## Hydraulic Cements for Construction: The Development of Cementitious Materials, Standards and Specifications, and X-Ray Analysis

Paul Stutzman National Institute of Standards and Technology Gaithersburg, MD USA

The beginning of portland cement concrete as a principal infrastructure construction material, the discovery of X-rays, and the development of standardized materials specifications all occurred around the beginning of the 20<sup>th</sup> century. While apparently unrelated, these materials, methods and institutions came together with a synergy that improved our understanding of the compositions of hydraulic cements, improved product specifications and standards, and fostered development of quantitative X-ray methods and instrumentation.

While light microscopy provided the initial insights on portland cement clinker phase composition, it required X-ray powder diffraction in the mid-1920s to unambiguously identify the phases, an issue of heated international dispute at that time. The adoption of X-ray fluorescence and X-ray powder diffraction for the characterization of raw materials, clinker, and finished cement improved their production through more rapid control and feedback. By the 1960's and 1970's these instruments were routinely used in research and some production applications, with standardized test protocols being developed to provide consistent analyses across the industry. Industry needs to understand cement production and cement hydration kinetics assisted in development of quantitative X-ray methods, environmental control for early powder diffractometers and automation of powder diffraction data collection and analysis.

ASTM Committee C01, Cement, worked toward standardizing test protocols and developing performance metrics for chemical and phase analysis with a novel approach which provides flexibility with a means of validating a lab's ability to properly conduct an analysis. These may be found in their work on performance specifications for XRF analyses and a standard test method for X-ray powder diffraction of portland cement clinker and portland cement and routine proficiency testing.

Over time, hydraulic cements have become more complex with the addition of alternative cementitous materials to the cement kiln and to the processed cements, the use of alternative and waste fuels, and in looking beyond the venerable calcium silicate-based cements of the past century. This evolution in the construction materials industry places a greater reliance on X-ray methods to uncover the materials chemistry and phase composition, reaction kinetics, and long-range durability of these materials to assist in establishing a confidence in their use and in developing specifications for their production and use in construction.