

Cement analysis by X-ray diffraction and X-ray fluorescence

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Cement is one of the most important materials for construction and reacts with water to form hydrates, which gradually condense and harden. This hardening is caused when the formed hydrates coat cement particles and bind them together. A detailed understanding of the hydrate formation process is expected to lead to the elucidation of the mechanism of condensation and hardening. X-ray diffraction (XRD) techniques can identify crystalline hydrates such as ettringite and monocarbonate in cement. We report on the observation of changes in the crystalline phase or ordinary Portland cement over time with a water/cement ratio of 25% using a liquid sample holder which can prevent the sample from drying in an ambient environment by covering the sample surface with a film. Fig. 1 exemplifies changes in the XRD pattern of cement during a hydration reaction.

In addition, X-ray fluorescence (XRF) techniques are used to control the chemical composition of cement products and interim products. Since the fusion method can eliminate sample heterogeneity problems, such as grain size and mineralogical effects, it is possible to obtain high accuracy for cement samples and to establish calibrations using a variety of materials. ASTM C114-18 covers chemical analysis of hydraulic cements. In this standard, mainly procedures of wet chemical analysis are described and XRF spectrometry is mentioned as an example of “Rapid Test Methods”. In practice, XRF spectrometry has been used for chemical composition analysis of cement owing to its simple sample preparation and high precision. We will show quantitative analysis for Portland cements by the fusion method according to ASTM C114-18, as displayed in Table 1, using a multi-channel simultaneous wavelength dispersive XRF (WDXRF) spectrometer, which enables the simultaneous measurement of all the elements in the sample under investigation. The counting time of the measurement was 40 seconds for twelve elements in cement.

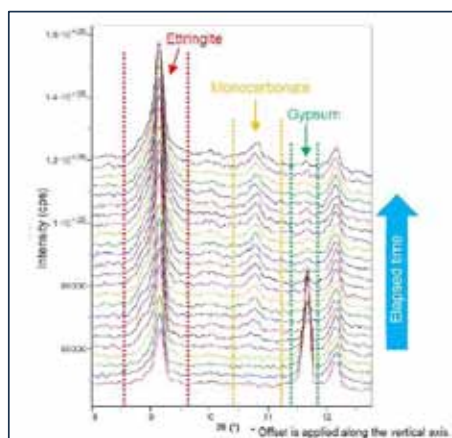


Figure 1. Overlaid X-ray diffraction patterns of hydrated cement.

Analysis component	Analysis range	Difference between duplicates		Qualified
		Limit (ASTM)	Maximum difference	
SiO ₂	18.637 – 22.38	0.16	0.05	✓
Al ₂ O ₃	3.875 – 7.06	0.20	0.01	✓
Fe ₂ O ₃	0.152 – 3.09	0.10	0.003	✓
CaO	57.58 – 67.87	0.20	0.14	✓
MgO	0.814 – 4.475	0.16	0.01	✓
SO ₃	2.086 – 4.622	0.10	0.01	✓
Na ₂ O	0.021 – 1.068	0.03	0.006	✓
K ₂ O	0.093 – 1.228	0.03	0.004	✓
TiO ₂	0.084 – 0.3663	0.02	0.003	✓
P ₂ O ₅	0.022 – 0.306	0.03	0.001	✓
ZnO	0.0048–0.107	0.03	0.0004	✓
Mn ₂ O ₃	0.0073–0.2588	0.03	0.001	✓

Table 1. Qualification test results (unit: mass%)