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Facile extractive separation studies of uranium(VI) assisted by dicyclohexano-18-crown-6 through green approach

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Abstract

A reliable precise analytical method has been developed for the extraction of uranium(VI) from 1 M ammonium thiocyanate and 1 M acetic acid with 0.001 M dicyclohexano-18-crown-6 in nitrobenzene. Various parameters like ammonium thiocyanate concentration, acetic acid concentration, reagent study, solvent study, strippant study and loading capacity were studied. Uranium(VI) were selectively extracted and separated from diverse ion and ternary mixture. The proposed method was also used for the determination of uranium(VI) from rock and monazite sand sample.

Keywords Analysis of rock sample · Extraction · Uranium(VI) · Dicyclohexano-18-crown-6

Introduction

Uranium is a naturally occurring significant radioactive metal. Nowadays uranium is used as a nuclear fuel in nuclear power generation. Uranium is a significant strategic resource [1]. Uranium resources are broadly employed in medicine, scientific research, national defence, industry, and other fields [2, 3]. The quick growth of nuclear technology has steadily increased the requirement of uranium mining and smelting products, resulting in a huge quantity of uranium tailings [1, 4]. Therefore time requires that it should be extracted and lastly in pure form.

It is significant to extract uranium from natural resources by environmental friendly approaches. Different strategies for the separation of uranium have been investigated. Along with solvent extraction is most often employed strategy. Different extractants have been used for the extraction of uranium such as dibenzo-24-crown-8 [5], dibenzo-18-crown-6 [6], trioctylphosphine oxide (TOPO), triphenylphosphine-oxide (TPPO), tri *iso*-octylamine [7], tributylphosphate [8], KROPHOS-18 [9]. Cyanex-272 [10], Tri-n-dodecylamine [11], 2-ethylhexylphosphonic acid (PC88A) [12], UTEVA [13], didodecylphosphoric acid (DDPA) [14], N,N,N′,N′-tetrabutylmalonamide [15], N,N-dialkylamide [16].

Calcium chloride ($CaCl_2$) roasting and nitric acid (HNO_3) leaching were used for the extraction of uranium [17]. Wen-Jiwang studied the distribution of Dicyclohexano-18-crown-6 in 1, 2-dichloroethane at two phases [18]. The potential analytical abilities of dicyclohexano-18-crown-6 is high, therefore it was used for extraction of U(VI) through solid-phase extraction [19], also, it was employed for extraction of U(VI) from mixed aqueous-organic solutions [20], its fate was applied for extraction of U(VI) with Re(VII) [21]. The N, N, N, N-tetra octyl diglycomide (TODGA) has been investigated for extraction of uranium in nitric acid medium [22].

According to the robustness of the work is concerned, in the former work, U(VI) was extracted with different extractants as given in Table 1. However, those necessitate a high concentration of mineral acid media, maximum concentration of extractant and more time of extraction etc. Whereas, in the projected system, the extraction was carried out in a mixture of 1 M acetic acid and 1 M ammonium thiocyanate medium, dicyclohexano-18-crown-6 concentration was 1×10^{-3} M, demonstrating the system is comparatively eco-friendly and a step ahead in the direction of green chemistry.

The goal of the present study was to extend a more specific and greener method for the extraction of U(VI). The extraction scheme has been optimized by studying various parameters. The uniqueness of the scheme lies in a smaller

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Table 1 Comparison of the current method with previous reported solvent extraction systems for extraction of uranium

Extractant	Solvent	Acidity	Strippant	Method of determination	Equilibrium time	References
Dibenzo-24-crown-8	Nitrobenzene	0.5–8.0 M HBr	2 M HNO_3	Spectrophotometry	10 min	[5]
Dibenzo-18-crown-6	Nitrobenzene	0.1–5.0 M NH_4SCN	1 M HNO_3	Spectrophotometry	10 min	[6]
Tributyl phosphate	Dodecane	3 M HNO_3	–	Spectrophotometry	10 min	[8]
KROPHOS-18	Kerosene	8 M HCl	10 M H_3PO_4	–	2 min	[9]
Cyanex-272	Xylene	2 M HNO_3	0.5 M Na_2CO_3	ICP-AES	5 min	[10]
Tri-n- dodecylamine	n-Hexane	7 M HCl	–	Spectrophotometry	24 h	[11]
PC88A	Dodecane	0.1 M Oxalic acid	3 M HNO_3	FTIR	30 min	[12]
Didodecylphosphoric acid	Toluene	1–7 M $HNO_3/HClO_4$	–	Spectrophotometry	3 h	[14]
N,N,N’,N’- tetrabutylmalonamide	Toluene	1 M HNO_3	$LiNO_3$	Spectrophotometry	10 min	[15]
N,N-dialkylamide	Toluene	4 M HNO_3	–	Spectrophotometry	30 min	[16]
DC-18-C-6	1,2-DCE	4 N HCl	Picric acid	–	–	[18]
DC-18-C-6	Toluene	7 M HNO_3	–	Spectrophotometry	40 min	[20]
DC-18-C-6	$CHCl_3$	3–8 M HNO_3	–	–	10 min and 30 min contact time	[21]
Dicyclohexano-18-crown-6	Nitrobenzene	1 M CH_3COOH	1 M HCl	Spectrophotometry	5 min	Proposed method

amount use of extractant concentration and utilizing of greener weak acid. The prettiness of the system is a recovery of solvent and does not need too much instrumentation.

Experimental

Equipments

A systronic spectrophotometer, digital pH meter (Model LI-120, Elico, India) and separating funnel were used for experimental work.

Reagents and solutions

The U(VI) solution was prepared by dissolving 2.166 g of uranyl nitrate hexahydrate (Anala R, BDH) in 1000 ml double distilled water and standardized gravimetrically [23]. The 100 µg/ml of U(VI) solution was equipped by suitable dilution of standard stock solution. Solution of dicyclohexano-18-crown-6 (Aldrich, USA) without further purification.

Extraction procedure

To an aliquot of solution containing 100 µg of U(VI) was mixed with 1 M acetic acid and 1 M ammonium thiocyanate in a total volume of 10 ml and then transferred to 125 cm^3 separating funnel. 10 ml of 0.001 M Dicyclohexano-18-crown-6 in nitrobenzene was added as an extractant and equilibrated for 5 min. The two phases were allowed to settle down and separate. Uranium(VI) from the organic phase was back-extracted with 1 M HCl as a strippant (3 × 10 ml) solution. The stripped solution containing U(VI) was concentrated to moist dryness and diluted with water. U(VI) in the aqueous phase was quantitatively calculated spectrophotometrically with PAR [24] at 530 nm using a calibration graph.

Results and discussion

Effect of Ammonium thiocyanate on extraction of uranium(VI)

The influence of ammonium thiocyanate concentration on the extraction of uranium(VI) assisted by dicyclohexano-18-crown-6 in nitrobenzene was studied. In this study, the concentration of ammonium thiocyanate was differed over the range 0.1–5 M, while 0.001 M dicyclohexano-18-crown-6 in nitrobenzene was kept constant and extraction of uranium(VI) was performed. The extraction of uranium(VI) was initiated at 0.1 M NH_4SCN and becomes quantitative at 0.8 M. From the range of 0.8–5 M NH_4SCN concentrations, extraction remains quantitative as shown in Fig. 1. Ammonium thiocyanate acts as a salting out effect which reduce free water molecule in the system, which increases the activity of the extracted species to increase the distribution ratio. There is no dissociation of complexed

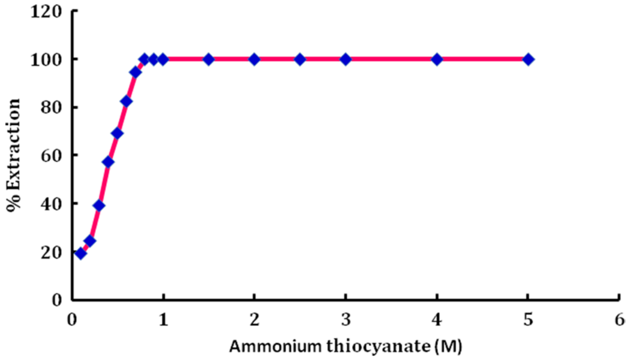

Fig. 1 Ammonium thiocyanate concentration

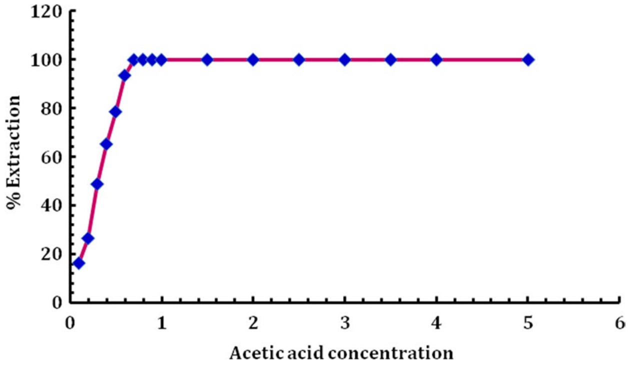

Fig. 2 Acetic acid concentration

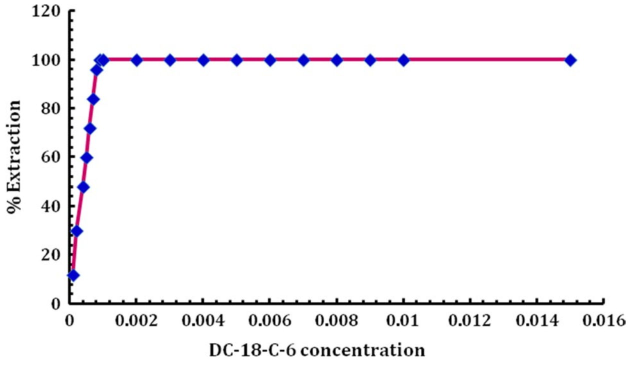

Fig. 3 Reagent concentration

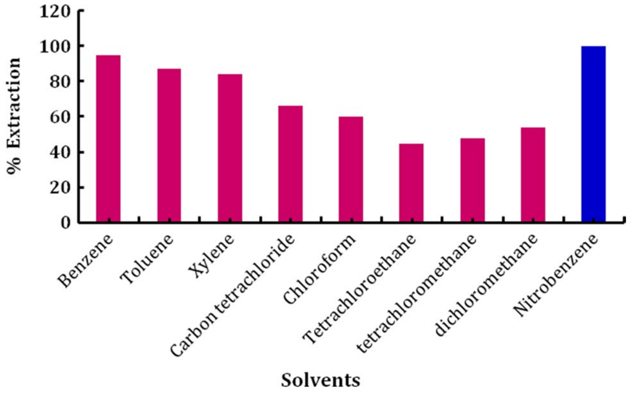

Fig. 4 Solvent study

ions in a higher concentration of NH_4SCN. Consequently, for further extraction study, 1 M NH_4SCN was used.

Impact of acetic acid concentration on the extraction of uranium(VI)

The extraction of uranium(VI) was studied from acetic acid in the range of 0.1–5 M as shown in Fig. 2. Acetic acid was used as a medium which helps to formation of uranium-dicyclohexano-18-crown-6 complex. It was examined that the extraction of uranium(VI) increases with an increase in acetic acid concentration in the range of 0.1–0.7 M and remains quantitative in the range of 0.7–5 M. Therefore, 1 M concentration of acetic acid was used throughout work.

Effect of dicyclohexano-18-crown-6 concentration

The concentration of dicyclohexano-18-crown-6 was varied in the range between 1×10^{-4} to 2×10^{-2} M in nitrobenzene to optimize extraction conditions of uranium(VI) at 1 M acetic acid and 1 M ammonium thiocyanate. The study showed that extraction of uranium(VI) increases from 1×10^{-4} to 9×10^{-4} M and becomes quantitative in the range of 9×10^{-4} to 2×10^{-2} M. Therefore based on study, we illustrate that 1×10^{-3} M of dicyclohexano-18-crown-6 was sufficient to getting quantitative extraction of uranium(VI). Therefore 10 mL of 1×10^{-3} M dicyclohexano-18-crown-6 in nitrobenzene was used in an actual experiment (Fig. 3).

Effect of solvents on extraction of uranium(VI)

The selectivity and extractability for the extraction of uranium(VI) by organic extractant are greatly influenced by the nature of the diluents. Thus the influence of nature of solvents viz. Tetrachloroethane, tetrachloromethane, dichloromethane, chloroform, carbon tetrachloride, xylene, toluene, benzene, and nitrobenzene (Fig. 4) was examined. The 0.001 M dicyclohexano-18-crown-6 dissolved in the above solvents and used for the extraction of uranium(VI). The study showed that the nitrobenzene diluents shows clear cut phase separation due to solubility of species and gives quantitative extraction of uranium(VI) while other solvents show incomplete extraction of uranium(VI). However, for further experimental study nitrobenzene was adopted as a solvent.

Effect of shaking time on extraction of uranium(VI)

The effect of contact time on the extraction of uranium(VI) was observed by using 0.001 M dicyclohexano-18-crown-6 in nitrobenzene and the aqueous solution of 100 μg of uranium(VI) in 1 M CH_3COOH and 1 M NH_4SCN. The equilibrium time has differed from 15 s to 30 min (Fig. 5). The results showed that the quantitative extraction of uranium was achieved in 5 min. Moreover, the prolonged contact time does not affect on quantitative extraction of uranium(VI). Therefore, for further study 5 min contact time was selected to ensure complete extraction of uranium(VI).

Strippant study

In the solvent extraction technique, the back extraction is the contrary phenomenon of extraction and it is the most significant step for uranium(VI) extraction in the organic phase. To back extract uranium(VI) from dicyclohexano-18-crown-6 in nitrobenzene organic phase. We have employed various stripping agents viz. HCl, HNO_3, NaOH, and NH_3. The study shows that uranium(VI) was extracted with dicyclohexano-18-crown-6 in nitrobenzene and was completely back-extracted with 0.5–1.5 M HCl and 2–3.5 M HNO_3 (Fig. 6). There was no complete back extraction with ammonia and sodium hydroxide solution. Hence, for the experimental study 1 M HCl was used as a strippant.

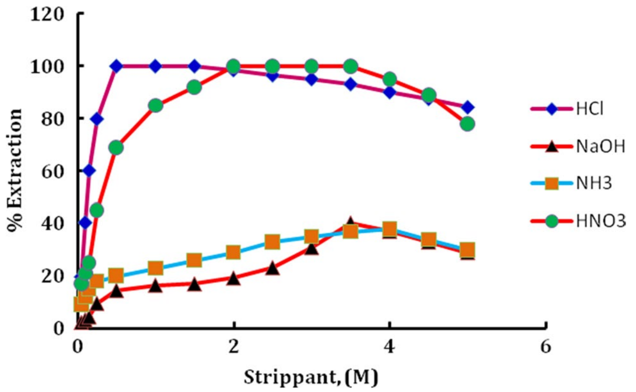

Fig. 6 Strippant study

Effect of uranium(VI) loading capacity on 0.001 M dicyclohexano-18-crown-6

The solvent extraction behaviour of uranium(VI) as a function of uranium(VI) loading capacity was examined at different concentrations of uranium(VI) between 100 and 1500 μg (Fig. 7). The examination showed that the quantitative extraction takes place between 100 and 1200 μg. It means that up to 1200 μg of uranium(VI) was tolerable in 10 mL of 0.001 M dicyclohexano-18-crown-6. After 1200 μg extraction decreases this indicates that there is a deficiency of dicyclohexano-18-crown-6. The results showed that 1200 μg of uranium(VI) was a loading capacity of 10 mL dicyclohexano-18-crown-6.

Probable mechanism of uranium(VI) extraction

Stock solution of uranyl nitrate forms aquo complex of uranium nitrate. In the presence weak acid medium salting out agent substituted the nitrate. Uranium forms complex species with crown ether as a neutral uncharged species, which was extracted in organic solvent.

$$UO_2(NO_3)_{2\ (aq)} + 2H_2O \rightleftharpoons [UO_2(NO_3)_2(H_2O_2)]_{aq}$$

$$[UO_2(NO_3)_2(H_2O)_2]_{(aq)} + 2NH_4SCN \xrightarrow{CH_3COOH} [UO_2(SCN)_2(H_2O)_2]_{(aq)} + 2NH_4NO_3$$

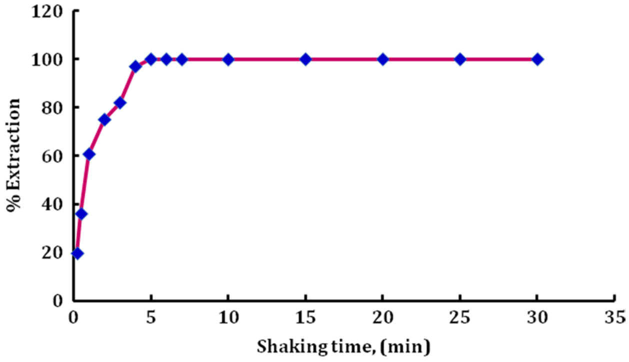

Fig. 5 Effect of shaking time

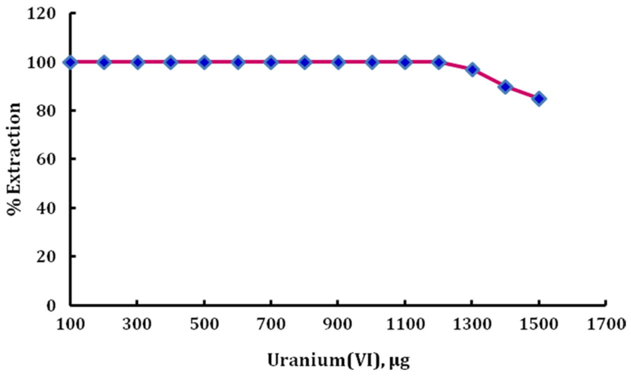

Fig. 7 Loading capacity

$$\mathrm{DC\text{-}18\text{-}C\text{-}6}_{(org)} + \left[\mathrm{UO_2(SCN)_2} \cdot \left(\mathrm{H_2O}\right)_2\right]_{(aq)} \xrightarrow{\mathrm{CH_3COOH}} \left[\mathrm{DC\text{-}18\text{-}C\text{-}6} \cdot \mathrm{UO_2(SCN)_2}\right]_{org} + 2\mathrm{H_2O}\ (aq)$$

Applications

Foreign ion study

To observe the versatility and applicability of the proposed uranium(VI) extraction method. The effect of foreign ions was checked. The uranium(VI)-dicyclohexano-18-crown-6 method is free from a large number of anions and cations. In the proposed method uranium(VI) was extracted with 0.001 M reagent concentration in nitrobenzene from 1 M NH_4SCN in the presence of a broad range of ions (Table 2). The extraction and determination of uranium(VI) from diverse ions were achieved by taking the benefit of the variation in the extraction conditions of the metal ions. Those metal ions showed interference in the extraction and determination of uranium(VI), they were masked with appropriate sequestering agents. The amount of added ions that gives an error less than ± 2% in the recovery of uranium(VI) known as the tolerance limit.

In this study solution of 100 µg of uranium(VI) was taken, another anion/cation was mixed and followed by the proposed methods procedure. Firstly the diverse ions mixed to uranium(VI) solution in large excess i.e. 100 mg for anions and 50 mg for cations. For finding the interference, the practical procedure was repeated with successively lesser amounts of added ion. Interference of very few Cu(II), Ni(II), Zn(II), Mn(II), Pb(II) and Th(IV) ions was registered and they were masked with respective masking agents as shown in Table 2.

Separation of uranium(VI) from multicomponent mixtures

To extend the applicability of the uranium(VI)-dicyclohexano-18-crown-6 extraction method. It was applied for the multicomponent mixture separation. Multicomponent mixture of uranium(VI) along with Fe(III), K(I), Li(I), Mg(II), Ca(II) were carried out. The extraction of uranium(VI) from added metal ion was quantitative as given in Table 3.

Table 3 Separation of uranium(VI) from multicomponent mixtures

Composition (µg)	Average recovery, %	R. S. D., %
U(VI) 100; Fe(III) 100;K(I) 100	99.91	0.04
U(VI) 100; Fe(III) 100;Li (I) 100	99.95	0.03
U(VI) 100; Fe(III) 100;Mg(II) 100	99.96	0.04
U(VI) 100; Fe(III) 100;Ca(II) 100	99.98	0.04
U(VI) 100; Fe(III) 100;Ba(II) 100	99.95	0.02
U(VI) 100; Fe(III) 100;Zn(II)[c] 100	99.92	0.04

[c] Masked by 10 mg of thiocyanate

Table 2 Separation of uranium(VI) from foreign ions

Ions	Added as	Tolerance limit (mg)	Ions	Added as	Tolerance limit (mg)
Na(I)	NaCl	25	Ce(III)	$CeCl_3 \cdot 6H_2O$	40
Rb(I)	RbCl	20	Sb(III)	$SbCl_3$	20
Cs(I)	CsCl	20	Y(III)	$Y(NO_3)_3$	30
Be(II)	$BeSO_4 \cdot 4H_2O$	25	Zr(IV)	$Zr(NO_3)_4 \cdot 4H_2O$	5
Mg(II)	$MgCl_2 \cdot 6H_2O$	40	V(IV)	$VOSO_4 \cdot 4H_2O$	5
Ca(II)	$CaCl_2$	25	Th(IV)[d]	$Th(NO_3)_4$	0.5
Sr(II)	$Sr(NO_3)_2$	30	Cr(VI)	$K_2Cr_2O_7$	5
Ba(II)	$Ba(NO_3)_2$	25	Mo(VI)	$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$	5
Cu(II)[a]	$CuCl_2 \cdot 2H_2O$	0.5	W(VI)	$Na_2WO_4 \cdot 4H_2O$	30
Ni(II)[c]	$NiCl_2 \cdot 6H_2O$	5	La(III)	$LaCl_3$	0.1
Mn(II)[b]	$MnCl_2 \cdot 4H_2O$	0.3	Al(III)	$Al(NO_3)_3 \cdot 9\ H_2O$	5
Zn(II)[c]	$ZnCl_2$	0.3	SCN^-	NaSCN	75
Pb(II)[a]	$Pb(NO_3)_2$	0.5	ClO_4^-	$HClO_4$	10
Fe(III)	$FeCl_3 \cdot 6H_2O$	5	CH_3COO^-	CH_3COOH	35
Cr(III)	$Cr(NO_3)_3 \cdot 9\ H_2O$	5	Tartrate	Tartaric acid	25

[a] Masked by 10 mg tartrate, [b] Masked by 10 mg citrate, [c] Masked by 10 mg of thiocyanate, [d] Masked by 10 mg fluoride

Table 4 Analysis of uranium(VI) in rock and sand sample

Sr. no	Sample	Certified value of uranium taken	Uranium found by proposed method	Relative standard deviation %
1	Syenite rock	280 ppm	278 ppm	0.71
2	Monazite sand	0.29%	0.285%	0.54

Extraction of uranium(VI) in syenite rock sample and monazite sand sample

The proposed method was applied to the measurement of uranium(VI) in real samples viz. Syenite rock sample and monazite sand. The rock and sand samples were brought in solution by the procedure [25]. An aliquot of rock and sand sample solution was separated and determined with 0.001 M dicyclohexano-18-crown-6 in nitrobenzene from 1 M NH_4SCN and 1 M CH_3COOH. The quantity of uranium getting in syenite rock was 278 ppm as against the reported value of 280 ppm and monazite sand was 0.285% as against the standard value of 0.29% as shown in Table 4.

Conclusions

1. The proposed method permits the separation of uranium(VI) at micro level concentration.
2. The concentration of dicyclohexano-18-crown-6 required is very low.
3. The method permits the separation of uranium(VI) from Fe(III), K(I), Li(I), Ba(II) and Ca(II) effectively.
4. The proposed system was employed for the analysis of uranium(VI) in different sand and rock samples.
5. The recommended method is reliable, rapid and simple for the separation of uranium(VI).

Author's contributions S B carried out the experimental studies, analysis, data interpretation. SZ wrote the manuscript under the supervision of SB. Authors read and approved the final manuscript.

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Declaration

Conflict of interest The authors declare that they have no conflict of interest.

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