

QUANTIFICATION OF SMALL AMOUNTS OF SWELLING MINERAL, SMECTITE, IN SYNTHETIC MIXTURES WITH KAOLINITE USING DIFFUSE REFLECTANCE INFRARED SPECTROSCOPY (DRIFTS)

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Processing problems associated with the presence of low quantities of smectite in kaolinite are caused by the difference in surface area between the non-swelling kaolinite (15–30 m²/g) and swelling mineral, smectite (700–850 m²/g). This surface area issue is a problem for the coatings industry, and for reservoir rock analysis. In the oil industry, treatment fluids are pumped into reservoir rocks to increase well productivity. Interactions of this treatment fluid with swelling minerals on the pore surface may catastrophically alter the fluid composition.

The aim of this study was to determine low quantities (0.1–30wt%) of swelling mineral, smectite, in mixtures with non-swelling mineral, kaolinite, using DRIFTS and the chemometric technique, partial least squares (PLS). A third component, calcite, was included in the training set to avoid collinearity. Infrared spectroscopy and in particular, DRIFTS, is an effective technique to determine small quantities of these minerals since it is a sensitive, fast and relatively inexpensive technique. PLS is a decomposition technique which carries out the simultaneous decomposition of spectral and concentration data to construct a calibration model (Clegg, 1998; Forsyth, 2001). It was thought that PLS could improve the detection limit determined by (Madejová et al., 2002) using peak fitting. For a smectite-kaolinite mixture (Madejová et al., 2002) could detect kaolinite at levels as low as 0.5wt%, while smectite could not be detected below 40wt%.

A quantitative determination of components in mixtures was obtained using two software programs and the results compared. OMNIC[®] software and Spectracalc software package GRAMS[®] (Galactic Industries Corporation, NH, USA) were used. The most accurate models for the set, containing 21 standards in this study, do not include the OH stretching region, but instead use only the wavenumber region below 2150 cm⁻¹.

The training set was then exposed to a chemical probe, dimethylformamide (DMF). A chemical probe is an organic

molecule that easily enters the interlayer space of smectites but much less easily into kaolins. When a mineral mixture or rock is exposed to the chemical probe, the interactions of the molecules with different clay minerals are exploited to detect the clay mineral content. Therefore, a probe was used with the expectation that more accurate quantitative results would be obtained regarding the quantities of different minerals in a mixture. It was thought that the presence of the peak at 1658 cm⁻¹, assigned to intercalated DMF, would improve the models predictive ability. However the peak was found to be present in the spectra of both smectite and kaolinite after exposure to DMF and its presence actually reduced the accuracy and precision of predictions.

Spectra of the training set, without DMF exposure, were then collected using ball milled KBr diluent and a sample compaction device. It was thought that the smaller particle size distribution and homogeneous compaction would improve model predictions. The model produced using this method did give the most accurate predictions.

Results indicate that the highest error in kaolinite prediction is ±2.2%, in smectite is ±3.8%, and in calcite is ±4.9%. In conclusion, it has been found that smectite can be detected at lower concentrations (0.2wt%) using PLS than can be detected visually (30wt%) from a peak on a spectrum, with an error of ±3.8wt%.

References

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