XRD Analysis of Illite-Smectite Interstratification in Clays from Oil Sands Ores

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The presence of clays in oil sands, especially swelling clays such as smectite, has an adverse effect on bitumen recovery and tailings management. Identification and quantification of clay minerals are therefore important to the oil sands industry. Two estuarine (E3 and E7) and two marine (M13 and M18) oil sands were used in this study. Separation of <2um clay solids was accomplished using centrifugation technique based on Stokes' law. Higher clay contents are observed in marine than estuarine ore[1]. XRD patterns were collected on oriented-mount specimens prepared from Ca-saturated <2μm clay suspensions treated under various sample preparation conditions including: air drying at controlled 54% relative humidity, ethylene glycol solvation, and heat treatments at 375 and 550 °C. Comparison of XRD patterns produced from air-dried (AD) and ethylene-glycolated (EG) preparations confirmed the absence of discrete smectite, while revealing the presence of interstratified illite-smectite (I-Sm) and kaolinite as the primary clay minerals in these ores. Minor amounts of chlorite were found in significant quantities only in the marine ores. The degree of expandability owing to the presence of swelling (smectite) layer in the interstratified I-Sm component was measured using the intensity ratio[3], Ir = $[I(001)/I(003)]_{AD}/[I(001)/I(003)]_{EG}$, involving integrated intensities (I) of (001) and (003) reflections from AD and EG preparations. The Ir values determined for samples E7, E3, M18 and M13 were 1.05, 1.26, 1.34 and 2.23, respectively. Full width at half maximum (FWHM) of the illite 001 diffraction peaks were obtained from AD preparations. The FWHM values were of ~0.39–0.54 for E7, E9, and M18 and of 0.94 for M13. The plotting of FWHM *vs.* Ir values was used to estimate percentage of swelling layers[4]. This indicated that the contents of swelling layers were 4–6% for M13, 2–4% for M18 and E3, and nearly none for E7. By comparing various methods of measuring FWHM and Ir values, we conclude that these parameters may be quantified in a robust manner in XRD patterns from AD and EG preparations of <2um clays from oil sands ore. As the groupings of samples identified here in plotting FWHM *vs.* Ir values correlate well with batch-extraction unit bitumen recovery measured for the four ores[1], we suggest that this approach may represent a convenient way to assess the processability of oil sands ore. To validate this methodology, as such investigations have never been done before for oil sands clays, this type of analysis shall be applied to a large number of oil sands ore samples.

[1] Mercier et al. (2008) Energy & Fuels, 22: 3174-3193. **[2]** Moore & Reynolds (1997). X-ray Diffraction and the identification and analysis of clay minerals, 2nd edition. Oxford University Press, Oxford, New York. **[3]** Srodon (1984) Clays and Clay Minerals, 32: 337-349. **[4]** Eberl & Velde (1989) Clay Minerals, 24: 571-577.