14311/APP.2024.47.0071

Published by the Czech Technical University in Prague

MONITORING DEGRADATION IN ALKALINE-ACTIVATED SLAG MATERIALS

Houssam Mahmoud*, Libor Topolář, Iveta Plšková, Luboš Jakubka, Luboš Pazdera

Brno University of Technology, Faculty of Civil Engineering, Veveří 331/95, 602 00 Brno, Czech Republic
* corresponding author: 172266@vutbr.cz

Abstract. This paper deals with determining degradation changes in alkaline-activated slag-based building materials. It describes methods for determining dynamic elastic moduli obtained by acoustic non-destructive methods, including the ultrasonic pulse method and the resonance method. A fine-grained mortar was chosen as the initial mix for the implementation of the experiment, the binder part of which was sodium hydroxide-activated blast-furnace granulated slag. Beams with dimensions of $40 \times 40 \times 160$ mm were chosen as test bodies, for which acoustic non-destructive methods monitored changes in the material structure during degradation processes.

Keywords: Degradation, alkali-activated slag materials, non-destructive testing, decalcification, compressive strength.

1. Introduction

The ultrasonic method has been used in the construction industry for many decades, especially for detecting cavities and caverns. It is one of the non-destructive methods of testing building materials, elements, components and structures. The main advantage is that it does not cause any damage to the material, so it cannot compromise the load-bearing capacity or serviceability of the building element. It is repeatable, so changes in the structure over time can be monitored. The essence of this method is the repeated transmission of ultrasonic pulses into the material under test. After the measured path has been traversed, the transducer removes the vibrations. The transit time of the ultrasonic waves, time t, is measured from the sending of the beating by the exciter to the recording of its impact on the sensor [1–3]. Alkali-activated systems are a modern alternative binder material against traditional Portland cement. Their main advantages lie in their environmental friendliness, where, with appropriate adjustment of the mixture composition, a 50–100 % reduction in carbon dioxide emissions can be achieved compared to Portland cement (approximately 1 t CO_2 is produced per 1 t of binder), thanks to the possibility of efficient use of so-called secondary raw materials of aluminosilicate nature, such as the blast furnace slag or high-temperature fly ash used in this work. Compared to Portland cement, they also have other advantages such as increased resistance to aggressive environments, faster onset of strength development or lower released hydration heat. The disadvantage is the high shrinkage compared to Portland cement-based materials [4–6].

2. Experimental setup

To measure the speed of ultrasound (ultrasound) signal passage through the test samples, we used the "Pulse analyzer 58-E4900". The measurement was performed according to ČSN 73 1371 [7]. The test bodies were placed on rubber pads (see Figure 1). A thin layer of plasticine was used as an acoustic binding agent. The measurements were carried out in a plane perpendicular to the compaction direction.

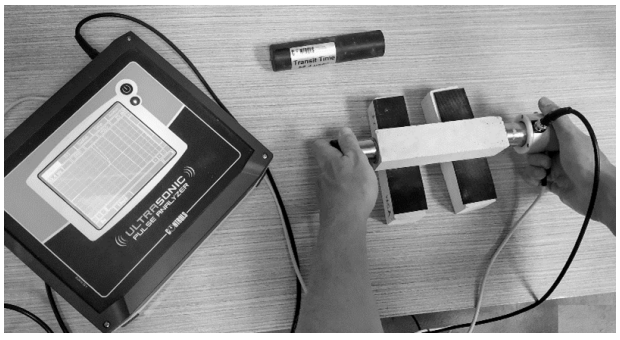

Figure 1. The demonstration of ultrasonic signal velocity measurement.

We recorded the transit time of the UZ wave, from the sending of the pulse by the exciter to recording its impact on the transducer. This speed depends on the type and properties of the material. From the average of the three measured times, the ultrasound propagation velocity v_L was then calculated. From the ultrasound wave propagation velocity, the value of the dynamic elastic modulus E_{bu} was determined using norm relations [1, 7]:

$$E_{bu} = \frac{\rho\, v_L^2}{k^2}\, 10^{-6}, \tag{1}$$

where is

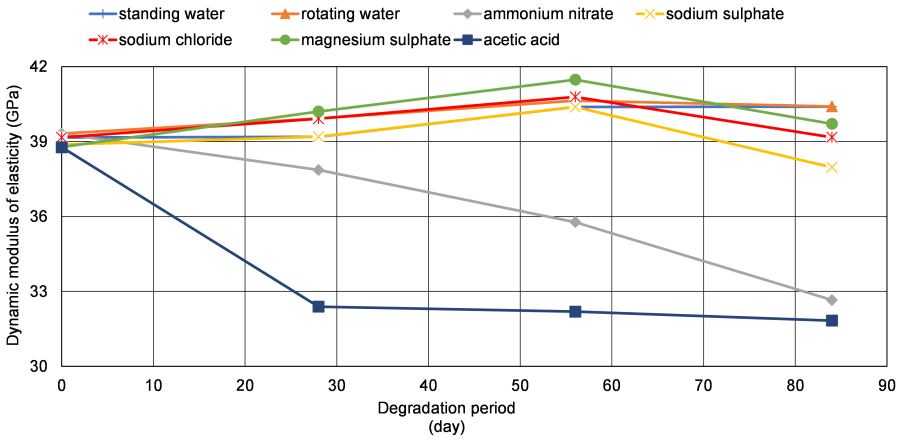

Figure 2. The change of dynamic modulus of elasticity over time for each aqueous solution.

Chemical composition wt. %									
CaO	SiO_2	MgO	Al_2O_3	SO_3	TiO_2	K_2O	MnO	Na_2O	Fe_2O_3
41.1	34.7	10.5	9.1	1.4	1.0	0.9	0.6	0.4	0.3

Table 1. VPS chemical composition by X-ray fluorescence (XRF).

ϱ bulk density of the concrete $[\mathrm{kg\,m^{-3}}]$,
v_L^2 pulse velocity of longitudinal UZ wave propagation $[\mathrm{m\,s^{-1}}]$,
k environmental dimensionality coefficient [-].

Destructive testing was conducted by determining the compressive strength according to EN 196-1 [8] on the Desttest 4310 Compact A (Beton Systém s.r.o.).

3. Composition of specimens and degradation solutions

The starting material was based on the alkaline activation of blast furnace slag with 50 % sodium hydroxide solution. The activator dosage per binder was 6 % Na_2O. A 1 % dose of a lignosulfonate-based plasticizer (ChrysoPlast 461) was used to improve processability. The water coefficient was set to 0.445. Standard aggregate with a maximum grain size of 2 mm was used for mortar preparation. The slag with a specific surface area of $400\,\mathrm{m^2\,kg^{-1}}$ (determined by the Blaine method) was supplied by Kotouč Štramberk s.r.o. The chemical composition of blast furnace slag (VPS) is shown in Table 1. The amorphous fraction of the slag is 84 %; the rest comprises crystalline phases such as acermanite, calcite or merwinite.

The samples were demolded after 24 hours and left to ripen in water storage. After 28 days, the actual testing of the effect of exposure of the test bodies to different degradation media on the monitored parameters was started. The following degradation media were chosen for the degradation study – standing and rotating water, 5 % solutions of selected salts (sodium chloride, sodium sulfate, magnesium sulfate), 6M ammonium nitrate solution and acetic acid solution at pH ≈ 3. The degradation media were changed after 14, 28 and 56 days of leaching of the test bodies. The pH was checked at regular 3-day intervals for the acetic acid solution and adjusted, if necessary, by adding concentrated acetic acid to the desired value. All the chemicals used were at least of a purity class described as pure, i.e., a minimum of 98 % content.

4. Measured results and discussion

From the values obtained by the actual measurements and using Equation 1, the dynamic modulus values were calculated and are shown in the graph in Figure 2. From the curves of the dynamic moduli degraded by the selected solutions, it can be seen that acetic acid and ammonium nitrate solutions had the most significant influence on the value of the dynamic modulus. The ammonium nitrate solution showed a decrease in dynamic modulus values throughout the monitoring period (84 days of degradation). In the case of acetic acid, a sharp reduction in the dynamic modulus values occurred after only 28 days of degradation, with a gradual decrease in the following monitoring period. In the case of the other solutions, there was a slight increase in the dynamic modulus values.

In the case of a different comparison, we arrive at another interesting finding that might not be entirely apparent from the graph in Figure 2. The graph in Figure 3 represents the relative values of the dynamic modulus relative to the initial value (100 %) measured before the samples were loaded into each solution. This shows an apparent decrease in the dynamic modulus values in the case of acetic acid (by more than 15 %) and in the case of ammonium nitrate (successively by 5, 10 and 15 % after 84 days of degradation). Let's look at the changes in the dynamic modulus values when the solutions of sodium sulphate, sodium chloride, and magnesium sulphate are treated. We

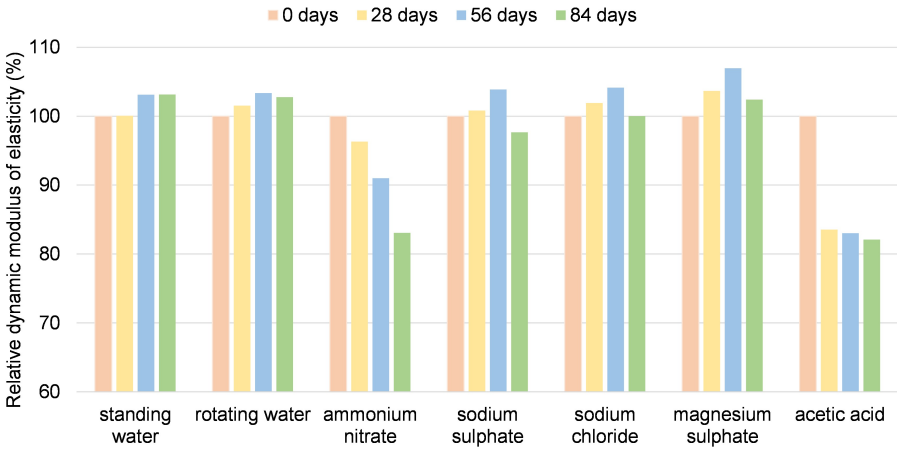

FIGURE 3. Values of relative dynamic modulus of elasticity for each aqueous solution.

Degradation solution	Degradation period (days) 0	28	56	84
standing water		25.2 ± 0.5	25.1 ± 0.2	25.4 ± 0.3
rotating water		22.4 ± 0.2	26.3 ± 0.5	26.5 ± 0.6
ammonium nitrate		22.3 ± 0.5	22.9 ± 0.5	21.2 ± 0.8
sodium sulphate	18.5 ± 0.5	24.0 ± 0.5	26.3 ± 0.5	26.4 ± 0.8
sodium chloride		22.9 ± 0.3	26.8 ± 0.2	27.0 ± 0.7
magnesium sulphate		22.2 ± 0.5	26.4 ± 0.6	27.5 ± 0.3
acetic acid		15.8 ± 0.1	19.6 ± 0.6	16.8 ± 0.2

TABLE 2. Values of compression strength in MPa.

find an increase in the weights after 28 and 56 days of exposure to the samples. When applied for 84 days, there is already a significant decrease and, in the case of sodium sulphate, even below the original initial value. In the case of water (standing and rotating), the resulting dynamic modulus values tend to increase (by 2 to 3 %). Table 2 shows the compressive strength values. It can be seen that for all the solutions studied, the compressive values have an increasing trend. Only in the case of ammonium nitrate, there is an initial increase (28 days) and then a subsequent stagnation (56 days) to decrease (84 days). Only in the case of acetic acid degradation is a decrease in compressive strength values.

The deterioration of mechanical properties of AAS due to leaching in acetic acid solution at pH 3 is related to the process of C-S-H (C-A-S-H) gel decalcification and the associated microstructural change. The released calcium ions react with the acetate anion to form calcium acetate, which is highly soluble and does not reduce the penetration rate due to the formation of a protective layer of a compound with low solubility (e.g. calcium sulphate). The decalcification rate depends on several parameters, the main ones being the porosity of the test bodies or the CaO/SiO_2 ratio, as outlined by Bernal [9]. The decalcification process of the C-S-H gel is also realized in the case of leaching in ammonium nitrate solution. However, its contribution is not as fatal as in the case of CH_3COOH. Another aspect explaining the advanced degradation rate is the decrease in the surface tension of the leaching solution due to the addition of acetic acid, which is associated with an increase in the penetration rate into the pore structure. The results obtained in this work agree with the publication [9], where a significant decrease in compressive strength due to degradation by CH_3COOH (pH 4.5).

5. CONSLUSION

These results indicate that fine-grained mortar based on sodium hydroxide (6 % Na_2O) activated blast furnace slag is most degraded by acetic acid solution (pH ≈ 3). The advanced degradation accompanied by a deterioration of the mechanical properties is closely related to the decalcification of the C-S-H gel and the accompanying microstructural changes. It is also evident that non-destructive testing results are in good agreement with destructive testing.as also observed.

ACKNOWLEDGEMENTS

The Czech Grant Agency financially supported this paper under the project GA 19-04703S, "The use of non-destructive methods for testing of the condition of degraded alkali-activated concrete" and project No. FAST-J-22-8007 supported by Faculty of Civil Engineering of Brno University of Technology.

References

[1] P. Schmid. *Základy zkušebnictví.* Vysoké učení technické v Brně, Stavební fakulta, 2004.

[2] L. Hobst, J. Adámek, P. Cikrle, P. Schmid. *Diagnostika stavebních konstrukcí.* CERM, FAST VUT v Brně, 2005.

[3] O. Anton. *Základy zkušebnictví: návody do cvičení.* CERM, 2002.

[4] K. L. Scrivener, R. J. Kirkpatrick. Innovation in use and research on cementitious material. *Cement and Concrete Research* **38**(2):128–136, 2008. Special Issue — The 12th International Congress on the Chemistry of Cement. Montreal, Canada, July 8-13 2007. https://doi.org/10.1016/j.cemconres.2007.09.025

[5] S. A. Bernal, R. Mejía de Gutiérrez, A. L. Pedraza, et al. Effect of binder content on the performance of alkali-activated slag concretes. *Cement and Concrete Research* **41**(1):1–8, 2011. https://doi.org/10.1016/j.cemconres.2010.08.017

[6] J. Zhang, C. Shi, Z. Zhang, Z. Ou. Durability of alkali-activated materials in aggressive environments: A review on recent studies. *Construction and Building Materials* **152**:598–613, 2017. https://doi.org/10.1016/j.conbuildmat.2017.07.027

[7] Nedestruktivní zkoušení betonu – ultrazvuková impulzová metoda zkoušení betonu [Non-destructive testing of concrete – Method of ultrasonic pulse testing of concrete]. Standard, Czech Standard Institute, Prague, 2011.

[8] Metody zkoušení cementu. Část 1: Stanovení pevnosti [Methods of testing cement – Part 1: Determination of strength]. Standard, Czech Standard Institute, Prague, 2016.

[9] S. A. Bernal, E. D. Rodríguez, R. M. de Gutiérrez, J. L. Provis. Performance of alkali-activated slag mortars exposed to acids. *Journal of Sustainable Cement-Based Materials* **1**(3):138–151, 2012. https://doi.org/10.1080/21650373.2012.747235