Application note | AN41505

X-ray diffraction

Quantification of amorphous cementitious materials based on CPL using ARL X'TRA Companion Benchtop XRD

Figure 1: ARL X'TRA Companion diffraction system

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Introduction

Amorphous materials play a pivotal role in cementitious products, influencing the strength, durability, and sustainability of concrete. These non-crystalline components, often present in supplementary cementitious materials (SCMs) like fly ash, slag, calcined clay and pozzolans, contribute to the unique properties of cements. The quantification and identification of amorphous phases are crucial for understanding cement's properties and performance. X-ray Diffraction (XRD) excels as a key analytical technique for this purpose.

X-ray diffraction provides valuable information about the mineralogical composition of a substance by analyzing the diffraction patterns produced when X-rays interact with a sample. Through this information, XRD enables the quantification of both crystalline and amorphous phases in cementitious materials. The current top XRD method for quantitative analysis of crystalline materials makes use of Rietveld refinements; to quantify amorphous content, methods based on internal or external standards are used. A more recent standardless approach[1–3] called PONKCS (partially or not known crystal structures method) is based on a list of calibrated peaks. Profex Rietveld software implements a similar method, based on a calibrated peaks list (CPL) derived from known mixtures to quantify unknown materials and amorphous content accordingly.[4]

As the cement industry strives for sustainability, the ability to understand and control amorphous content through XRD analysis will contribute to the development of eco-friendly cements, allowing the industry to align with global initiatives for greener construction practices. In decarbonized cement, which emphasizes reduced environmental impact, the use of SCMs rich in amorphous content is prevalent. Traditional slag and pozzolan cements leverage this aspect, incorporating materials like blast furnace slag and volcanic ash.

Instrument and software

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (Figure 1) is a simple, easy-to-use benchtop 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 coupled with 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. Thanks to its state-of-the-art solid state pixel detector (55 x 55 µm 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

Powdered mixtures of clinker and amorphous slag (nominal 20, 30, 50, 70 and 80 weight % slag) were measured in reflection using an ARL X'TRA Companion with Cu Kα radiation (10 min) (see Figure 2). Analysis of the slag yielded an amorphousness of 97% and the amorphous content levels of the mixtures were calculated accordingly. Phase quantification was performed with Profex (BGMN algorithm).[4] using a fundamental parameters approach. Quantification of the amorphous content was carried out using CPL with the 48.5% amorphous content mixture used as the reference sample (50% slag).

Results

Quantifying the amorphous content in certain slag mixtures using CPL shows results within a deviation of 1% absolute error (see Figure 3) and a linear correlation of 0.999.

Conclusion

Using CPL in Profex on data measured with the ARL X'TRA Companion allows quantification of the amorphous content within 1% absolute error without the need for an internal standard. Therefore, the ARL X'TRA Companion is an ideal solution for any routine analysis task, especially decarbonized cements.

References

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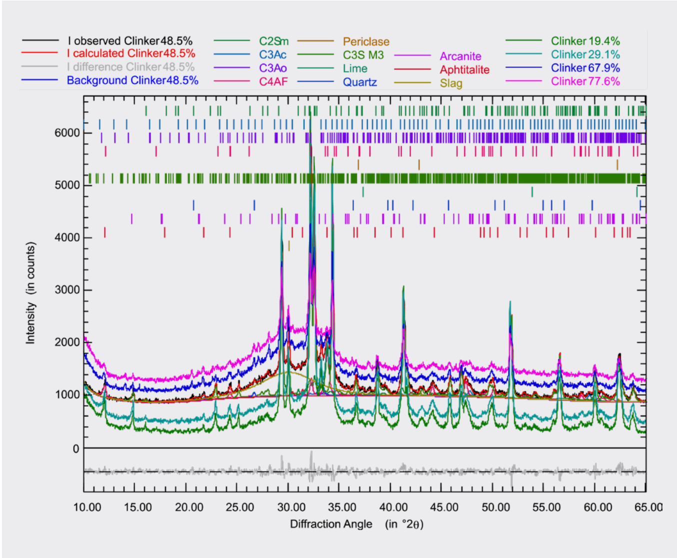

Figure 2: Rietveld fit of 48.5% amorphous content clinker mixture (center; black) and additional mixtures below and above; green 19.4%, turquoise 29.1%, blue 67.9% and purple 77.6% amorphous content.

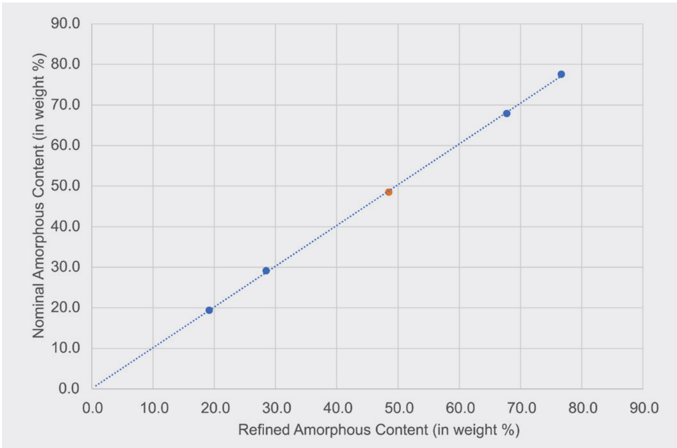

Figure 3: Nominal vs refined amorphous content with trend line; reference sample in orange.

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