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Structural Studies of Athabasca Oil Sands Minerals, Hydrotreating Catalysts and Cement Stabilized Chromium Compounds using Synchrotron X-ray Powder Diffraction

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Beamline(s): X3B1

Introduction: The high resolution and flux offered by a synchrotron x-ray source (powder diffraction) enable accurate refinement of crystalline mineral structures in Athabasca oil sands. The refined structures provide a baseline for greater quantification accuracy of diffraction data obtained from laboratory source x-ray. High resolution is also necessary for defining the structural changes that accompany thermal treatments of hydrotreating catalysts [1]. Understanding these structural changes is essential to designing catalysts with the proper pore structures to accommodate the different organic molecules in synthetic crude oil. The structure of two chromium compounds produced when chromium wastes are stabilized in Portland cement are elucidated. These compounds are essential to understanding the mechanism of chromium immobilization in Portland cement matrix [2].

Methods and Materials: Randomly oriented aggregates were investigated in flat plates and capillaries.

Results: Figure 1 shows ion-exchanged mordenite that had undergone de-alumination after acid washing and subsequent steam treatment. While the framework remained intact, the de-alumination process produced mesopores (55 – 115 Å) with uniform size distributions. The structures of the de-aluminated species are being refined to determine the pore structure directly from x-ray diffraction data. The reaction of calcium hydroxide with chromium (III) salts and chromium (VI) oxide produce the diffraction patterns shown in Fig. 2. Both of these products are observed during hydration of Portland cement used for stabilizing chromium wastes. The compounds determine the long-term leachability of chromium in cement matrices. The structures are being solved to facilitate an understanding of the reaction mechanisms. Figure 3 shows the diffraction pattern of a limestone deposit (Canadian McMurray formation) used for producing gypsum. The gypsum is used as coagulant for reclaiming the fluid fine tails resulting from the extraction of bitumen from oil sands. The important parameter is the Ca/Mg/Fe ratio in the calcite and ankerite minerals (carbonates) in the limestone. These ratios are important for modeling the water chemistry of the extraction process.

Conclusions: While the diffraction data are still being analyzed, preliminary assessments show that the structure determination of the chromium compounds and refinements of the mordenites and carbonates can be accomplished.

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References:

[1]. H. Yang, M. Wilson, C. Fairbridge, Z. Ring and O. Omotoso, “Engineering Pore Size of Mordenite by Acid and Hydrothermal Treatment,” (poster presentation), 17th North American Catalysis Society Meeting, Toronto, ON, Canada 2001.

[2]. O. Omotoso, “Mechanisms of Chromium Stabilization in Tricalcium Silicate,” Ph.D.Thesis, U. of Alberta, AB, Canada, 1996.

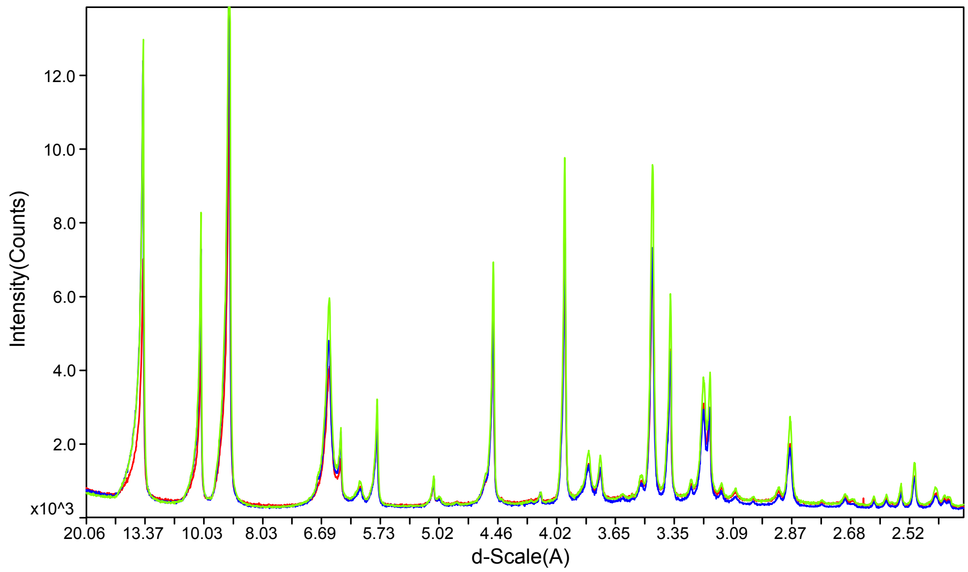
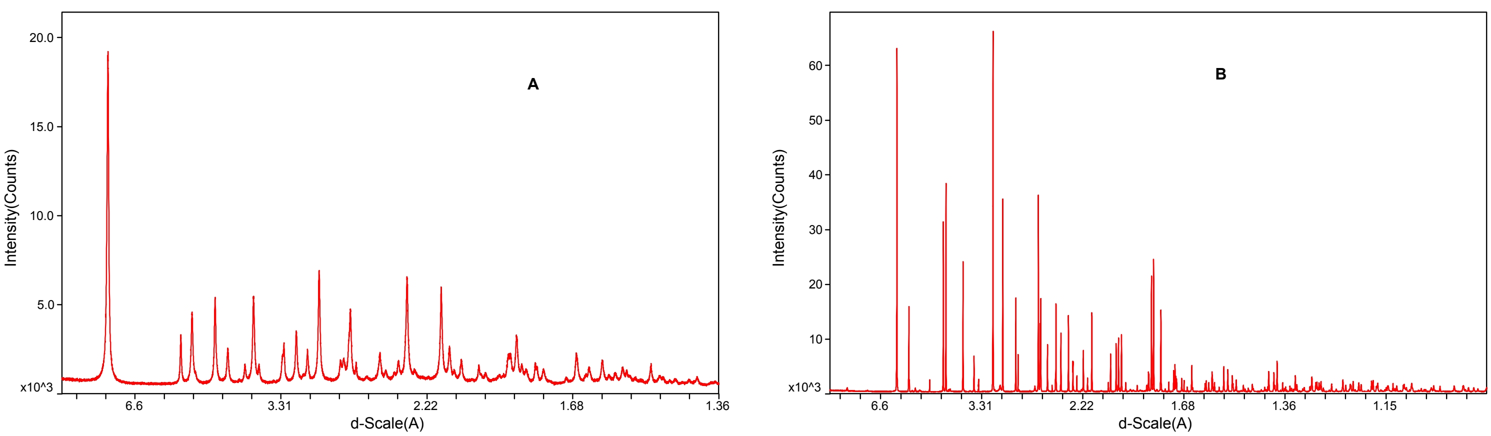


Fig. 1 – X-ray diffraction patterns of a zeolite (red) that was subjected to acid treatment (blue) and further hydrothermal



treatment (green)

Fig. 2 – XRD patterns of a Cr(III) analogue of ettringite (A) and a dicalcium chromate - $Ca_2CrO_5.3H_2O$ (B). Both are new structures identified in chromium wastes immobilized in cement.

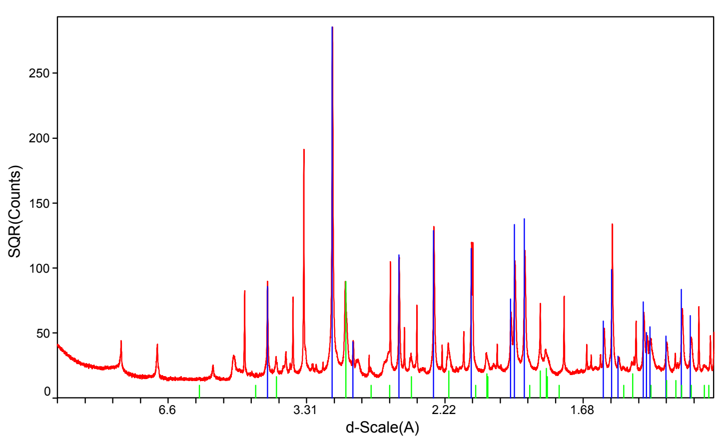


Fig. 3 – Limestone deposit containing calcite – $(Ca,Mg)CO_3$ (blue) and ankerite – $(Ca,Fe,Mg)_2(CO_3)_2$ (green)