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Application News

No. X263

X-ray Diffraction

X-ray Diffraction Analysis of Cement (2)

- Quantitative Analysis of Compounds Using the Rietveld Method -

Cement is manufactured through the processes of crushing and mixing raw materials, calcination, and finishing. The crushing and mixing process uses raw materials such as limestone, clay, silica stone, and iron oxide and these materials contain alite (C3S), belite (C2S), aluminate (C3A), and ferrite (C4AF). Since the ratios of these compounds differ according to manufacturer and product type and significantly affect product performance, cement is analyzed using X-ray fluorescence and X-ray diffraction. However, even with X-ray diffraction, which is capable of qualitatively analyzing powder samples, quantitative analysis of samples consisting of multiple components is known to be difficult due to considerable overlapping of diffraction lines.

This article introduces an analysis example of a cement powder sample which is known the component composition and the ratio, using Siroquant software (Sietronics Pty. Ltd.) applying Rietvelt analysis method which is the one of the profile fitting method. Siroquant enables identification of composition components and quantitative analysis in addition to the element analysis using X-ray fluorescence in quality control of cement.

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Sample

Cement reference material for X-ray fluorescence analysis: 601A (Japan Cement Association)

Qualitative Analysis Using the XRD-6100

Measurement was performed using the XRD-6100 and detection using the OneSight wide-range high-speed detector.

Table 1 lists the measurement conditions and Fig. 1 shows the X-ray diffraction pattern of the cement powder sample. We can see from the qualitative analysis results the six types of composition components which are alite, belite, ferrite, aluminate, hemihydrate gypsum, and periclase. We can also see overlapping of the diffraction peaks of the main components, alite and belite, in the 2θ = 32° to 34° range. Table 2 lists the qualitative analysis results and the notation of the composition components that correspond to the diffraction peaks in Fig. 1.

Table 1 Measurement Conditions

Instrument	:	XRD-6100, OneSight
X-ray tube	:	Cu target
X-ray condition	:	30 kV – 50 mA
Monochromatization	:	Ni filter
Divergence slits	:	0.5 deg.
Scan range	:	10 – 80 degree
Scan mode	:	Step scan - Standard
Scan speed	:	10 deg./min
Rotation	:	30 rpm

Table 2 Qualitative Analysis Results

Chemical Formula *		Composition Component	
$Ca_3(SiO_4)O$	:	C3S	alite
Ca_2SiO_4	:	C2S	belite
$Fe_2O_3Al_2O_3(CaO)_4$	:	C4AF	ferrite
$Ca_3Al_2O_6$	:	C3A	aluminate
$Ca(SO_4)(H_2O)_{0.5}$	:	Ca	hemihydrate gypsum
MgO	:	MgO	periclase

* Chemical formula notations correspond to that of the ICDD database.

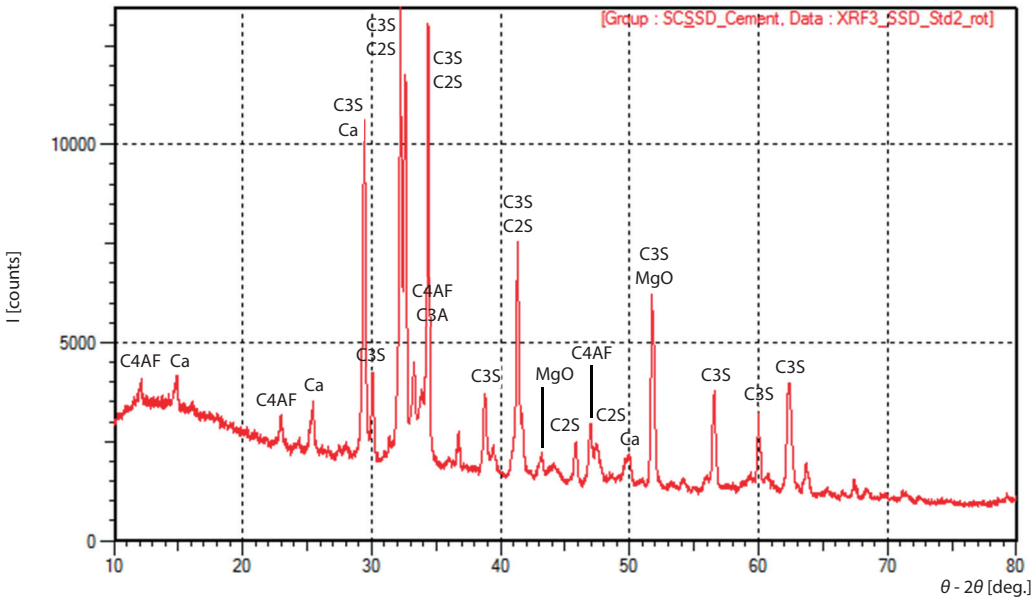

Fig. 1 X-ray Diffraction Pattern of Cement Powder

Quantitative Analysis Using the Siroquant Software

Quantitative analysis using a calibration curve is difficult for samples that contain multiple crystal phases with overlapping diffraction peaks as in Fig. 1. In such a case, Rietveld method which quantitatively analyzes from the theoretical diffraction pattern of the database and the measured diffraction pattern, using the profile fitting method by least squares method, is the best. The Siroquant software performs quantitative analysis of crystal compounds using the Rietveld method and executes calculations using parameters including the lattice constant, theoretical profile, and diffraction peak height as variables.

Quantitative analysis was performed with Siroquant to determine the ratios of the composition components listed in Table 2. Fig. 2 shows the fitting screen displayed by Siroquant. Green represents the actual diffraction pattern and red represents the calculated diffraction pattern. The deviation between the green and red diffraction patterns is shown below in blue. We can see very little deviation and a fit that exhibits good accuracy (performance index indicating degree of fit: x^2 = 16.04). Since actual measurement data can also be registered to the Siroquant database, substances with wide full width at half maximum (FWHM) values and high background noise due to low crystallinity as well as clay materials with strong orientation can be analyzed with favorable accuracy.

Table 3 and Fig. 3 show the quantitative analysis results. As shown, quantitative analysis results can be output in pie graph format or as numerical values.

Table 3 Quantitative Analysis Results

Unit: wt%

C3S	alite	72.6
C2S	belite	12.9
C4AF	ferrite	7.1
C3A	aluminate	4.9
Ca	hemihydrate gypsum	1.6
MgO	periclase	0.9

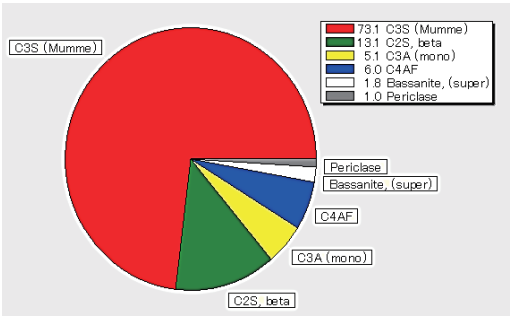

Fig. 3 Pie Graph Display of Quantitative Analysis Results

Element Analysis Using X-ray Fluorescence (EDX)

Element analysis information is required to perform qualitative and quantitative analysis of compounds using X-ray diffraction. On the sample used for X-ray diffraction, element analysis was performed with EDX. Fig. 4 shows the quantitative analysis result screen using the fundamental parameter (FP) method when element is assumed to be oxide. The sample in powder form was measured in a sample container as it was.[*1]

By EDX analysis, the constituent element of the composition component by X-ray diffraction shown in Table 2 was detected. This shows that the elemental analysis result by EDX is useful information for the qualitative and quantitative analysis of X-ray diffraction when analyzing an unknown sample.

Analyte	Result	
CaO	66.815	%
SiO2	20.722	%
Al2O3	4.143	%
SO3	3.295	%
Fe2O3	2.333	%
MgO	1.734	%
K2O	0.454	%
TiO2	0.240	%
MnO	0.066	%

Fig. 4 Results of Quantitative Analysis Using the FP Method and Qualitative Analysis

*1 Shimadzu Application News No. X247

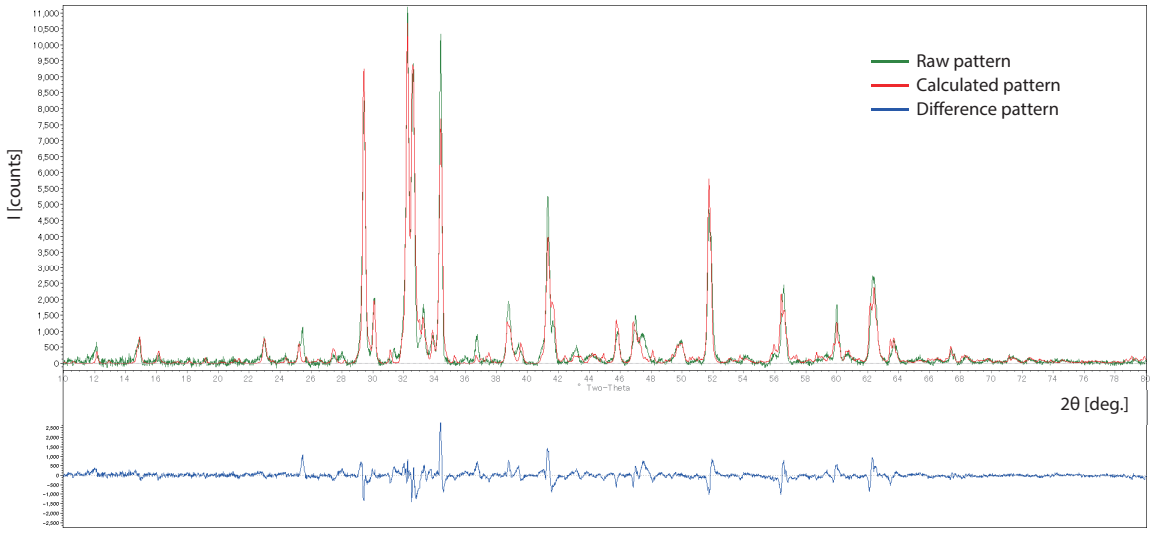

Fig. 2 Profile Fitting Screen for Cement

First Edition: Sep. 2017

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