Characterization of Intercalated Smectites Using XRD Profile Analysis in the Low-Angle Region

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Abstract: X-ray diffraction (XRD) characterization of natural and intercalated smectites is usually limited to the apparent *d*-value estimated from the peak maxima in the raw data. This can lead to the misinterpretation of the measured data. In the case of XRD, the interference function is modulated by instrumental factors (Lorentz-polarization factor, diffraction geometry) and physical factors (structure factor, surface roughness effect). These effects lead to diffraction profile distortions, depending on the diffraction angle and peak full width at half maximum (FWHM). As a result, the diffraction profiles for structures with large line broadening (FWHM > 1 °) exhibit a significant peak shift ($\Delta d \sim 1.5$ Å), especially at low angles ($2\theta \leqslant 10$ °). The present work deals with the detailed analysis of all these effects, their corrections and their consequences for the interpretation of diffraction patterns (including possible errors in determining lattice parameters or the structure model). The investigated materials were montmorillonites (MMT) intercalated with hydroxy-Al polymers. Diffraction profile analysis revealed the corrected *d*-values and showed that the intercalated sample is not a mixed-layered structure. As a result a structural model of the interlayer is presented.

Key Words: Montmorillonite • Profile Analysis • Smectite • XRD

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