

Commissioning of curved image plate for powder X-ray diffraction (Charge Density Study on Powder Diffraction Data)

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The actual experimental period is from Feb. 26 to Mar. 4. The main purposes of this time is to (1) install the driving motors for the alignment conveniently (2) calibrate the zero shift of angle and (3) collect the low temperature data of KNiF₃ for charge density analysis because the previous experiment on KNiF₃, carried out on Oct. 16 in 2002, was indicated the structure factors ($h+k+l = \text{odd}$) affected by TDS¹ (Temperature Diffuse Scattering). Thus, the low temperature experiment is necessary for charge density analysis.

For the moment, there are four motors used to align the collimator, two motors used to align the vertical and horizontal of the optical table, one used to align the beam stopper, and one used to do translation of image plate. After the recalibration, the highest resolution of the measurement of LaB₆ is about $2\theta = 0.04$ with the incident wavelength 0.51 Å. The result is displayed in the Figure 1. In this figure, we can find the FWHM = 2 pixels corresponded to 0.04 degree because each pixel is corresponded to 0.02 degree. In the most famous BL02B2, the same standard sample is measured with 0.5 Å and the resolution is 0.02 degree. In comparison with the result in BL02B2, our result indicates that the resolution of our image plate is lower than BL02B2. At the moment, the present resolution is the limitation of our image plate. Thus, if we want to get compatible data as BL02B2, we have to change the resolution of image plate. This also implies we that to change the IP reader of the present machine.

The temporary refinement result of low temperature KNiF₃ is displayed in the Figure 2. The agreement factors are listed as following: $R_f = 0.07215$, $R_1 = 0.07743$, and $R_p = 0.04346$. At the present, the background subtraction is still not good. A further analysis is under processing.

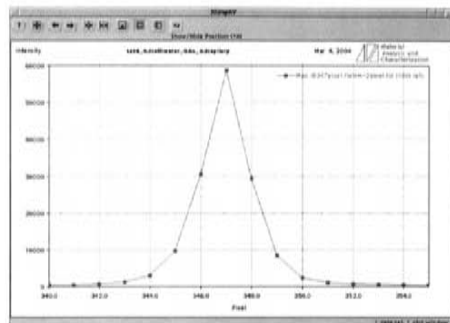


Figure 1

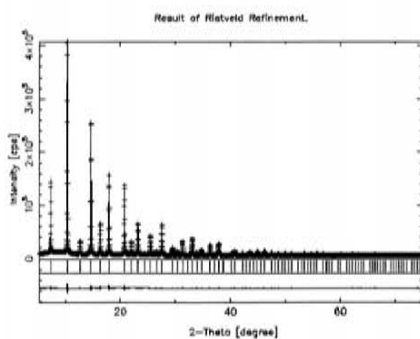


Figure 2

Isomerization of *n*-Butane over Heteropoly Acids Supported on Mesoporous Materials

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Heteropoly acid (HPA) is a strong acid comparable to 100% sulfuric acid, and is used as a catalyst in various acid-catalyzed reactions. 12-tungstophosphoric acid, H₃PW₁₂O₄₀ (HPW), is one of the most acidic heteropoly acid with the most widely studied structure — the Keggin structure. The main factor that determined the catalytic activities of the HPA is the surface area and thus active site contents on the catalysts. Since the surface area of bulk heteropoly acids is very low (<5 m²/g). For this purpose, supported heteropoly compounds, where heteropoly acids are dispersed on porous materials such as MCM-41 or SBA-15, are important for applications in *n*-butane isomerization.

The EXAFS results provide the nature of W atoms positioned in the heteropoly anions, and help us to estimate the dispersy of HPW clusters and the thermal stability of heteropoly anions on the support.

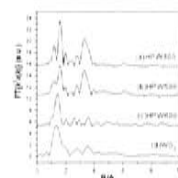


Fig. 1. Fourier transform of $k^3 \cdot \chi(k)$ associated with the W L_{III}-edge of bulk HPW treated at different temperatures: (a)300 °C , (b)500 °C , (c)600 °C , and (d)WO₃ treated at 600 °C .

Fig. 1. shows Fourier-transformed EXAFS spectra at W L_{III}-edge of bulk HPWs which were treated at different temperatures. After heating to 300 °C or 500 °C in air, the Keggin structure of HPA was still retained, due to the similar radial structure function compared with previous literatures^{1,2}(Fig. 2). In Fig. 1, the peak at 1.6 Å is attributed to the average W-O distance in adjoining octahedron in the Keggin structure, and the peak at 3.3 Å is associated with the W-W distances in the sample. While heating to 600 °C, the Keggin unit might be distorted and transition to WO₃ structure.

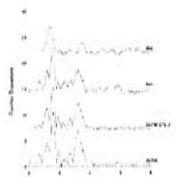


Fig. 2. Fourier transform of $k^3 \cdot \chi(k)$ associated with the W L_{III}-edge of WO₃, PW, 10PW, and 10PW pretreated for 2 h in flowing He at 300 °C .

Fig. 3 and Fig. 4 show W L_{III}-edge EXAFS data of different HPW loading MCM-41 and SBA-15 (denoted as x wt% HPW-M41 and x wt% HPW/SBA-15). The local structure of tungsten in the both supports appear similar structure with bulk HPW, indicating that the majority of W atoms remained in the Keggin structure of the catalysts. In our EXAFS analysis, the decompose of Keggin units to form tungsten oxide was not observed, even increasing HPW content up to 50wt%.

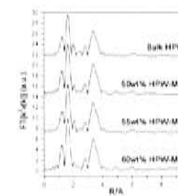


Fig. 3. W L_{III}-edge FT EXAFS spectra of several HPW loaded MCM-41 and compared with bulk HPW.

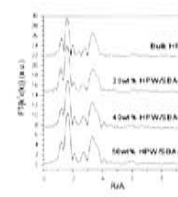


Fig 4. W L_{III}-edge FT EXAFS spectra of several HPW loaded SBA-15 and compared with bulk HPW.

The further EXAFS analysis will be carried out to provide deep insight into the relationship between crystallites of the HPWs on porous materials and the catalytic activity in *n*-butane isomerization.

References:

1. B. B. Bardin, R. J. Davis, Appl. Catal. A: General, 200, (2000) 219
2. B. B. Bardin, R. J. Davis, Appl. Catal. A: General, 185, (1999) 283