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Synthesis of Zeolites LiX and LiAgX

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PREFACE OR ACKNOWLEDGEMENTS

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EXECUTIVE SUMMARY

In this work, we produced approximately 50 grams of zeolite LiX and 50 grams of zeolite LiAgX from commercially available zeolite X. The production of the two specialized zeolite phases was accomplished via a series of ion exchange steps followed by washing, drying, and calcining to produce the final materials. The ion exchange was confirmed by several methods including inductively-coupled plasma emission spectroscopy of exchange solution aliquots, gravimetric analysis, energy dispersive x-ray spectroscopy, and X-ray diffraction.

We synthesized approximately 115 grams of zeolite LiX in a large-scale production step after completing a proof-of-concept experiment demonstrating the methodology. The production of zeolite LiX from zeolite X yielded an ion exchange ($Li^+ \rightarrow Na^+$) of approximately 82%.

The production of zeolite LiAgX from the previously synthesized zeolite LiX was targeted to exchange approximately 20% of the Li^+ ions with Ag^+ ions. We first performed a proof-of-concept experiment demonstrating the reaction pathway and selectivity of the Ag^+ ion, and then performed a large-scale synthesis of zeolite LiAgX using approximately 50 grams of the zeolite LiX. Our final exchange percentage of silver into the zeolite LiX matrix was approximately 20.8%.

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LIST OF ABBREVIATIONS

AgAce	Silver acetate, $AgC_2H_3O_2$
DI	De-ionized
FAU	Faujasite
HDPE	High-density polyethylene
ICP-ES	Inductively-coupled plasma emission spectroscopy
IEX	Ion exchange
M/V	Mass to volume ratio
POC	Proof of concept
SRNL	Savannah River National Laboratory
XRD	X-ray diffraction
EDS	Energy Dispersive X-ray Spectroscopy

1.0 Introduction

In this work, we produced zeolite LiX and LiAgX from a commercially and commonly available zeolite X powder (Sigma Aldrich, Zeolite 13x – $Na_{86}Al_{86}Si_{106}O_{384}\cdot xH_2O$ (nominally)). Zeolite X has a faujasite (FAU) framework type and has remarkable gas adsorption properties related to the openness of the lattice (Figure 1) compared to other common zeolite frameworks[1] as shown in Figure 2.

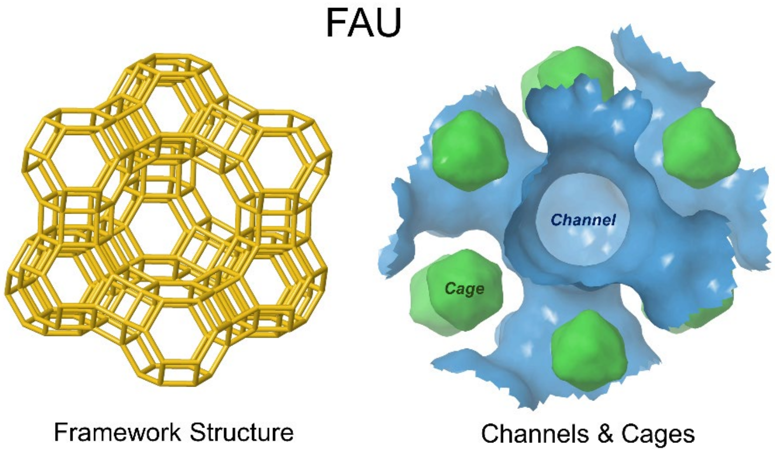

Figure 1: Crystal structure of zeolite X.

One of the primary uses of zeolite X in industry today is for air separations, particularly of oxygen and nitrogen as a replacement for cryogenic distillation technologies. The large pore size of zeolite X makes the mineral ideal for hosting both small and large guest species ranging from nitrogen to neopentane[2].

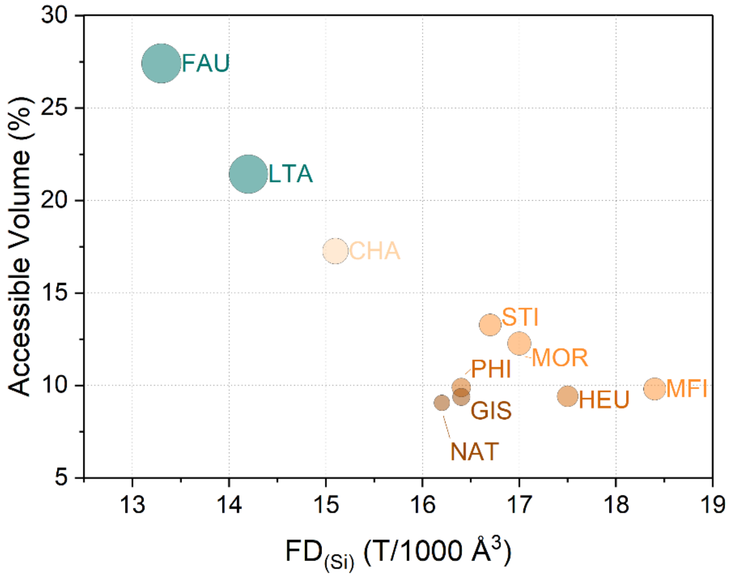

Figure 2: The faujasite framework has a significantly more open structure than other common, industrial zeolites including zeolite A (LTA), chabazite (CHA), phillipsite (PHI), natrolite (NAT), stilbite (STI), ZSM-5 (MFI), clinoptilolite (HEU), zeolite P (GIS), and mordenite (MOR).

Further modification of the zeolite chemistry via ion exchange (IEX) can enhance the materials' efficacy at trapping various species and/or increase the selectivity of the zeolite for one species over another. In this work, we first substituted Li for Na in the zeolite X structure, which is hypothesized to enhance the material's ability to trap N_2 gas. This enhancement is due to the high polarizing power of the Li^+ ion[3]. Further separation efficacy can be achieved by modification of the cationic zeolite species with transition metals such as Ag^+ and Cu^+. These modifications enable the exploitation of a phenomenon known as π-complexation in which the adsorption of guest molecules is through covalent bonding rather than the weaker van der Waals forces associated with physisorption (physical adsorption)[4].

In this work, we sought to synthesize zeolite LiX and zeolite LiAgX using a method reported by Yang, et. al[5]. The two zeolites were produced in sequential steps from the starting material zeolite X following a proposed reaction pathway of $Z\text{-}Na+Li^+/Z\text{-}Li+Na^+$ and then $Z\text{-}Li+Ag^+/Z\text{-}Li\text{-}Ag+Li^+$. The selectivity of zeolite X is given as Ag >> Na >> Li[6]; therefore, complete exchange of Li for Na is difficult (i.e., 100% exchange)[5]. In order to achieve a high fraction of Li exchange in zeolite X, we employed solutions with strong exchange conditions – in particular, LiCl solutions with an ionic strength greater than or equal to 2.5 mol/kg.

Given the selectivity of the Ag ion relative to Na and Li in the zeolite X system and our desire to only achieve 20% exchange of Ag^+, we controlled this exchange by targeting a mass-to-volume ratio (M/V) of zeolite to exchange solution volume that would yield a 20% exchange.

After each of the exchanges were completed, the exchange solutions were characterized by inductively-coupled plasma emission spectroscopy (ICP-ES) to determine the extent of ion exchange. A dehydration step was also used to remove crystalline water from the ion exchanged zeolites, which further increases the materials' efficacy at gas adsorption.

This report outlines in detail the scalable step-by-step process to create large quantities of zeolite LiX and zeolite LiAgX for future use in various applications as well as describes the results of the characterization techniques that ensured the quality of the final materials.

2.0 Synthesis of Zeolite LiX

2.1 Proof of concept (POC) for zeolite LiX synthesis

The zeolite LiX was synthesized first in a small quantity to ensure the methodology for exchange and then in a larger quantity to produce the desired amount for testing and for the synthesis of zeolite LiAgX. We first prepared a 2.5 M LiCl solution to ensure sufficient chemical potential for exchange given the aforementioned selectivity of zeolite X. We prepared two experiments in which two grams of crushed zeolite X[a] were added to 50 ml Corning centrifuge vials, and then we added 30 ml of the 2.5 M LiCl solution to these tubes (M/V = 0.07 g/ml). Both tubes were placed on a turntable to provide mixing/agitation throughout the course of the exchange.

After approximately two hours, we centrifuged the vial at 3500 rpm for 5 minutes and decanted the initial exchange solution from one of the tubes and replaced that solution with a "spike" of fresh, 2.5 M LiCl solution. The vial with the fresh solution was again placed on the turntable and the mixing/agitation continued.

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[a] We used zeolite X in the form of beads, which were crushed via mortar and pestle to a powder-like consistency for these initial exchange experiments. After these first proof-of-concept experiment, we started using zeolite X in a 2 μm powder form.

After approximately 24 hours, both vials were removed from the turntable and centrifuged at 3500 rpm for 5 minutes to separate the exchanged zeolite material from the exchange solution. Aliquots of the exchange solutions were collected for analysis and the remainder of the exchange solutions were discarded. The exchanged zeolite materials were then washed twice with de-ionized (DI) H_2O by manual agitation, and after each wash, the vials were centrifuged at 3500 rpm for 5 minutes and an aliquot was sampled for analysis. The remainder of the wash solutions were discarded. A third wash with DI H_2O was conducted for an hour by placing the vials on the turntable for agitation, and again, aliquots of the wash solutions were collected for analysis.

The washed zeolite LiX materials were then dried. The solution aliquots that were collected from the original exchange and the washes were analyzed using (ICP-ES).

2.2 Results of zeolite LiX synthesis proof of concept

ICP-ES analysis of the ion exchange and subsequent wash solutions demonstrated the exchange of Li for Na in the zeolite X structure for both the unspiked and spiked solutions. As expected, the amount of Li and Na in the system decreased with each wash. The results of the initial experiments' ICP analysis are shown in Table 1. A control sample was included to determine the amount of Li and/or Na that would be present in a pure DI H_2O solution without the added LiCl driving ion exchange. The zeolite phase used in these initial POC experiments was identified as $Na_{88}Al_{88}Si_{104}O_{384}\cdot172.1H_2O$ via X-ray diffraction (XRD) (Figure 3). This zeolite chemistry was used to determine the anticipated Na exchange for the POC experiments given in Table 1. We assumed the displacement of sodium by protons was minimal throughout the course of this work.

Table 1: ICP-ES results of the initial proof-of-concept experiment of $Li^+ \rightarrow Na^+$ ion exchange in zeolite X.

Sample ID	**Anticipated Na at 50% exchange (mg/l)**	**Anticipated Na at 100% exchange(mg/l)**	**Li**	**Na**
LiX - 24 - super 1	4071	8142	14250	4850
LiX - 2-24 - super 1	4071	8142	14800	1535
LiX - control - super 1	4071	8142	2	19
LiX - 24 - wash 1	4071	8142	922	251
LiX - 2-24 - wash 1	4071	8142	455	80
LiX - control - wash 1	4071	8142	<1	29
LiX - 24 - wash 2	4071	8142	71	19
LiX - 2-24 - wash 2	4071	8142	68	6

LiX - control - wash 2	4071	8142	<1	205
LiX - 24 - wash 3	4071	8142	30	10
LiX - 2-24 - wash 3	4071	8142	34	4
LiX - control - wash 3	4071	8142	<1	57

Inspection of Table 1 reveals that the amount of Na possible for exchange was approximately 8142 mg/l, which was determined via Equation 1:

$$C_i^{max} = \frac{m_z x_i}{V_s} \tag{1}$$

Where:

C_i^{max}= the maximum concentration in solution of the ith element of the zeolite
m_z = the mass of zeolite in solution (mg)
x_i = the weight fraction of the ith element in the zeolite
V_s = the volume of the solution (l)

If we sum the amount of Na in the solution at the end of the 24 hour aliquot with the amount that was observed in the respiked solution at the end of 24 hours, we get an extraction of approximately 6385 mg/l of Na from the zeolite X into the solution assuming that the kinetics of this ion exchange are sufficiently fast that nearly all the exchange occurs within two hours of solution/zeolite contact. This equates to an exchange of approximately 78%. We suspect that had we analyzed the aliquots of the initial exchange solution prior to spiking, the actual exchange may have been even higher. Regardless, a nearly 80% exchange was deemed sufficient to proceed to the large-scale synthesis.

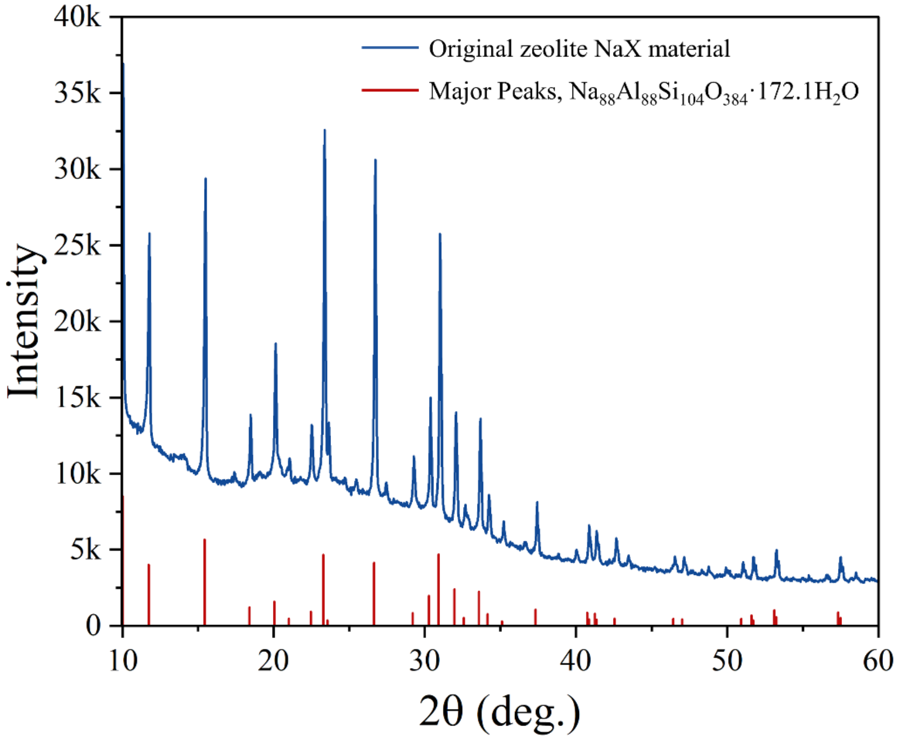

Figure 3: Diffraction pattern of the original zeolite NaX material that was used in the initial zeolite LiX POC experiment. The red lines show the major peaks associated with the zeolite having the stoichiometric formula $Na_{88}Al_{88}Si_{104}O_{384}\cdot 172.1H_2O$.

2.3 Large batch (>100 g) LiX synthesis

Upon confirmation of the synthesis pathway for zeolite LiX with the POC experiment, we proceeded to make an upscaled production run using a slightly lower ionic strength exchange solution than in the POC experiment. In the large-scale batch, we added 127.17 g of LiCl to 1.5 L of DI H_2O to yield a 2 M LiCl solution. We added approximately 116 grams of 2 µm powder zeolite X to 1160 ml of this solution in 4-L high-density polyethylene (HDPE) bottle and the solution was agitated by manual shaking for approximately 30 seconds. The exchange solution and zeolite were then allowed to sit for approximately 4 hours.

After the initial, 4-h exchange, the original exchange solution was decanted, aliquots of this solution were sampled, and 1 L of a fresh 2.0 M LiCl solution was added to the 4-L bottle with the zeolites. A magnetic stir bar was added to the bottle and the solution was agitated via stirring for an additional ~48 hours after the spike. We used longer times in the large-scale synthesis relative to the POC experiments to ensure we were exchanging the maximum amount of Li possible given the significantly higher mass of zeolites compared to the initial POC experiments.

After the 48-hour exchange had completed, the spike solution was decanted from the 4-L bottle, aliquots were sampled for ICP-ES analysis, and approximately 1 L of fresh DI H_2O wash solution was added to the 4-L bottle. After 24 hours, the wash solution (wash 1) was decanted and a fresh wash solution was added to the 4-L bottle. This wash cycle was repeated once more at a 24-hour interval for a total of three washes over 72 hours. After 72 hours, at the anticipated end of the third wash, we observed continued suspension of some of the zeolite solids in solution. We took a small aliquot of the solution and allowed this system to settle for an additional 24 hours prior to decanting the remainder of the solution and moving on to the drying steps.

The remainder of the now zeolite LiX solids were removed from the 4-L HDPE bottle and placed into glass beakers for an initial oven drying at 110 °C for 24 hours. This drying was done to remove all of the non-crystalline wash water from the sample in a timely fashion. After the oven drying, half of the remaining zeolite LiX powder was apportioned for LiAgX exchange, and the remaining mass was placed into a Pt-Rh crucible and "calcined" at 400 °C for approximately 6 hours to dehydrate the zeolite.

2.4 Results of LiX large batch synthesis

The results of the ICP-ES analysis for the large-scale exchange are shown in Table 3. The nominal empirical formula for the powdered zeolite X is given by the manufacturer as $Na_{86}Al_{86}Si_{106}O_{384}{\cdot}xH_2O$ – the value of x was determined by two gravimetric analyses in which 1.00 g and 2.657 g of zeolite X were placed into Pt crucibles and calcined at 800 °C for 2.5 hour to remove crystalline water. The data for this gravimetric analyses are shown in Table 2.

Table 2: Gravimetric analyses data for determining crystalline water amount.

	Run 1	Run 2
Zeolite mass (g)	1.00	2.66
Zeolite mass after calcine (g)	0.97	2.58
Mass Loss (%)	3.0	3.0

This data was used to determine the stoichiometric crystalline water, and the analyzed empirical formula, $Na_{86}Al_{86}Si_{106}O_{384}{\cdot}23H_2O$, was used to determine the "anticipated Na exchange" at the various percentages shown in Table 3.

Given the stoichiometry of zeolite X, this 1:10 g/ml M/V ratio would provide approximately 12200 mg/l of Na available for exchange in the solution.

Table 3: ICP-ES results of the large-scale synthesis of zeolite LiX.

Sample ID	**Anticipated Na at 50% (mg/l)**	**Anticipated Na at 100% (mg/l)**	**Li**	**Na**
Na-Exchanged Initial Exchange (4 hr)	7146	14292	14233	7932
Na-Exchanged Secondary exchange (48 hr) POST SPIKE	7146	14292	14667	3713
Wash 1	7146	14292	3672	818
Wash 2	7146	14292	1023	213
Wash 3 (72 h)	7146	14292	268	56
Wash 3 (96 h)	7146	14292	268	56

After the first 4 hours of exchange, over 50% of the total available Na in the zeolite X had been exchanged (anticipated at 50%: 7146 mg/l; measured: 7932 mg/l). The analysis of Na in the "post-spike" aliquot indicated an additional 3713 mg/l of Na in the solution. Addition of the Na present in these two samples yields 11645 mg/l of Na that was present in the solutions throughout the course of the synthesis. Therefore, the percent $Li^+ \rightarrow Na^+$ in the large-scale LiX synthesis is given by taking the total amount of Na measured in the exchange solutions and dividing that by the total amount of Na present in the zeolite initially:

$$\frac{11645 \text{ mg/l}}{14292 \text{ mg/l}} \times 100\% = 82\% \text{ exchanged}$$

Based on the results of the ICP-ES analysis, we concluded that we had exchanged sufficient amounts of Li^+ for Na^+ in the zeolite X structure to continue with the preparation and synthesis of zeolite LiAgX.

3.0 Synthesis of zeolite LiAgX

3.1 Proof of concept for zeolite LiAgX synthesis

Approximately 2 grams of the large-scale zeolite LiX material were used for a POC zeolite LiAgX synthesis experiment. We used silver acetate, $AgC_2H_3O_2$ (AgAce), as the solute to prepare the silver-containing, ion exchange solution. The solubility limit of AgAce is 1.04 g/100 ml or 0.06 mol/l. To maximize the exchange in the initial experiment, we prepared a 0.05 mol/l AgAce solution by adding 0.835 g of AgAce to 100 ml of DI H_2O.

We then prepared two ion exchange experiments by adding 1 g of zeolite LiX to 40 ml of the 0.05 mol/l AgAce solution in 50 ml centrifuge tubes. The centrifuge tubes were placed on a rotating turntable as in the LiX synthesis. After approximately 2 h of the initial exchange, the tubes and zeolite samples were centrifuged at 3500 rpm for 5 min. Aliquots were collected from the centrifuged tubes, and the remaining supernate from each tube was decanted and replaced with a fresh, 0.05 mol/l solution. After approximately 24 h, the centrifuge tubes were removed from the turntable, and the tubes were centrifuged at 3500 rpm for 5 min. Aliquots were again collected of the spike exchange supernate and the remnant exchange solutions were decanted and discarded. Each zeolite material was then washed three times with DI H_2O with a procedure that was similar to that described in Section 2.1.

The solids from each tube were then combined into a glass beaker and dried at 110 °C to remove all of the non-crystalline, remnant wash water from the zeolitic materials. After drying, the solids were analyzed via X-ray diffraction (XRD) to determine the zeolite structure after this exchange as shown in Figure 4.

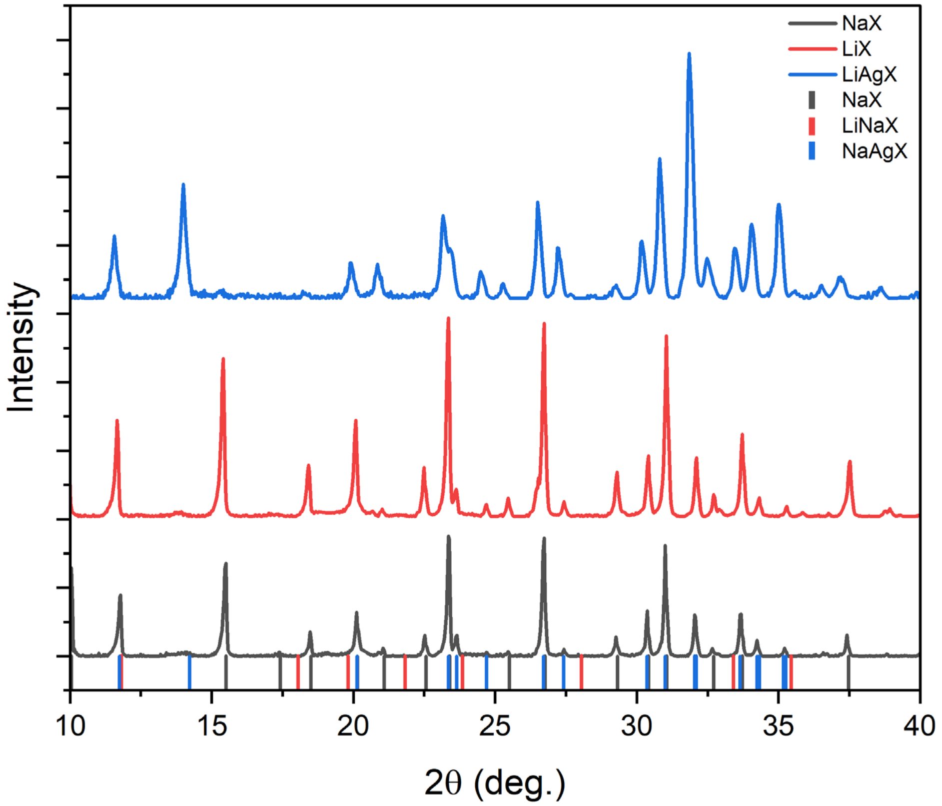

Figure 4: XRD patterns for the various zeolites (NaX, LiX, and LiAgX) used or synthesized in this work.

3.2 Results of the POC zeolite LiAgX synthesis

The results of the ICP-ES analysis of the POC zeolite LiAgX ion exchange and wash solutions are given in Table 4. A "blank" experiment was included with the exchange series to ensure that the silver was remaining in solution throughout the exchange process. This centrifuge tube contained only the 0.05 mol/l AgAce solution and was void of zeolite materials.

Table 4: ICP-ES Results of the POC zeolite LiAgX ion exchange solutions

Sample ID	**Ag (mg/l)**	**Anticipated Li at 100% (mg/l)**	**Li (mg/l)**	**Anticipated Na at 100% (mg/l)**	**Na (mg/l)**
PRE-spike	<1	975	305	665	111
POST-spike	63	975	235	665	323
WASH-1	2	975	8	665	11
WASH-2	<1	975	<1	665	1
WASH-3	<1	975	<1	665	1
blank	5463	975	<1	665	<1

Using the data in Table 3, we determined the approximate percentage of $Ag^+ \rightarrow Li^+$ and $Ag^+ \rightarrow Na^+$ exchange in the POC experiments by summing the pre- and post- spike Li and Na concentrations and comparing to the anticipated exchange concentrations:

$$\frac{(305\ \text{mg/l} + 235\ \text{mg/l})}{975\ \text{mg/l}} \times 100 = 55\%\ \text{exchange of available Li}$$

$$\frac{(111\ \text{mg/l}+323\ \text{mg/l})}{665\ \text{mg/l}} \times 100 = 65\%\ \text{exchange of available Na}$$

With this demonstration of Ag^+ exchange for the Li and the remnant Na in the zeolite structure, we determined that we could proceed with the large-scale zeolite LiAgX synthesis. Based on the calculated exchange of $Li^+ \rightarrow Na^+$ from the large-scale zeolite LiX synthesis and the $Ag^+ \rightarrow Li^+$ and $Ag^+ \rightarrow Na^+$ percentages above, the new stoichiometric zeolite LiAgX formula would be: $Ag_{48.85}Li_{31.73}Na_{5.42}Al_{86}Si_{106}O_{384} \cdot 23H_2O$.

3.3 Large batch zeolite LiAgX synthesis (> 50 g)

We based the large-scale synthesis of zeolite LiAgX on the method described in Section 3.1 of this report. However, in order to more closely align with testing reported previously in literature, we lessened the amount of Ag used in the exchange to target a specific $Ag^+ \rightarrow Li^+$ exchange of 20%. The concentration of the silver acetate exchange solution was determined by assuming a 100% exchange of Li in the initial zeolite LiX synthesis such that the stoichiometric formula of the zeolite would be $Li_{86}Al_{86}Si_{106}O_{384} \cdot 23H_2O$.

We prepared 0.01 M $AgC_2H_3O_2$ solutions by dissolving the material in DI H_2O. In order to achieve the appropriate M/V ratio for the desired exchange target, the mass of zeolite LiX was split into two batches. The exchanges were conducted in 4-L HDPE bottles in which 25 grams of zeolite LiX was added to each bottle, and 2.5 L of the 0.01 M $AgC_2H_3O_2$ solution were added to each bottle. Each bottle was agitated, and the exchange systems were allowed to sit overnight. Since we were targeting a specific exchange percentage by only providing sufficient Ag to yield that exchange amount, we were not concerned with controlling the kinetics of the exchange via time or temperature control – we assumed, based on the previous POC experiment, that 24 h was sufficient time to achieve the desired exchange.

After the exchange period had concluded, the complete contents of each of the 4-L HDPE exchange bottles were separated into 10, 250 mL HDPE bottles. These bottles were then centrifuged at 3500 rpm for 10 min. Aliquots of the supernate, IEX solutions were collected for ICP-ES analysis, and the remainder of the IEX solutions were discarded.

We then proceeded to wash the contents of each of the 10, 250-mL HDPE bottles in the same fashion as previously described and centrifuging each wash solution at 3500 rpm for 10 minutes. Aliquots of each wash solution were collected for analysis with ICP-ES, and the remainder of the solutions were discarded.

A smaller scale, witness experiment was conducted using 100 mL of the 0.01 M $AgC_2H_3O_2$ exchange solution and 1 g of zeolite LiX to maintain a consistent M/V ratio. This experiment was conducted to assist in the mass balance calculation to ensure the appropriate amount of $Ag^+ \rightarrow Li^+$ exchange. Centrifuging, washing and aliquot sampling of the small-scale experiment followed the same schedule as the larger batch synthesis.

3.4 Results of the large batch zeolite LiAgX synthesis

The solution analysis results of the two 25 gram/2500 ml exchange systems along with the smaller scale witness system are given in Table 5.

Table 5: The ICP-ES analysis of the large-scale zeolite LiAgX ion exchange and wash solutions.

Sample ID	Ag (mg/l)	Li (mg/l)	Max Amount Li (mg/l)	Na (mg/l)	Max Amount Na (mg/l)
Ag→LiX Zeo Exchange Soln A	<1	81	390	23	266
Ag→LiX Zeo IEX (A) Wash 1	<1	10	390	3	266
Ag→LiX Zeo IEX (A) Wash 2	<1	4	390	1	266
Ag→LiX Zeo IEX (A) Wash 3	<1	2	390	1	266
Ag→LiX Zeo Excahnge Soln B	<1	80	390	22	266
Ag→LiX Zeo IEX (B)	<1	81	390	22	266
Ag→LiX Zeo IEX (B) WASH 1	<1	9	390	3	266
Ag→LiX Zeo IEX (B) WASH 2	<1	3	390	1	266
Ag→LiX Zeo IEX (B) WASH 3	<1	2	390	<1	266
Ag→LiX Zeo Exchange Soln Small Scale Test (Blank)	1233	<1	390	<1	266
Zeolite LiAgX (c) - IEX Soln	<1	81	390	22	266
Zeolite LiAgX (c) - Wash 1	<1	10	390	4	266
Zeolite LiAgX (c) - Wash 2	<1	3	390	1	266
Zeolite LiAgX (c) - Wash 3	<1	1	390	<1	266

Using the stoichiometric formula for zeolite LiX determined in Section 2.4, we calculated the percentage of Li and remnant Na exchanged with Ag. The percentage of Li exchange in this synthesis evolution was then found by dividing the mass of Li measured in the solution by the maximum amount of Li in the sample per liter of solution (i.e., Max amount of Li):

$$\frac{81\,mg/l}{390\,mg/l} x100\% = 20.8\%$$

And the amount of the remnant Na that was exchanged was found in the same fashion:

$$\frac{22\,mg/l}{266\,mg/l} x100\% = 8.3\%$$

As such, we determined that the final aluminosilicate (non-crystalline-water components) stoichiometry for the zeolite LiAgX that we prepared was approximately $Li_{56}Na_{14}Ag_{16}Al_{86}Si_{106}O_{384}{\cdot}23H_2O$. The stoichiometry was confirmed via electron dispersive X-ray spectroscopy (EDS). Ten EDS spectra were recorded at different locations on a zeolite LiAgX powdered sample. The spectra are shown in Figure 5.

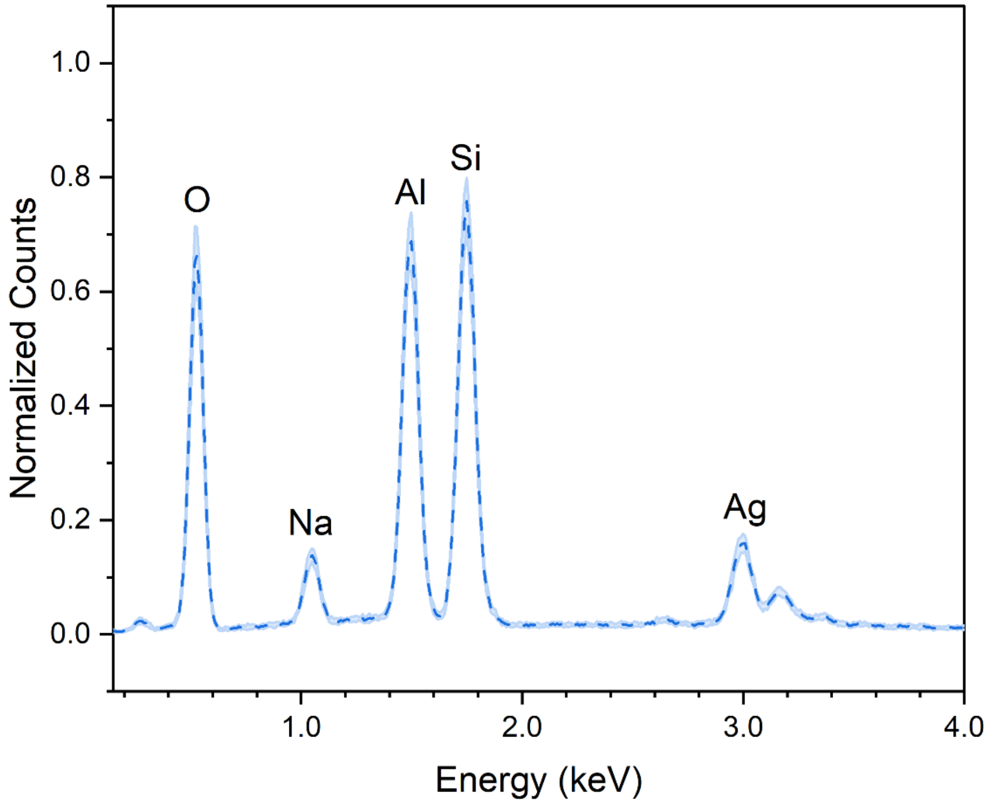

Figure 5: EDS spectra taken from ten different locations a powder zeolite LiAgX sample. The dashed line represents the average value of the ten measurements and the light blue shaded region shows the uncertainty.

The atomic percentages of the various elements in each measurement were then used to compare the ratio of Ag to Al and Si in the sample. The results of these calculations are shown in Table 6. The "nominal" values for Ag/Si and Ag/Al of 0.15 and 0.19, respectively, were calculated from the stoichiometric formula derived from the mass balance. The atomic percentages of each of these elements from each EDS spectrum were used to calculate an Ag/Si and Ag/Al ratio for each measurement, and then the average and standard deviation (St. Dev.) of each measured ratio was determined. The results of this analysis confirm the final zeolite stoichiometry calculated from the ICP-ES measurements of the ion exchange solutions throughout the course of the synthesis work.

Table 6: The results of the ten EDS measurements used to determine the stoichiometric ratios of Ag, Al, and Si in the zeolite LiAgX.

Spectrum #	Ag/Si	Ag/Al
1	0.16	0.18
2	0.17	0.21
3	0.11	0.13
4	0.12	0.15
5	0.14	0.16
6	0.15	0.18
7	0.15	0.18
8	0.15	0.18

9	0.16	0.19
10	0.17	0.20
Average	0.15	0.18
St. Dev.	0.02	0.02
Nominal	0.15	0.19

4.0 Summary

We prepared approximately 50 grams of zeolite LiX and 50 grams of zeolite LiAgX utilizing the ion exchange phenomenon with LiCl and silver acetate solutions, respectively. We first performed POC experiments to ensure that the reaction pathways were as expected for each zeolite type and then moved into large-scale production (>50 grams). In the large-scale zeolite LiX synthesis, we had an ion exchange yield of approximately 82%. In the large-scale zeolite LiAgX exchange, we targeted a 20% ion exchange and determined, via mass balance, that the actual exchange yield was approximately 20.8%. The final calculated and confirmed stoichiometries of the zeolite LiX and zeolite LiAgX materials were, $Li_{71}Na_{15}Al_{86}Si_{106}O_{384}\cdot xH_2O$ and $Li_{56}Na_{14}Ag_{16}Al_{86}Si_{106}O_{384}\cdot xH_2O$, respectively and where x is dependent on the hydrated state of the zeolite.

5.0 References

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