

Extraction of Scandium(III) Using Ionic Liquids Functionalized Solvent Impregnated Resins

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ABSTRACT: Ionic liquids (ILs) functionalized solvent impregnated resins (SIRs) were prepared using IL modified Merrifield resin as the polymeric supports by impregnation of extractant for extraction of Sc(III). The ILs modified Merrifield resin were prepared via covalent anchoring of imidazolium salts onto Merrifield resin. The polymeric supports with imidazolium chloride group (RCl) and imidazolium hexafluorophosphate group (RPF₆) were characterized by FTIR, TGA, and elemental analysis. It was found that RCl and RPF₆ had tunable hydrophilicity and hydrophobicity, different acid stability, and swelling behaviors in solvents. The effect of Cyanex 923 extractant or [C₈mim][PF₆] IL impregnated on RCl and RPF₆ were studied. The results showed Cyanex

923 and [C₈mim][PF₆] exhibited stronger affinity to RPF₆ than to RCl. RPF₆ with Cyanex 923 was found to be effective in Sc(III) extraction. The extraction mechanisms of SIRs containing RPF₆ and Cyanex 923 with or without [C₈mim][PF₆] were cation exchange and neutral complexation, respectively. [C₈mim][PF₆] acted as an extraction media and was involved in the cation exchanged extraction reaction. Sc(III) can be selectively separated from Tm(III), Yb(III), and Lu(III) by the SIRs. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 120: 3284–3290, 2011

Key words: solvent impregnated resins; scandium(III); Cyanex 923; polymeric supports; ionic liquid

INTRODUCTION

Scandium presents as a minor element in many minerals,¹ and is generally recovered as a by-product from residues, effluents, and slags of other mineral processes.^{2,3} Scandium is an important element from an industrial point of view, and widely used in astronavigation, electronic, nuclear energy, and metallurgical industries.^{4,5} Therefore, the recovery and purification of Sc(III) have been attracting a lot of attention. Solvent extraction is the most common technique to extract scandium from various resources.^{3,4} However, the use of a large amount of volatile organic compounds in solvent extraction processes are harmful to environment. Moreover, the solvent

extraction processes are often accompanied with the multi-stage cycles, extractant loss and formation of stable emulsions, etc.^{6,7} The application of impregnated resins can overcome these problems. There are two types of impregnated resins. One is solvent impregnated resin (SIR) prepared by adsorption of extractant onto polymeric support,⁸ and the other is Levextrel-type resin prepared by polymerization of styrene and divinylbenzene in the presence of extractant.⁶

SIR has been shown to be a simple and effective tool to selectively separate metal ions from aqueous solution.^{9–11} It is possible to prepare different SIRs tailored for particular ions in solution using various combinations of polymeric supports and extractants.¹² However, some SIRs have relatively low stability because small amount of extractant may leach from the supports during cycles. Most SIRs used styrene-divinylbenzene copolymers as supports for separation of metal ions, e.g., Amberlite XAD series resins.^{9,13} The sorption of the extractant by the polymeric support is generally achieved through hydrophobic interaction and sometimes partly through polar or electrostatic forces.^{14,15} The stability of SIR depends mainly on the support used and the organic reagent retained, when the impregnating

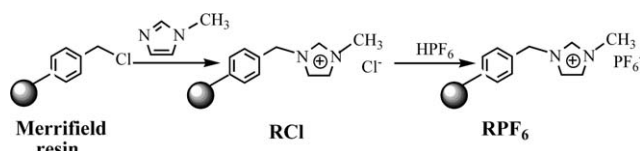
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Scheme 1 Preparation of modified Merrifield resin.

extractant exhibits strong affinity to the support.⁹ To try to enhance the interaction between the extractant and the support, active supports with reactive pyridine groups or ionic polymers were studied.^{16–19} Strong acid-base interaction was established between the metal chelator di(2-ethylhexyl) dithiophosphoric acid and poly(4-vinylpyridine) resin.¹⁸ It was attempted in this study by selection of an ionic polymer support carrying imidazolium salts.

In this study, the ILs modified Merrifield resin were prepared through covalent anchoring of imidazolium salts onto Merrifield resin (Scheme 1). The modified resins with imidazolium chloride group (RCl) and imidazolium hexafluorophosphate group (RPF₆) were studied as polymeric supports by impregnation of extractant for extraction of Sc(III). RCl and RPF₆ were characterized by FTIR, TGA, and elemental analysis. Luis and coworkers reported when Merrifield resin was modified with imidazolium salts (cationic fraction of ILs), the resin essentially maintained the same polarity of bulk ILs.²⁰ Compared with commonly used inert polymeric supports, the ILs modified Merrifield resin with active imidazolium salt groups were ionic polymers. They had tunable surface characteristics, which were benefit for adjusting the affinity of the extractant to the support. The SIRs based on RCl and RPF₆ impregnated with neutral Cyanex 923 extractant or [C₈mim][PF₆] IL were studied in this text. The SIRs were investigated in batch extraction experiment, and the equilibrium time and extraction mechanism were discussed. The selective separation of Sc(III) from other rare earth (RE) elements were also studied.

EXPERIMENTAL

Materials

Merrifield resin (3.5 mmol Cl/g, 1% DVB, 200–400 mesh) was purchased from Tianjin Nankai Hecheng Science and Technology Co., Ltd. (Tianjin, China). The extractant Cyanex 923, a mixture of straight chain alkylated phosphine oxides (Scheme 2) was kindly supplied by CYTEC Canada Inc. and used without further purification. HPF₆ (60%) was purchased from Dongyang Flysun Fluoro Chem. Co., Ltd. (Zhejiang, China). *N*-Methylimidazole was purchased from Aldrich (USA). [C₈mim][PF₆] (Scheme 2) used in this study was synthesized by

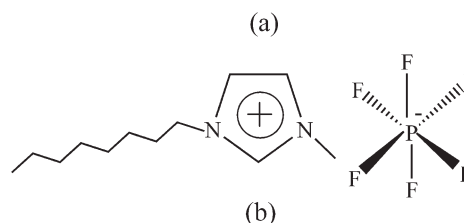
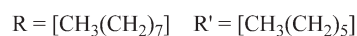
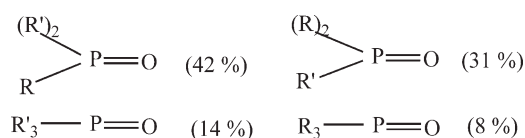
previous method.²¹ The stock solution of Sc(III) was prepared by dissolving 0.15 g of Sc₂O₃ (99.99%) in 1 mL of 95% HNO₃. After removal of excess acid by evaporation, the solution was diluted to 100 mL. The concentration of the stock solution of Sc(III) was determined by titration using Zn standard solution and xylene orange as indicator. The preparation of other REs(III) solution was similar with Sc(III). The other chemicals were analytical grade reagents.

Methods

FTIR analysis was conducted with a Bruker VECTOR 22/N spectrometer (German). Thermogravimetric (TG) and differential thermogravimetric (DTG) data were recorded with Thermal Analysis Instrument (SDTQ600, TA Instruments, America). Measurements were conducted by heating Merrifield resin and RPF₆ from 50 to 1000°C in air at a heating rate of 10°C/min, and RCl from 50 to 800°C. Elementary analysis was performed by VarioEL (German). The concentrations of anions were determined by Ion Chromatography (Dionex ICS-1500, America). UV-visible spectra were measured with a Shimadzu UVmini-1240 spectrophotometer (Japan).

Preparation of modified Merrifield resin

RCl and RPF₆ were synthesized according to the modified method by Wang et al.,²² and the preparing procedure is shown in Scheme 1. A 5.0 g sample of Merrifield resin in 30 mL of DMF was stirred for 2 h. Then, 1.65 g of *N*-methylimidazole was added and the mixture was stirred for no less than 36 h at 80°C. The resin was filtered and washed with ethanol. The resin in 30 mL of ethanol was stirred for another 24 h at 65°C. The resin was filtered and dried in a vacuum to afford RCl. Then it reacted with 4 mL of HPF₆ after 13 mL of deionized water



Scheme 2 The structure of major Cyanex 923 components and composition of the mixture (a), and the structure of [C₈mim][PF₆] (b).

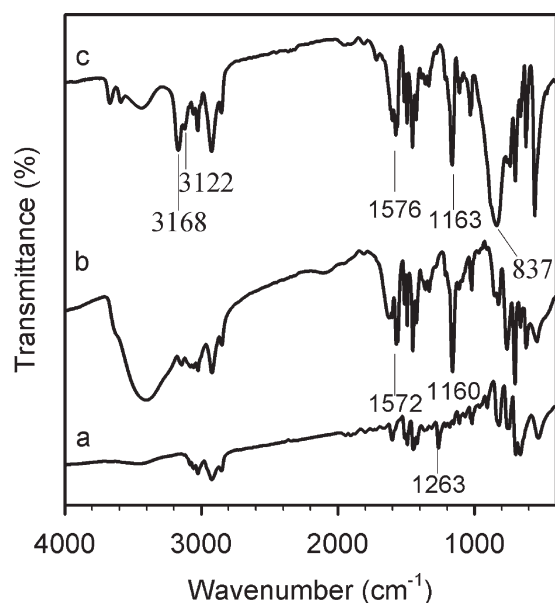


Figure 1 FTIR spectra of (a) Merrifield resin, (b) RCl, and (c) RPF₆.

was added. The synthesized resin was washed neutral with deionized water to afford RPF₆.

Impregnation process

A known amount of Cyanex 923 and/or [C₈mim][PF₆] were dissolved together in EtOH, and RPF₆ prepared above was added in the mixture and stirred. EtOH was removed under reduced pressure, and the resultant materials were dried in a vacuum. The SIR in which only Cyanex 923 was impregnated on RPF₆ was denoted as RPF₆-923, the SIR in which only [C₈mim][PF₆] was impregnated on RPF₆ was denoted as RPF₆-IL, and the SIR in which [C₈mim][PF₆] containing Cyanex 923 was impregnated on RPF₆ was denoted as RPF₆-IL-923. The compared materials of RCl-923 and RCl-IL were prepared according to the above method except that RPF₆ was displaced by RCl.

Extraction procedure

The uptake of Sc(III) was measured by contacting 5 mL of Sc(III) solution (4.046×10^{-4} – 4.616×10^{-4} mol/L) and 200 mg of SIRs under constant stirring for 30 min at 25°C. The aqueous phase was filtered through a 0.45- μ m PTFE syringe filter. The concentration of Sc(III) in aqueous phase was determined by colorimetry with Arsenazo III as color developing agent. 3 mL ClCH₂COOH-NaOH buffer solution (pH 2.8), 2 mL 0.05% Arsenazo III, and different amount of Sc(III) solution were added to volumetric flask and diluted to 25 mL with deionized water. The absorbance of this solution was measured at

677 nm after 15 min. The determination of other REs(III) concentrations was the same as Sc(III) at their corresponding maximum absorption wavelengths. The extraction efficiency (*E*), distribution ratio (*D*), and separation factor (β) were defined as follows²³:

$$E(\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

$$D = \frac{C_0 - C_e}{C_e} \frac{V_a}{M} \quad (2)$$

$$\beta = \frac{D_1}{D_2} \quad (3)$$

where *C*₀ and *C*_{*e*} represent the initial and equilibrium concentration (mol/L) of REs(III) in aqueous phase, respectively, *V*_{*a*} represents the volume of aqueous phase, *M* is the mass of SIR.

RESULTS AND DISCUSSION

Characterization and properties of RCl and RPF₆

Confirmation of the structure of RCl and RPF₆

RCl and RPF₆ were synthesized through a simple and swift method. The structure of RCl has been confirmed.²⁴ The FTIR spectra of Merrifield resin, RCl, and RPF₆ were determined to ensure the structure of RPF₆ (Fig. 1). The characteristic band at 1263 cm⁻¹ —CH₂— bending vibration of —CH₂Cl was found in Merrifield resin but not found in RCl and RPF₆. The bands at 3168 cm⁻¹ and 3122 cm⁻¹ were C—H stretching vibration of imidazolium ring. The bands at 1576 and 1163 cm⁻¹ for RPF₆ and 1572 and 1160 cm⁻¹ for RCl were framework vibration and C—H in-plane bending vibration of imidazolium ring. The band at 837 cm⁻¹ was distortion vibration of P—F of PF₆⁻, which implied Cl⁻ exchanged with PF₆⁻. According to elemental analysis of nitrogen, 7.22 wt % for RCl and 5.66 wt % for RPF₆, the yield of RPF₆ was no less than 97%. The composition was calculated to be 2.02 mmol mimPF₆ per gram RPF₆.

Thermogravimetric analysis

Merrifield resin, RCl, and RPF₆ decomposed in two, four, and three steps, respectively, (Fig. 2). Table I shows the degradation stages and the weight loss for the above-mentioned materials. RPF₆ began to decompose at 304°C, and lost almost all of their masses at about 787°C. RPF₆ was thermally more stable than Merrifield resin which began to decompose at 270°C. RCl contained about 6.5% physically adsorbed water (weight loss before 114°C) and began to decompose at 220°C. The order of thermal stability was RPF₆ > Merrifield resin > RCl.

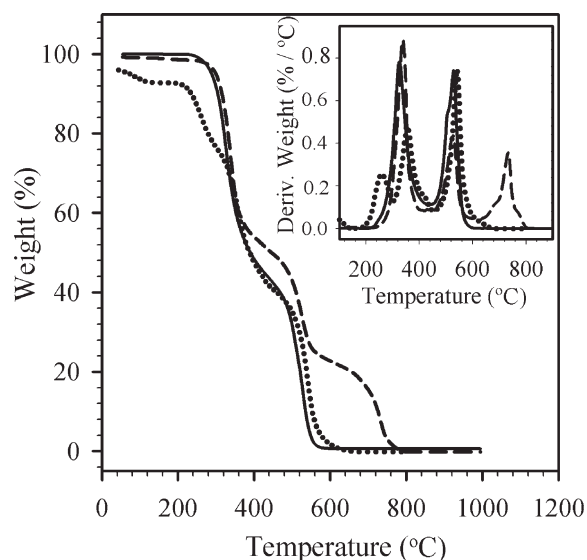


Figure 2 TG and DTG curves of Merrifield resin (—), RCl (· · ·), and RPF₆ (---).

Swelling properties

The swelling volumes were measured in a 5-mL syringe with a thin, fine polypropylene filter using 500 mg resins and 5 mL solvent after vortex 30 s and equilibrating for 1 h at 25°C.²⁵ The swelling extents of RCl, RPF₆, and Merrifield resin were different in some solvents, as shown in Table II. All the three dry microporous materials could be swollen in polar aprotic solvents (e.g., DMF and ethyl acetate). RCl was found to have good swelling property in more polar protic solvents, such as EtOH, H₂O, and CH₃OH. However, RPF₆ cannot be swollen in these solvents. The hydrophilicity of RCl was much higher than RPF₆ because RCl can be swollen in H₂O.

Chemical properties of RCl and RPF₆

As reported previously, some properties such as hydrophilicity/hydrophobicity and thermal stability of RCl and RPF₆ were different. The chemical properties of RCl and RPF₆ were also studied. RCl (200.8 mg) was mixed with nitric acid (5 mL, pH 4.3) under constant agitation for 10 min. The aqueous phase

TABLE II
Volumes of the Swollen Merrifield Resin, RCl, and RPF₆ (mL/g)^a

Solvent	Merrifield resin	RCl	RPF ₆
EtOH	\ ^b	4.8	\
H ₂ O	\	3.7	\
CH ₃ OH	\	6.1	\
Ethyl acetate	4.1	2.4	3.2
DMF	4.6	3.2	6.1

^a The dry volumes of Merrifield resin, RCl, and RPF₆ were 1.6, 1.7, 1.2 mL/g, respectively.

^b “\” meant no swelling property was found in the solvent.

was detected by Ion Chromatography. Few NO₃⁻ (4.70 mg/L) and a large amount of Cl⁻ (46.59 mg/L) were found in the aqueous solution, which indicated that RCl reacted with dilute nitric acid (pH 4.3). However, RPF₆ hardly reacted with acid under the experimental condition. 202.7 mg of RPF₆ and 5 mL of nitric acid (1 mol/L) were mechanically shaken for 30 min. 28.84 mg/L F⁻ and small unreacted Cl⁻ were detected in aqueous phase by ion chromatography. All F⁻ of RPF₆ was about 46.5 mg, and only 0.31% was detected in aqueous phase. RPF₆ was chemically stable and hardly reacted with dilute acid solution compared with RCl.

The effect of Cyanex 923 impregnated on RCl and RPF₆

RCl-923 (Cyanex 923: 1.7 wt %) was prepared to study the effect of RCl as polymeric support of SIRs with Cyanex 923 extractant. 200 mg of RCl-923 was mixed with 5 mL of Sc(III) (4.046 × 10⁻⁴ mol/L) under constant agitation for 30 min, and the extraction efficiency of Sc(III) was only 5.5%. Namely, Cyanex 923 cannot be immobilized on RCl stably. When RCl-923 was contacted with the aqueous solution, most Cyanex 923 was removed from RCl and the extraction hardly proceeded on the resin phase. RCl was unable to adsorb Cyanex 923 because the polar imidazolium chloride sites were too hydrophilic to be compatible with the hydrophobic Cyanex 923. Therefore, RCl-923 impregnated resin

TABLE I
Thermogravimetric Data for Merrifield Resin, RCl, and RPF₆

Sample	Weight loss [W (%)] and temperature range [ΔT (°C)]		
	Stage I	Stage II	Stage III
Merrifield resin	ΔT _I = 270–501 W _I = 49.3%	ΔT _{II} = 501–575 W _{II} = 46.2%	
RCl	ΔT _I = 220–315 W _I = 17.2%	ΔT _{II} = 315–503 W _{II} = 37.4%	ΔT _{III} = 503–568 W _{III} = 36.6%
RPF ₆	ΔT _I = 304–499 W _I = 50.8%	ΔT _{II} = 499–687 W _{II} = 29.0%	ΔT _{III} = 687–787 W _{III} = 14.8%

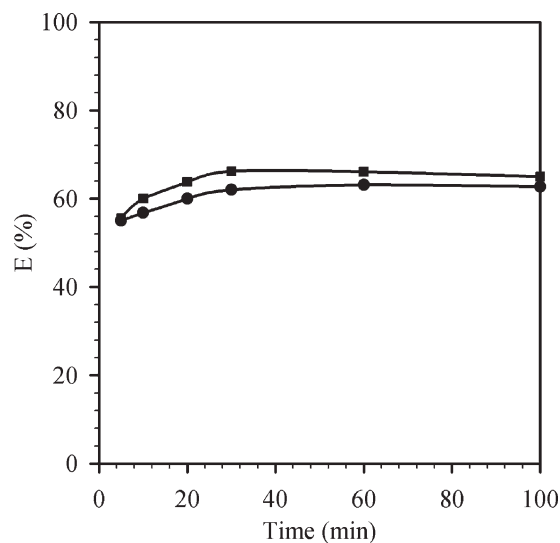


Figure 3 Effect of equilibration time of aqueous solution of Sc(III) with RPF₆-923 (square) and RPF₆-IL-923 (circle). (Initial concentration of Sc(III), 4.616×10^{-4} mol/L; resin amount, 200 mg; volume of Sc(III) solution, 5 mL; pH, 4.3)

was very unstable and RCl was not suitable to be used as polymeric support of SIRs.

RPF₆-923 with 2.0 wt % Cyanex 923 was mixed with the aqueous solution of Sc(III) under constant agitation for different time from 5 to 100 min. The results obtained are presented in Figure 3. The extraction rate was fast, and 30 min was needed to reach equilibrium for RPF₆-923. The extraction efficiency of Sc(III) was 66.6% when the contact time was 30 min. RPF₆-923 could effectively extract Sc(III) from aqueous solution, which meant Cyanex 923 exhibited relatively stronger affinity to RPF₆ than to RCl. The interactions between Cyanex 923 and RPF₆ were nonpolar interaction due in part to the nonpolar groups and electrostatic interaction. Therefore, RPF₆ was more stable than RCl and suitable to be used as polymeric support of SIRs.

RPF₆-923 with different content of Cyanex 923 (0–10.0 wt %) were prepared. As shown in Figure 4, the extraction efficiency of Sc(III) increased with increasing content of extractant. The extraction efficiency could attain 98.0% with the extractant content of 10.0 wt %. Much more Cyanex 923 could be stably immobilized on RPF₆ due to the strong affinity between them. However, under the same condition, RPF₆ could only extract 7.5% of Sc(III) when 200 mg of RPF₆ was mixed with 5 mL of Sc(III) solution under constant agitation for 30 min.

The effect of [C₈mim][PF₆] IL impregnated on RCl and RPF₆

RCl and RPF₆ had IL fractions of methylimidazolium chloride or methylimidazolium hexafluorophosphate.

Perhaps there would be some interactions between the polymeric supports with IL fractions and IL (e.g., [C₈mim][PF₆]). RCl-IL (IL: 9.6 wt %) and RPF₆-IL (IL: 20.3 wt %) were prepared to identify the possibility. 200 mg of RCl-IL was mixed with 5 mL of deionized water under constant agitation for 30 min, and 90% IL losses of attached IL were determined in aqueous phase by UV adsorption at 211 nm. Hydrophobic [C₈mim][PF₆] could not be immobilized on hydrophilic RCl either. When 200 mg of RPF₆-IL was mixed with 5 mL of deionized water under constant agitation for 30 min, the IL lost in the aqueous phase was only 7.7% of its attached IL. IL was relatively stably immobilized on RPF₆ compared with 90% IL losses from RCl. The interactions between RPF₆ and [C₈mim][PF₆] were mainly electrostatic force between the ions of [C₈mim][PF₆] and the mimPF₆ ionic groups of RPF₆, polar force due to the functional surface on RPF₆ essentially maintained the same polarity of bulk [C₈mim][PF₆]²⁰ and hydrophobic interaction. Unavoidably, a small quantity of IL lost into the aqueous phase when mechanical shaking because the external layer of IL had weak interaction with RPF₆ and the solubility losses. Therefore, RPF₆ could be used as polymeric support of SIRs to immobilize the extractant and solvent such as Cyanex 923 and [C₈mim][PF₆].

Extraction mechanism of RPF₆-923 and RPF₆-IL-923

IL was usually used as alternative extraction media, and some extractant necessitated to be added in IL when separating metal ions.²⁶ Our group studied the solvent (IL) impregnated resin for solid-liquid

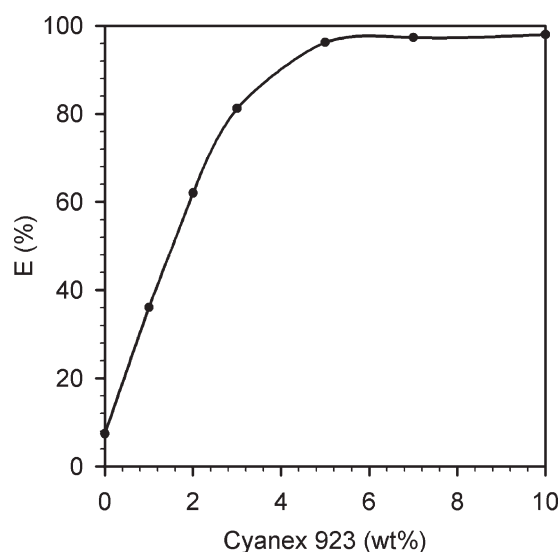


Figure 4 Effect of Cyanex 923 content of RPF₆-923 on the extraction of Sc(III). (Initial concentration of Sc(III), 4.616×10^{-4} mol/L; resin amount, 200 mg; volume of Sc(III) solution, 5 mL; pH, 4.3; equilibration time, 30 min)

TABLE III
Sc(III) and NO₃⁻ Partitioning Between
Aqueous Phase and SIRs^a

SIRs	%E _{Sc} ^b	%E _{NO₃⁻} ^c	Mechanism
RPF ₆ -923	66.18	68.16	extraction of neutral complex
RPF ₆ -IL-923	63.06	0.70	cation exchange

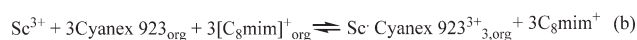
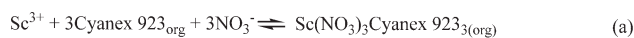
^a 200 mg of SIRs and Sc(III) solution (5 mL, 4.616 × 10⁻⁴ mol/L) were shaken for 60 min.

^b Determined by colorimetry.

^c Determined by Ion Chromatography. Each experiment was done duplicated, and the results were given as averages.

extraction of RE, in which [C₈mim][PF₆] containing Cyanex 923 were immobilized on XAD-7.²³ We also tried to study the effect of RPF₆ as support of solvent ([C₈mim][PF₆]) impregnated resin. RPF₆-923 (Cyanex 923: 2.0 wt %) and RPF₆-IL-923 (Cyanex 923: 2.0 wt %, [C₈mim][PF₆]: 20.3 wt %) were prepared, and the extraction behaviors were studied. Under the same condition, the extraction efficiency of Sc(III) for RPF₆-923 and RPF₆-IL-923 was similar, as shown in Figure 3.

The attached IL of RPF₆-IL-923 was not helpful for the extraction efficiency, but it changed the extraction mechanism. For RPF₆-923, the amount of NO₃⁻ extracted was almost equal to the amount of extracted Sc³⁺, as shown in Table III. The extraction mechanism was extraction of neutral complex [Scheme 3(a)], which was consistent with the extraction in conventional organic solvent.² As we know, electroneutrality was required to be maintained when a metal ion was extracted from an aqueous phase into an organic solvent.²⁷ For neutral extractant Cyanex 923, this was achieved by coextraction of anions into the organic solvent. In the extraction of Sc³⁺ from nitrate media by Cyanex 923 onto RPF₆-923, the neutral complex Sc(NO₃)₃Cyanex923_{3(org)} was formed. However, for RPF₆-IL-923, the amount of NO₃⁻ extracted was far less than the amount of extracted Sc³⁺ (Table III), and was vastly insufficient to produce a neutral complex. During the extraction, [C₈mim]⁺ ion was detected in the aqueous solution. The extraction mechanism was mainly cation exchange [Scheme 3(b)], consistent with the extraction in bulk IL. In that report, the cation exchange mechanism was proposed in the extraction of Sc(NO₃)₃ from aqueous solution into [C₈mim][PF₆] system by Cyanex 925.²¹ For RPF₆-IL-923, the attached IL on RPF₆ acted as an extraction media, in which Cyanex 923 and Sc³⁺ formed a complex, Sc(Cyanex



Scheme 3 The extraction mechanisms of (a) extraction of neutral complex and (b) cation exchange.

TABLE IV
The Extraction Efficiency and Separation Factors of
REs(III) by RPF₆-923^a

Extraction efficiency (%)				Separation factor		
E _{Sc}	E _{Tm}	E _{Yb}	E _{Lu}	β _{Sc/Tm}	β _{Sc/Yb}	β _{Sc/Lu}
66.9	30.9	27.4	31.1	4.50	5.36	4.46

^a The initial concentrations of Sc(III), Tm(III), Yb(III), and Lu(III) were 4.616 × 10⁻⁴, 4.600 × 10⁻⁴, 4.640 × 10⁻⁴, and 4.630 × 10⁻⁴, respectively. The initial pH of Sc(III), Tm(III), Yb(III), and Lu(III) were all 4.3.

923)₃⁺. The complex exchanged with [C₈mim]⁺ of the attached IL, and [C₈mim]⁺ was exchanged into the aqueous phase. The extraction process proceeded through the IL layer attached on the support.

Separation of Sc(III) from other REs(III)

The separation of Sc(III) from Tm(III), Yb(III), and Lu(III) using RPF₆-923 was studied in individual RE(III) systems. A 200 mg sample of RPF₆-923 (Cyanex 923: 2.0 wt %) was contacted with 5 mL of Sc(III), Tm(III), Yb(III) or Lu(III) solution and shaken for 30 min. The extraction efficiency and selectivity of RPF₆-923 are shown in Table IV. RPF₆-923 had higher extraction efficiency for Sc(III) but lower extraction efficiency for Tm(III), Yb(III), and Lu(III). The separation factors of Sc/Tm, Sc/Yb, and Sc/Lu were 4.50, 5.36, and 4.46, respectively. The β values were so high that Sc can be selectively extracted from the Sc/Tm/Yb/Lu mixtures.

CONCLUSION

RCl and RPF₆ were prepared via covalent anchoring of imidazolium salts onto Merrifield resin. The properties of the polymeric supports were tunable through varying the anion of imidazolium salts. RCl can be swollen in protic solvents such as EtOH, H₂O and CH₃OH, and was more hydrophilic than RPF₆. The thermal and acid stability of RPF₆ was higher than RCl. RPF₆ could effectively immobilize Cyanex 923 and [C₈mim][PF₆], and was suitable to be used as polymeric support of SIRs. However, RCl could not be used as polymeric support of SIRs. Both RPF₆-923 and RPF₆-IL-923 could effectively extract Sc(III) from aqueous solution. The attached IL on RPF₆ acted as an extraction media and was involved in the cation exchanged extraction reaction. The extraction mechanisms of Sc(III) by RPF₆-923 and RPF₆-IL-923 were extraction of neutral complex and cation exchange, respectively. Sc(III) can be selectively separated from Tm(III), Yb(III) and Lu(III) by RPF₆-923.

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