



# Extraction of uranium from non-saline and hypersaline conditions using iminodiacetic acid chelating resin Purolite S930 +

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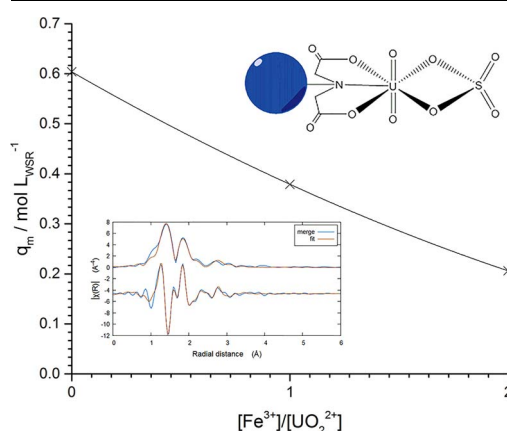
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## HIGHLIGHTS

- No observed impact of chloride on uranium recovery.
- IDA functionality binds  $\text{UO}_2^{2+}$  in a tridentate fashion.
- Chelation ion exchange mechanism for  $\text{UO}_2^{2+}$  recovery proposed.
- $\text{Fe}^{3+}$  has a large impact on  $\text{UO}_2^{2+}$  recovery from saline media.

## GRAPHICAL ABSTRACT



## ARTICLE INFO

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## ABSTRACT

Uptake behaviour of uranium from aqueous, acidic sulfate and chloride media on iminodiacetic acid chelating resin Purolite S930+ has been studied. Experiments have followed  $\text{UO}_2^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Fe}^{3+}$  uptake behaviour with respect to acidic and ionic media type and concentration. Uptake suppression of all metals was observed at  $[\text{H}^+] > 0.1 \text{ M}$  sourced from  $\text{H}_2\text{SO}_4$  and  $\text{HCl}$ . In contrast, significant uptake of  $\text{Fe}^{3+}$  was observed from solutions with  $[\text{HCl}] > 2 \text{ M}$ . Suppression of  $\text{UO}_2^{2+}$  uptake (up to 15%) was observed upon increasing  $[\text{SO}_4^{2-}]$  to 4 M, whilst negligible  $\text{UO}_2^{2+}$  uptake suppression was observed with  $[\text{Cl}^-]$  up to 6 M. The impact of  $\text{Fe}^{3+}$  concentration on  $\text{UO}_2^{2+}$  extraction under hypersaline conditions ( $[\text{Cl}^-] = 22.6 \text{ g L}^{-1}$ , 0.64 M) has been studied and behaviour fit to Langmuir and Dubinin-Radushkevich isotherms. Extended X-ray absorption fine structure (EXAFS) studies have been performed to assess the effect of salinity on the uranium coordination environment on the resin and therefore the mechanism of uptake. No change in surface species was observed, with the fit species being uranyl bound by the iminodiacetic acid functional group in a tridentate motif, with an associated bidentate sulfate group. An isotherm model based on this surface species has also been derived. It has been shown that at pH 2 there is little impact of increasing chloride and sulfate concentrations on the extraction behaviour of metals onto Purolite S930+ under the conditions tested. Rather, uranium uptake is more affected by the presence of

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$\text{Fe}^{3+}$  in solution. As  $[\text{Fe}^{3+}]/[\text{UO}_2^{2+}]$  is increased from 0 to 2,  $\text{UO}_2^{2+}$  uptake is reduced by up to 66% at aqueous equilibrium.

## 1. Introduction

Uranium mines consume vast quantities of water, and many of these mines are located in arid regions with either poor access to fresh water or access only to lower quality (saline) water sources [1,2]. As an example, it is estimated that the Olympic Dam mine site in South Australia consumes 42 ML of fresh water per day, with a planned expansion of this site pushing this figure up to 200 ML per day [3]. This consumption puts a strain on fresh water supplies and increases the overall cost of the mining operation. The majority of this fresh water is used to extract soluble uranium from its ore, this liquor is then treated with ion exchange (IX) and/or solvent extraction (SX) processes as part of the milling strategy. The use of untreated bore water and/or seawater instead of fresh water in these extraction steps may address both the environmental and economic issues of using such large quantities of fresh water. However, if such waters are used in uranium extractions, the effect of  $\text{Cl}^-$  on the chemistry of the system must be fully understood. For the purposes of this paper, the  $\text{Cl}^-$  concentrations have been classified into three categories (with respect to liquors used in common uranium IX processes), conventional, saline and hypersaline liquors, as outlined in the schematic diagram (Fig. 1) below.

After a uranium ore has been mined, screened and crushed it undergoes leaching. This leaching process involves the solubilising of uranium to produce a pregnant leach liquor (PLL). The leach liquor used depends on the chemical makeup of the ore, but it generally employs sulfuric acid as it is more environmentally friendly and cheaper than hydrochloric or nitric acid. A carbonate leach can be used if the ore consumes large amounts of acid. Ores containing tetravalent uranium need an oxidant, commonly sodium chlorate or manganese dioxide, to produce soluble hexavalent uranium which can be sent for further processing. Extraction of uranium from the PLL most commonly employs IX or SX, although nanofiltration has seen some use [4]. Ideally, the extraction medium, whether SX or IX, will selectively remove the uranium from the PLL. Therefore the choice of extractant depends heavily upon the chemistry of the PLL. The development of new extraction processes that are more tolerant to the presence of  $\text{Cl}^-$  may allow greater flexibility in the composition of the PLL, thus allowing the use of lower quality waters relative to fresh water.

Although SX is the major workhorse technology for uranium recovery there are still some general drawbacks to this process [5,6]. These include the need for an extractant molecule soluble in the organic phase, potential for solvent loss, phase disengagement in multiple contact stages, third phase formation and the generation of large volumes of secondary organic waste. IX processes, as the sorbent is solid, remove all of the difficulties of handling large volumes of organic solvent. They also often have faster kinetics than SX systems and are more effective when extracting uranium at lower concentrations [6].

The majority of uranium processing circuits are sulfate-based [7]. Processes employing IX resins for the separation/concentration of uranium typically use anion exchange resins with strong base (SBA) functional groups [8]. Those employing SX generally use long chain tertiary amines as extractants [9]. Both techniques are sensitive to the presence of  $\text{Cl}^-$  in solution, which causes suppressed uranium uptake.

Similarly to IX resins, it has been shown that tertiary amines in SX can work via an IX mechanism [10]. In both cases the suppression is due to the decreased exchange of the uranium-sulfate species due to competing  $\text{Cl}^-$  [9,11]. This is compounded by the increased exchange of iron and other impurity elements. This reduced  $\text{UO}_2^{2+}$  affinity in high saline conditions can be overcome in IX systems by the application of a chelating resin. Functionalities on the resin surface employ the chelate effect through multidentate coordination, forming thermodynamically stable complexes.

In this paper the application of an iminodiacetic acid (IDA) chelation resin, Purolite S930+, to the extraction of uranium from simulant uranium process liquors has been explored. Though there are many examples of effective, novel sorbents for uranium, including layered silicates, nanocomposites, functionalised chitosan and functionalised silica (among numerous others), the Purolite S930+ resin has been selected due to its commercial availability and therefore relative ease of implementation into a traditional uranium recovery process [12–15]. The iminodiacetic acid functionality has been specifically chosen due to the fact that it offers both a strong acid uranium elution option, as  $\text{H}^+$  can effectively compete with uranium at low pH, and chelation strength usually increases with high ionic strength [16].

## 2. Experimental

### 2.1. Reagents and stock solutions

For all experimental studies, the commercially available chelating IX resin, Purolite S930+, was directly supplied by Purolite. The general specification data for this resin and other commercial equivalents, for comparative purposes, are given in Table 1 [17]. Prior to experimentation, the S930+ resin was preconditioned by contacting 1 mol  $\text{L}^{-1}$  HCl or  $\text{H}_2\text{SO}_4$  at a resin volume:acid volume ratio of 1:10 on an orbital shaker for 24 h at room temperature. The resin was then washed three times with 3 bed volumes (BV) of deionised water (18 M $\Omega$ ) before use. The structure of the IDA functionality of Purolite S930+ resin is given in Fig. 2.

All solutions used in loading experiments were prepared using deionised water (18 M $\Omega$ ) and analytical grade reagents. These uptake solutions were formulated to simulate a uranium leach liquor by the dissolution of metallic cation species commonly found in these liquors, in their sulfate form. For studies assessing the effect of iron,  $\text{Fe}^{3+}$  was added in molar ratios of 1 and 2 relative to  $[\text{UO}_2^{2+}]$  (Table 2).

### 2.2. Batch extractions from sulfate and chloride media

All batch extractions as a function of acid concentration were carried out as single contacts with 2 mL of wet settled resin (WSR) contacted with 50 mL of aqueous simulant feed. The addition of either NaCl or  $(\text{NH}_4)_2\text{SO}_4$  to increase  $\text{Cl}^-$  or  $\text{SO}_4^{2-}$  levels in the simulant feed was carried out as part of the simulant feed production to prevent increases in experimental solution volume.  $\text{UO}_2^{2+}$ ,  $\text{Cu}^{2+}$  and  $\text{Fe}^{3+}$  were spiked into the solution at 0.42, 1.57 and 1.79 mM, respectively (100 ppm of each metal). The resin and aqueous feed were continuously mixed for a

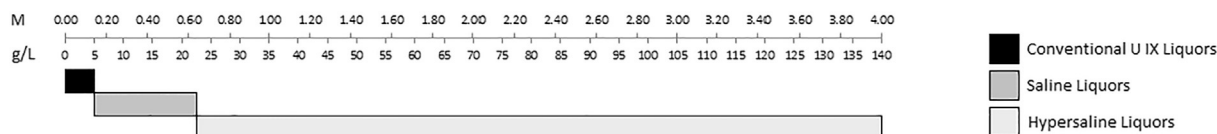


Fig. 1. Chloride concentrations in saline and hypersaline liquors.

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