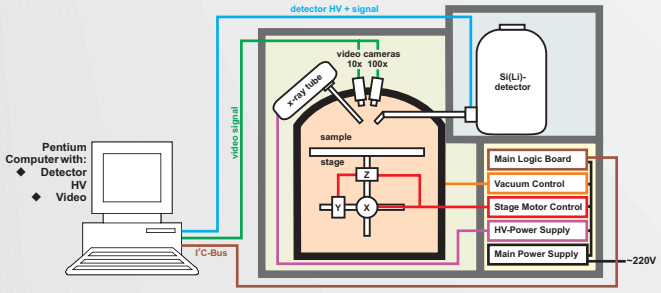
The Micro X-Ray Fluorescence Analysis (MXRF) for the Analysis of Concrete

Method

With micro X-ray fluorescence analysis the chemical composition of an inorganic material can be determined in a fast and easy way. A single analysis lasts no longer than some 50 seconds. Sample specimen can have various shapes from flat to fractured surfaces.

The major difference to the standard X-ray fluorescence spectroscopy is based on two features:

- The sample stage is controllable, that means the sample on the stage can be moved in all three directions.
- The X-ray beam is focusable by micro capillary optics. So it is possible to vary the spot size of the X-ray beam on the sample surface in-between 35 and 200 µm.



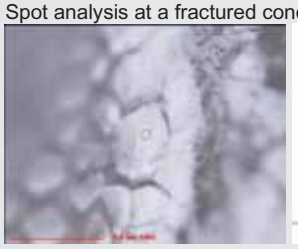
Modes of Analysis

The MXRF is primarily used for the determination of the chemical components in the texture of a building material. The instrument is able to analyze elements starting with the atomic number 11 (sodium), all simultaneously. The detection of lighter elements (e.g. C, N, O) is not possible. Two video cameras with 10x and 100x magnification help to position the sample under the incident X-ray beam with high precision.

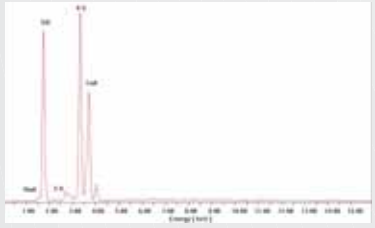
In essence the MXRF enables an elemental spot analysis of the sample surface. Since the stage and therefore the analyzed spot can be controlled the following analytical modes are possible:

- Spot analysis - analysis at a single spot on the sample surface (flat and uneven surfaces).
- Line scan - analysis of multiple equidistant spots along a line (flat surfaces only). The distance on the line is plotted against the intensity of one element.
- Elemental mapping - analysis of multiple equidistant spots along a dot matrix (flat surfaces only). The intensity of one element is displayed in two dimensions. The instrument is capable to measure areas of 70 x 60 mm^2 in a single run.

Spot analysis at a fractured concrete surface



Reaction product in a pore of a concrete.

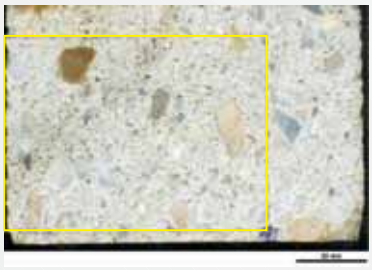


The element spectra of the reaction product indicates an alkali-silica gel.

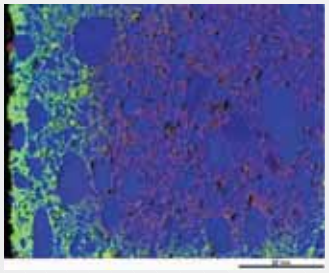
Example for the Elemental Mapping of Concrete

Concrete cubes (100x100x100 mm^3) with two different w/c ratios of 0.71 and 0.80 and a low cement content were immersed in a sulfate solution containing 30 g/l sodium sulfate for 1 year. The sample cubes were cut and subjected to analysis by MXRF.

First elemental maps of Al, Si, S, K, Ca and Fe with a resolution of 512 x 400 points were acquired from sample cross sections. A second step included the analysis of a matrix of 70 x 50 points with quantification of each spectra. The quantification was carried out by fundamental parameter analysis of each spectra.

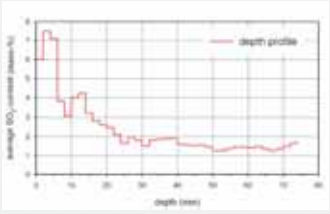


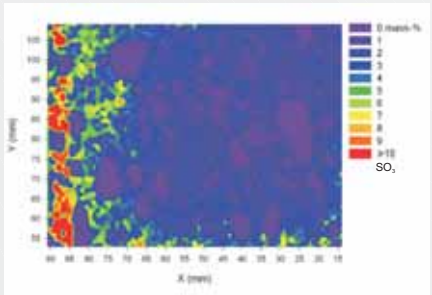
Cross section of the sulfate exposed concrete (w/c = 0.71). The yellow outline indicates the scanned area. The original concrete surface is on the left side.



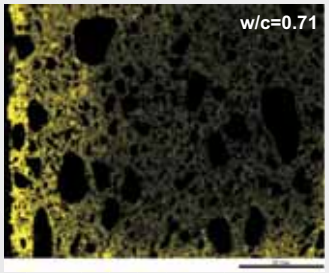
Overlay of the elemental maps of **sulfur**, **calcium** and **silicon** with a resolution of 512x400 points. The intensity of the color gives an indication of the concentration of elements present. Clearly discernible is the chemical profile of sulfur in the sample.

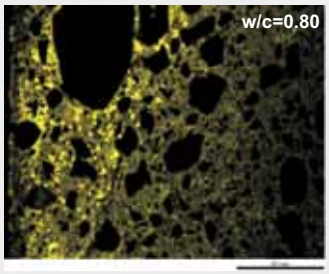
Depth profile of the average SO_3 content of the concrete sample. The data were extracted from the elemental map of sulfur.





Results of the measurement and quantification of a 70x50 point matrix. Though the resolution is much lower compared to the elemental maps, instead of intensity values quantitative data can be acquired.





Elemental maps of sulfur of the concretes with the two different water/cement ratios. The concrete with w/c=0.71 exhibits a more distinctive profile with an enrichment of sulfur only in the first 7 millimeters followed by a 15 mm deep zone with a moderate sulfur content. The concrete with w/c=0.80 exhibits a deeper ingress of sulfur down to a depth of 50 mm with a more inhomogeneous distribution.

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