Quantitative Phase Analysis of Samples Containing Low Crystallinity Components by Using the Direct Derivation (DD) Method

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The direct derivation (DD) method for quantitative phase analysis (QPA) can derive weight fractions of individual crystalline phases in a mixture from sets of observed intensities, measured in a wide 2θ range, and chemical composition data [1 – 3]. In the present study, it has been incorporated into the whole-powder-pattern fitting (WPPF) procedure to conduct QPA of samples, powder diffraction patterns of which are hard to be analyzed by ordinary procedures of the decomposition methods or the Rietveld method. In Rietveld QPA, structure parameters such as atomic coordinates are required to delineate the diffraction patterns of individual components, and adjusted scale parameters for individual phases are used to calculate the weight fractions. The scale parameters represent, in this case, sample-dependent observed quantities determined by XRD experiments. In the DD method, total sums of scattered intensities from individual phases are used to represent the observed quantities, expressed in a single unit of measurement with intensity counts. Therefore, any form such as the sum of integrated intensities and/or integrated profile intensities can be used irrespective of whether they are multiplied by the scale parameters or not. In the case of degraded diffraction patterns, for example, of clay minerals, observed profile intensities can be used instead of calculated intensities, and they can directly be fitted together with the diffraction patterns of other phases as has been done by a full-pattern fitting technique [4]. The integration of background-subtracted profile intensities of the diffraction pattern provides a quantity, which is equivalent to the sum of integrated intensities of individual diffraction lines. In the DD method, the molecular weight and the sum of squared numbers of electrons belonging to atoms in the chemical formula unit replace the sum of squared structure amplitudes, used in Rietveld QPA. Therefore, the DD method can be applied to QPA of any mixture consisting of known structure, unknown structure, and high and low crystallinity components. QPA of mixtures consisting of α-quartz, albite and kaolinite will be shown to demonstrate the capability of the present procedure.

References

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