Texas A&M Transportation Institute

PROJECT SUMMARY

Texas Department of Transportation

0-6941: Direct Determination of Cement Composition Using X-ray Diffraction

Background

The Bogue calculation is the most widely used method to estimate potential phase composition of ordinary portland cement (OPC). In this method, the elemental oxide wt% of OPC determined by x-ray fluorescence is used to estimate cement phase contents using the Bogue formulas. But the Bogue method, being an indirect estimation technique, cannot accurately quantify the main cement phases and cannot identify and quantify sulfate phases and determine limestone and amorphous contents in cement. Quantitative x-ray diffraction (QXRD) can not only identify and quantify the actual cement phases but also their polymorphic variants. This more complete accounting of the actual phase compositions helps relate mineralogical composition to performance characteristics and improves the predictive capability of cements. QXRD is also effective for identification and quantification of different sulfate polymorphs in cement and determination of limestone and supplementary cementitious materials (e.g., fly ash and slag) contents in blended cements.

One of major challenge of the QXRD method is getting the same results when the same sample is analyzed in different laboratories or by different instruments/ personnel. Sample preparation errors were identified to be the major cause of this variability in QXRD, but not much effort was taken to address this insufficiency.

This project advanced the use of the QXRD method for cement phase quantification by:

- Determining the barriers preventing the progression of QXRD in production testing and solutions to overcome these barriers through extensive literature survey.
- Developing protocols for determining cement composition by QXRD for ASTM C150 and C595 types of cements.
- Determining the effects on cement production if QXRD is used to quantify the cement phases, especially relating to C3A-gypsum optimization and the influence of polymorphs.
- Providing recommendations to the current specification based on QXRD-based phase quantification information rather than Bogue based.

What the Researchers Did

Researchers made the QXRD results more repeatable and stable between different laboratories and made QXRD more accessible for routine quality control. With ASTM C1365 as the foundation, the individual steps in QXRD execution (sampling, sample preparation, measurement, and analysis) were examined for improvement, and a list of issues or gaps in existing protocols were identified. Solutions to some of these problems were generated experimentally, such as optimum fineness, preferred orientation, extraction methods, etc., while other solutions were generated from the literature survey. With the gaps addressed, researchers performed experimental investigations on improving the repeatability and reproducibility of the QXRD in

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three laboratories, and a consistent QXRD sample preparation technique was idealized. This technique, called pressed pellet QXRD, was validated for its accuracy and effectiveness through microscopic quantification of the same samples. A step-by-step procedure detailing the whole process from sample preparation to Rietveld refinement analysis was developed for replication in the Texas Department of Transportation (TxDOT) laboratory. Researchers generated the phase limits for the important phases by modeling a set of 320 cement samples with corresponding Bogue and QXRD information. Effective implementation recommendations on how to further this research were also proposed.

What They Found

Researchers found:

- The variability involved in sample preparation and lack of standard calibration were found to be the main barrier for obtaining reproducible QXRD results. Making QXRD more reliable and easier to use by addressing these barriers can significantly increase the interest in QXRD adoption for online quality control. Pelletization was successfully attempted as a quickly applicable sample preparation technique for QXRD with good repeatability and reproducibility.
- Improved accuracy of Bogue to QXRD conversion leads to accurate derivation of QXRD phase quantification from the Bogue phase quantification to apply ASTM C150 specifications.
- Experimental validation of the accuracy of the amorphous content determination by QXRD was found through the PONKCS or pseudo-phase method.
- QXRD was found to be very effective for accurate determination of limestone content, fly ash content in blended cements (ASTM C595), and quantification of the different types of sulfate phases for all ASTM C 150 and C 595 types of cements.
- Through in-house performance testing and industrial collaboration, researchers found areas of influence of QXRD and documented the effects in each zone of cement production if QXRD is employed for testing.

What This Means

This project generated a consistent methodology to perform QXRD quickly and get results with reasonable accuracy. The protocols and specifications were developed and provide a:

- Direct and highly accurate technique for performing cement phase quantification that is an improvement on a widely accepted technique (the Bogue method).
- Means to predict the performance of cements (e.g., strength and durability [e.g., alkali silica reaction]) even before application in the field.
- Model to convert the Bogue quantification to QXRD with better accuracy than a similar model available in the current ASTM C1365 protocols.
- Motivation to cement producers to procure x-ray diffraction and promote the use of QXRD for routine cement quality control.

The results derived using data from one cement plant were very encouraging to improve and validate the relationship between QXRD and Bogue. This work needs to be continued using reliable data from multiple cement laboratories or research and development laboratories to develop a more reliable predictive model to convert Bogue to QXRD, which will help TxDOT apply QXRD to routine quality control of cement, avoid issues during construction, ensure long-lasting, durable concrete, and save money (taxpayer dollars).

For More Information

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