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IN-SITU INVESTIGATION OF THE INTERACTIONS BETWEEN GAS ADSORBATES AND POROUS SORBENTS BY DRIFTS

Figure 1. The Harrick Praying Mantis™ diffuse reflectance accessory.

Figure 2. The Harrick High Temperature Reaction Chamber.

INTRODUCTION

Good understanding of the molecular interactions between gas adsorbates and porous sorbent materials is key for the development and advancement of diverse applications, ranging from catalysis and gas storage to sensing.

From all the characterization techniques available on a laboratory for analyzing the interactions between gas and solids, such as gas sorption isotherms or thermogravimetric analysis (TGA), *in-situ* FTIR prevails due to the ability of retrieving time-resolved and highly precise data under controlled conditions of environment and temperature. The precise control over the conditions required for gathering relevant data can be achieved by using Harrick's DRIFT cell.

The ability of FTIR for simultaneously live visualizing, in a glimpse (even seconds), the nature of all the chemical bonds present on a given gas-solid system, both quantitatively and qualitatively, allow us to determine gas uptakes and diffusion rates. FTIR also allows us to determine mutual interactions between them, i.e., bond strength and coordination of gas adsorbates within active porous materials.

Here, we show an example of how FTIR enables to determine an adsorption isotherm at a given temperature via measuring different CO_2 concentrations in the gas phase adsorbed on a porous material (solid phase).

EXPERIMENTAL

Infrared spectra were acquired by using the Praying Mantis diffuse reflectance accessory installed in a commercial FTIR. The HVC reaction chamber was heated at a given temperature under controlled environment containing various gas adsorbates (i.e., CO_2, and air). All spectra were recorded at 8 cm^{-1} resolution, 64 scans with a gain of 8 and an aperture of 100 using a DTGS detector. ZnSe windows were used on the DRIFT dome.

First, the background was recorded by filling the DRIFT cup with neat KBr and purging the cell for 15 min under dry air at the temperature selected for the experiment (i.e., 40 °C). Second, the sample was prepared by diluting the porous material in KBr (i.e., 10 wt%), and filling the DRIFT cup with the resulting grinded fine powder mixture. The sample was then heated at the experiment temperature and purged with gas containing specific amounts of CO_2 in dry air for 15 min. Third, varying CO_2 concentrations in dry air were also evaluated to obtain several points of an isotherm for this adsorbate at the given temperature.

RESULTS AND DISCUSSION

As shown in Figure 3, CO_2 adsorbed in the material exhibits an IR absorption band centered at 2340 cm^{-1} attributed to the CO_2 asymmetric stretching. Increasing concentrations of CO_2 in dry air leads to increasing intensity of the IR band due to higher adsorption of CO_2 on the porous material. The area of the bands can be afterwards translated to uptake of adsorbed CO_2 by correlating the collected IR band areas to gravimetric data obtained by CO_2 sorption isotherms and/or thermogravimetric analysis (TGA).

The generated CO_2 calibration curve allows the determination of CO_2 uptakes under different temperatures and concentrations. More information can be retrieved from this type of experiments, such as adsorption kinetics by analyzing the temporal evolution of the IR band during the equilibration at the different CO_2 concentrations.

CONCLUSIONS

The DRIFTS technique allows the determination of gas uptakes and sorption kinetics on solid porous adsorbents. *In-situ* infrared diffuse reflection studies are definitively the way to go towards reaching more deep understanding of gas-solid systems.

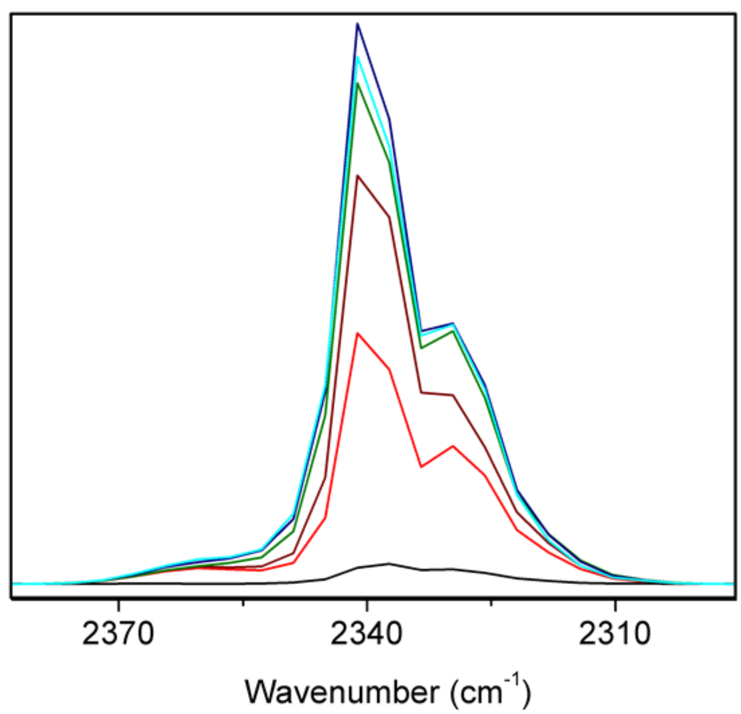

Figure 3. Example of the FTIR spectra of varying concentrations of CO_2 adsorbed on a porous material measured in a DRIFT cell. Y-axis is represented in Kubelka-Munk units.

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