

Hydrothermal Preparation of Zeolite Li-A and Ion Exchange Properties of Cs and Sr in salt waste

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1. Introduction

An advanced spent fuel management process that were based on Li reduction of the oxide spent fuel to a metallic form will generate a LiCl waste.

Zeolite A has been reported as a promising immobilization medium for waste salt with CsCl and SrCl₂[1-2]. However, Sodium is accumulated as an ionic form (Na⁺-ion) in molten salt during ion exchange step between Na⁺-ion in zeolite A and Li⁺-ion in the molten salt. Therefore, zeolite Na-A need to be replaced by the Li-type zeolite for recycling the salt waste by removing the Cs and Sr ions.

In this study, the hydrothermal preparation of zeolite Li-A was performed in 350ml pressure vessel by P. Norby method [3]. The preparation characteristics of zeolite Li-A was investigated. And the ion exchange properties of Cs and Sr in molten LiCl salt were investigated under the condition of 923K using zeolite 4A and prepared zeolite Li-A.

2. Material and Methods

Zeolite Li-A(BW), LiAlSiO₄-H₂O was prepared hydrothermally from zeolite 4A as following 2-steps by P. Norby method,

1) batch ion exchange step
[150ml, 1.6M LiCl solution + 10g zeolite Na-A]
mix at room temperature until uniform slurry,

2) Crystallization step

- Vessel: 350ml, S.S 304 pressure vessel
- Time: 72-93hr, - Temperature: 180-285°C

After two steps, samples wash until chloride-free and then, dry at 110°C. The prepared zeolite zeolite Li-A was analyzed by XRD.

To study the ion exchange properties of Cs and Sr in salt waste, the S.S vessel with a mixture of LiCl, CsCl, and SrCl₂ powders was placed within the bath ion exchanger under Ar atmosphere. The reactor was heated to 923 K and maintained for more than 30 min. After ion exchange reaction, zeolite was washed free of residual salt on the zeolite surface, and dried at 383 K.

The experimental apparatuses for the hydrothermal preparation of zeolite Li-A(left side) and immobilization of salt waste(right side) are as follows;



Figure 1. Experimental Apparatuses for the preparation of zeolite Li-A and immobilization of salt waste.

3. Results and Discussion

Hydrothermal conversion of zeolite Na with an aqueous solution of LiCl at temperature from 180

to 285°C results in formation of Zeolite Li-A(BW), $\text{LiAlSiO}_4 \cdot \text{H}_2\text{O}$. Several experiments were carried out to determine the optimal conditions(temp. et al.) Good results were obtained at the following conditions.

- 1) Experimental condition 1:
 - Ion exchange : LiCl solution for 48hr
 - crystallization : 200 °C for 72 hr
- 2) Experimental condtion 2
 - Ion exchange : LiCl solution for 72hr
 - crystallization : 200 °C for 93 hr

XRD patterns for the prepared zeolite Li-A at upper two conditions are shown at Figure 2.

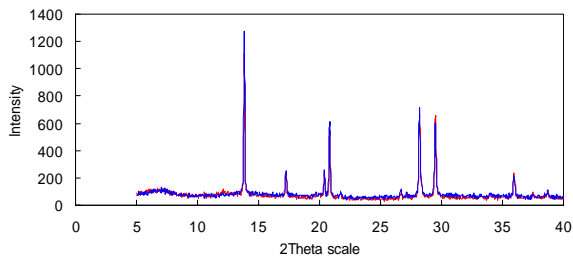


Figure 2. XRD patterns of prepared zeolite Li-A

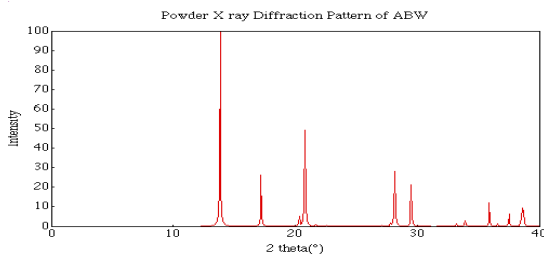


Figure 3. XRD patterns for standard zeolite Li-A

The XRD patterns of prepared zeolite Li-A was compared with that of standard zeolite Li-A(BW) in Figure 3. As shown in XRD patterns in Figure 2 and Figure 3, the prepared zeolite Li-A has similar XRD pattern with that of the standard zeolite Li-A. Based on these results, the prepared zeolite Li-A seems to be almost replaced by Li ion.

As shown in Fig. 3, an uptake of Cs reached an equilibrium state very quickly and kept constant, ion exchange characteristic of Cs by prepared zeolite has similar trend with that of zeolite 4A. Uptake of Cs and Sr ions in LiCl molten salt reaches nearly constant value after 2 to 4 hr of ion exchange reaction.

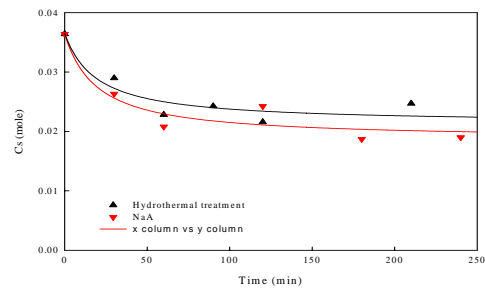


Figure 3. Cs ion exchange with zeolite Na-A and prepared zeolite Li -A

4. Conclusions

Hydrothermal preparation of Zeolite Li-A(BW) was performed from bead type zeolite 4A. The prepared zeolite Li-A has similar XRD pattern with standard zeolite Li-A(BW). Based on these results, Na^+ -ion in zeolite A seems to be almost replaced by Li ion in LiCl solution, the prepared zeolite seems to be Zeolite Li-A(BW). Uptake of Cs and Sr ions in LiCl molten salt reaches nearly constant value after 2 to 4 hr of ion exchange reaction.

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