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Quantification of minor phases in growth kinetics experiments with powder X-ray diffraction

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ABSTRACT

Minor amounts of clay minerals precipitated from aqueous solution can be rapidly identified and quantified in a mineral mixture with powder X-ray diffraction using a rotating-anode source and a position-sensitive detector. For the case of gibbsite precipitated on a kaolinite powder substrate we demonstrate a simple method having a minimum detection limit of 0.1 wt%, using pure gibbsite as the intensity reference in mechanical mixtures of gibbsite and kaolinite. The amount of gibbsite precipitated onto kaolinite at 80 °C, pH 3 is higher when determined from solution chemistry than from the X-ray method, and the difference in amounts increases with increasing Al concentration in solution. This discrepancy can be explained by assuming that a fraction of the precipitated material is effectively invisible to the X-ray diffraction technique, either due to a small diffracting domain size along the gibbsite [001] direction or formation of an Al-phase that is amorphous to X-rays. This method should be generally useful for a range of mineral mixtures where at least one intense reflection for the phase of interest is not obscured. The ability to identify, characterize, and quantify trace phases by X-ray diffraction, especially when combined with surface analysis by electron or atomic force imaging, is an important complement to the conventional approach of monitoring solution composition in growth kinetics experiments.