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X-ray microdiffraction as a tool for quantitative phase analysis

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This work aims to verify if data obtained via laboratory-based X-ray microdiffraction are suitable for mineralogical quantitative analyses and to determine the best instrumental parameters for quantitative phase analyses using powder diffraction.

A X-ray microdiffractometer needs few milligrams of powder material to obtain a complete X-ray spectrum and can directly work on thin sections and bulk matrices; this routine technique could satisfy the claims of a lot of experiments where small samples are used and mineralogical quantitative determinations are required.

Different ratios of solid binary mixtures of quartz, calcite, kaolinite, and illite were prepared by analytical balance: 5/95, 25/75, 50/50, 75/25, 95/5. These powder mixtures were analyzed using a Rigaku Rint 2200 instrument with a conventional Bragg-Brentano geometry equipped with a copper tube and a graphite crystal monochromator. The data were collected in the angular range 3-100° 2θ with steps of 0.02° and 5 s/step. The data collections were converted into GSAS files for EXPGUI software and quantitatively refined by Rietveld method. These refinements and weight amounts were used as references for the following data collections.

The same powder mixtures were analyzed by a X-ray microdiffractometer Rigaku D-Max Rapid, equipped with an image plate detector, a flat graphite monochromator, a microscope for the positioning of the sample in the path of the X-ray beam, operating at 40 kV and 30 mA with CuKα radiation. Each powder sample was collected with different beam collimators (beam diameters: 0.03, 0.05, 0.10, 0.30 mm), different collection times, two fixed values of omega angle (3° and 6°), five angular ranges of rotation of phi angle (0°, 30°, 90°, 180°, 360°). The XRD data were collected as two-dimensional images and converted into $2\theta - I$ profiles; further conversions into GSAS files for EXPGUI software were carried out, and the data were quantitatively refined by Rietveld method.

The results of these refinements were compared with the weight amounts and the results obtained by the other X-ray technique, highlighting X-ray microdiffraction appears to be suitable for mineralogical quantitative analyses using different instrumental parameters.