Extraction of Uranium from Aqueous Solution of Nitric Acid and Organic Solvent Using Ionic Liquid

Pradeep Kumar[1,*] and Ashok Khanna[2]

[1]Department of Chemical Engineering, Institute of Engineering and Technology, Lucknow-226021, India
[2]Department of Chemical Engineering, Indian Institute of Technology Kanpur, Kanpur-208016, India

*Corresponding author: E-mail: pradeepkumar@ietlucknow.ac.in

In present study, tri-*n*-butylphosphate (TBP), a classical complexing agent for metallic cations, has been studied for the extraction of uranium into ionic liquids (ILs): 1-butyl-3-methylimidazolium-*bis*(tri-fluoromethylsulfonyl)imide ([BMIM][TF2N]) and trihexyl-tetradecylphosphonium-*bis*(tri-fluoromethylsulfonyl)imide ([P$_{(14)666}$][TF2N]). Increasing HNO$_3$ acidity of aqueous solution from 0.01 to 1 M the distribution ratio, D$_U$ decreases from 16 to 1.2 for 1.1 M TBP in [BMIM][TF2N] and the corresponding extraction efficiency (% E) varies from ~94 to 55. In the acidic range of 1 to 8 M D$_U$ and % E shows reversal trend of giving a local maximum at 8 M. This behaviour is compared and validates by literature. In contrast, on increasing in aqueous acidity from 0.01 to 8 M the extraction of uranium into [P$_{(14)666}$][TF2N] ionic liquid, D$_U$ enhances from 2 to 39 and (%E) goes up from ~ 51 to 95. Since, [P$_{(14)666}$][TF2N] works better than [BMIM][TF2N] in the acidic range > 0.1. This ionic liquid has been used for selective separation of uranium from strontium giving a D$_U$ of 63 and D$_{Sr}$ of 2 with 98% E of uranium at 8 M acidity. To confirm the various species and groups in the formed complex, FTIR studies have been conducted.

Keywords: Distribution ratio, Extraction efficiency, Ionic liquid, Tributyl phosphate, Solvent extraction.

INTRODUCTION

Separation of uranium ion from the aqueous solution is a major challenge in chemical, metallurgical and nuclear process industries. Solvent extraction has been a favoured choice [1]. Conventional solvent extraction process uses organic solvents such as octanol, hexane, *n*-dodecane which are not environment friendly because of their toxicity, flammability and volatility [2,3]. Extraction of uranium and plutonium from spent nuclear fuel which contains 95.6% uranium, 0.9% plutonium, minor actinides 0.1%, stable short lived fission products 3%, major fission products cesium and strontium 0.3% and long-lived fission products iodine and technetium 0.1% is conventionally carried out by the PUREX process [3] requiring the dissolution of spent fuel in aqueous nitric acid using 30% solution of tri-*n*-butyl phosphate (TBP) in *n*-dodecane [4] or in kerosene [5]. Similarly, in SREX process, strontium extraction is achieved by using crown ether as an extractant which could either be dicyclohexyl-18-crow*n*-6 (DCH18C6) or 4',4',(5')-di-(*t*-butyl-dicyclo-hexano)-18-crow*n*-6 in 1-octanol [5,6]. Though, the processes are well established but they generate large volumes of liquid radioactive waste; also volatility and ammability being a risk to the process. With growing interest in sustainable, "green" chemistry ionic liquids (ILs) has potential to replace these volatile organic solvents [7]. Ionic liquids are organic salts that are liquid in their pure state near ambient conditions; they are regarded as potentially environmentally benign solvents [8] due to their low melting point, non-flammability, negligible vapour pressure, high polarity, thermal and radiation stability [9,10]. Several solvent extraction studies of U(VI) using various ionic liquids [11-16] are available in literature. Imidazolium based ionic liquids have been used for most of the U(VI) extraction with TBP or TTA or CMPO as extractant [13,17-19]. Solvent extraction of uranium from a acidic medium into an imidazolium based ionic liquids gave higher extraction efficiencies in comparison to organic solvents such as *n*-dodecane [20]. Visser and Rogers [21] reported that extraction of Pu^{4+}, Th^{4+} or UO$_2^{2+}$ into 1-butyl-3-methylimidazolium hexafluoro-phosphate ([C4MIM][PF$_6$]) ionic liquids by a mixture of octyl(phenyl)-*N,N*-diisobutylcarbamoylmethylphosphine oxide (CMPO) and tri-*n*-butylphosphate (TBP) is at least an order of magnitude higher than that obtained for the same

extractant concentrations in dodecane. The extraction process with ionic liquids depends on several factors such as hydrophobic verses hydrophilic character of ionic liquid, nature and concentration of extractant, pH or the aqueous acidity [22-25]. Bell and Ikeda [26] reported that cationic exchange predominates at low acidity whereas at high acidity anionic exchange takes over. Additionally, as reported by Dietz and Stepinski [27] for long chain alkyl groups on imidazolium, distribution ratio is lower than short chains. Extraction of U(VI) at higher acidity with ionic liquids has not been exploited. This study investigates the influence of nitric acid and TBP extractant concentration alongwith effect of cation on distribution ratio for [BMIM][TF2N] and [P$_{(14)666}$]-[TF2N] ionic liquids. To confirm the various species and groups in the U-TBP complex: FTIR studies have been conducted on [P$_{(14)666}$][TF2N], 50% TBP in [P$_{(14)666}$][TF2N] and $UO_2(NO_3)_2 \cdot 6H_2O$-TBP in [P$_{(14)666}$][TF2N] system. Quantitative analysis of TBP-uranyl nitrate complex in ionic liquids in the regions of P=O and U=O stretching vibrations is discussed in agreement with a previous studies. Also, the selective extraction of uranium over strontium has been studied.

EXPERIMENTAL

All experiments were performed at room temperature. All samples were prepared with ultrapure water. Strontium nitrate was obtained from Laboratory Reagents & Fine Chemicals (LOBA Chemie Pvt Ltd. Mumbai, India). Tributyl Phosphate (99%), 1-Butyl-3-methyl-imidazolium-*bis*-(trifluoromethyl-sulfonyl)imide ([BMIM][TF2N])(98%) [P$_{(14)666}$][TF2N] (95%) and Arsenazo III were all obtained from Sigma-Aldrich Chemie GmbH (Riedstr.2, Germany) and were used as received. The $UO_2(NO_3)_2 \cdot 6H_2O$ was obtained from Nuclear Fuel Complex, Hyderabad, India. Sulphamic acid and nitric acid (65%) was obtained from (Merck, India).

Extraction experiments: All extraction studies were carried out at room temperature. Extraction experiments involve 2:1 volume ratio of aqueous to organic phase. An aqueous phase consists acidic solution of nitric acid (0.01 to 8 M) containing uranium (0.005 M) and organic phase contains TBP in Ionic liquids ([P$_{(14)666}$][TF2N] and [BMIM][TF2N]). Organic and aqueous phases were equilibrated by magnetic stirring of the mixture at 2500 rpm for 0.5 h, followed by centrifugation for 0.5 h at 2000 rpm to achieve phase separation. The top layer contained the aqueous solution and the bottom layer contained the organic ionic liquid phase. For uranium detection in a sample an aliquot from the aqueous phase after extraction and an aliquot from the starting uranium nitrate solution were taken for the UV spectrophotometric analysis. The distribution ratio D_U for uranium and D_{Sr} for strontium and the percent extraction (% E) into the ionic liquid phase was determined using the following formula:

$$D_{Sr} \text{ or } D_U = \left(\frac{C_i - C_f}{C_f} \right) \times \frac{V_{aq}}{V_{org}} \tag{1}$$

$$\%E_{Sr} \text{ or } \%E_U = \left(\frac{C_i - C_f}{C_i} \right) \times 100 \tag{2}$$

where, C_i and C_f represent the initial (before extraction) and the final concentration (after extraction) in the aqueous phase. V_{aq} and V_{org} indicate the volume taken for extraction *i.e.* aqueous phase and organic (ionic liquid) phase volume.

Spectrophotometric determination: Hitachi UV-2900 spectrophotometer was used for absorbance measurements. Uranium amount in a sample was determined spectrophotometrically by using arsenazo III metal indiacator. To prepare 0.1% Arsenazo III solution, 0.1 g of Arsenazo III and 0.25 g of sodium acetate were dissolved into 100 mL of double distilled water [28]. The solution was kept in an amber bottle. For uranium detection in a sample 1 mL of sample containing uranium were aliquotted in a flask followed by the addition of 2 mL of 1.5 M sulphamic acid and 2 mL of arsenazo III (0.1%). The volume was then made up to 10 mL using 4 M HNO_3. Uranium concentration was calculated from calibration graph between uranium amounts (µg/mL) *versus* absorbance. The absorbance of uranium was observed at a wavelength of 655 nm. The method is found to be quite suitable for the direct estimation of uranium (1-15 µg/mL). For strontium detection an aliquot from the aqueous phase after extraction and an aliquot from the starting strontium nitrate solution were taken for the atomic absorbance spectroscopy (AAS) analysis.

RESULTS AND DISCUSSION

Effect of various parameters such as influence of acidity, influence of extractant concentration on the distribution ratio and extraction efficiency of U(VI) and comparison of distribution ratio D_U of [P(14)666][TF2N] to dodecane is discussed in the following section.

Influence of acidity on distribution ratio D_U: Extraction of heavy metal from acidic medium is important from a practical point of view. Wastewater from the nuclear power plants contains heavy metals and has high acidity. Acidity affects the extraction process. The extraction of U(VI) by [BMIM][TF2N] and [P$_{(14)666}$][TF2N] ionic liquids was studied as a function of nitric acid concentration. From Fig. 1, it has been observed that the D_U increases from ~ 2 to ~39 in the case of [P$_{(14)666}$][TF2N], when the concentration of nitric acid was varied from 0.01 to 8 M.

In the range of high acidity of interest to PUREX process, 0.1 to 3 M of the uranium extraction by the [P$_{(14)666}$][TF2N], is significant (~ 18 at 3 M and ~39 at 8 M). In case of ionic liquid [BMIM][TF2N] the D_U decreases from ~ 16 to 2 with increase in nitric acid strength from 0.01 to 1 M and then increases from ~ 2 to ~13 for further increment in nitric acid concentration. In [BMIM][TF2N] nitric acid is easily dissolved [29] and transferred to organic phase (ionic liquid phase) during phase equilibration. The U shape as depicted in Fig. 1 for [BMIM]-[TF2N] is because of a change of extraction mechanism: (1) at low aqueous acidity cation exchange prevails with the formation of neutral complex $[UO_2(NO_3)_2.2TBP]$ [27] and (2) *via* anion exchange at high acidities forming $UO_2(NO_3)_3(TBP)_n]^-$ and Tf2N$^-$ [30]. On comparing the two ionic liquids, it is observed from Fig. 1 that the effect of cation is more prominent on metal ion than that of anion. On increasing the chain length from butyl ([BMIM][TF2N]) to ([P$_{(14)666}$][TF2N]) in ionic liquid a significant change has been observed in distribution
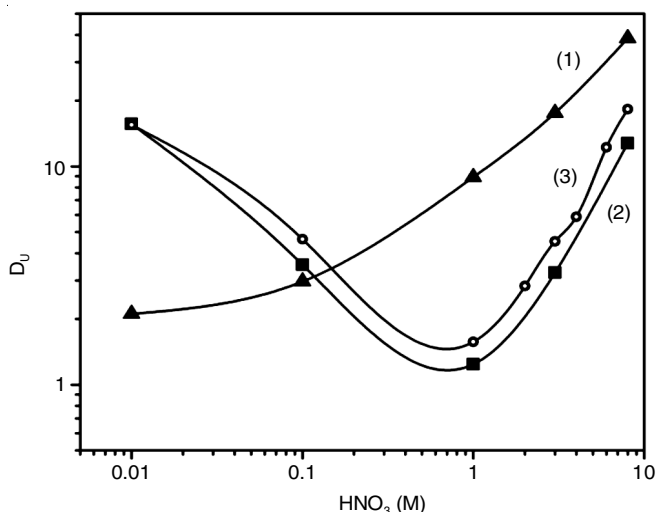
Fig. 1. Dependency of D_U on acidity at 1.1 M TBP in: (1) [P$_{(14)666}$][TF2N] (2) in [BMIM][TF2N] and (3) in [BMIM][TF2N] Billard *et al.* [30]

ratio D_U. The significant change in D_U can be attributed to the phosphonium group attached to the cation that is why for high acidity beyond 0.1 M [P$_{(14)666}$][TF2N] is better than [BMIM][TF2N].

Comparison of D_U on varying [TBP] at low and high acidity: In most of the studies on uranium extraction with ionic liquids [13,26], 30% TBP has been used. To see the effect of TBP in [P$_{(14)666}$][TF2N] on D_U, the TBP concentration varied from 10 (wt%) to 50 (wt%) in ionic liquid for the extraction experiments. It has been observed that acidity is important factors for the extraction process so two ranges of acidity have been selected for further study. The two ranges for aqueous solution are having low acidity that is 0.01 M and high acidity that is 8 M is chosen for the experiments. It has been observed in Fig. 2 that the distribution ratio D_U increases with increase in TBP concentration in ionic liquid for 8.0 M acidity and very less nearly constant for 0.01 M acidity.
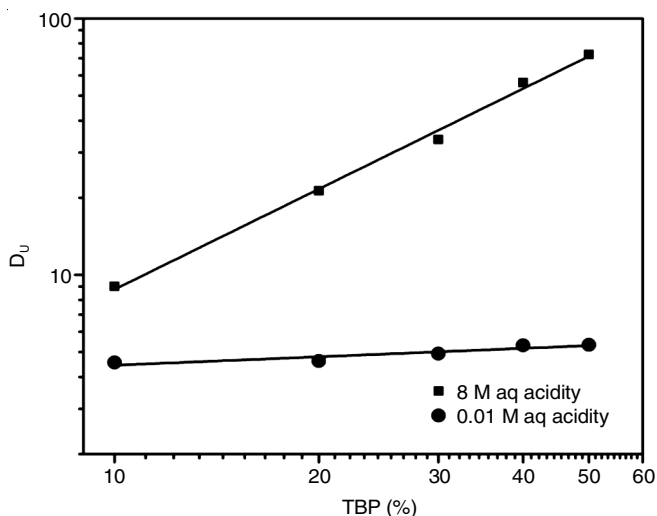

Fig. 2. Dependency of D_U, on [TBP] in [P$_{(14)666}$][TF2N]

Comparison of D_U with different [TBP] in ionic liquid to pure ionic liquid: As observed from Fig. 3 that addition of
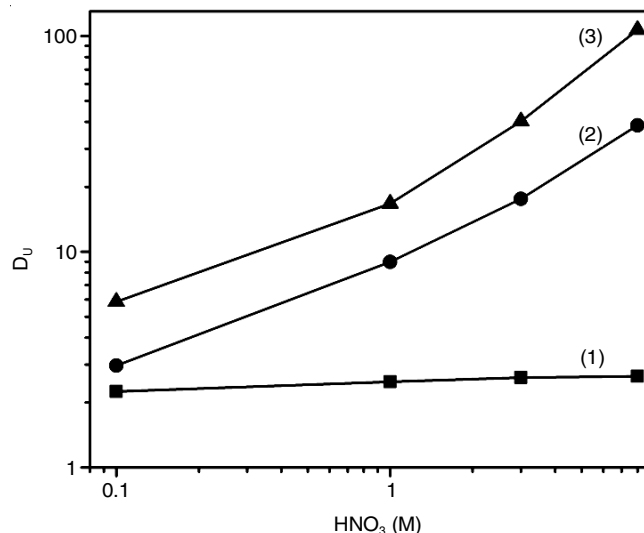

Fig. 3. Dependency of D_U, on acidity in ionic liquid ([P$_{(14)666}$][TF2N]) at different [TBP]: (1) pure ionic liquid (2) 30% TBP (3) 50% TBP

TBP to [P$_{(14)666}$][TF2N] improved the distribution ratio. From pure ionic liquid to addition of 50 % TBP improved the distribution ratio from nearly 3 times to 40 times at low and high acidity, respectively.

Similarly from Fig. 4, on comparing both the ionic liquids insignificant extraction of uranium was observed for pure [BMIM][TF2N] whereas little extraction has been observed for pure [P$_{(14)666}$][TF2N].
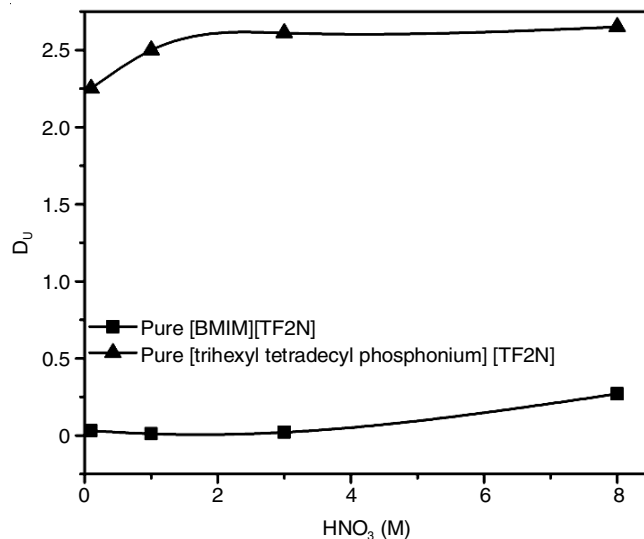

Fig. 4. D_U comparison of pure ionic liquid without [TBP] with varying acidity

Comparison of distribution ratio D_U of [P$_{(14)666}$][TF2N] to dodecane: Enhancement of distribution ratio is observed as shown in Fig. 5 on comparing [P$_{(14)666}$][TF2N] to dodecane. Increasing TBP concentration in [P$_{(14)666}$][TF2N] with varying HNO$_3$ increases D_U as discussed earlier. Distribution ratio of dodecane increases from 0.2 to 18 with varying acidity. On comparing the enhancement of distribution ratio; ~ 6 times at low acidity that is 0.1 M HNO$_3$ acidity and ~ 32 times at high acidity at 8 M HNO$_3$.
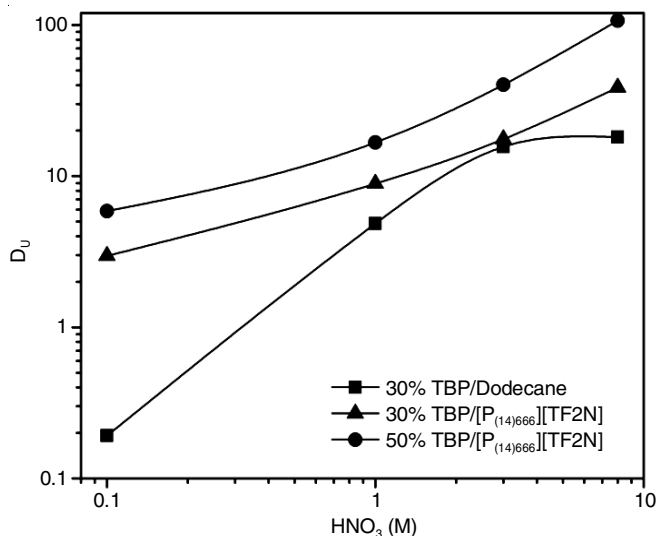
Fig. 5. Comparison of distribution ratio D_U of ionic liquid ($[P_{(14)666}][TF2N]$) with varying acidity to dodecane

Fig. 6. FTIR of: (1) $[P_{(14)666}][TF2N]$ (2) TBP/$[P_{(14)666}][TF2N]$ (3) TBP-U/ $[P_{(14)666}][TF2N]$

Fourier transform infrared spectroscopy (FT-IR) analysis: FTIR analysis of the ionic liquid [trihexyl tetradecyl phosphonium] [TF2N], the loaded organic phase TBP/ionic liquid and loaded $UO_2(NO_3)_2.6H_2O$-TBP/ionic liquid were carried out in order to identify the species present and their corresponding infrared bands with TBP and TBP-U was shown in Figs. 6 and 7. The infrared spectrum (1) of Fig. 6 represents $[P_{(14)666}][TF2N]$ shows several bands at 1469, 1142, 1029 and 738 cm^{-1} attributes to the P-C vibrations. The C-H stretching region show contributions between 2800 and 3000 cm^{-1}; C-H stretching of CH$_3$ at 2963 cm^{-1}, aliphatic C-H stretching at 2932 cm^{-1}, C-H stretching at 2859 cm^{-1}. CF$_3$ stretching frequency is at 1192 cm^{-1}, C=S stretching frequency at 1351 cm^{-1} and –SO$_2$–N– stretching frequency is at 1056 cm^{-1}. The IR bands assignments are consistent with those in the literature [32,33]. The infrared spectrum (2) (Fig. 6) represents the loaded organic phase that is 50% TBP in $[P_{(14)666}][TF2N]$ ionic liquid the phosphoryl region involving stretching vibration of P=O is at 1273 cm^{-1} it represents the TBP cordination with HNO$_3$ and represents the formation of the TBP-HNO$_3$ complex, the P-O-C vibration is at 1030 cm^{-1} the results are consistent with the literature [34,35].

The infrared spectrum (3) of Figs. 6 and 7 represents the loaded organic phase $UO_2(NO_3)_2·6H_2O$-TBP in $[P_{(14)666}][TF2N]$ the U=O stretching frequency of uranyl ions is observed at 945 cm^{-1} because of the complexation the P=O vibration band of uranium TBP complex is shifted to1192 cm^{-1} from 1273 cm^{-1} the shift is consistent with the results observed for the uranyl complexes with TBP [35,36].

Acidity effect on distribution ratio for selective extraction of U and Sr: The selective extraction of uranium and strontium from the mixture of both has been carried out with 50% TBP in $[P_{(14)666}][TF2N]$. The composition of simulated mixture that is the mixture of uranium and strontium has been taken as 99.7 % uranium (0.05 M) and 0.3 % strontium (0.0005 M) from $UO_2(NO_3)_2·6H_2O$ and $Sr(NO_3)_2$ salt respectively. The influence of acidity on D_U and D_{Sr} is shown in Figs. 8 and 9 it
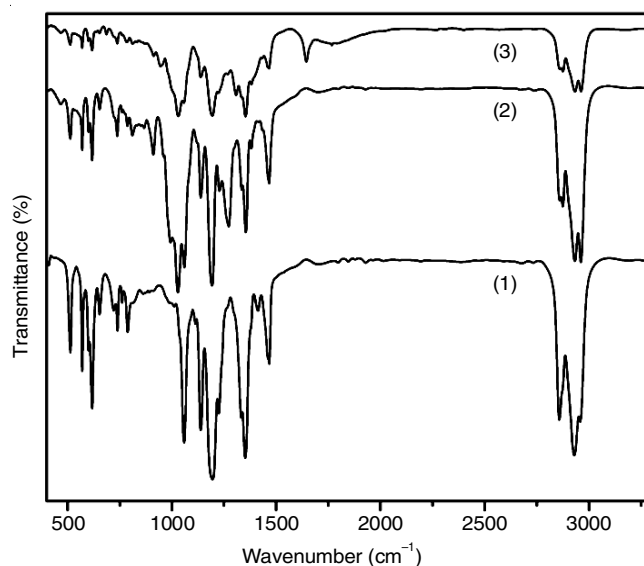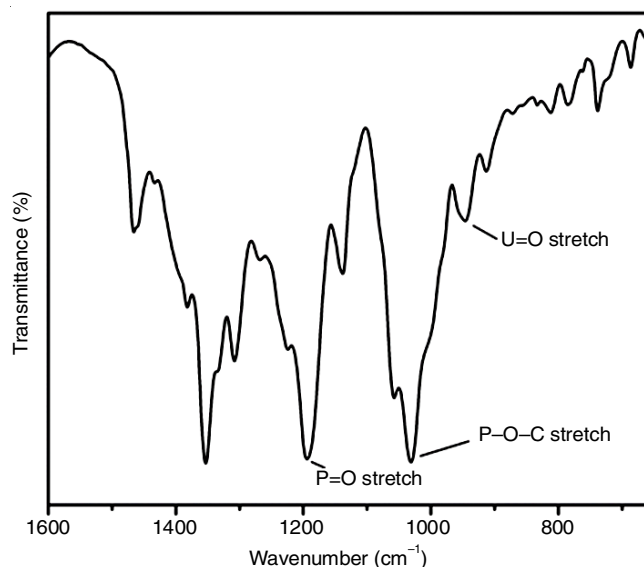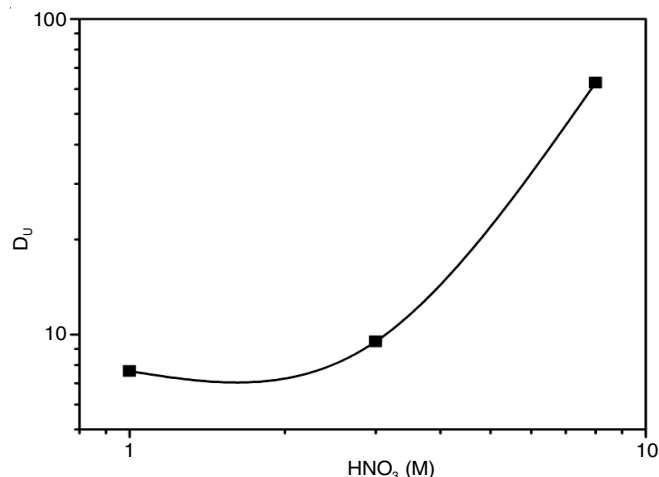

Fig. 7. FTIR of TBP-U/$[P_{(14)666}][TF2N]$

Fig. 8. Dependency of D_U on acidity at 50% TBP in ($[P_{(14)666}][TF2N]$ from mixture U + Sr
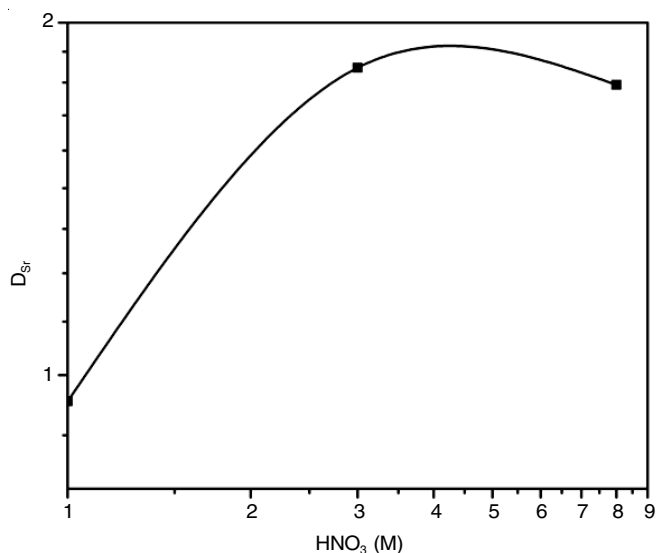
Fig. 9. Dependency of D_{Sr} on acidity at 50% TBP in ([P$_{(14)666}$][TF2N] in U + Sr mixture

has been observed from Fig. 8 that the D_U increases from ~ 8 to ~63 when the concentration of nitric acid varies from 1 to 8 M similarly in Fig. 9 D_{Sr} varies from ~ 0.95 to ~1.77.

In the range of high acidity of interest the uranium extraction from the simulated mixture by the 50% TBP in [P$_{(14)666}$][TF2N] is significant (~ 10 at 3 M and ~63 at 8 M).

Conclusion

The extraction of U(VI) by tributyl phosphate (TBP), a classical complexing agent, has been studied into the ionic liquid (IL) 1-butyl-3-methylimidazolium *bis*(trifuoromethyl-sulfonyl)imide ([BMIM][TF2N]) and [P$_{(14)666}$][TF2N]. The results are compared for 30% TBP, 50% TBP in ionic liquid as a solvent. Increasing the HNO$_3$ acidity of aqueous solution from 0.01 M to nearly 1 M the distribution ratio D_U decreases from 16 to 1.2 for 1.1 M TBP in [BMIM][TF2N] and the corresponding extraction efficiency (% E) varies from ~94 to 55. In the acidic range of 1 to 8 M the distribution ratio and extraction efficiency (% E) shows reversal of trend that is it increases to give a local maximum at 8 M. In contrast, for [P$_{(14)666}$][TF2N] ionic liquid it is observed that with increase in acidity from 0.01 to 8 M distribution ratio keeps enhancing from 2 to 39 and the corresponding extraction efficiency (%E) varies from ~ 51 to 95. Since [P$_{(14)666}$][TF2N] works better than [BMIM][TF2N] in higher acidic range so this ionic liquid is used for selective separation of uranium and strontium form the mixture of both. Nearly 98% of extraction efficiency (%E) and 63 distribution ratio (D$_U$) of uranium is achieved at 8 M acidity. Infrared studies of [P$_{(14)666}$][TF2N], 50%TBP in [P$_{(14)666}$][TF2N] and UO$_2$(NO$_3$)$_2$·6H$_2$O-TBP in [P$_{(14)666}$][TF2N] system are reported. Quantitative analysis of TBP-uranyl nitrate complex in ionic liquids in the regions of P=O and U=O stretching vibrations is discussed in agreement with a previous studies.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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