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B-XRD1147 - High-precision quantitative analysis of clinker mineral polymorphs by Rietveld refinement

Introduction

Properties of cement—such as curing time and strength—depend on the components in the clinker minerals and the ratios of the amounts of polymorphs. There are two typical polymorphs of clinker minerals, known as M1 and M3 types of alite, and cubic and orthorhombic forms of aluminate. The amount ratio of polymorphs can be evaluated easily and quickly by Rietveld refinement because the diffraction patterns vary depending on which polymorphs are present. Precise analysis is required for high-precision cement characterization. We evaluated the precise quantity of the clinker minerals by Rietveld refinement by performing ten times iterative measurements (5 min. each) using a benchtop X-ray diffractometer equipped with a high-speed 1D X-ray detector.

Measurements and results

Ordinary Portland Cement (OPC) was used for the measurement. Rietveld refinement was performed using the SmartLab Studio II integrated X-ray analysis software with a template that allows multiple data analyses under the same conditions. Identical crystal structure information, initial values of refinement parameters, and the refinement procedure were employed throughout the refinement. Table 1 shows the average quantitative value and standard deviation (1 σ (n = 10)) for each component. The standard deviations of the quantitative values are very small. It is concluded that Rietveld refinement using a template against multiple datasets enables high-precision quantitative analysis of cement samples.



Figure 1: Result of profile fitting of OPC by Rietveld refinement

Table 1: The average quantitative value and standard deviation for each component in OPC (Units: mass%, Standard deviation: 1σ (n = 10))

ltem	∑Alite	M1	M3	Belite	∑Aluminate		Orthorhombic	C₄AF	Others
Average	68.00	23.56	44.44	13.60	5.34	1.83	3.51	9.45	<2.00
σ	0.15	0.12	0.16	0.18	0.05	0.05	0.05	0.09	<0.1

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