Application note

Quantitative analysis of C3S M1/M3 in CEM I using ARL X'TRA Companion benchtop XRD

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Figure 1. ARL X'TRA Companion diffraction system

Introduction

X-ray diffraction (XRD) is a widely used analytical technique in the industrial analysis of cement-based materials. Cement is a vital component in the construction industry and is used to make a wide range of products, including concrete, mortar, and grout. The quality and performance of these materials depend on the crystalline structure and chemical composition of the cement used. XRD is a non-destructive technique that provides valuable information about the crystallographic phases present in cement-based materials, enabling engineers and scientists to optimize the production process and improve the performance of cement-based materials.

This application note will provide an overview of the application of XRD in the industrial cement analysis of CEM I, including the quantification of C3S M1 and M3 polymorphs. This application note will be useful for anyone involved in the cement industry, including researchers, engineers, and quality control professionals.

Instrument

The Thermo Scientific™ ARL™ X'TRA Companion (c.f. Figure 1) is a simple, easy-to-use bench top XRD system for process control and more advanced applications.

The ARL X'TRA Companion uses a θ/θ goniometer (160 mm radius) in Bragg-Brentano geometry and a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available as an option.

Due to the state-of-the art solid state pixel detector (55x55µm pitch) the ARL X'TRA Companion provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS.

Experimental

A CEM I powder sample was measured in reflection using an ARL X'TRA Companion with Cu Kα radiation (Ni filter). The sample was measured in eleven repeats of 5 min to calculate the reproducibility. Phase quantification was performed with Profex [1] (BGMN algorithm) using a fundamental parameters approach. Reference structures were selected according to Aranda et.al (2012).[2]

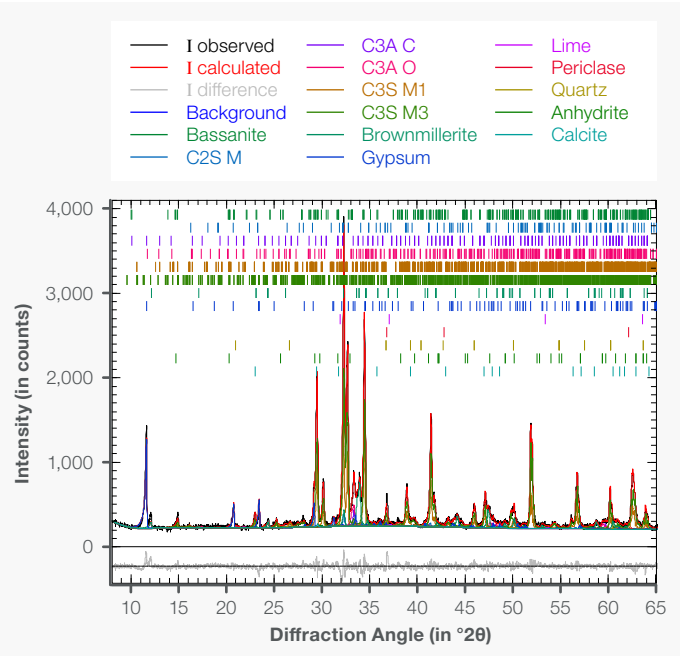

Figure 2. Rietveld fit of CEM I (5 min acquisition)

Results

A CEM I sample was measured (11 x 5 min; c.f. Figure 2) and a Rietveld refinement was carried out consecutively using a fundamental parameter approach.

Standard deviations shown in Table 1 indicate the good quality of data and refinement.

The sample contains no amorphous additives. Quantification of C3S M1 and M3 modifications is possible and results in a standard deviation of 1.2% (1σ) (c.f. Figure 3).

The composition agrees with specifications made in ASTM C150 norm on OPC Type 1. It is noteworthy that C3S (73.5% and C4AF (10.0%) are high, with low C2S (2.2%) and C3A (6.4%), while the sum of calcium sulphates equals 6.3% (c.f. Table 1). All values are still in agreement with ASTM C150.

Conclusion

The ARL X'TRA Companion XRD can collect data on OPC Type 1 in 5 min which allows quantification of C3S M1 and M3 in parallel with all other relevant cement phases. The one-click Rietveld refinement, based on a fundamental parameter approach, is an extremely robust method with high reproducibility which yields reliable results even for C3S M1 and M3. Because of these qualities the ARL X'TRA Companion is the perfect solution for any process control task in cement industry.

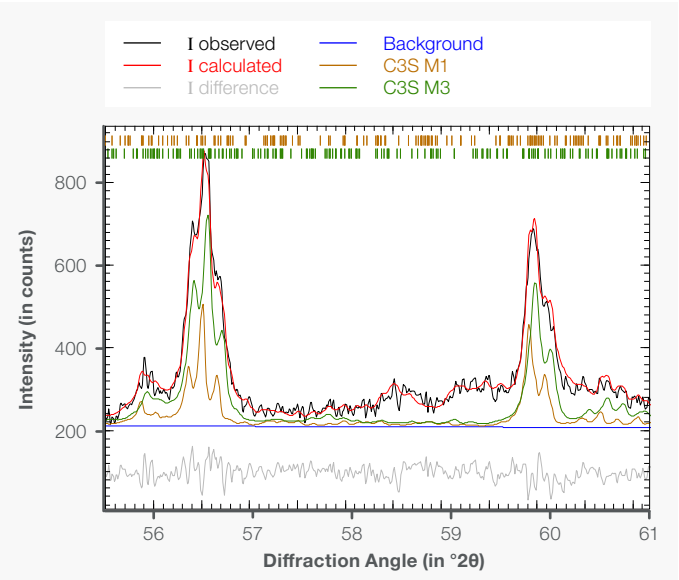

Figure 3. Zoom of CEM1 measurement (55-61° 2θ); Contribution of C3S M1 (brown) and C3S M3 (green) is shown and in good agreement with shape and intensity of the peaks.

	Average values	STDEV	ASTM
	(in %)	(1σ in %)	C150
C3S M1	22.64	1.17	
C3S M3	50.87	1.17	
C3S Total	73.52	0.39	Min 52%
C4AF	10.01	0.21	Max 10%
C2S β	2.21	0.34	Max 28%
C3A Cubic	1.85	0.50	
C3A Ortho	4.52	0.51	
C3A Total	6.37	0.24	Min 6%
Anhydrite	0.34	0.10	
Bassanite	0.30	0.22	
Gypsum	5.69	0.35	
Sulphates Total	6.32	0.33	Max 10%
Calcite	0.47	0.20	
Lime	0.24	0.10	Max 1%
Periclase	0.19	0.05	
Quartz	0.67	0.06	

Table 1. Results of 11 consecutive refinements of CEM I (values in weight percent)

References

1. N. Döbelin, R. Kleeberg, J. Appl. *Crystallogr.* **2015**, *48*, 1573-1580.
2. M.A.G. Aranda, A.G. De la Torre, L. León-Reina, *Rev. Mineral. Geochem.* **2012**, *74*, 169-209.

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