

# Purolite S957 Ion Exchange Resin for Uranium Removal From Effluents Generated During a Spent Catalyst Treatment Process: Real Waste Uptake and Elution Studies

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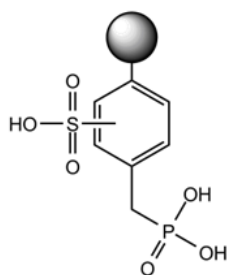
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## 1. Introduction

A large volume of problematic uranium waste in the form of a spent catalyst is in temporary storage in Korea. Recently the Korea Atomic Energy Research Institute (KAERI) has developed an appropriate method for the treatment, segregation and ultimately safe disposal of this waste.[1-5] This research has led to the development of a process involving multiple unit steps including heat treatment, dissolution, selective precipitation and filtration. The process relies on the treatment of generated effluents via a uranium precipitation method.[1,4] While effective, recording decontamination factors greater than 8000, the generation of large floc volumes is not ideal for final volume reduction. Further, there is currently no back-up system in the event of mal operation should there be a process failure during uranium precipitation.

Recently we have reassessed, in collaboration with the University of Manchester (UoM), the treatment options available for the generated effluents. The goal was to explore alternative treatment methods and develop a back-up strategy based on ion exchange (IX) in the event of mal operation. It is shown that Purolite S957 (**Figure 1**), a phosphonic sulfonic acid chelating/anion exchange resin, outperforms all other tested resins during screening trials.[6] In this study we present the results from real waste tests focusing on laboratory scale uranium removal and elution. We have assessed the viability of S957 for the selective removal of uranium from the real process effluent.



S957

Fig. 1. Structural diagram of Purolite S957.

## 2. Experiment

### 2.1 Materials

Purolite S957 is a macroporous styrene DVB 8%

crosslinked phosphonic sulfonic acid chelating/anion exchange ion exchange resin. The resin was supplied by Purolite. The effluent was produced during the treatment of the catalyst and stored in 20 L plastic drums before use. All other chemicals were purchased from Sigma-Aldrich and used as supplied. A complete description of the spent uranium catalyst and dissolution process has been previously reported.[1]

### 2.2. Method

**U Removal:** Dynamic tests with the real waste (~30 ppm U) were conducted at the laboratory scale using variable flow rates (12.5, 7.4, 4.7 or 1.3 BV Hr<sup>-1</sup>) and 1 mL of WSR loaded in a sealed plastic column in an mounted in an upflow configuration.

**U Elution:** Elution tests were performed under the same configuration as the removal tests. The elution media was passed through the resin at 1.3 BV Hr<sup>-1</sup>. Four eluents were tested, 0.1 M H<sub>2</sub>SO<sub>4</sub>, 2.5 M H<sub>2</sub>SO<sub>4</sub>, 0.1 M Na<sub>2</sub>CO<sub>3</sub> in 2 M Na<sub>2</sub>SO<sub>4</sub> and 0.1 M FeCl<sub>3</sub>. Five BV of deionised water was passed over the resin bed prior to the eluent media. A total of 100 BV of eluent was used. Uranium concentration was determined via ICP-OES analysis in a 5% nitric acid carrier-media.

## 3. Results and discussion

### 3.1 Screening studies & IXR Selection (UoM)

Uranium loading tests showed Purolite S957 to be the most effective at high [H<sup>+</sup>]; synonymous to the real waste. Purolite S957 was also found to have the highest loading capacity of all the resins tested (96.15 mg g<sup>-1</sup>) in batch and dynamic testing. The superior performance of S957 also carried across to the dynamic testing studies with the simulant waste. Purolite S957 was found to show the highest uranium breakthrough capacity at the slower flow rate of 88.73 mg g<sup>-1</sup> at a flow rate of 7.5 BV hr<sup>-1</sup>. [6]

### 3.2 Real waste tests – Uranium Removal (KAERI)

Tests with the real waste were performed with Purolite S957. Removal of uranium was successful across all flow rates tested (**Figure 2**). However, the breakthrough capacity of Purolite S957 under real waste conditions is significantly lower at 12.2 mg g<sup>-1</sup> under a flow of 1.3 BV Hr<sup>-1</sup> when compared to 88.73 mg g<sup>-1</sup> at a flow of 7.5 BV Hr<sup>-1</sup> for the simulant waste.

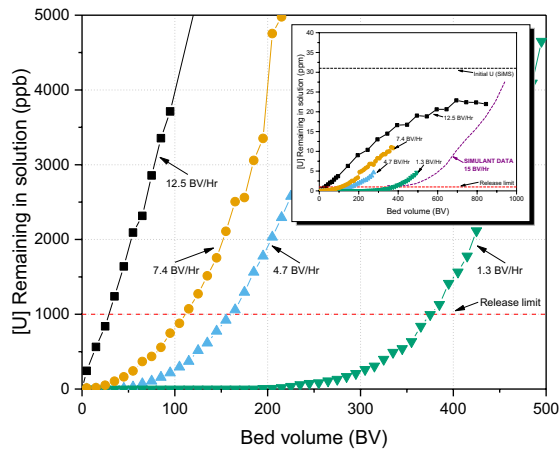


Fig. 2. Breakthrough curves for S957 at various flow rates.

### 3.3 Real waste tests – Uranium Elution (KAERI)

Elution of uranium was achieved with four different elution media (**Figure 3**). Sulfuric acid, regardless of concentration, was poor for the elution of uranium. At 2.5 M  $H_2SO_4$  after 100 BV residual uranium remained on the resin which accounted for approximately 20% by difference (**Figure 3 B**). Iron chloride showed a significant improvement with an initial sharp rise in eluted uranium, but this was followed with a long and drawn out tail (**Figure 3 D**). A mix of sodium sulfate and sodium carbonate yielded the most promising results with a sharp elution profile over 15 - 20 BV (**Figure 3 C**). It is well known that uranium forms highly soluble carbonate species, a fact which is leveraged for the purposes of elution in this study.

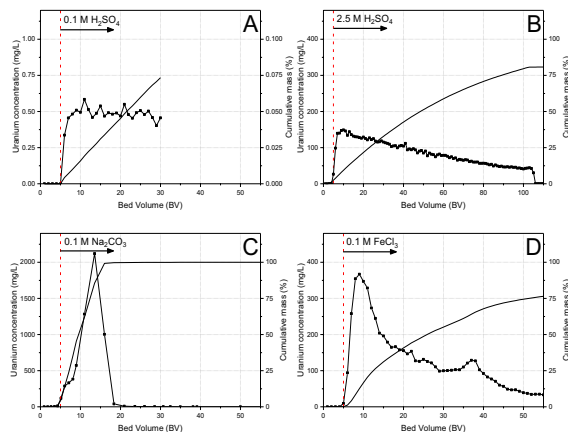


Fig. 3. Uranium elution curves for S957 using various eluents.

The use of a carbonate-based elution media offers the possibility of recycling the S957 resin, thus enabling significantly more effluent through the resin before final exhaustion and disposal. Such an approach would reduce costs and potentially overcome the issue of reduced breakthrough capacity as seen compared to the simulant studies.[6] However, not only is the uranium eluted off the column but so too are the other major contaminants (**Figure 4**). This has potential to further complicate effluent handling post-column as the effluent would require final treatment.

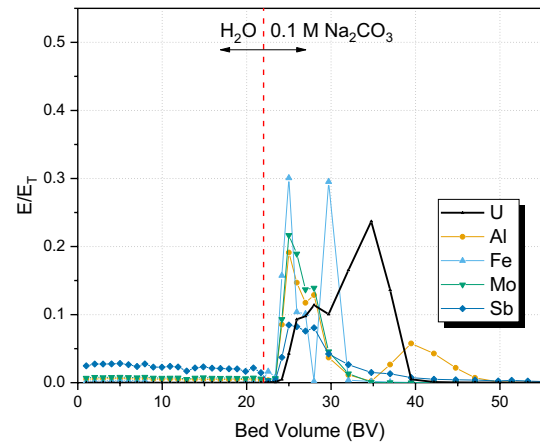


Fig. 4. Elution profile of all contaminants with  $H_2O$  and  $Na_2CO_3$ .

## 4. Conclusions

Despite promising results with the simulant S957 showed poorer performance under real waste tests with approximately 376 BV being treated before breakthrough at the lowest flow rate of  $1.3 \text{ BV Hr}^{-1}$ . This was accompanied by a significantly lower breakthrough capacity of only  $12.2 \text{ mg g}^{-1}$ . Evidence suggests uranium elution is possible with a carbonate elution media, however, this is accompanied by the elution of the other contaminants which poses issues for final effluent treatment. Therefore, implementing a uranium absorption column loaded with S957 for the treatment of the effluent as a direct replacement for the phosphate precipitation method[4] would not be recommend. However, there is evidence to suggest that incorporating such a column into the process flow sheet in the event of mal-operation is still possible.

## REFERENCES

- [1] K. W. Kim *et al.*, "The development of a process for the volume reduction of uranium catalyst waste used for production of acrylonitrile", In Press, (2018).
- [2] K.-W. Kim *et al.*, "Treatment method of spent uranium catalyst", U.S. Patent 9181605 B2, 2015.
- [3] R. I. Foster *et al.*, "The removal of uranium from aqueous effluents via meta-ankoleite formation", KRS, (2017).
- [4] R. I. Foster *et al.*, "The Selective Removal of Uranium via Phosphate Addition for the Treatment of Uranium Laden Effluents", In Press, (2018).
- [5] H. H. Sung *et al.*, "An Evaluation on the Glass-Ceramic Solidification Characteristics of Uranium catalyst waste by Green body Pressure", KRS, (2017).
- [6] R. I. Foster *et al.*, "Ion-Exchange as an Alternative Treatment Method for Uranium Effluents Generated as part of a Spent Uranium Catalyst Treatment Process", International Conference on Ion Exchange, Indonesia, (2018).