EDS-Based Phase Analysis of Alkali Activated Slag

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Alkali-activated materials are considered third-generation binders after lime and ordinary Portland cement (OPC). There are two main categories of alkali activated binders: the first one is the activation of industrial by-products such as fly ash and blast furnace slag with a mild alkaline solution and calcium silicate hydrate (C-S-H) as the main reaction product. The second one is the alkali activation of natural aluminosilicates such as metakaolin with medium to high alkaline solutions. Alkali-activated materials have been known as an alternative to Portland cements due to the higher compressive strength (exceeding 100 MPa in 28 days), durability and environmental benefits [1,2]. However, to understand the development of mechanical strength and durability, an understanding of the phase formation and chemical composition of these phases it required. [6]

Blast furnace slag is a highly heterogeneous material. It is well known that its chemical composition affects the physical and mechanical properties of the alkali activated material, however there is little work on how these inhomogeneities affect the final microstructure. In previous research, a correlation between the low Ca/Si ratio in slag and its resistance to aggressive chemical attack by acids, chlorides and sulphates has been shown [3,4,5]. In traditional Portland cement materials, the main reaction product is calcium silicate hydrate (C-S-H) gel with $1.5 \le \text{Ca/Si} \le 2$. In alkali activated materials, it has been proposed that one of the main products is (C-A-S-H) gel, in which Al partially substitutes for Ca.

This study explores EDS-based phase characterization of two specimens; unactivated slag and slag activated with KOH. Backscattered electron (BSE) images were acquired at approximately 5kX for unactivated slag and 2kX for activated slag using a 15kV beam voltage. The phase analysis shows two phases in unactivated slag: the first phase represents homogenous particles with Ca/Si ratio 1-1.2, while the second phase corresponds to a much broader Ca/Si ratio 0.6-1.6. On the other hand, activated slag has four phases: one with homogenous particles with Ca/Si ratio 1.06-1.15 and three other phases, one being too small to analyze. The two analyzed phases have Ca/Si ratio of 1-1.25 and 0.7-0.8. The elemental quantification indicates that calcium and silicon increases in the activated particles comparing to the one in the unactivated slag, while the aluminum and magnesium remain constant in both, but the oxygen decreases in the activated slag particles. According to the Myers et al study, calcium is entering the tetrahedral Al or Si sites as ions, this could result in the observed decreased of oxygen. [7]

References:

- [1] C Li, H Sun, and L Li, Cement and Concrete Research 40 (2010), p. 1341.
- [2] F Pacheco-Torgal, J Castro-Gomes, and S Jalali, Construction and Building Materials **22** (2008), p. 1305.
- [3] H Xu, et al, ACI Materials Journal **105** (2008).
- [4] C Shi, AF Jiménez, and A Palomo, Cement and Concrete Research 41 (2011), p. 750.

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- [5] R San Nicolas, et al, Cement and Concrete Research 65 (2014), p. 41.
- [6] SE Moghaddam, et al, Journal of Materials Chemistry A (2017).
- [7] RJ Myers, et al, Journal of the American Ceramic Society 98 (2015), p. 996.

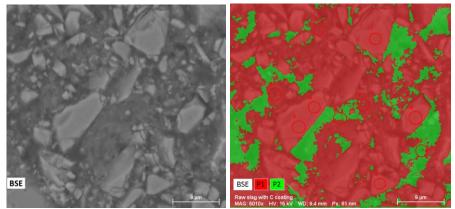


Figure 1. Unactivated slag BSE image showing two different phases, P1 and P2. The circles in the right hand figure indicate areas where quantitative analysis was done.

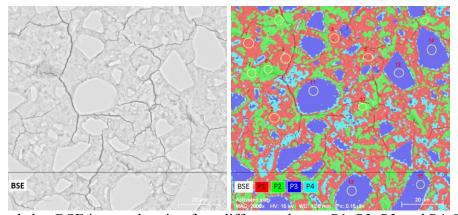


Figure 2. Activated slag BSE image showing four different phases, P1, P2, P3 and P4. The circles in the right hand figure indicate areas where quantitative analysis was done.