Application note | AN41509

X-ray diffraction

Analysis of natural pozzolan in cement (CEM IV)

ARL X'TRA Companion X-ray Diffraction System

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Introduction

Natural pozzolans are commonly used as additives in CEM IV cement to enhance its properties and reduce the carbon footprint. CEM IV cement is a blend of Portland cement and cementitious materials, with natural pozzolans being derived from volcanic ash or other naturally occurring sources. X-ray diffraction (XRD) is a valuable quality control (QC) tool used through all steps of the cement manufacturing process. XRD helps identify and quantify crystalline and amorphous phases ensuring consistent quality and performance.

The use of natural pozzolans in CEM IV cement significantly reduces its carbon footprint. By incorporating natural pozzolans, a portion of the Portland cement is replaced, resulting in lower carbon dioxide emissions during production. Additionally, natural pozzolans often require less energy for activation, further reducing greenhouse gas emissions associated with cement manufacturing. The utilization of natural pozzolans in CEM IV cement, analyzed using XRD as a QC tool, promotes sustainable construction practices by improving performance and reducing environmental impact.

Instrument & software

The Thermo Scientific™ ARL™ X'TRA Companion XRD System (c.f. Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion XRD System 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 the state-of-the art solid state pixel detector (55 x 55 µm pitch), the ARL X'TRA Companion XRD System provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (Laboratory Information Management System).

Figure 1. ARL X'TRA Companion Diffraction System.

Experimental

In a first step, natural pozzolan was mixed with ~20 wt % ZnO as internal standard to determine the amorphousness. This information was used in a second step to determine a calibrated peak for standardless quantification of the amorphous content. Pozzolan samples and CEM IV with added pozzolan were measured in reflection mode using Cu Kα (1.541874 Å) radiation for 10 minutes. The samples were prepared in top loading sample cups and acquisition was performed with spinning sample. (c.f. Figure 2 and 3).

Results

Using ZnO as an internal standard, an amorphous content of 22.87 wt% was determined for the natural pozzolan sample (cf. Table 1). The total pozzolan content in the CEM IV sample amounts to 40.7 wt% including amorphous content derived from a calibrated peak (cf. Table 2). In the combined refinement, the presence of C3S M1/M3 and various Ca-sulphate species was also determined. All values fall within the typical range for CEM IV cement.

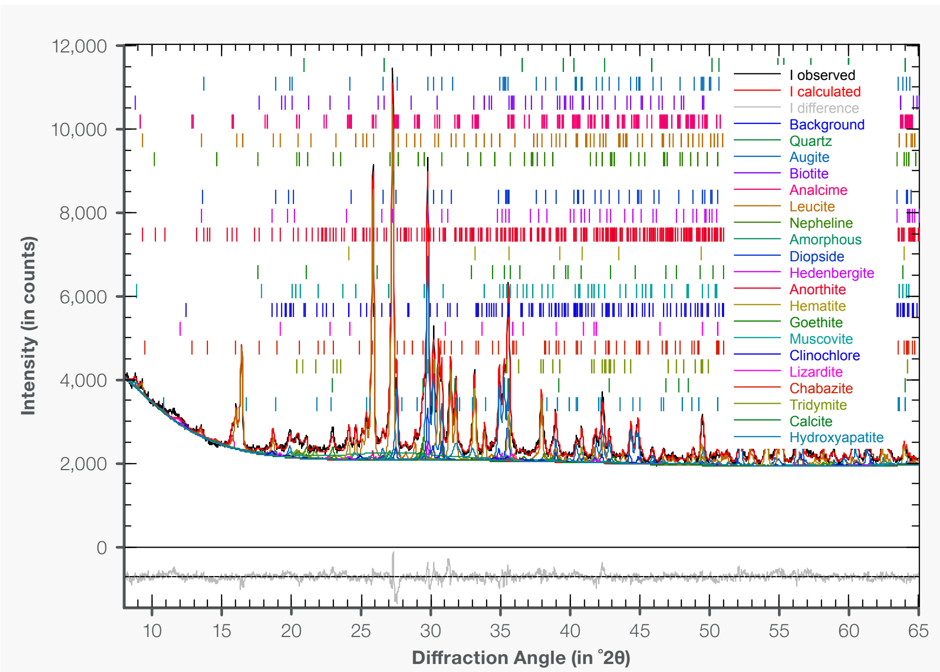

Figure 2. Measurement (10 minutes) of natural pozzolan (calibrated peak for amorphous content).

Phase	Augite $CaMg_{0.75}Fe_{0.25}Si_2O_6$	Biotite $KMg_2FeAl\ Si_3O_{12}$	Analcime $NaAlSi_2O_6$	Leucite $KAlSi_2O_6$	Nepheline $NaAlSiO_4$	Amorphous	Diopside $CaMgSi_2O_6$	Hedenbergite $CaFeSi_2O_6$
Wt %	25.15	0.42	0.68	25.93	4.10	22.87	5.30	0.71
Phase	**Hematite Fe_2O_3**	**Goethite FeO(OH)**	**Muscovite $KAl_3Si_3O_{10}(OH)_2$**	**Clinochlore $Mg_5Al_2Si_3O_{10}(OH)_8$**	**Lizardite $Mg_3Si_2O_5(OH)_4$**	**Tridymite SiO_2**	**Calcite $CaCO_3$**	**Hydroxyapatite $Ca_5(PO)_4OH$**
Wt %	3.34	0.15	3.07	1.96	1.35	1.39	0.66	2.94

Table 1. Results of Rietveld refinement of Pozzolan (Amorphous content by calibrated peak).

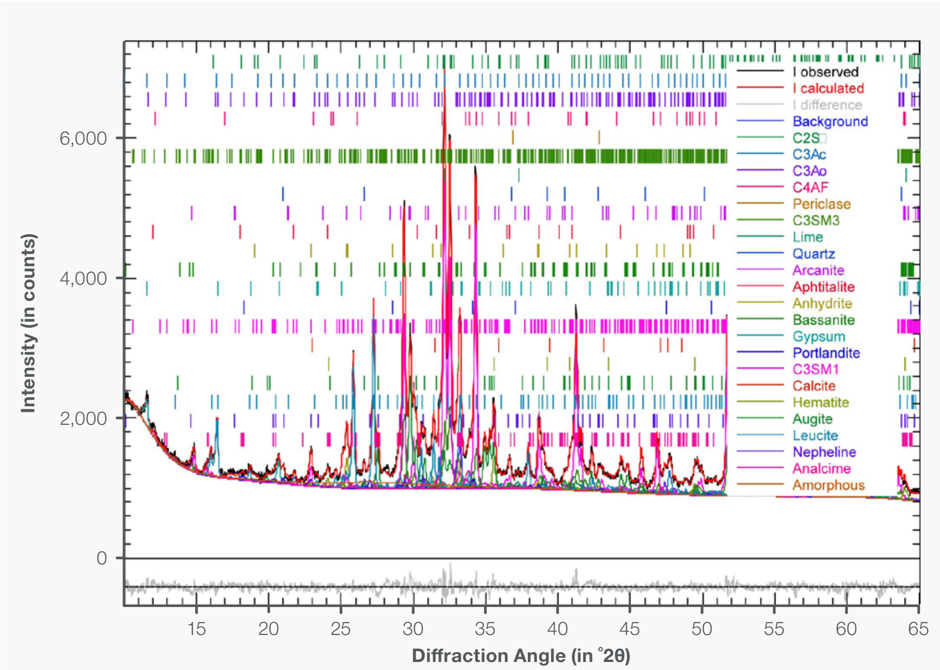

Figure 3. Measurement (10 minutes) of Cem IV with pozzolan.

Phase	C2S ß Ca_2SiO_4	C3A c $Ca_3Al_2O_6$	C3A o $Ca_{2.797}Fe_{0.15}Na_{0.292}Al_{1.725}Si_{0.125}O_6$	C4AF Ca_2FeAlO_5	C3SM1 Ca_3SiO_5	C3SM3 Ca_3SiO_5	Lime CaO	Quartz SiO_2	Arcanite K_2SO4
Wt %	6.37	3.68	2.78	1.87	25.64	11.92	0.11	0.07	0.28
Phase	**Bassanite $CaSO_4·0.5H_2O$**	**Gypsum $CaSO_4·2H_2O$**	**Anhydrite $CaSO_4$**	**Periclase MgO**	**Calcite $CaCO_3$**	**Aphtitalite $K_{1.5}Na_{0.5}SO_4$**	**Portlandite $Ca(OH)_2$**	**Pozzolan total**	
Wt %	1.00	2.93	1.28	0.43	0.29	0.28	0.36	40.70	

Table 2. Results of Rietveld refinement of CEM IV (Amorphous content by calibrated peak).

Conclusion

The ARL X'TRA Companion XRD System is perfectly suited to determine the total pozzolan content in CEM IV during Rietveld refinement, utilizing a calibrated peak for quantifying the amorphous content. The instrument is configured with one-click analysis functionality to enable easy operation.

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