VOL 18 NO 3

Nitrogen Adsorption on Lithium Exchanged X Zeolite (Li-X) at Multiple Temperatures Using the ASAP 2050

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Introduction

When gas physically adsorbs onto a sample, temperature plays a very significant role in the amount of gas that the sample is able to adsorb. Performing analyses on a sample at multiple temperatures allows the correlation between temperature and adsorbance to be seen. Zeolite Li-X (lithium exchanged X Zeolite) is a very good sample for the purpose of temperature-controlled analyses as it is highly dependent on temperature. Useful information can be extracted from the adsorption data at multiple temperatures; for example, finding a temperature which maximizes efficiency of adsorption on a large scale. Heat of adsorption data can also be extracted. The heat of adsorption is the amount of energy that is released when the adsorbate adsorbs onto the sample. The ASAP 2050 software includes a heat of adsorption report to allow easy calculation of the data. Heat of adsorption reports require at least two separate isotherms at different temperatures over the same pressure range to report accurate and repeatable results. The gas compressibility factor can be changed based upon the temperature and the thermochemical properties of the gas. The compressibility factor relates to the behavior of the gas at certain temperatures when compared to an ideal gas. Changes in the compressibility factor over a small temperature range are usually small, but have a noticeable impact on the adsorption data; therefore they cannot be ignored.

Preparation

Approximately 1.2 grams of Li-X was manually degassed on the analysis port of the ASAP 2050 at 350 °C for at least two hours in a steel sample tube. The sample was degassed on the analysis port to prevent atmospheric exposure after degas and, thereby, reduce potential causes for discrepancies.

Analysis

After degassing, the sample was analyzed at six different temperatures (-5 °C, 0 °C, 10 °C, 20 °C, 30 °C, and 40 °C) to obtain a range of temperatures for computation of a heat of adsorption isotherm.

The analysis was conducted over a pressure range of 0 mmHg to 7500 mmHg with both adsorption and desorption data being taken. To keep the sample free of helium during the analysis process, free space was measured after each analysis as a separate analysis file. Isothermal jackets and a Dewar were not used for the analysis since temperature was controlled through a thermoelectric cooling device. The device uses a thermoelectric cooler, also known as a Peltier, to cool the block in which the sample tube fits. The Peltier holds the temperature at a stable, precise temperature during the analysis.

ASAP 2050 Xtended Pressure Sorption Analyzer

Results

The Li-X uptake of nitrogen is highly dependent upon temperature as is shown in Figure 1. As temperature increases, nitrogen adsorption decreases; it can be seen in the isotherms that a small variation in temperature causes a noticeable variation in the amount of nitrogen adsorbed. Because of the temperature's effects on the isotherms, the thermoelectric cooling device was used to maintain a stable temperature.

From the multiple isotherms at different temperatures, the isosteric heat of adsorption isotherm can be calculated. Figure 2 shows the isosteric heat of adsorption calculated from the nitrogen adsorption data given in Figure 1. The data show the energy associated with the N_2 adsorption onto the Li-X sample. The negative sloping sections are where energy is released as the N_2 adsorbs. The upward sloping sections are where work has to be performed by the gas to adsorb onto the surface. Nitrogen adsorbs readily onto the sample; the only time that work is performed is when high quantities of nitrogen are adsorbed. The beginning and end of the heat of adsorption graph are not as stable as the middle section because the higher quantity adsorbed section is more dependent on lower temperatures and the lower quantity adsorbed section is more dependent on higher temperatures. Overall, the most noticeable attribute of this heat of adsorption plot is that Li-X has an unusually high amount of energy released for a sample undergoing physical adsorption of nitrogen.

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Figure 1:

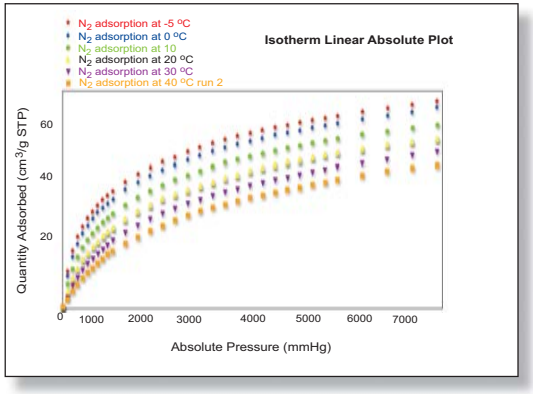

Figure 2: