Application Note

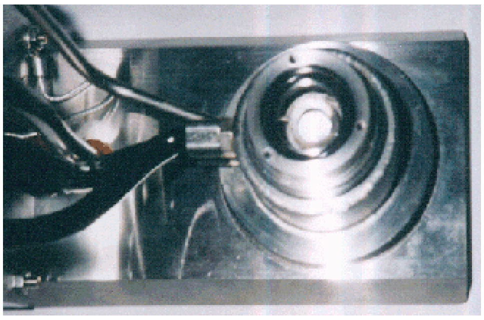
240-DR-0167

Analysis of Catalyst Reactions using Vacuum Thermal Diffuse Reflection Accessory

<Introduction>

Several studies have examined catalytic reactions in an attempt to clarify reaction processes. Spectroscopic methods are essential in determining the mechanism by which organic reactions occur on the surface of solid catalyst. One effective method for analyzing the surface of solid catalyst involves the use of a vacuum thermal diffuse reflection accessory. This reflection accessory enables powdered samples to be measured under heating. The cell of this accessory not only enables gases that limit adsorptivity to be purged, but also allows a vacuum to be created so that the adsorption gas can be eliminated. In addition, this accessory supplies a constant volume of gas to the sample surface by connecting the cell to a closed circulating device, and the status of the sample surface can be monitored in real time.

<System>



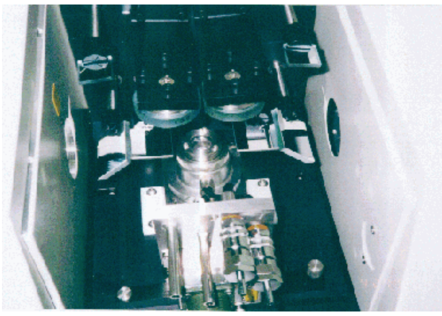


Figure 1. Cell of vacuum thermal reflection accessory

Figure 1 shows a photograph of the cell of the vacuum thermal diffuse reflection accessory used in the present experiment and Figure 2 shows a photograph of the closed circulating device. Three types of vacuum thermal diffuse reflection accessories are available, based on the required temperature control range. The temperature specifications are for a vacuum environment; the actual temperature on the sample surface is slightly lower than the temperature specified by the user.

Figure 2. Closed circulating device

<Instrument>

FT/IR-620 FT/IR Spectrometer

DR-600A, B, C vacuum thermal reflection accessories

(A : 1000 ºC, B: 800 ºC, C : 600 ºC)

Closed circulating device

<Experimental>

Adsorption of pyridine on the surface of zeolite

1. Place a solid catalyst (zeolite powder) on the cell of the vacuum thermal diffuse reflection accessory.
2. Create and maintain a vacuum in the cell for approximately one hour at 500 ºC. Then measure baseline (background spectrum) at room temperature.
3. Using the closed circulating device introduce a constant volume of pyridine into the cell.
4. Create and maintain a vacuum in the cell for approximately one hour at 200 ºC in order to eliminate physical adsorptive activity. Then measure spectrum at room temperature.

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<Conclusion>

Acid receptor sites were found on the surface of zeolite. These acid receptor sites function as active sites for the catalyst activity of zeolite. Spectroscopic analysis enables the adsorption of probe molecules at the active sites to be determined. If base molecules, such as pyridine, are adsorbed at an active site, the site can be further classified as B acid (Bronsted acid) receptor sited or L acid (Lewis acid) receptor sites, according to the adsorption. Conversely, an L acid receptor site may be changed into a B acid receptor site by hydration. Figure 3 shows respectively the diffuse reflection spectra for the adsorption of pyridine obtained both before and after the application vacuum conditions. Adsorptive pyridine at a B acid receptor site, where H^+ was accepted, is changed into PyB (pyrindium ion), having an absorption peak at 1542 cm^{-1}. However, the amount of adsorptive PyB at the B acid receptor site is very limited, because zeolite was heated to a high temperature before adsorption. Adsorption PyL at the B acid receptor site shows absorption at 1450 cm^{-1} and strong peak intensity can be observed immediately after adsorption. Both the 1445 and 1595 cm^{-1} peaks represent hydrated pyridine(PyH). Figure 4 shows the status of chemical adsorption on the solid surface.

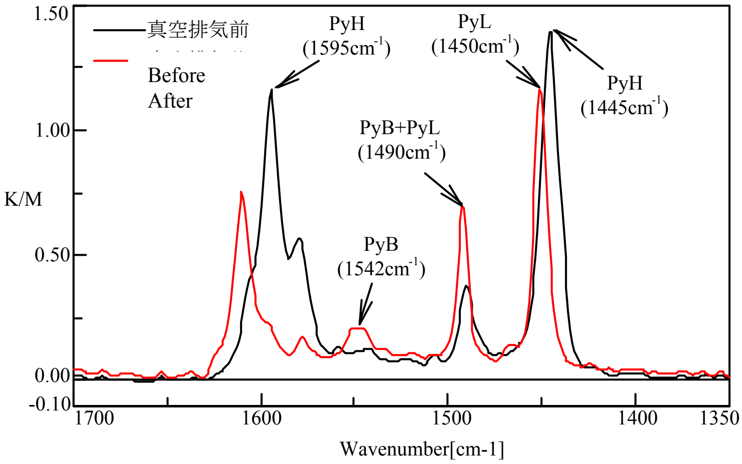

Figure 3. Diffuse reflection spectra obtained, respectively before and after vacuum conditions

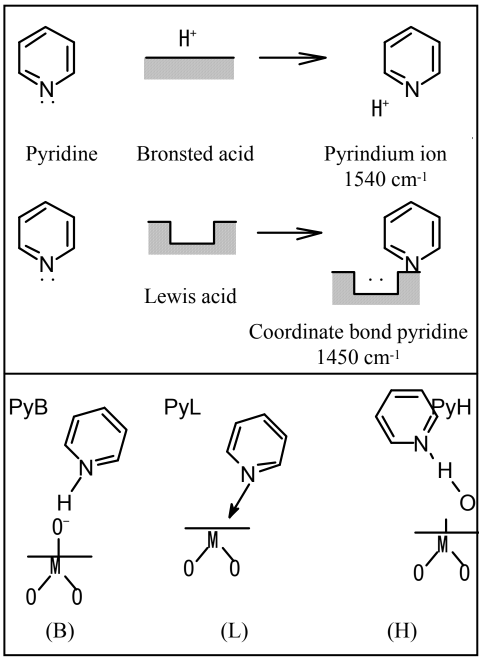

Figure 4. Status of chemical adsorption on the solid surface