Experimental report

13/09/2019

Proposal: 7-05-488 **Council:** 4/2018

Title: INS study of ethene on Ag+ cationsexchanged on Chabazite and Faujasite Zeolites

Research area: Chemistry

This proposal is a new proposal

Main proposer: Teresa BLASCO

Experimental team: Miguel PALOMINO
Teresa BLASCO
Joaquin MARTINEZ ORTIGOSA
Fernando REY

Local contacts: Monica JIMENEZ RUIZ

Samples: Faujasite zeolite (Si/Al=40), Ag+ exchanged
Chabazite zeolite (Si/Al=40), with Ag+exchange
Chabazite zeolite(Si/Al=5), Ag+ exchanged
Chabazite zeolite (Si/Al=5), Ag+ exchanged

Instrument	Requested days	Allocated days	From	To
IN1 LAG	5	3	20/09/2018	23/09/2018

Abstract:

Many chemicals and polymers relies on the production of ethene that exceeded 144 million tons in 2015, mostly by steam cracking. The separation of ethene from other hydrocarbons is actually carried out by the cryogenic distillation. The high energy demand of this technology makes desirable the development of new processes, being the zeolites based separation the most promising. This alternative route takes advantage of the molecular sieving properties of zeolites and the possibility of tuning their polarity. One approach to adjust the polar character of silicoaluminate zeolites is by the presence of cations in the voids channels and cavities. Here, we propose to study the interaction of ethene adsorbed on small pore zeolite Chabazite (CHA) and a large pore zeolite Faujasite (FAU) with Si/Al ratios of 5 and 40 and having as counter-cations silver. We think that INS is the ideal spectroscopic technique for understanding the mechanism of ethene-zeolite interaction, particularly when combined with advanced Solid State NMR studies and theoretical calculations.

EXPERIMENTAL REPORT OF THE PROPOSAL: 7-05-456

In this project we proposed to study the adsorption of ethene at varying loading on two zeolites of different structures (small pore CHA and large pore FAU) with two different Si/Al molar ratios (5 and 40). The main purpose is to study the influence of the pore size and the framework polarity on the adsorption of ethene.

Therefore, the zeolites proposed in this experiment are:

1. Ag-CHA-40 is an Ag^+ exchanged Chabazite zeolite with a Si/Al ratio of 40.
2. Ag-CHA-5 is an Ag^+ exchanged Chabazite zeolite with a silica ratio of 5.
3. Ag-FAU-40 is an Ag^+ exchanged Faujasite zeolite with a Si/Al ratio of 40.
4. Ag-FAU-5 is an Ag^+ exchanged Faujasite zeolite with a Si/Al ratio of 5.

The experiment was carried out by using the manual gas handing system at ILL that allows dosing variable amounts of gas on the same sample. In the allocated time we were only able to make experiments on zeolite Ag-CHA-40 and Ag-FAU-5. We measured the volume of the manifold, the transfer line and the probe by measuring pressure variations by expanding He gas.

First, we studied **Ag-CHA-40 zeolite** previously degassed at 673 K that was transferred into the insert under an Ar atmosphere using a dry box available at ILL. The sample was cooled down to 10 K and the spectrum was recorded. Then, zeolite Ag-CHA-40 was taken out from the spectrometer and heated to room temperature to proceed to the adsorption of ethene.

We made two experiments in order to adsorb an ethene/Al =0.5 and ethene/Al =2 molar ratios and the spectra were recorded at 10 K. We did not observe any pressure drop when the gas was contacted with the sample at room temperature in any case, but we cooled down the sample and recorded the spectra at 10 K to check if there the presence of ethene in the sample. However, under these conditions we did not observe any signal of ethene.

Subsequently, we increased the pressure to get an ethene/Al = 5,2 molar ratio and we detected only a very weak signal which could indicate the presence of ethene in the zeolite.

We made experiments using **Ag-FAU-5 zeolite** following a similar procedure. First, we recorded the spectrum of the bare zeolite at 10 K. Then we adsorbed ethene corresponding to an ethene/Al = 4 molar ratio and recorded the spectrum at 10 K (red line in Figure 1). The spectrum shows a series of bands, which according to bibliography data can be due to solid ethene, and to physisorbed and chemisorbed ethene on the zeolite. In an attempt to observe only chemisorbed ethene, we increased the temperature of the sample to 212 K and degassed the sample in the temperature range 212 K – 273 K during 15 min, while we detected the gas desorption. After that, we cooled the sample to 10 K and recorded the spectrum (green line in Figure 1).

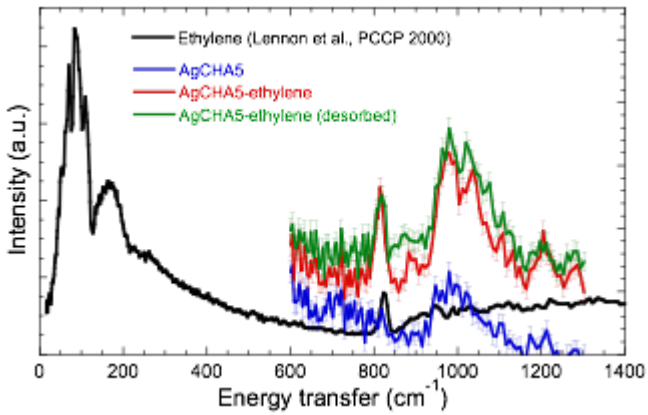

Figure 1