

μ XRF-XSI for Tracking Chloride Ion Infiltration in Concrete

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A variety of methods exist for tracking externally supplied chloride ions as they infiltrate concrete specimens. The most common method, particularly useful for laboratory tests, involves spraying silver nitrate onto the specimen, and observing the areas of silver chloride precipitate (Collepari, Marcialis, & Turruzani). A new method, based on micro X-ray fluorescence X-ray spectrum imaging (μ XRF-XSI), for analyzing chloride ion infiltration has been developed. While the method is slower than the silver nitrate test, μ XRF-XSI can yield both qualitative and quantitative information from the derived infiltration profile, and provides far more information than a simple qualitative test.

Samples for the study were prepared, as part of a larger study (Bentz, Peltz, Snyder, & Davis), by soaking concrete cylinders in a solution of water and potassium chloride. Cylinders were removed from the soak bath after 1 month, 3 months, 6 months and 12 months, and then split along the centerline (see Figure 1). Full spectra were acquired at each point in an area extending from the outer surface to the middle of the cylinder. From the X-ray spectrum image, the depth of chloride ion penetration was determined by analyzing the chlorine elemental images (Figure 2), and the profile of chloride ion concentration was derived from the background subtracted X-ray intensity data. The concrete samples, as prepared, represent a significant challenge for X-ray microanalysis. First, the chloride ions are highly soluble in water, meaning that polishing the split, exposed surfaces will require oil or other non-polar solvents. As a porous material, cement is difficult to polish and clean when oil is used to prepare the surface. The end result is that oil preparations generally cause the sample to be unusable for any analytical techniques requiring vacuum. Second, when a vacuum is applied to the sample, significant desiccation occurs, removing water from the surface of the sample. This removal of water will drive water soluble ions, such as chlorine, into the specimen. By depleting ions from the surface, the apparent concentration profile changes, but the change simply an artifact of sample analysis conditions. Third, infiltration of ions is usually tracked over centimeters, meaning that the microanalytical technique must have the ability to map over a large spatial scale and have sufficient chamber space to accommodate a specimen with dimensions of 50 mm x 100 mm x 50 mm. Solutions to these problems were necessary in order to correlate the X-ray data to the larger study.

For measuring chloride ion profiles in concrete specimens, μ XRF-XSI is uniquely qualified to provide efficient, reliable results. Due to a combination of spectrometer geometry and the polychromatic nature of the incident X-ray beam, imaging applications require far less sample smoothness than what is required for electron imaging techniques (Janssens). In addition, the samples may be run at atmospheric pressure, eliminating the drying issue. By using μ XRF-XSI, chloride ion profiles in concrete were derived on unpolished, wet samples at atmospheric pressure almost immediately after they were removed from the water bath and split. This talk will focus on the error analysis, background modeling and mapping parameters derivation as a method for studying a broad range of cement and concrete durability problems with μ XRF-XSI.

References

- Bentz, D. P., Peltz, M. A., Snyder, K. A., & Davis, J. M. (2009). VERDICT: Viscosity Enhancers Reducing Diffusion in Concrete Technology. *Concrete International*, 31-36.
- Collepari, M., Marcialis, A., & Turriziani, R. (1970). Kinetics of Penetration of Chloride Ions into the Concrete. *Il Cemento*, 4, 157-164.
- Janssens, K. (2000). Comparison with Other Microanalytical Techniques. In K. Janssens, F. Adams, & A. Rindby, *Microscopic X-ray Fluorescence Analysis* (pp. 215-218).

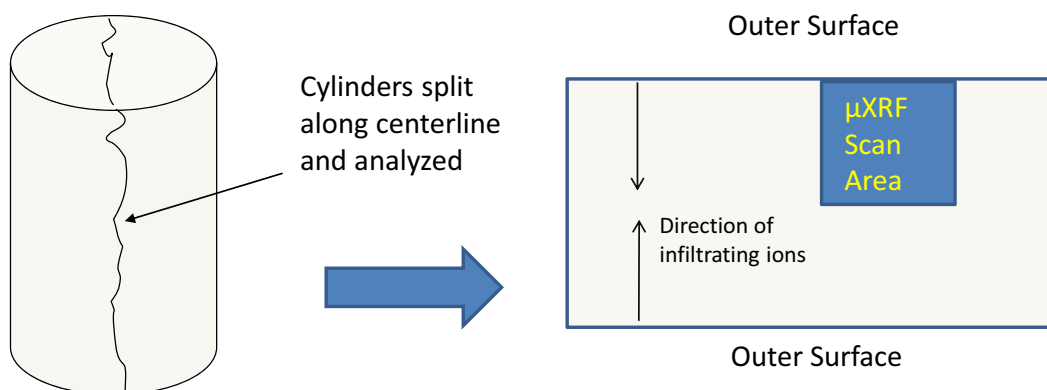


Figure 1: Illustration of the sample preparation process. The original samples, cylinders measuring 150 mm in height and 50 mm in radius were removed from a chloride ion bath and split along the centerline. One of the exposed halves was rotated and a scan area was set up to measure the concentration of ions from the outside of the cylinder to the midpoint.

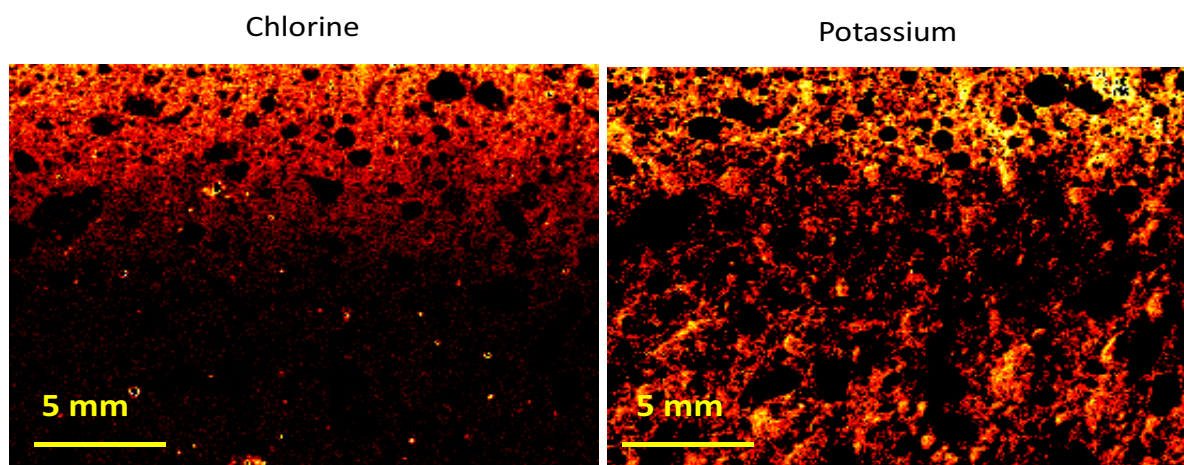


Figure 2: Derived Region of Interest Maps for Cl and K (thermal scale). Shown here, the derived region of interest maps for Cl and K are useful for determining limits of infiltration of both externally supplied ions. The gradient and full extent of the infiltration is apparent in the Cl image.