Application Note 139

Micropore Analysis of Zeolites Using the ASAP 2420

Introduction

Six zeolite samples were analyzed simultaneously using the ASAP 2420 equipped with the Micropore option. This option enables you to perform micropore analyses on all six ports at the same time, comparable to the High Throughput mode. These analyses were performed using argon as the adsorptive at 87 K. Typical micropore analyses using nitrogen may take as long as five to seven days. By using argon, the analyses can be shortened to as little as two days.

Materials

The following samples were used in the analyses:

Zeolite Type	Trade Name	SiO_2 / Al_2O_3
H-Y (FAU)	CBV600	5.2
H-Y (FAU)	CBV760	60
H-Y (FAU)	CBV901	80
H-ß (BEA)	CP 811C-300	300
H-ß (BEA)	CP 811E-75	75
ZSM-5 (MFI)	CBV3020	30

Preparation

First, the samples were prepared on the degas ports using the automated degas control. All samples were heated to 400 °C at 10 °C per minute and held at that temperature for two hours. Second, the samples were transferred to the analysis ports, where they were manually degassed at a temperature of 200 °C for one hour using lace-up heating mantles. Between the two degas procedures, each tube was weighed and the sample mass was calculated.

Analysis

The micropore analyses were performed with the low-pressure dosing option, and each sample tube was equipped with an isothermal jacket and seal frit. The Dewars were initially filled to a level that was satisfactory as measured by the depth gauge. After the analyses were completed, the Dewars were refilled to a comparable level and a second series of single-point analyses was performed to determine the free space. A full isotherm was also obtained for each sample (see Figures 1 and 2).

Data

The linear and logarithm isotherm plots are shown in Figures 1 and 2. Results from the analyses show an interesting trend, which was expected to some degree. The H-Y samples all align very closely, as seen on the isotherm log plot. Interestingly, the H-β samples behaved somewhat differently from each other. The ZSM-5 also has a unique shape, which is to be expected considering it belongs to yet another class of zeolites. Shown in Figure 3 are the Horvath-Kawazoe differential plots giving the Saito-Foley cylinder pore volume.

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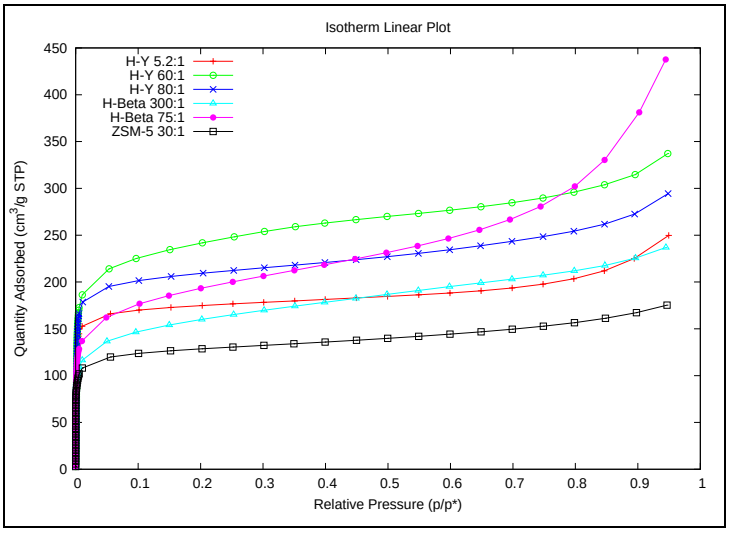

Figure 1. *Isotherm with linear pressure axis. Samples are listed in the same order as in the table on page 1.*

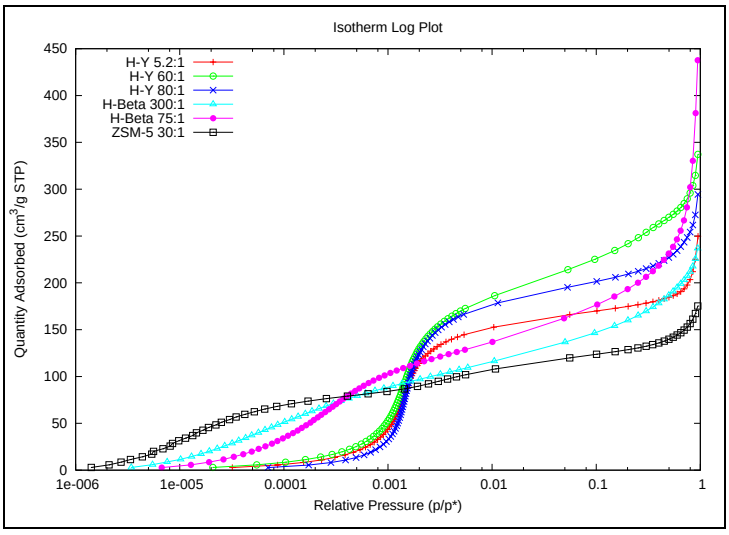

Figure 2. *The same data as shown in Figure 1 with the pressure in log scale.*

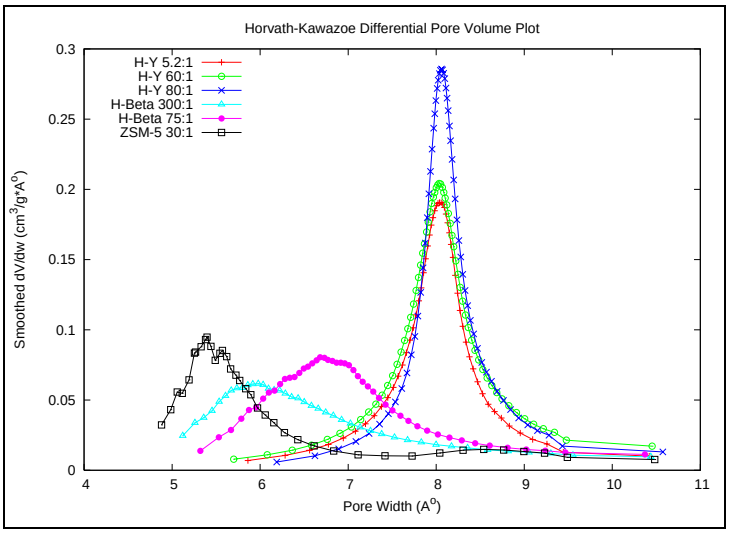

Figure 3. *The Horvath-Kawazoe pore volume distribution.*