

Abstract No. char774

Synchrotron X-Ray Diffraction Analysis: Applications in Catalysis Research

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Beamline(s): X3B1

Introduction: Analysis of materials by Synchrotron X-ray Powder Diffraction (SXRD) provides more and higher quality structural information than by sealed tube X-ray diffraction (XRD). In this project, data were collected on various catalysts for which high quality data were unattainable using regular XRD. This type of added knowledge is key in trying to understand the physical properties and reactivity of catalysts in chemical processes.

Methods and Materials: Powdered catalysts used in fluidized catalytic cracking (FCC) and natural gas conversion were investigated using SXRD at 1.540Å and 0.699Å wavelength. Measurements were conducted using two sample-mounting techniques: top loading with a zero background holder, and sealed quartz capillary.

Results: Fig. 1 shows low angle XRD and SXRD spectra of three well-dispersed mesoporous MCM catalysts. Their low crystallinity prevented lab measurement of mesopore channel dimensions beyond 45Å. SXRD yielded well-resolved peaks for the 45Å MCM's and observation of a corresponding to a 55Å channel aperture for a MCM that could not be measured on a lab instrument. The SXRD data were also used to evaluate metal dispersion.

Recently, we initiated a project to understand the performance of Y-zeolite FCC catalysts with respect to aging and metal deposition. These features are related to process lifetime, an important operational parameter with major cost impact in an industrial application. We prepared a series of Y-zeolite catalysts steamed at various temperatures to correlate structural and physical property data. It was decided to use SXRD since it yields narrower peaks resulting in more accurate cell parameters and reproducible diffracted intensities. Fig. 2 shows the intensity variation of one of the zeolite diffraction peaks as a function of temperature. As expected, the crystallinity of Y-zeolite decreases with steaming temperature. Fig. 3 shows the linear correlation between surface area and crystallinity based on the sum of integrated SXRD peak intensities. Fig. 4 shows also the correlation linking surface area to the cell parameter of these cubic zeolites (the Y-axis on Fig. 4 plots difference values obtained from subtraction of the cell parameter of starting Y zeolite).

Acknowledgments: This study was funded by the Natural Resources Canada synchrotron committee. This XRD project was carried out at the National Synchrotron Light Source, Brookhaven National Laboratory, which is supported by the U.S. Department of Energy, Division of Materials Sciences and Division of Chemical Sciences.

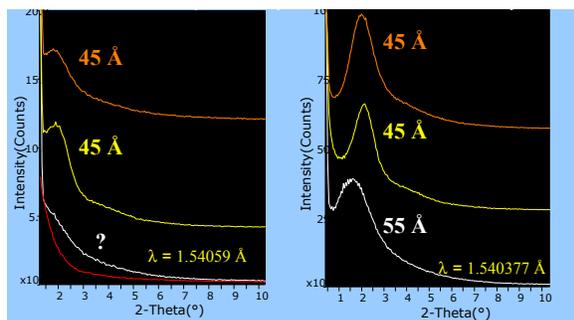


Fig. 1 Comparison of lab XRD (left) and SXRD data (right)

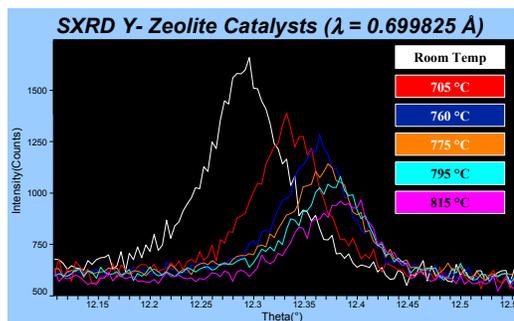


Fig. 2 SXRD region of Y-zeolites heated at various temperatures

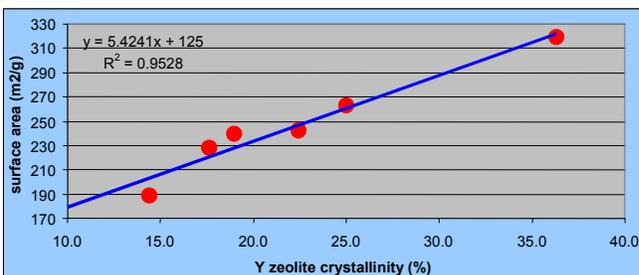


Fig. 3 Correlation of Y zeolite crystallinity with surface area

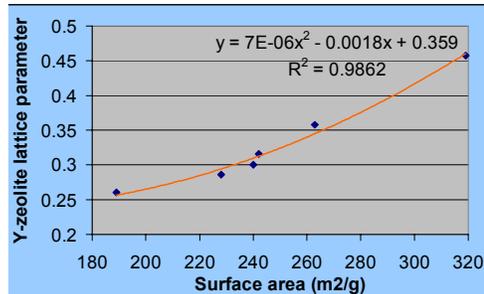


Fig. 4 Correlation of zeolite lattice parameter with surface area