X-RAY POWDER DIFFRACTION ON HEATED SUPPORT: NEW METHOD IN CLAY DIAGNOSTICS

BEZDICKA, P., GRYGAR, T., HRADIL, D., HRUŠKOVÁ, M.

Institute of Inorganic Chemistry AS CR [Ústav anorganické chemie AV ČR], Řež, 250 68, Czech Republic
E-mail: hradil@iic.cas.cz

Thermal behaviour of clays mirrors phase composition and structure. In multicomponent clay mineral mixtures, their dehydration and further transformation could be used as one of the fundamental diagnostic features reflecting structure and mineralogy. The interlayer space in expandable structures sensitively reflects temperature and humidity variations. The influence of the temperature on sorption and ion exchange processes on clays is discussed, for example, when evaluating clay barriers in the radioactive waste disposal. Dynamic behaviour of the interlayer space is not possible to monitor by conventional powder diffraction measurements of preheated samples, also because of full or partial reversibility of the process up to 300°C.

The powder diffraction measurements were performed in high-temperature XRD cell (Anton Paar HTK 16) with thin layers of samples deposited as methanol or water suspension of wet samples with diffractometer PANalytical X'Pert PRO, CoKα (30 kV, 45 mA) and X'Celerator detector. For identification of dehydration of clay minerals we performed thermal scan with step of 5°C starting from ambient temperature up to 300°C. At each step we measured one diffractogram in the angular range from 4 to 40° 2θ and examined the development of positions and integral intensities of basal reflections of expandable structures during the sample heating.

We have measured reference clay minerals, natural bentonites and sedimentary clays. In the example of smectite from Brodce dehydration of interlayer space occupied by different cations is compared. In all cases a continual reduction of the interlayer results in movement of the 001 diffraction towards d ≈ 10 Å. Differences are in the final temperature and mechanism of dehydration. Ca-Mg smectite contains water in two molecular layers and therefore intermediate single-layer structure is clearly visible at d ≈ 12 Å. When comparing monovalent cations, Na-interlayers dehydrate much easier than Li-interlayers; it could be explained by higher density of surface charge of Li^+.

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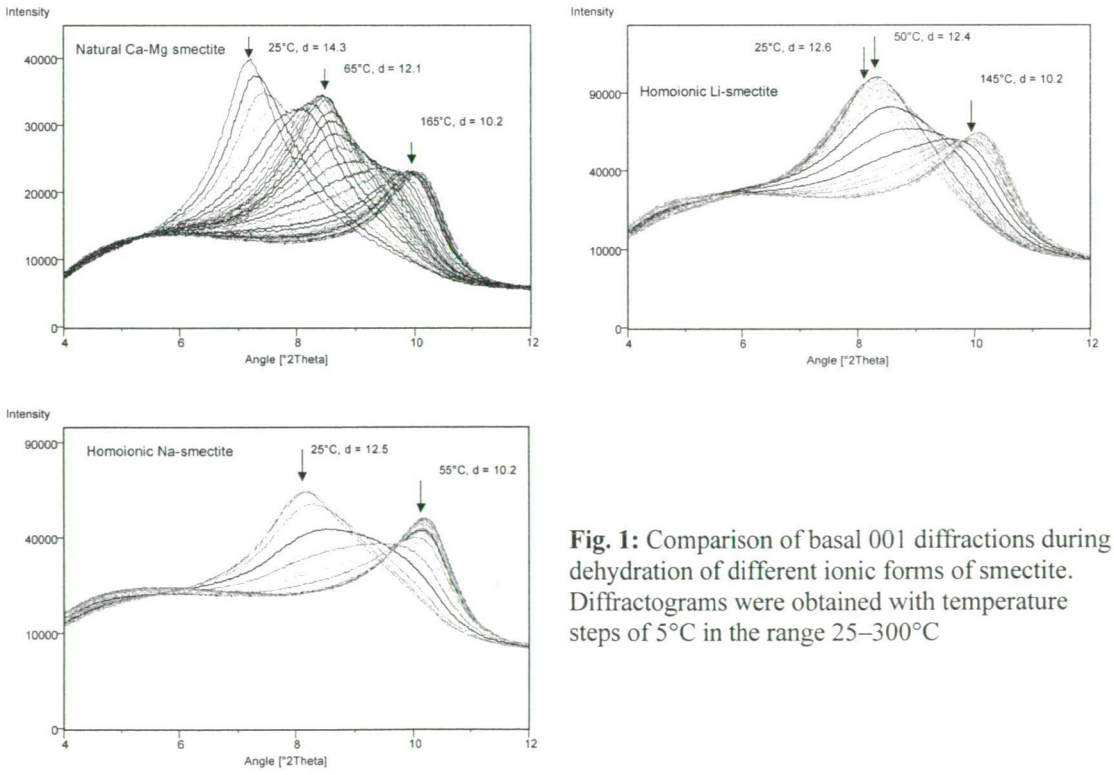

Fig. 1: Comparison of basal 001 diffractions during dehydration of different ionic forms of smectite. Diffractograms were obtained with temperature steps of 5°C in the range 25–300°C