

The use of backscattered electron (BSE) imaging for the microstructural quantification of cementitious based materials

Author: Mohsen Ben Haha
Contact: bauchemie@empa.ch

Introduction

Scanning electron microscopy (SEM) is a powerful technique for examining the cement hydration process and the resulting hydration products.

The SEM backscattered electron (BSE) images can be analyzed quantitatively to get volume fractions of phases in cement pastes, mortars and concrete, based on the segmentation principle methods (grey level, morphology, etc.).

Image analysis (IA) is an additive tool that enables to study the evolution of cementitious materials and to quantify the microstructural features in order to define microstructure–macrostructure properties relationships.

Relevance for Our Field

- BSE-IA allows to study:
- The microstructure of cementitious composites (the anhydrous components (OPC, Fly ash (FA) and slag particles), the main hydration products and capillary porosity).
 - The development of microstructure associated with atypical hydration processes.
 - The degradation process in cementitious composites materials or in concrete (leaching, Sulphate attacks, Alkali silica reaction (ASR), crack propagation, etc...)

- It enables
- To understand the degradation mechanisms and thus to develop realistic models.
 - To transfer knowledge acquired from laboratory accelerated tests to the field.

Measurement Principle

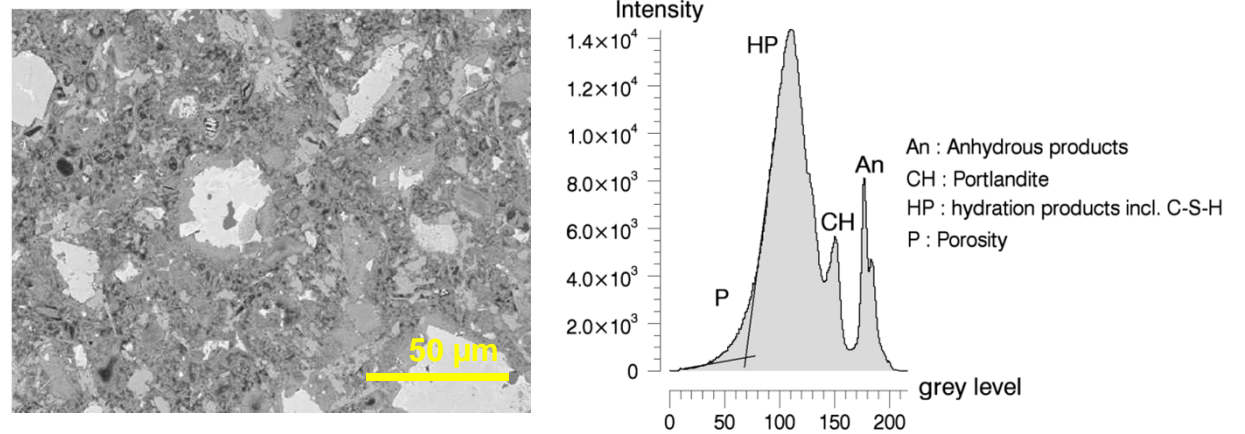


Figure 1: SEM image of OPC hydrated paste at 28d and its corresponding grey level histogram
The images are acquired in 8 bits. 256 grey level values are included in the image, ranging from 0, black (porosity) to 255, white. Therefore, the typical constituents of cement pastes in concrete can be distinguished by their grey levels:

- anhydrous phases (An) appear bright,
- calcium hydroxide (CH) light grey
- HP and C-S-H grey
- porosity (P) black.

Example

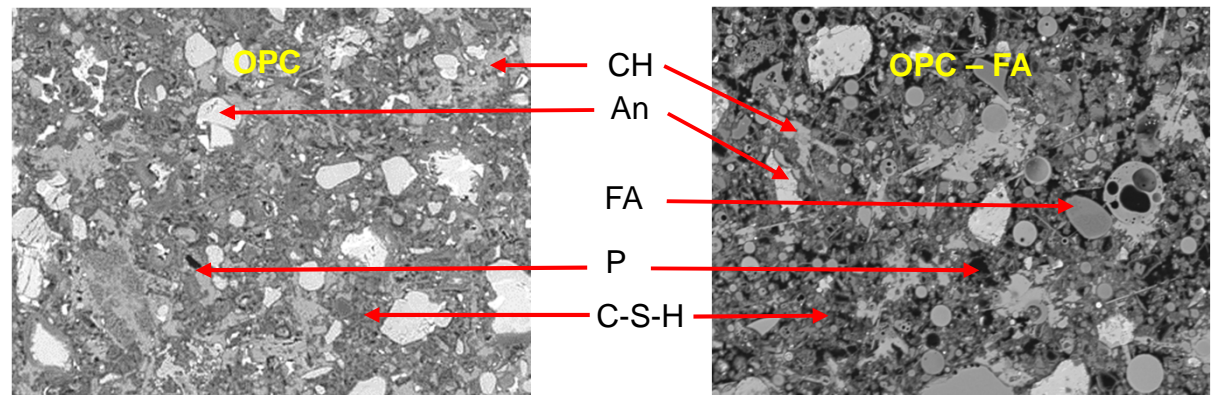


Figure 2: SEM image of OPC and OPC-FA hydrated paste at 28d

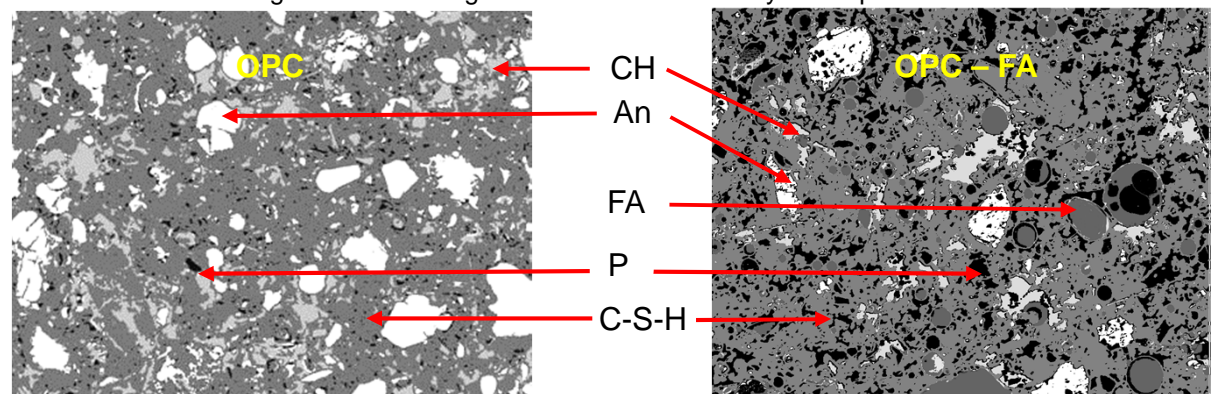


Figure 3: corresponding segmented image of OPC and OPC-FA hydrated paste at 28d of figure 2

Applications & Potentials

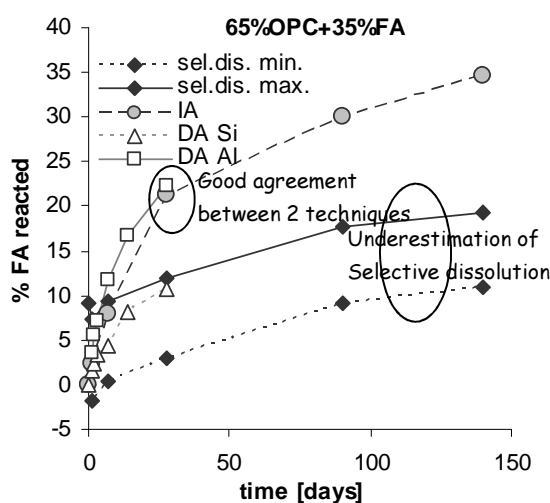


Figure 4: Quantification of the degree of reaction of FA (comparison between different techniques)

- The measurement of the degree of reaction of the anhydrous species.
- The link between macrostructural properties and microstructural observations e.g, strength vs. porosity.

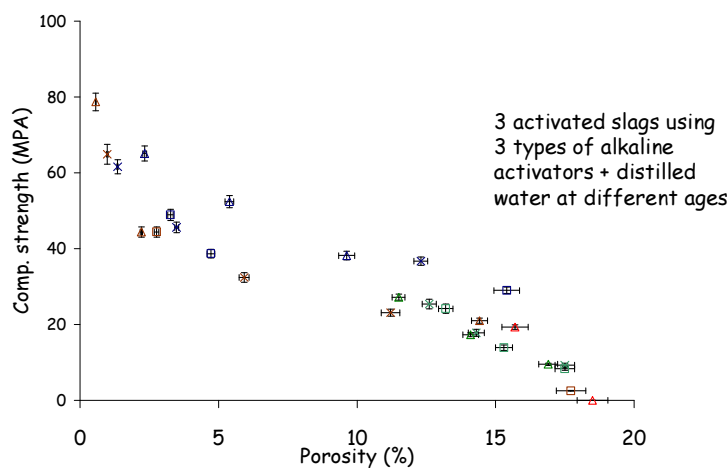


Figure 5: Compressive strength vs. porosity(%) of different slags using different alkaline activators

Limitations

Size limitations:

- The maximum pixel size is dependent of the magnification. As threshold for porosity involves an averaging effect whereby some pixels counted as porosity in fact contains some solids and vice versa.

Phase identification:

- CH peak is well defined in histograms, but its quantification with other phases (calcite + others) is less reliable than results obtained by more traditional techniques (TGA or XRD).
- The grey level differences between the different hydration products (C-S-H, ettringite, monosulfate, etc.) are too small to be individually distinguished by SEM-IA.