SnT 2025
8 SEPTEMBER
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CTBT: SCIENCE AND TECHNOLOGY CONFERENCE

P3.2-074

An alternative silver-exchanged zeolite for noble gas collection and separation

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INTRODUCTION AND MAIN RESULTS

Ag-zeolites have recently allowed scientific breakthroughs in xenon collection and separation. Two synthetic Ag-zeolites are used in new generation noble gas stations in the IMS. Other Ag-zeolite options could help ensure long-term availability and may further enhance the xenon collection and separation process.

First results on a silver-exchanged natural chabazite, with and without a surface homogenization treatment, demonstrate a Xe adsorption capacity that is about two times lower than the currently used Ag-zeolites, but ten times higher than activated carbon.

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Introduction

Noble gas monitoring systems are used in the IMS for the verification of the CTBT. They monitor the atmosphere for traces of radioxenon released by clandestine nuclear weapon tests. Radioargon is used as a complementary tracer during OSI to search for underground tests. Efficient collection and purification of noble gases in porous adsorbents is essential for the detection capability of these systems.

In the last decade, Ag-zeolites have been investigated for these applications. Currently, two synthetic Ag-zeolites (Ag-ETS-10 and Ag-ZSM-5) are used in the new generation radioxenon monitoring systems as part of the collection and separation process. Ag-ETS-10 has been investigated for argon collection and separation during OSI. Each Ag-zeolite has specific noble gas adsorption properties, which are driven by the presence of silver. Other Ag-zeolites might provide further developments for radioxenon and radioargon measurements.

In this work, we investigate different modified mineral chabazites, exchanged with silver, for their application in CTBT verification, by characterizing their xenon collection and separation properties.

Adsorbents

Mineral chabazites from a deposit in Arizona (USA) were used in this study. The samples were taken from a section of the deposit with high Na cation content (essentially Ca-free).

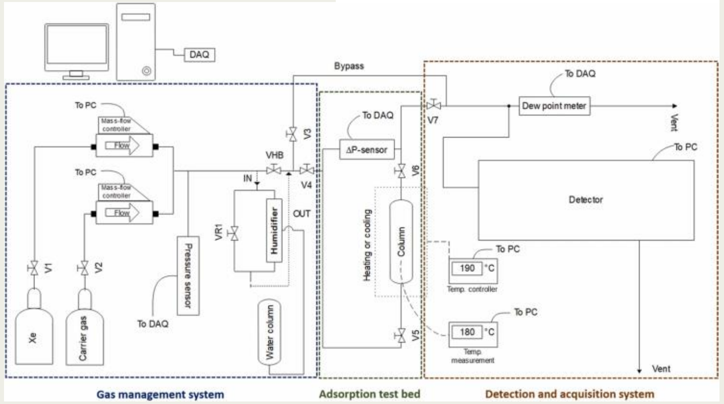
Even though high purity samples were used, internal surfaces are inherently inhomogeneous. To homogenize the crystal surfaces, samples were reacted in mild-alkaline-silicate solutions at relatively low temperatures following the procedure in Kuznicki et al., 2007. To finalize the surface treatment, the samples were aged for 72 h at 70 to 80 °C, washed and dried for 16 h at 80 °C. In total, six reactions were tested for surface treatment. The surface-treated (ST) sample providing the most promising results (Fig. 1) is reported here.

After surface homogenization, samples were exchanged with $AgNO_3$ and subsequently washed and dried. The samples were then activated in an adsorption column at about 160 °C for at least 12 hours under nitrogen for subsequent adsorption measurements.

The results on these Ag-chabazite samples are compared to measurements performed on activated carbon, Ag-ZSM-5 and Ag-ETS-10.

Fig. 1 – Granules of a surface treated Ag-Chabazite sample.

Fig. 2 – Schematic representation of the experimental setup.



Adsorption measurements

The adsorption measurements were performed using a gas breakthrough experimental setup (Fig. 2) described in Gueibe et al., 2022. The chosen gas mixture was passed over an adsorption column containing the adsorbent and the outlet was monitored with a mass spectrometer to collect breakthrough curves.

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References:
Kuznicki et al. Clays and Clay Minerals. 2007;55(3):235-238.
Gueibe et al. Process Safety and Environmental Protection. 2022;158:576-588.

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Xe adsorption in nitrogen at 294 K

Breakthrough curves of Xe in N_2 were measured on a ST Ag-Chabazite sample. As shown in Fig. 3, an increase of about 30% was observed in Xe adsorption capacity at 50 ppm for the ST sample. At 500 ppm, the breakthrough curves were very similar.

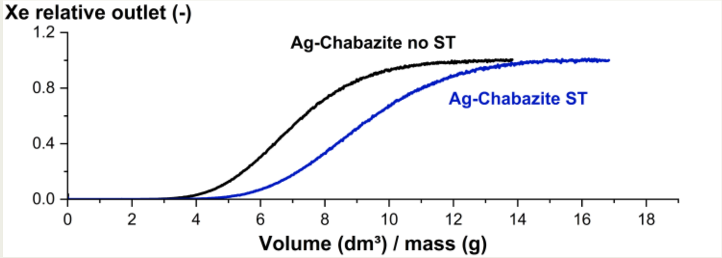

Fig. 3 – Xe breakthrough curves at 50 ppm on the surface treated and untreated Ag-Chabazite samples.

As shown in Fig. 4, rapid equilibration was obtained at 50 ppm, whereas slower equilibration, with potentially two adsorption mechanisms, was observed at 500 ppm. The intermediate equilibration point for 500 ppm was used to calculate the adsorption capacity.

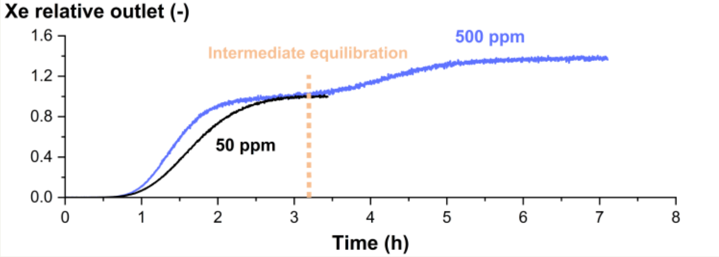

Fig. 4 – Xe breakthrough curves on the surface treated Ag-Chabazite sample.

Compared to activated carbon, the Xe adsorption capacity (Fig. 5) is 11 times higher at 50 ppm (~ 5 Pa). The adsorption capacity is still about 2 times lower than that of the synthetic Ag-ETS-10 and Ag-ZSM-5.

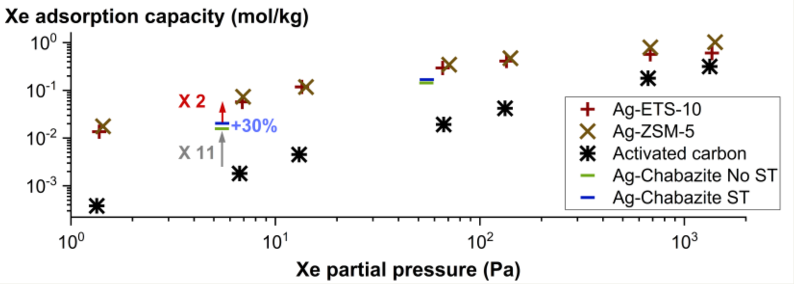

Fig. 5 – Xe isotherm measurements at 294 K on Ag-chabazite samples compared to other adsorbents.

Xe adsorption in air

Breakthrough of Xe in air (87 ppb Xe) on the treated and untreated samples was very similar (±10%). This might be due to an increased competition with air in the ST sample. Xe breakthrough on the untreated sample is compared to activated carbon, Ag-ETS-10 and Ag-ZSM-5 in Fig. 6. Based on the midpoint of the breakthrough curve, the Ag-Chabazite ST can retain Xe 10 times longer then activated carbon. The Ag-ZSM-5 can retain Xe 2.5 times longer than the Ag-Chabazite.

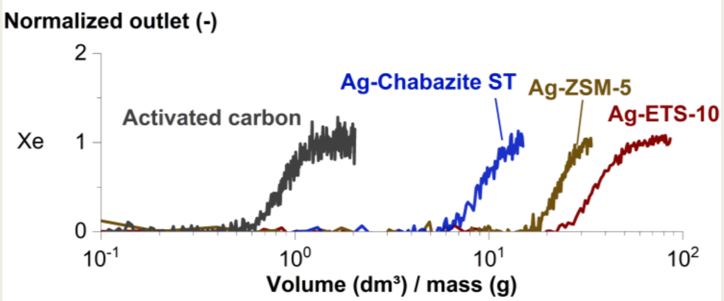

Fig. 6 – Xe breakthrough curve from air on the ST Ag-Chabazite compared to other adsorbents.

The Xe/Kr selectivity (110) in air on this Ag-Chabazite is higher than on activated carbon (17), but significantly lower than on Ag-ZSM-5 (430) and Ag-ETS-10 (1450). Similar conclusions apply to the Xe/air selectivity.

Conclusions

Preliminary findings show that Xe adsorption in air is 2 to 4 times lower in the natural Ag-chabazite, even with surface treatment, compared to currently used Ag-zeolites. The surface treatment enhances Xe adsorption in N_2 by about 30% at low Xe pressure. This improvement is not observed in air, likely due to competitive adsorption effects. Further investigation is necessary to better understand the slow equilibration at 500 ppm and the increased competition in air.

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