EXPERIMENTS WITH X-RAY DIFFRACTION FOR SMECTITE PRESENCE ON MIXTURES WITH KNOWN COMPOSITION: IDENTIFICATION, QUANTIFICATION AND LIMIT OF DETECTION FOR ARTIFICIAL "SANDSTONE"

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ABSTRACT

X- ray diffraction (XRD) is an essential tool for the analysis of clay minerals, especially smectites, wherever they occur and sandstones are not exception. Smectites give basal (001) and (00l) type peaks with different d-values, depending on chemical composition. Interlayer hydration, the number H2O molecules in hydration shells, is also an important factor regarding chemical composition, besides cation content of the interlayer space. Using (0k0) and (hk0) peaks also, the (00l) are most important for the possible differentiation between smectite compositional types, or even smectite species. There are several specimen dependent contributions expected in XRD measurments. Smectite basal peaks are usually extremely broadened, thus overlapping peaks for different smectites are problematic to recognize. This issue is further deepened by the small amount of smectites, e.g. what peak intensity should we expect at ~ 5% total smectite content? And also other questions arise like, can we separate smectite peaks by line decomposition? And if we do, is the result not controlled only by interlayer hydration state?

In our experiments we have used minerals in pure state to prepare mixtures of known composition, similar to sandstone composition. All minerals were individually characterized by X-ray powder diffraction (XRD), X-ray fluorescence spectrometry (XRF) or inductively coupled plasma optical emission spectrometry (ICP-OES), Fourier-transform infrared spectrometry (FTIR) and thermal analysis (TA) where needed. For smectite content we have used: smectite from Kopernica (Slovakia) bentonite; hydrothermal smectite from andesite (Mátra Mts., Hungary); smectite+talk from metaserpentinites (Sopron Mts., Hungary). Non-clay components are: quartz, calcite, dolomite, feldspars, muscovite and biotite. The other than smectite clay minerals used for clay fraction and rock simutation were: dickite from coal-andesite interaction of the Mecsek Mts (Hungary) and diagenetic clinochlore (Bükk Mts., Hungary). Mixtures similar to sandstone were prepared with +/- 10% of single mineral variation (several sets) around a basic composition (50% quartz, 10% glass, 10% feldpsars, 10% micas, 5% of calcite+dolomite, 5% dickite, 10% of total smectite).

Quantitative analysis of clay minerals in sandstones is a requirement of many investigations and the potential of XRD in this respect is unsurpassed. The theory that underlies a large set of the published methods of quantitative analysis by XRD is outlined. Two common methods of preparing samples for clay analysis were applied, namely the analysis of oriented clay diffractions and the analysis of whole-rock samples as random powders. Issues of sample preparation, measurement of peak intensities, validation and uncertainty, and lower limits of detection will be discussed for each method and illustrated by examples. Studies of both oriented clay fractions and whole- rock random powders provide complementary information.

***Keywords**: X-ray diffraction, smectite, swelling, sandstone, quantitative analysis*