

# INTERFACIAL TRANSITION ZONE BETWEEN AGGREGATE AND ALKALI-ACTIVATED BLAST FURNACE SLAG – A SCANNING ELECTRON MICROSCOPY

*MEC, Pavel, GEMBALOVÁ, Lucie*

*Institute of Clean Technologies for Mining and Utilization of Raw Materials for Energy Use, VŠB – Technical university of Ostrava, 17. listopadu 15/2172, Ostrava, Czech Republic,*

[pavel.mec@vsb.cz](mailto:pavel.mec@vsb.cz), [lucie.gembalova@vsb.cz](mailto:lucie.gembalova@vsb.cz)

## ABSTRACT

Alkali-activated binders are currently a widely-researched material. Thanks to the use of secondary raw materials such as slag from metallurgical production and ash from combustion, it appears to be a more promising and more environmentally friendly material than conventional cement concrete. Considerable attention is paid to the bonding phase itself, but only a few works deal with the binder-aggregate interaction. With cement concrete, much more attention is paid to this issue. This paper deals with the possibility of observation using electron microscopy and the information that can be obtained by this method. The problems of sample preparation and difficulties in the course of our own observation are observed.

**Keywords:** Alkali-activated binders, Cement concrete, Scanning electron microscopy

## 1 INTRODUCTION

Alkali activated materials have been known for more than sixty years, but the research intensified in last two decades. It is especially because the production of ordinary Portland cement is very environmentally inefficient, and many researchers try to find out an alternative binder. Many studies have been reported on alkali activated binders, mortars and concretes [1, 2, 3, 4]. The main focus is nowadays placed on chemistry and microstructure of various types of alkali-activated materials (AAM) [2]. The interaction of binder matrix and aggregate was very intensely studied in ordinary Portland cement materials [5, 6, 7]. However, in alkali-activated binders this area is not well explored [8]. Because the interaction of alkali-activated materials and aggregate is not the same as in Portland cement, research in this topic is needed.

## 2 MATERIALS AND METHODS

The alkali-activated materials were prepared in the same way as cement mortar for strength testing. Because there is no standard for testing alkali-activated materials, the standard for cement testing was used [9]. Standard samples of dimensions 160x40x40mm were prepared from alkali-activated binder and aggregate prepared from various types of rock. The granulometry of aggregate was prepared according to standard for cement testing. These samples were cut to smaller sizes. These small samples were used for electron microscopy observation. In previous research the broken samples after strength were used for microscopy observation. Due to its mechanical destruction, the results were not satisfactory.

### **Alkali-activated blast furnace slag**

The fine milled blast furnace slag (BFS) was used for preparation of alkali-activated binder. The chemical composition of BFS is in Table 1. Activator was prepared from sodium water glass with modified silicate modulus to  $M = 2.0$ . Modification of modulus was done by addition of 50% sodium hydroxide solution. Chemical composition of used water glass is in Table 2. The amount of added hydroxide solution was 11.9 ml to 100 ml of water glass.

**Table 1. Chemical composition of blast furnace slag**

Chemical element	[%]
Ca	30.054
Si	20.293
Fe	0.259
Al	6.109
Mg	1.832
S	0.653

**Table 2. Chemical composition of water glass**

Oxid	[%]	M (SiO <sub>2</sub> / Na <sub>2</sub> O)
Na <sub>2</sub> O	7.53	3.42
SiO <sub>2</sub>	24.94	

### Rock types and aggregate preparation

Used rocks for preparation of aggregate were chosen due to industrial usage in concrete preparation in the Czech Republic. Four types of rock were used. The rock type and mineralogical composition is in Table 3. Aggregate prepared from rock was crushed and sieved to the same size as standard sand for preparation of samples for cement strength testing.

**Table 2. Mineralogical composition of used rocks**

Amphibolite		Limestone		Travertine		Granite	
Mineral type	%	Mineral type	%	Mineral type	%	Mineral type	%
Calcite	4.7	Calcite	93.2	Calcite	98.2	Calcite	4.3
Biotite	2.1	Quartz	6.8	Quartz	1.8	Quartz	31.5
Chlorite	6.5					Microcline	17.0
Actinolite	33.1					Biotite	5.2
Andesine	53.7					Chlorite	5.3
						Oligoclase	46.7

### Electron microscopy observation and sample preparation

Scanning electron microscopy was used for monitoring the surface of samples, their composition and structure. Due to selected method, it was necessary to prepare smaller samples than the original beams prepared according to standard for cement testing. Samples were cut from the original 160x40x40mm to a size of 10 x 10mm and then a polished surface on one side of sample was prepared. Grinding was performed on grain papers of grain size 220 and 1000. After the cut was made, the sample was polished with Al<sub>2</sub>O<sub>3</sub>. The oxide residues were washed with water and the samples were dried, because electron microscopy does not allow analyses of wet samples. The drying process is not very appropriate for hydrated binder materials. It is because the thermal destruction of hydrated phases of binder. Solution of this problem will be taken into account in next research.

Despite this preparation of the samples, it was not possible to achieve a sufficient vacuum in the vacuum chamber as they were porous materials. Further surface treatment of the specimen was the staining of the sample surface with a thin layer of chromium approximately 30 µm. This ensures the filling of the pores on the surface of the samples, but also the discharge of the electrical charge.

Conditions of measuring:

Scanning Electron Microscope FEI Quanta 650 FEG

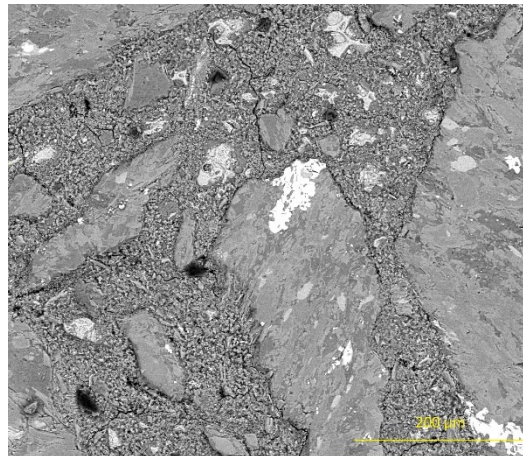
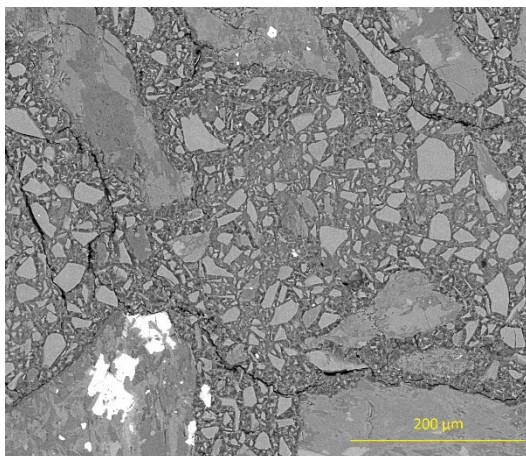
Acceleration voltage: 20 kV

Current: 8 - 10 nA

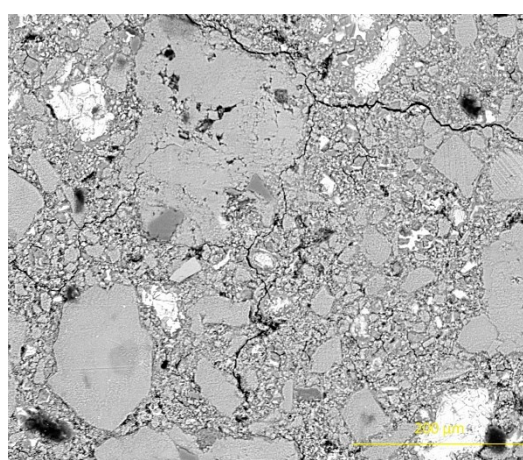
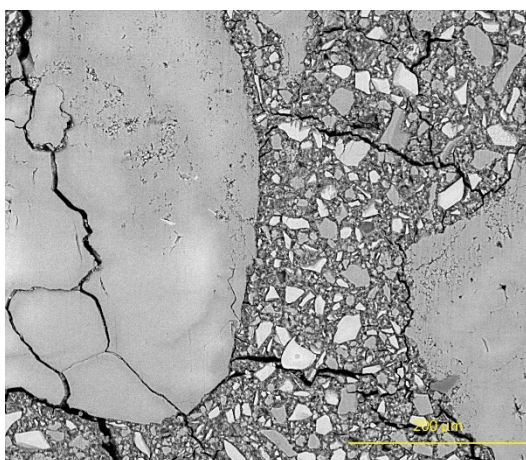
Pressure in vacuum chamber: 50 Pa

Electron beam diameter: 6 µm

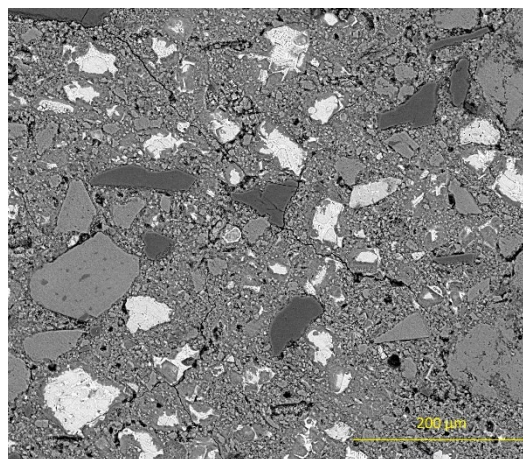
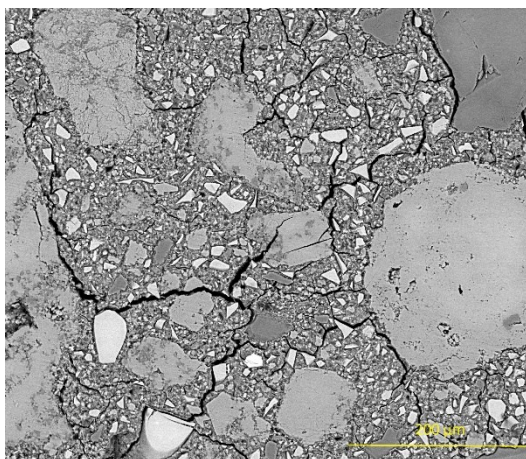
### 3 RESULTS AND DISCUSSION



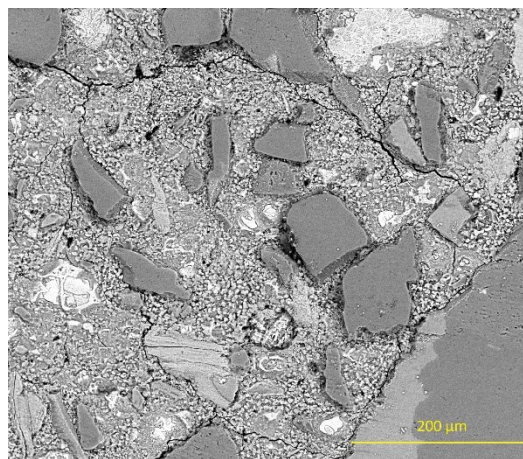
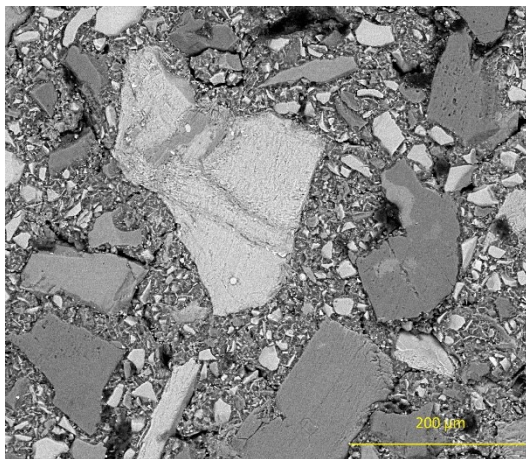
**Figure 1 Amphibolite with alkali activated grounded blast furnace slag (left) and with Portland cement (right)**



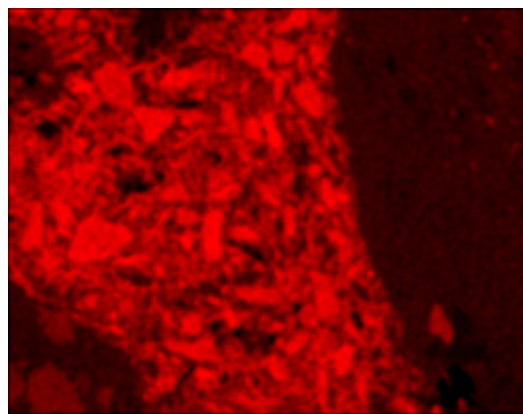
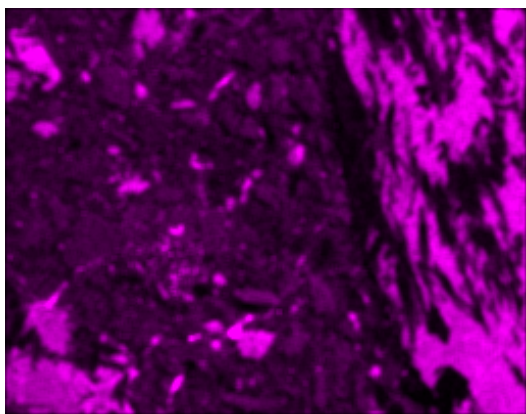
**Figure 2 Travertine with alkali activated grounded blast furnace slag (left) and with Portland cement (right)**



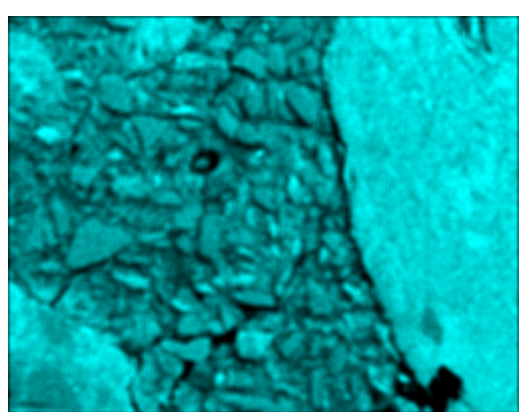
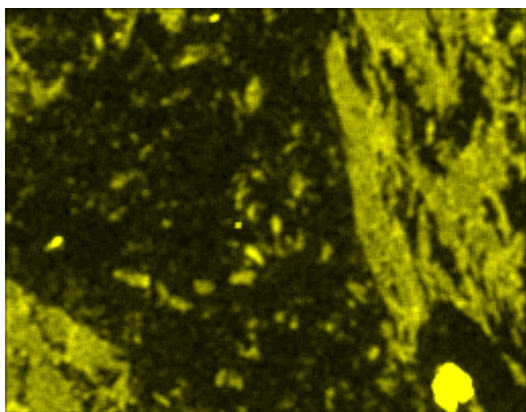
**Figure 3 Limestone with alkali activated grounded blast furnace slag (left) and with Portland cement (right)**



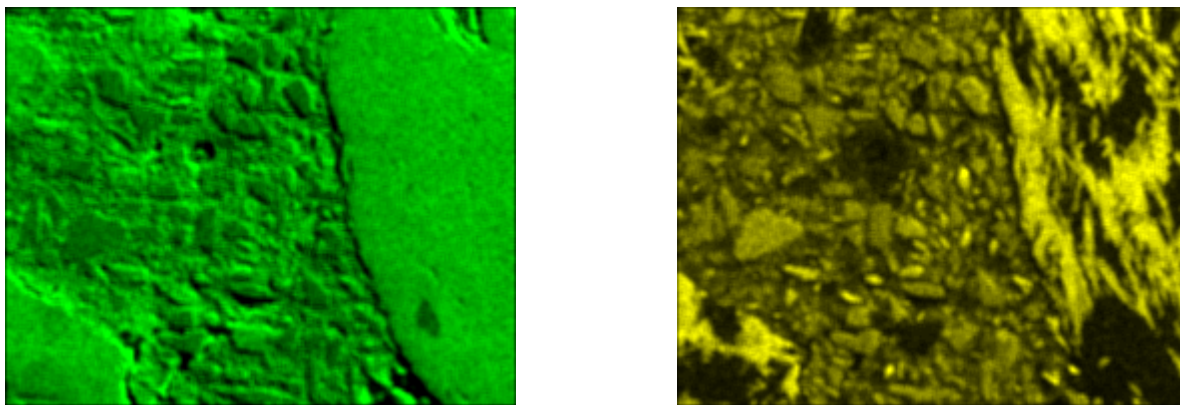
**Figure 4 Granite with alkali activated grounded blast furnace slag (left) and with Portland cement (right)**



**Figure 5 Energy-dispersive X-ray mapping the distribution of elements: Al (left) and Ca (right) on the surface of the amphibolite with alkali activated grounded blast furnace slag**



**Figure 6 Energy-dispersive X-ray mapping the distribution of elements: Fe (left) and Si (right) on the surface of the amphibolite with alkali activated grounded blast furnace slag**



**Figure 7 Energy-dispersive X-ray mapping the distribution of elements: Mg (left) and O (right) on the surface of the amphibolite with alkali activated grounded blast furnace slag**

As can be seen from Figures 1 - 4, despite the demanding preparation of samples for microscopic observation, cracks and small fragments appear on the surface of specimens and it hinders clear visibility of the grain boundaries. Cracks spread not only by the binder but also through the aggregate, especially in brittle materials such as limestone and travertine (Figure 2 and 3 left). Alkali activated grounded blast furnace slag as a binder contains larger particles, while the Portland cement has a finer character, which can result in better strength properties, and there is no such scaling of the surface when treated. For this reason, it is difficult to determine the dependence of the elements on the distance from the aggregate grain in the samples. Energy-dispersive X-ray mapping of the distribution of elements in Figures 5 – 7 provides evidence that investigated material is inhomogeneous.

#### 4 ACKNOWLEDGEMENT

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