CO adsorption on HKUST-1 at RT in drifts

Date: 2023-09-01
Tags: HKUST-1 DRIFTS
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Sample: HKUST-1 (sample Nr. 22 in the Database)

General description of the experiment:

To evaluate the adsorption bands of CO (1 atm) on HKUST-1 at RT (30 °C)

Detailed procedure:

Preparation: Put the water trap and trap containing active carbon into a oven (425 °C) overnight to remove all water and carbonyls.

KBr powders,

Run a background in an Ar (1 atm, 50 ml/min) atmosphere

Background parameter: 256 scans, 2 cm-1

heat 500 °C in Ar for 1 hour and cooled down in Ar to 30 °C

Run a background in an Ar atmosphere to compare the water/solvent content.

At rt switch to CO (1 atm, 200 ml/min), measure CO adsorption as a function of time up to 1 h.

parameter: 8 scans, 2cm-1

Switch to Ar, flush out CO.

Change sample to HKUST-1,

Run a background in an Ar atmosphere (1 atm, 50 ml/min)

parameter: 256 scans, 2cm-1

heat 100 °C in Ar for 1 hour and cooled down in Ar to 30 °C

Run a background in an Ar atmosphere to compare cleanliness of the sample

At rt switch to CO (1 atm, 200 ml/min), measure CO adsorption as a function of time up to 1 h.

parameter: 8 scans, 2cm-1

Switch to Ar, flush out CO.

Clean the sample chamber.

Settings

File settings:

Computer: for drift experiment
Folder: D:\data\shuangchen\2023---HKUST-1
Spectra names: SC0001---

Opus settings:

Resolution	2
Phase resolution	4
Acquisition Mode	Double Sided, Forward-Backward
Aperture	8mm
Mode	Absorbance
Detector	LN MCT Narrow [Internal Pos.2]

Gain	Automatic
ADC-counts	
Polarization	no
Scanner velocity	40

Protocol of the experiment:

Action/Spectrum	Name	Temperature□°C□	Time	Comments	
Action	Take traps out of the oven and cool down to RT				
Action	Load the KBr into the DRIFTS cell				
Action	Check for leaks using Ar (1 atm/ 50 ml/min)				
Action	test an initial background of KBr in Ar				
Action	teat the sample in Ar at 500 °C for 1h (to clean sample)				
Action	Cool down the sample in Ar to 23 °C				
Action	test a background of KBr in Ar (after heating to check cleanliness)				
Action	Run the experiment of CO (1 atm/ 200 ml/min) adsorption for 1 hours at 23 °C				
Action	Switch to Ar flush 10min to remove CO				
Action	Change sample to HKUST-1 and put it into the DRIFTS cell				
Action	test a background of HKUST-1 in Ar (same parameter)				
Action	Heat the HKUST-1 in Ar at 100 °C for 1h				
Action	Cool down the HKUST-1 in Ar to 30 °C				
Action	test a background of HKUST-1 in Ar after heating to check cleanliness				
Action	Switch to CO and run the experiment of CO adsorption on HKUST-1 for 1 hours at 30 °C				
Action	Flow Ar for 10min to remove CO				
Action	Remove sample and clean out DRIFTS cell with water				

Results :

Comments:

Steps

- Put traps into the oven / clean at 425°C over night
- Load the KBr into the DRIFTS cell
- Check for leaks using Ar (1 atm/ 50 ml/min)
- test an initial background of KBr in Ar
- Heat the sample in Ar at 500 °C for 1h (to clean sample)
- Cool down the sample in Ar to 30 °C
- test a background of KBr in Ar (after heating to check cleanliness)
- Run the experiment of CO (1 atm/ 200 ml/min) adsorption for 1 hours at 30 °C
- Switch to Ar flush 10min to remove CO
- Change sample to HKUST-1 and put it into the DRIFTS cell
- test a background of HKUST-1 in Ar (same parameter)
- Heat the HKUST-1 in Ar at 100 °C for 1h
- Cool down the HKUST-1 in Ar to 30 °C
- test a background of HKUST-1 in Ar after heating to check cleanliness
- Switch to CO and run the experiment of CO adsorption on HKUST-1 for 1 hours at 30 °C
- Flow Ar for 10min to remove CO
- Remove sample and clean out DRIFTS cell with water

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