

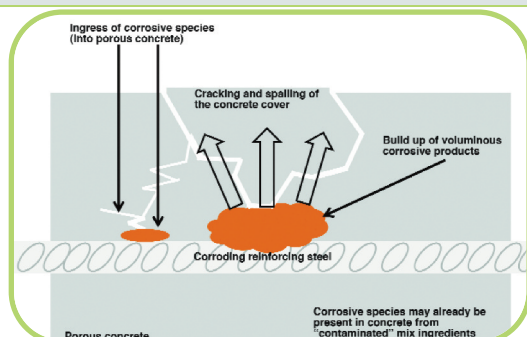
Determining variations in concentrations of chlorine prepared cement paste, using Micro-XRF

Materials Challenge

Concrete structures are designed with the expectation of a 100 year service life, however there are many factors which negatively influence their durability, including physical and chemical deterioration. One example of this is the penetration of anionic salts and subsequent degradation of the underlying rebar support structure from corrosion, which will result in a reduction in years of service and costly repairs.

Ions filter into the concrete from various external sources including sea water and de-icing salts. These salts penetrate into the concrete and can attack the surface of the reinforcing steel locally inside it. Once the electrochemical reaction has begun there is a volume increase in the reaction products at the surface of the reinforcing steel that creates internal stresses and ultimately cracking of the concrete.

Once these cracks reach the surface of the concrete it is easier for water to enter and cause more corrosion or other deterioration.



Comparison with Existing Solutions

Current test methods use chloride titrations of the powder extracted from concrete adhering to ASTM standard C1152. This testing process is costly and lengthy when considering core extraction all the way to core sub sectioning and pulverization of the cement for testing. An illustration of that process is shown in Figure 1.



The Orbis PC Micro-XRF System

Micro-XRF is a technique that measures the presence and concentration of elements in a sample. It is similar to bulk XRF except that it focuses the incident x-ray beam to an approximately $30\mu\text{m}$ spot resulting in a higher energy flux, which creates greater physical and analytical resolution. Micro-XRF also offers large area elemental mapping of a sample with little to no sample preparation. Using this technique it is possible to quickly detect low and varying concentrations of various ionic species including chlorine. The user can also increase the accuracy and greatly reduce the steps and time needed to investigate cores in concrete. The steps necessary to investigate the chloride content in concrete are summarized below. With micro-XRF the cores need only to be extracted and split before analysis is possible.

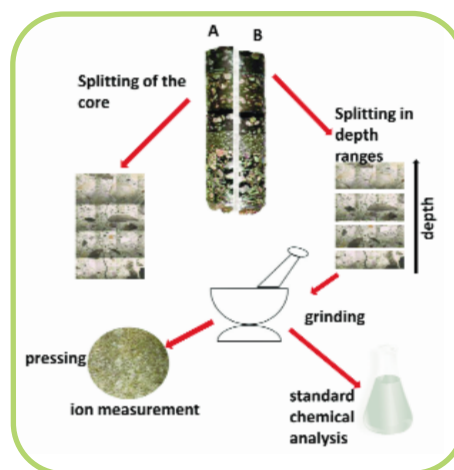


Figure 1

Microanalysis Results

To illustrate the capability of the tool to determine chlorine concentrations, samples were prepared with known levels of chlorine in the paste. The resulting samples were a homogenous mixture of concentrations which were 1%, 0.5%, 0.1% and 0.05% by weight chlorine.

The equipment test setup used a poly capillary optic focusing the incident x-rays to a 30 μ m spot using an aluminum primary beam energy filter, which further enhanced the chloride photon emissions. Each sample was analyzed using a feature, which maps and counts elements in a large area, then using Fundamental Parameters the chlorine weight percent was determined.

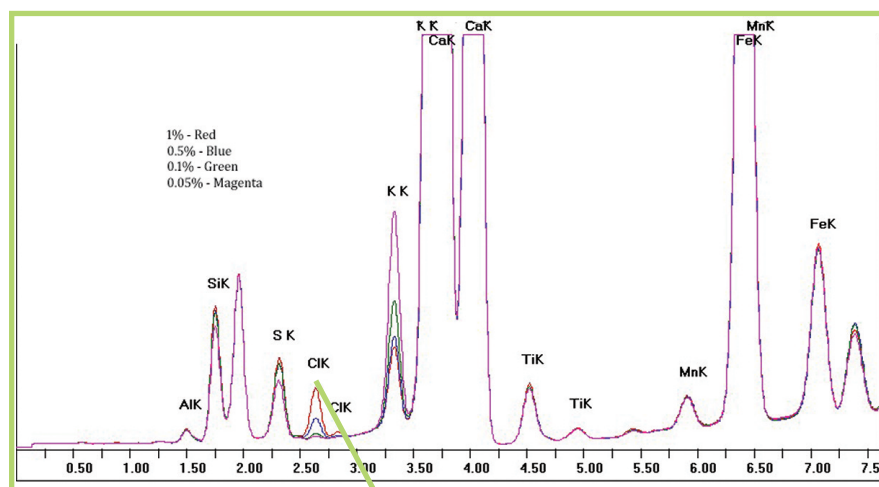


Figure 2: Four concentration overlay

Quantification results using micro-XRF	
Known chlorine concentration	Measured in weight %
1%	0.90%
0.50%	0.45%
0.10%	0.09%
0.05%	0.05%

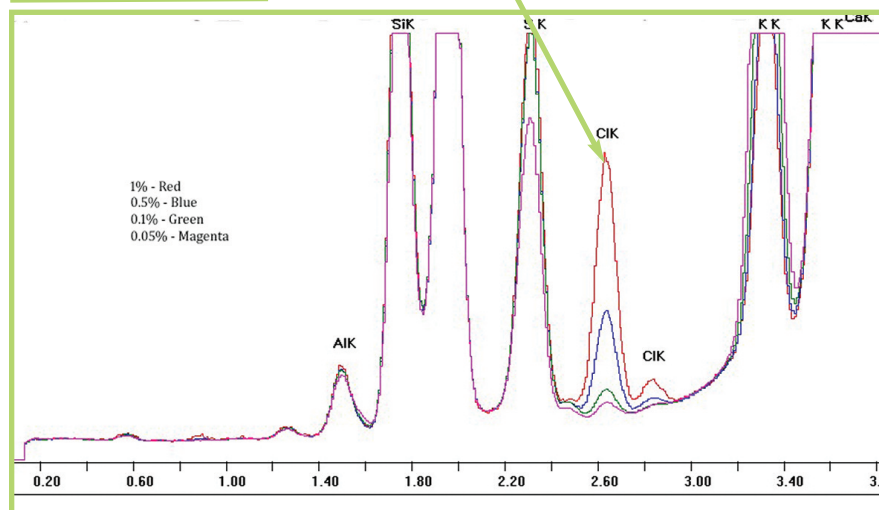


Figure 3: Four concentration overlay with zoom on chlorine

Conclusion

The spectra in Figure 2 and Figure 3 represent each sample illustrating counts versus energy emission. The interest here is in the chlorine spectral K line at 2.622eV. Note peak heights follow sample concentrations. Using the quantitative functions following Fundamental Parameters routines, the weight percent calculations are generated.

These test results confirm micro-XRF as a competitive technique for qualitative and quantitative analysis with a significant reduction in testing time and steps required.

Recommended Equipment

- Orbis PC Analyzer comes with the Vision Software suite including Microsoft operating system and Microsoft Office
- PC workstation comes standard with flat panel LCD display, dual core Intel Core 2 Duo processor, 1G RAM, 256MB video RAM, 160GB hard drive
- Precision automated stage
- Chamber vacuum pump
- Dual color CCD cameras
- 6 primary beam energy filters
- X-ray optics: 30um poly capillary, 1mm and 2mm collimators
- Silicon Drift Detector
- If your interest is primarily chlorine detection, we recommend the molybdenum x-ray tube, otherwise the standard rhodium in conjunction with the primary beam energy filters is applicable and standard.