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Orthogonal array design for the optimization of stripping Sr(II) from ionic liquids using supercritical CO_2☆

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ABSTRACT

The strontium ions extracted from the aqueous phase into 1-ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide ($C_2mimNTf_2$) with dicyclohexyl-18-crown-6 (DCH18C6) was stripped effectively by supercritical CO_2 (sc-CO_2). Hexafluoroacetylacetone (HFAA)–acetonitrile was found to be an excellent modifier of sc-CO_2 to enhance the stripping efficiency. In the orthogonal array design (OAD), OA_{25} (5^5) matrix was employed to optimize the stripping of Sr(II) from the DCH18C6–$C_2mimNTf_2$ system. Effects of five experimental factors: temperature, pressure, concentration of HFAA, static and dynamic extraction times as well as each factor at five-levels on the stripping of Sr(II) were optimized. The effects of these parameters were treated by the analysis of variance (ANOVA). The results showed that Sr(II) could be nearly 100% extracted from the IL phase at 308 K, 30 MPa, 40 min of dynamic extraction and 60 $mmol\cdot L^{-1}$ HFAA in acetonitrile, respectively. Finally, the stripping mechanism was studied by ESI-MS.

1. Introduction

The long lifetime strontium ion is one of the main high heat load, highly radioactive fission products in high level liquid wastes (HLLW) during the reprocessing of spent nuclear fuel. Many advantages were shown to remove Sr(II) from HLLW before the final geological disposal [1]. So far, solvent extraction has been a main method of separating Sr(II) from HLLW, and the ionic liquids (ILs), holding numerous excellent properties and becoming environmentally benign "green solvents" alternative to volatile organic solvents [2–7], have been employed in the extraction of Sr(II).

Dai *et al.* [8] first used ILs as solvents and DCH18C6 as extractant to extract Sr(II) from the aqueous phase. The distribution coefficient (D) reached 1.1×10^4, which was much higher than those of the conventional extraction systems. For example, the D values of the extraction systems with $C_6H_5CH_3$, $CHCl_3$, 2-octanol and 1,1,2,2-tetrachloroethane as diluents were only 0.76 [8], 0.77 [8], 6.5 [9] and 10.8 [10], respectively. Dietz and Dzielawa [11] proposed that the extraction of Sr(II) by IL systems was based on a cation exchange mechanism. Our research group studied the effect of the structure of ionic liquid, the acidity and the addition of inorganic salt in the aqueous phase on the extraction efficiency of Sr(II) by the DCH18C6–$C_nmimNTf_2$ ($n = 2, 4, 6$) system, and further confirmed the cation exchange mechanism [1]. In addition, we found that the γ-radiation stabilities of ILs were excellent during the extraction of Sr(II) with DCH18C6 [12,13], which is conducive to fit to the high irradiation environment in the spent nuclear fuel reprocessing.

Although ILs have lots of advantages towards the extraction of metal ions, the stripping, which is as important as the extraction process in the application, still remains a challenge. Our research group tried to study the stripping of Sr(II) with K_2SO_4 and the stripping efficiency reached 99% [1]. But there are two serious obstacles: cross contamination between two phases and loss of ILs. Electrodeposition is another method to recover metal ions from the IL phase [14,15]. However, this method applies at many strict conditions, *i.e.*, only for highly electropositive elements, and requiring the electrochemical window of an IL matching the reduction potential of a metal ion.

Supercritical CO_2 (sc-CO_2) is considered as another "green solvent", because CO_2 can be recycled and generates no wastes. Sc-CO_2 has many advantages including enhanced diffusivity, chemical inertness, non-flammability and low cost [16–18]. Sc-CO_2 has been employed to extract various metal ions such as lanthanides, actinides, alkali metals from aqueous or solid matrices [19–32]. Due to the limited mutual solubility between sc-CO_2 and IL, that is, the insolubility of IL in sc-CO_2 and the solubility of sc-CO_2 in IL [33–35], a feasible method to recover metal ions from IL phase by sc-CO_2 has been put forward. Some metal ions were stripped effectively by sc-CO_2 from IL phase [36–40]. However, the mechanism of stripping by sc-CO_2 has been rarely reported. Our research group recently used ESI-MS to study the mechanism of stripping U(VI) from the CMPO–$C_2mimNTf_2$ system by TOPO-modified sc-CO_2 [40].

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To the best of our knowledge, although DCH18C6 as the extractant in $C_2mimNTf_2$ performs excellently for the extraction of Sr(II), there have been no effective methods so far to strip it from the DCH18C6–$C_2mimNTf_2$ system with high stripping efficiency, no cross-contamination, and no loss of ILs.

In this study, we aim to use sc-CO_2 to strip Sr(II) from the DCH18C6–$C_2mimNTf_2$ system. Due to the strong polarity of DCH18C6·Sr^{2+} but nonpolarity of CO_2, the solubility of crown ether complexes in sc-CO_2 is very small. It is well known that fluorinated metal chelates are CO_2-philic [41], thus utilizing fluorinated anion to combine crown ether–metal cation as ion-pair is a favorable method. Wai *et al.* [23] successfully extracted Sr(II) from aqueous media into sc-CO_2 employing DCH18C6 and a fluorinated anion, *i.e.*, $CF_3(CF_2)_6CO_2^-$ ($PFOA^-$) or $CF_3(CF_2)_6CF_2SO_3^-$ ($PFOSA^-$). Another method to improve the solubility of crown ether complexes is adding cosolvent to sc-CO_2, such as acetonitrile, methanol, and acetone to enhance its polarity. Considering the two aspects in favor of the solubility of crown ether complexes in sc-CO_2, we chose a fluorinated metal chelate hexafluoroacetylacetone (HFAA) diluted in acetonitrile as

The mean value of each stripping efficiency for the respective factors and at each level was calculated according to the assignment of the experiment (Table 1). For example, the stripping efficiency of the five trials at 308 K was evaluated as mean value of the corresponding five runs (Trial number 1-5). The mean values of the five-levels of each factor reveal how the stripping efficiency changes when changing the level of that factor. Fig. 2 shows stripping efficiency as a function of levels of the studied factors. In all instances, it should be noted that each calculated stripping efficiency is the average of five measurements, and in each of which the interested parameter was kept constant, and all the other parameters were changed (Table 1).

The ANOVA results for calculated models are shown in Table 2. The ANOVA indicates that the temperature and pressure of sc-CO_2 play key roles and the dynamic time plays important role in the stripping of Sr(II) from the IL phase, whereas, in the selected range the static time and the concentration of HFAA do not have significant effects on the stripping of Sr(II).

3.2. Effect of extraction temperature

The variation of temperature during stripping affects the density of sc-CO_2, the volatility of the analytes and desorption of the analytes from the IL phase. At higher temperatures, the density of sc-CO_2 decreases and analytes become more volatile leading to lower efficiency as shown in Fig. 2(a).

The ANOVA of the results shows that the temperature plays a key role in the extraction of Sr(II). Therefore, we ought to select a lower extraction temperature (308 K) as the optimum temperature.

3.3. Effect of extraction pressure

Solubility of a solute in sc-CO_2 depends on a balance between sc-CO_2 density and solute vapor pressure, both of which are controlled by the pressure of sc-CO_2. As can be learnt from the ANOVA of the results, the pressure of the sc-CO_2 also plays a key role in the stripping of Sr(II) from the IL phase. This means that the stripping efficiency is enhanced by an increase in the pressure. As shown in Fig. 2(b), the optimized pressure is 30 MPa.

3.4. Effect of static and dynamic extraction times

In order to achieve high stripping efficiency, the static extraction can make the penetration of sc-CO_2 in the IL phase better than the dynamic extraction. The dynamic extraction follows static extraction to enhance the solubility of analyte in sc-CO_2.

Based on the ANOVA calculations, the dynamic time plays an important role in the stripping of Sr(II) from the IL phase. According to Fig. 2(d), when the dynamic time is increased, the stripping efficiency of Sr(II) firstly increases and then decreases, and the best dynamic time is 40 min. On the contrary, the static time has no significant effects on the stripping efficiency. For a rapid extraction of Sr(II) from the IL phase and on the basis of economy principle, one can select a shorter static time (15 min).

3.5. Effect of the concentration of HFAA

Based on the ANOVA calculations, the concentration of HFAA has no significant effects on the stripping of Sr(II) from the IL phase in the selected range (60–140 mmol·L^{-}

as the optimum concentration of HFAA according to the economy principle.

On the basis of the above results, the optimum values of the selected factors (temperature, pressure, static and dynamic extraction times and concentration of HFAA) for stripping of Sr(II) are 308 K, 30 MPa, 15 min, 40 min and 60 mmol·L^{-1}, respectively. Further experiment was performed under the proposed conditions and the stripping efficiency was about 100%.

In order to obtain the better conditions, we further optimized static stripping time and concentration of HFAA. When the static time was 15, 10, 5 and 0 min respectively at 308 K, 30 MPa, 40 min of dynamic stripping time and 60 mmol·L^{-1} HFAA in acetonitrile, the stripping efficiency of Sr(II) always reached 100%. The effect of the concentration of HFAA is shown in Fig. 3. The stripping efficiency is reduced when the concentration of HFAA decreases. So we can obtain the optimized conditions, namely, no static stripping time and the concentration of HFAA is 60 mmol·L^{-1} at 308 K, 30 MPa, 40 min of dynamic stripping time.

3.6. Stripping mechanism

The above results demonstrate that Sr(II) can be effectively stripped from the IL phase into the sc-CO_2 phase with HFAA–acetonitrile solution as the modifier. Acetonitrile as a co-solvent can enhance the polarity of CO_2 to increase the solubility of the complexes of Sr(II) in the modified sc-CO_2.

The stripping efficiency of Sr(II) from the IL phase can reach 100% and less than 5% of IL is lost during the stripping experiment under the optimal condition: 308 K, 30 MPa, 40 min of dynamic stripping time and 60 mmol·L^{-1} HFAA in acetonitrile.

We investigated the original IL phase, the recovered IL phase, and the stripping product by ESI-MS (Fig. 4(a-c)), respectively. The compositions of the fragments and their corresponding complexes are listed in Table 3.

Cationic complexes [C_2mim-DCH18C6-NTf_2-Sr-2H_2O]$^{2+}$, [Sr-NO_3-H_2O]$^{+}$, [Sr-NO_3-FAA-Sr]$^{2+}$, [DCH18C6-Sr-FAA]$^{+}$, [C_2mim-NTf_2-DCH18C6-FAA-Sr]$^{+}$ and [DCH18C6-Sr-NTf_2]$^{+}$ are observed in the ESI spectrum of the stripping product (Fig. 4(c)), which indicates that various kinds of the complexes of Sr(II) were formed in the stripping process. FAA^{-} not only competes with DCH18C6 to associate with Sr^{2+}, but also strips Sr^{2+} with DCH18C6 synergistically. According to the above results, one can infer that the affinity of Sr^{2+} with FAA^{-} is larger than that with DCH18C6, and the complexes of Sr(II) associating with FAA^{-} become more soluble in sc-CO_2. Therefore, with FAA^{-} as the modifier, sc-CO_2 can strip strontium from the DCH18C6–C_2mimNTf$_2$ system effectively. With respect to the recovery of Sr(II) from the chelate, two methods could be applied. If the recovery of complexing agents is not necessary, $Sr(NO_3)_2$ aqueous solution could be obtained via the digestion of the stripping product. If the recovery of ligands is obligatory, $SrSO_4$ precipitate could be collected by washing the organic solution of the stripping product with the aqueous solution containing SO_4^{2-}.

The cationic complexes [DCH18C6-Na]$^{+}$ and [DCH18C6-NH_4]

Table 3
The compositions of the fragments and their corresponding complexes of the original IL phase, the recovered IL phase and stripping product

m/z	Cationic complex
The original IL phase	
167.4	$[Sr\text{-}NO_3\text{-}H_2O]^+$
251.1	$[C_2mim\text{-}NTf_2\text{-}C_2mim\text{-}H]^{2+}$
390.3	$[DCH18C6\text{-}NH_4]^+$
411.2	$[DCH18C6\text{-}K]^+$
443.3	$[C_2mim\text{-}NTf_2\text{-}DCH18C6\text{-}Sr\text{-}2H_2O]^{2+}$
502.1	$[C_2mim\text{-}NTf_2\text{-}C_2mim]^+$
740.1	$[DCH18C6\text{-}Sr\text{-}NTf_2]^+$
773.5	$[2C_2mim\text{-}2NTf_2\text{-}2DCH18C6\text{-}H_2O\text{-}2H]^{2+}$
The recovered IL phase	
167.4	$[Sr\text{-}NO_3\text{-}H_2O]^+$
251.1	$[C_2mim\text{-}NTf_2\text{-}C_2mim\text{-}H]^{2+}$
411.2	$[DCH18C6\text{-}K]^+$
502.1	$[C_2mim\text{-}NTf_2\text{-}C_2mim]^+$
773.5	$[2C_2mim\text{-}2NTf_2\text{-}2DCH18C6\text{-}H_2O\text{-}2H]^{2+}$
The stripping product	
167.4	$[Sr\text{-}NO_3\text{-}H_2O]^+$
222.4	$[Sr\text{-}NO_3\text{-}FAA\text{-}Sr]^{2+}$
390.3	$[DCH18C6\text{-}NH_4]^+$
395.2	$[DCH18C6\text{-}Na]^+$
443.3	$[C_2mim\text{-}NTf_2\text{-}DCH18C6\text{-}Sr\text{-}2H_2O]^{2+}$
502.1	$[C_2mim\text{-}NTf_2\text{-}C_2mim]^+$
667.1	$[DCH18C6\text{-}Sr\text{-}FAA]^+$
740.1	$[DCH18C6\text{-}Sr\text{-}NTf_2]^+$
1058.2	$[C_2mim\text{-}NTf_2\text{-}DCH18C6\text{-}FAA\text{-}Sr]^+$

In the ESI spectrum of the recovered IL phase (Fig. 4(b)), the signals of the cationic complexes $[C_2mim\text{-}NTf_2\text{-}C_2mim]^+$ and $[C_2mim\text{-}NTf_2\text{-}C_2mim\text{-}H]^{2+}$ are observed, which are consistent with those in the ESI spectrum of the original IL phase (Fig. 4(a)). And the cationic complexes $[Sr\text{-}NO_3\text{-}H_2O]^+$ exist in the recovered IL phase, but the intensity is very weak. This indicates that Sr(II) has been stripped almost completely, which is in agreement with the results of ICP-AES. Thus, highly efficient stripping of Sr(II) from the DCH18C6–$C_2mimNTf_2$ system was achieved in this work.

Combined with our previous work about the extraction of Sr(II) by DCH18C6–$C_2mimNTf_2$ [1], a complete cycle including extraction and stripping processes for Sr(II) with both IL and sc-CO_2 can be established. The whole process is illustrated in Fig. 5.

Finally, we evaluated the other three fluorinated metal chelates HTTA, HPOD, and HFPOA, respectively, diluted in acetonitrile at the optimized conditions of HFAA, and the stripping efficiencies are 66%, 87% and 91% (Fig. 6). They can also be chosen as modifiers and their stripping efficiencies could be further optimized by OAD.

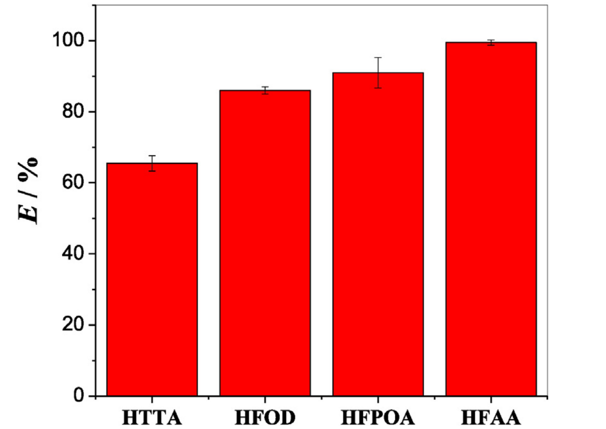

Fig. 6. Effect of different modifiers diluted in acetonitrile on the stripping of Sr(II) from DCH18C6–$C_2mimNTf_2$.

4. Conclusions

The stripping of Sr(II) from the DCH18C6–$C_2mimNTf_2$ system using the modified sc-CO_2 was studied. It was found that HFAA–acetonitrile is an efficient modifier to facilitate the stripping of Sr(II) from the IL phase. By the method of OAD and ANOVA, nearly 100% of Sr(II) could be successfully stripped off under the following conditions: 308 K, 30 MPa, 40 min of dynamic stripping time and 60 mmol·L^{-1} HFAA in acetonitrile.

The present work demonstrates the feasibility of employing sc-CO_2 to strip and promises a highly efficient extraction–stripping cycle for IL-based Sr(II) recovery process.

Nomenclature

C	concentration of HFAA, mmol·L^{-1}
P	pressure, MPa
T	temperature, K
t_d	dynamic extraction time, min
t_s	static extraction time, min

Subscripts

d	dynamic extraction
s	static extraction

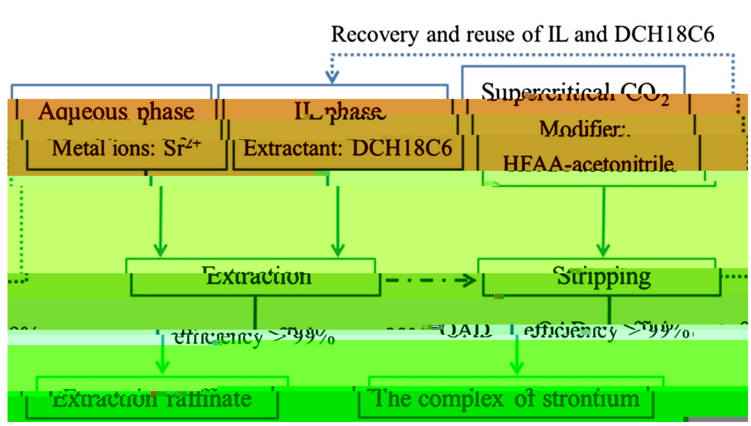

Fig. 5. A diagram showing the extraction–stripping procedure for strontium.

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