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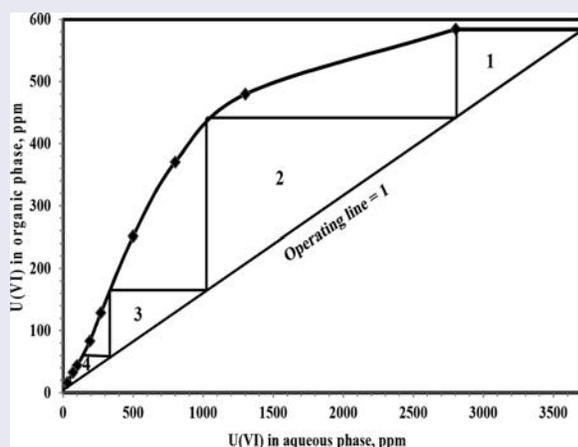
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ABSTRACT

In the present study, the use of 5% Aliquat-336/kerosene was evaluated in the liquid–liquid extraction of Uranium(VI) and some impurities of Thorium(IV) and rare earth elements (REEs(III)). Experiments were carried out to determine the factors affecting U(VI) and some impurities of Th(IV) and REEs(III) extraction. McCabe–Thiele diagram was constructed to define the theoretical stages for extraction mixer settler process. The effect of stripping factors from the loaded extractant was also tested. Uranium cake was finally obtained from the strip solution and a workable flow sheet was then formulated.

GRAPHICAL ABSTRACT



ARTICLE HISTORY

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KEYWORDS

Aliquat-336/kerosene;
egyptian monazite;
extraction; stripping;
Uranium(VI)

1. Introduction

Solvent extraction is an effective method for the recovery, separation, and purification of metals and it is used for the hydrometallurgical refining of uranium and the reprocessing of spent nuclear fuel. In recent years, solvent extraction has been attempted for uranium recovery from alkaline medium.^[1] The recovery and separation of uranium and thorium are accomplished through a variety of methods after chemical attacking of the mineral with sulfuric acid or sodium hydroxide.^[2] Hammad et al. proposed two individual extraction processes for separating thorium and uranium from Egyptian monazite alkaline hydrous oxide cake concentrate.^[3]

Aliquat-336 was studied to extract uranium from alkaline media using different diluents such as benzene, toluene, xylene, methyl-isobutyl ketone (MIBK), and carbon tetrachloride. It was found that different kinds of diluents had a significant effect on uranium extraction.^[4,5] The extraction behavior of several

elements using Aliquat-336, such as Sc, Fe, Zr, some lanthanides, and some actinides, and nitric acid was studied and it was reported that Aliquat-336 shows high extraction for tetravalent elements and anions: the extraction of multivalent cations was found to decrease in the order $M^{4+} > MO^{2+} > M^{3+}$.^[6] Selective method was developed for extraction chromatographic studies of U(VI) with Aliquat-336 (liquid anion exchanger) coated on silica gel as a stationary phase.^[7] El-Nadi et al.^[8] investigated the kinetics of uranium extraction from the alkaline carbonate medium by Aliquat-336 in kerosene.

The liquid–liquid extraction of uranium in HCl media by Aliquat-336 was performed in a microsystem to demonstrate the potential of miniaturization in radiochemistry compared to protocols (decrease in volumes, analysis time, radiations received, and feasibility of automation).^[9]

The present work is directed to investigate the uranium extraction from the selective carbonate leaching liquor

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of hydrous oxide cake from Egyptian monazite by Aliquat-336/kerosene in order to obtain information on the factors affecting the extraction rate of U(VI) and some impurities of Th(VI) and REEs(III) in this system and the subsequent stripping rate of U(VI) to propose a mechanism describing the extraction process.

2. Experimental

2.1. Chemicals and reagents

Several reagents that were used in this study were used as received. These are mainly Arsenazo III and Arsenazo I obtained from SIGMA. Aliquat-336 was obtained from Cognis.co. Kerosene was supplied from Misr Petroleum Co. The monazite was supplied by the Nuclear Materials Authority, Egypt, as 85% concentrate.

2.2. Preparation of aqueous solution

The monazite sample was treated with sodium hydroxide (50 wt%) solution and boiled for 4 hours at 140°C. The solution was diluted up to 20 wt% NaOH concentration and boiled again for another hour, then filtered at 80°C. The solid residue was washed several times with water, dried, and treated with an alkaline carbonate solution composed of 40 vol% 1 M Na₂CO₃, 50 vol% 1 M NaOH, and 10 vol% (30%) H₂O₂ mixture at 60°C in order to separate the maximum content of uranium selectively by forming tricarbonato complex. Since our solid residue was expected to contain uranium hydrous oxide rather than U₃O₈ making, it is easier to be dissolved in the above mixture. The leached carbonate solutions (50 ml for the first five steps, followed by 25 ml for the last two), which treated the hydrous oxide cake sample (10 g), were diluted and uranium was measured spectrophotometrically.

2.3. Analytical technique

All samples were weighed using an analytical balance produced by AND (Japan) having a maximum sensitivity of 10⁻⁵ g and an accuracy of ±0.01 mg. The hydrogen ion concentration of different solutions was measured using a digital pH-meter of the type Orion research model 701 N/digital ion analyzer, USA, within an error of ±0.1. The spectrophotometric analyses of U(VI), Th(IV), and REEs(III) were performed with a single-beam UV-VIS recording Metertech Ino model Sp-5001 spectrophotometer using Arsenazo III and Arsenazo I. Environmental scanning electron microscope (ESEM), Philips XL30, was used for the characterization of U(VI) products after precipitation with hydrogen peroxide and ammonium hydroxide. To identify the absorption bands of the solvent before and after extraction, FTIR instrument (4100 Jasco- Japan) was used at Cairo University.

2.4. Solvent extraction procedure

Extraction of uranium from selective carbonate leach liquor was carried out at 25°C ± 1°C (except when studying the effect of temperature). In this concern, equal volumes (ml) of the

aqueous phase and an organic solution of (5%) Aliquat-336, after converting it to sulfate form^[10] dissolved in kerosene or other diluents used, were equilibrated by contacting for 5 minutes.

3. Results and discussion

3.1. Extraction of uranium from the leach liquor solution

The selective carbonate leach liquor of Egyptian monazite was analyzed and was found to assay 0.378% U(VI), 0.013% Th (IV), and 0.039% REEs(III) so Th(IV) and REEs(III) present in solution as impurities. Therefore, to obtain high-purity uranium, the extraction of U(VI) from the leach solution by Aliquat-336/kerosene was investigated. The use of Aliquat-336 as an extractant is based on anionic complexes with carbonate. Further, Aliquat-336 behaves as a liquid anion exchanger. Therefore, the extraction parameters were investigated.

3.1.1. Effect of different diluents

The effect of diluents in the organic phase is quite significant on the extraction of metals because both physical and chemical interactions exist between the diluent and extractant. The diluents can affect the economy of a solvent extraction process; thus several experiments were carried out using different diluents, namely benzene, toluene, o-xylene (as aromatic diluents), carbon tetra chloride, n-hexane, cyclo-hexane, chloroform, and kerosene (as aliphatic diluents). The other experimental parameters were fixed at equilibrium pH 9, an A/O ratio of 1:1, 5% concentration of Aliquat-336/kerosene (containing 5% octanol as modifier), and the contacting time was 10 minutes at room temperature. It's clear from the results presented in Figure 1 that benzene and kerosene extract U(VI) more efficiently than other diluents, but kerosene was selected for economic and practical reasons.

3.1.2. Effect of pH

The effect of solution's pH was investigated in the pH range of 8–12 in these experiments, 5% Aliquat-336/kerosene was used at the phase ratio A/O 1:1 for about 10 minutes, at room

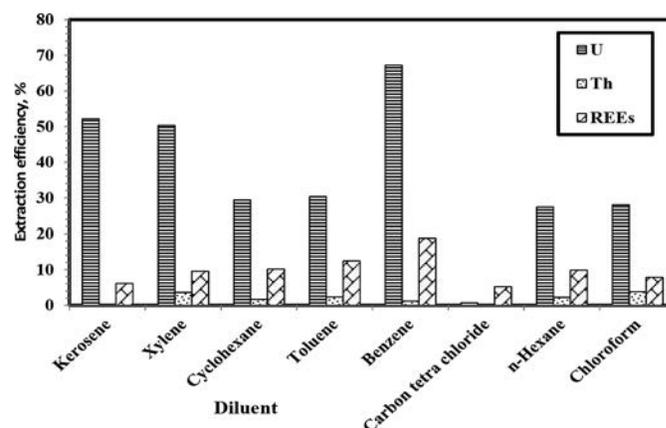


Figure 1. Effect of different diluents on the extraction of U(VI) and some impurities of Th(IV) and REEs(III).

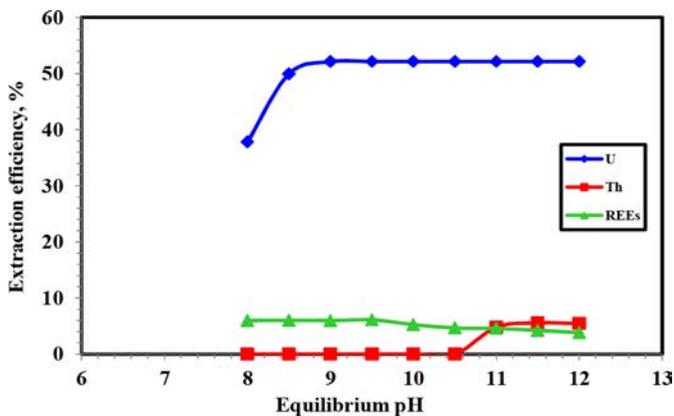


Figure 2. Effect of equilibrium pH on the extraction of U(VI) and some impurities of Th(IV) and REEs(III).

temperature. From the results shown in Figure 2 it was found that by using Aliquat-336/kerosene as an extractant, the extraction efficiency of U(VI) increased from 37.83% at pH 8 and reached the maximum (52.14%) at pH 9 and then plateaued until pH 12, which is due to the formation of $\text{UO}_2(\text{CO}_3)_3^{4-}$. On the other hand, it was found that Th(IV) was not extracted from pH 8 to 10.5. However, at pH 11.5, Th(IV) extraction efficiency reached a maximum value of only 5.56% and then plateaued. REEs(III) extraction efficiency gradually decreased from 6% at pH 8 till it reached a minimum value of 3.85% at pH 12.

3.1.3. Effect of aliquat-336/kerosene concentration

The effect of Aliquat-336/kerosene concentration on extraction of U(VI) and some impurities of Th(IV) and REEs(III) was carried out using various Aliquat-336/kerosene concentrations, in the range from 0.5% to 6.0% on equilibrium pH 9 and phase ratio A/O = 1/1, 10 minutes contacting time, and at room temperature. Based on the results presented in Figure 3, it was observed that the increase of Aliquat-336/kerosene concentration from 0.5% to 5% leads to an increase in the extraction efficiency of uranium from 1.08% to 52.14%; however, on a further increase in the Aliquat-336/kerosene concentration up to 6% the extraction of U(VI)

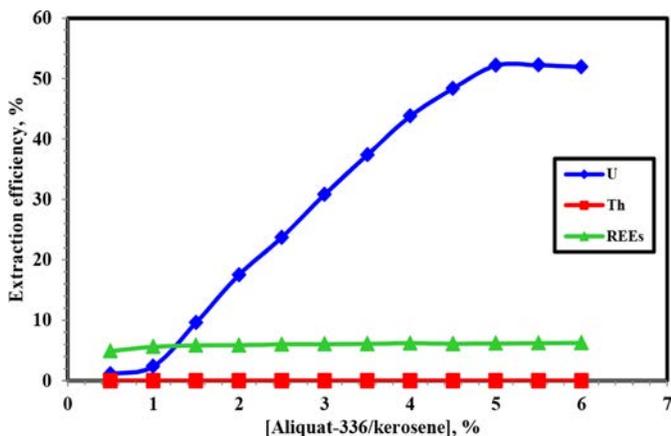


Figure 3. Effect of Aliquat-336/kerosene concentration on the extraction of U(VI) and some impurities of Th(IV) and REEs(III).

showed a plateau. The extraction efficiency of REEs(III) didn't exceed 6.3% by increasing the concentration of Aliquat-336/kerosene from 0.5% to 6%, and no extraction of Th(IV) was detected in this experiment. The linear plot of $\log D$ versus $\log [\text{Aliquat-336/kerosene}]$ shows the straight line ($R^2 = 0.975$) with a slope of 2.0256, confirming the association of two moles of Aliquat-336/kerosene with each of the extracting metal ions in the extracted species. The reason for the very low uranium maximum loading of Aliquat-336 from the alkaline solution is probably due to the presence of 1-octanol. The adverse effect of alcohol on uranium extraction with cetyl-dimethyl-benzyl-ammonium chloride was previously reported by Sardina et al.^[11] As the uranium concentration in leach solutions is generally low, the drawback of low loading capacity should not prevent the Aliquat-336/1-octanol system from application.^[12]

3.1.4. Effect of contacting time

Series of experiments have been performed to determine the effect of contacting time on U(VI) and some impurities of Th(IV) and REEs(III) extraction from 5 to 30 minutes, using 5% Aliquat-336/kerosene from the working leach liquor, under conditions of pH 9, and an A/O ratio of 1:1 at room temperature. From the results in Figure 4, it was obvious that a contacting time of 10 minutes would be enough to attain maximum extraction efficiency of U(VI) (52.14%). Increasing the contacting time to 30 minutes had no effect on the extraction efficiency. However, the extraction efficiency of REEs(III) increased from 1.24% to 14.25% by increasing the contacting time. On the other hand, there was no Th(IV) extracted by the Aliquat-336/kerosene under these conditions. Therefore, the following experiments were performed by contacting the two phases for 10 minutes.

3.1.5. Effect of temperature

The extraction efficiency of U(VI) and some impurities of Th(IV) and REEs(III) by 5% Aliquat-336/kerosene is studied at different temperatures (298–318 K) under optimum conditions, equilibrium pH 9, and an A/O phase ratio of 1:1 for 10 minutes. It is clear that from Figure 5 the extraction

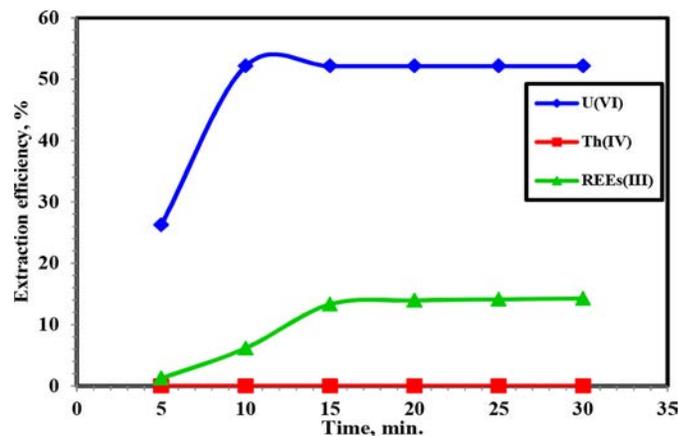


Figure 4. Effect of contact time on the extraction of U(VI) and some impurities of Th(IV) and REEs(III).

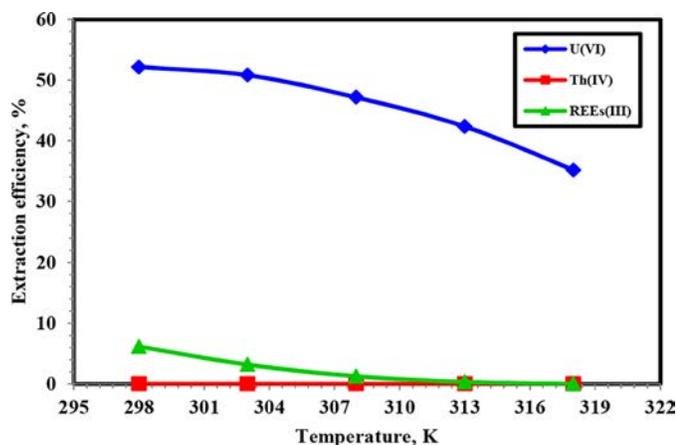


Figure 5. Effect of temperature on the extraction of U(VI) and some impurities of Th(IV) and REEs(III).

efficiency of U(VI), Th(IV), and REEs(III) decreased as the temperature increased; the percentage extraction of U(VI) decreased from 52.14% to 0% and from 6.16% to 0% for REEs(III) when the temperature was increased from 298 K to 318 K and no extraction occurred in Th(IV).

The effect of temperature on the complex extraction could be evaluated in terms of their thermodynamics values calculated from the following relations:

$$\text{Log } D = \frac{-\Delta H}{2.303RT} + C \quad [1]$$

$$\Delta G = -2.303RT \log D \quad [2]$$

$$\Delta G = \Delta H - T\Delta S \quad [3]$$

where D is the distribution coefficient, ΔH is the enthalpy change for the extraction reaction, R is the universal gas constant (8.314 J/mol K), and C is an integration constant, which includes the equilibrium constant for the extraction reaction. These values were used to obtain the corresponding free energy (ΔG) and entropy changes (ΔS) by using Equations (2) and (3), respectively. From Eq. (1), the respective enthalpy variation (ΔH) is evaluated. The free energy (ΔG) is obtained by applying Eq. (2) at standard state, 298 K. Also, the entropy variations (ΔS) were given by applying Eq. (3). The calculated values of ΔH , ΔG , and ΔS were found equal to 26.075 kJ/mol., 212.26 kJ/mol., and -0.624 J/mol K, respectively. The positive ΔH value obtained for the complex formation indicates the endothermic character of the extraction process, while the negative ΔS values indicate that the extraction is less random in nature.

3.1.6. Effect of A/O phase ratio on the extraction of U(VI)

The effect of the A/O ratio upon U(VI) extraction efficiency was studied in the range 1/5–5/1 using 5% Aliquat-336/kerosene, at room temperature for 10 minutes after adjusting the aqueous solution to pH 9. From the obtained results plotted in Figure 6, as the concentration of organic decrease the extraction efficiency decreases due to the reduction of relative amount of Aliquat-336 in the organic solution.

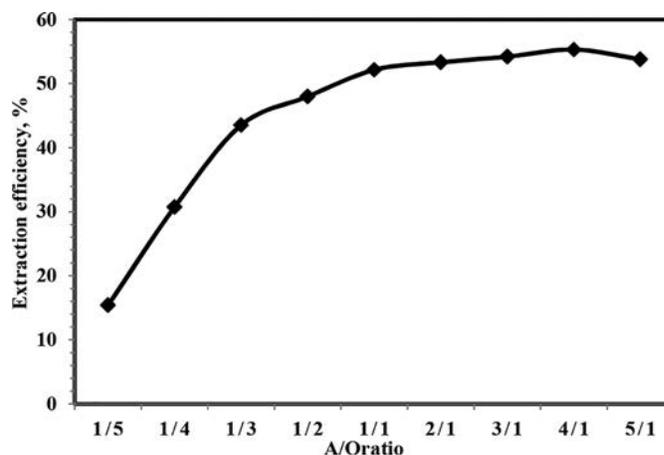


Figure 6. U(VI) extraction percent at different A/O ratios.

3.1.7. Extraction isotherm

At equilibrium, only partial transfer of metallic cations occurs. Thus, several stages of contact should be used in order to recover the maximum values of these species. In this work, the McCabe–Thiele construction is used to determine the number of theoretical stages required for achieving this separation, as shown in Figure 8a. As indicated in this figure, the number of theoretical stages required is four stages of U(VI) extraction with A/O ratio 1/1.

3.2. Stripping investigations

Stripping is the reverse of extraction; it depends on a number of factors such as different stripping agents, contacting times, and the effect of the A/O phase ratio and the construction of the McCabe–Thiele diagram of the stripping process.

3.2.1. Determination the proper stripping agent

Among the different stripping agents [1 M NaOH, 1 M Na₂CO₃, (1 M NaCl + 0.01 M NaOH), (50 g/l NaOH + 0.15% H₂O₂), (0.8)M H₂SO₄, and 1 M (NH₄)₂SO₄], high concentration of H₂SO₄ was chosen for the stripping of U(VI) from the loaded organic phase. In this respect, different concentrations of H₂SO₄ in the range from 0.2 to 1.4 mol/l were used and it was found that a concentration of 1 mol/l is quite efficient for the stripping of uranium (87%), as shown in Figure 7.

3.2.2. Stripping isotherm

Based on the laboratory experimental results, the stripping isotherm (equilibrium curve) was constructed and a McCabe–Thiele diagram was drawn to determine the theoretical number of stages and the volumetric feed ratio of the liquor and solvent for continuous experiments. The equilibrium curve was constructed according to the successive contacts technique using a phase ratio A/O of 1/1; the McCabe–Thiele diagram is shown in Figure 8b. As indicated from the figure, it was found that from the loaded organic phase, four stages were achieved for stripping U(VI) with 1 mol/l H₂SO₄.

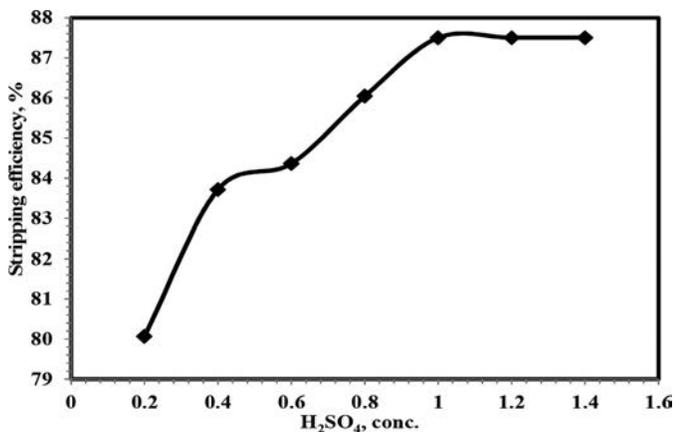


Figure 7. Effect of H₂SO₄ concentration on the stripping efficiency of U(VI).

3.3. FTIR study

In order to define the mechanism of U(VI) recovery by the pre-treated Aliquat-336 in sulfate form as a ligand, it was necessary to subject the studied compound to FTIR analysis. This analysis includes the prepared extractant (Aliquat-336/kerosene), that is, before U(VI) extraction (freshly prepared in their free state) as well as after their extraction (loaded-extractant) as shown in Figures 9 and 10. From the obtained results, it has been realized that some characteristic peaks representing the major groups of the studied Aliquat-36/kerosene such as the aliphatic C-H (stretch), -CH₃,-CH₂- (bend), and C-N groups have a sharp intensity, which turned to medium intensity after the extraction experiments. This indicates that the studied extractant is

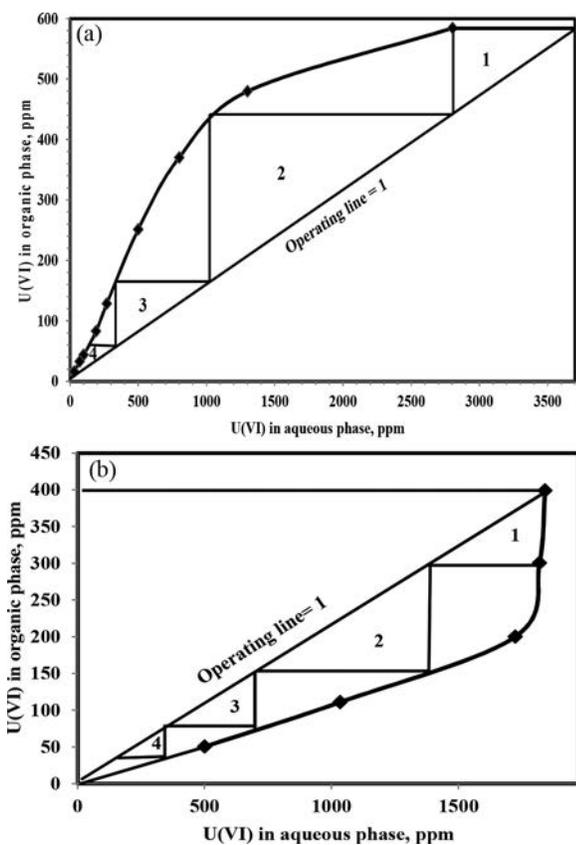


Figure 8. McCabe–Thiele diagram for U(VI) extraction a) and stripping b).

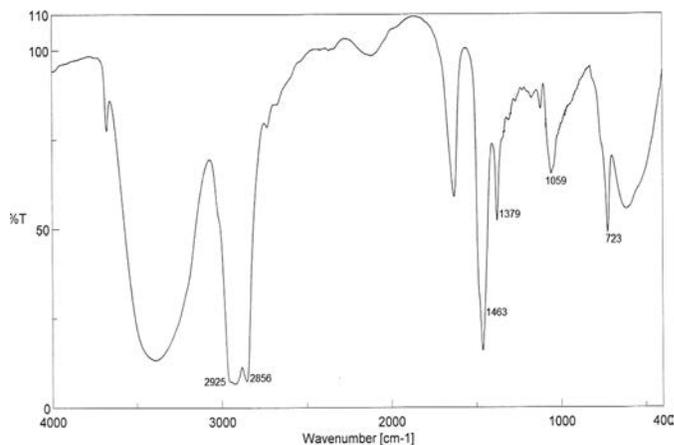


Figure 9. FTIR analysis of Aliquat-336/kerosene.

able to form a complex with U(VI) metal ions in the selective carbonate leach liquor. Accordingly, two kinds of bands are observed at around 2925 cm⁻¹ and 2856 cm⁻¹ assigned to anti-symmetric and symmetric stretch vibration absorption of CH₃, CH₂, and CH,^[13] and two other bands are observed at 1463 cm⁻¹ and 1379 cm⁻¹ assigned to bending vibration of CH₃, CH₂, and CH. On the other hand, there is a characteristic band at 1059 cm⁻¹ assigned to C-N stretching. In addition, the band observed at 720 cm⁻¹ comes from the special vibration absorption of quaternary ammonium salt.^[14] It is thus possible to mention that Aliquat-336/kerosene exhibits, indeed, good affinity toward the U(VI) metal ions in selective carbonate leach liquor. The FTIR showed new absorption bands that weren't assigned in the spectrum of Aliquat-336/kerosene; a characteristic peak of UO₂²⁺ has been clearly observed at 965 cm⁻¹.^[15–20] U(VI) metal ions have replaced the sulfate ions in the extraction step and to be released by stripping with a strong acid. It has also to be mentioned that it is clearly evident that, the formed complex between U(VI) metal ions and extractant would place by the reaction of uranium metal ions and sulfate ions. This reaction can be represented as follows:

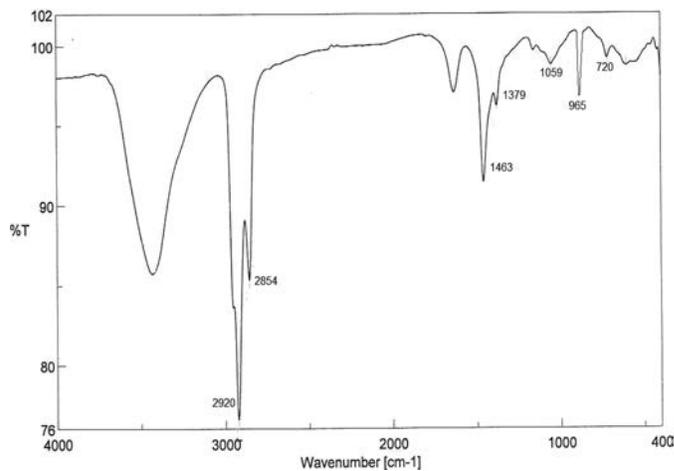
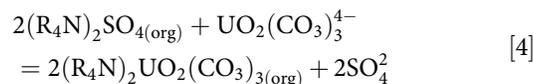


Figure 10. FTIR analysis of (U-Aliquat-336) complex.

Table 1. SEM elemental analysis of the dried U(VI) precipitates formed from the acidic stripped solution of the working Aliquat-336/kerosene.

Element	U(VI) by H ₂ O ₂ , wt%	U(VI) by NH ₄ OH, wt%
U	95.8	96.6
K	0.61	1.25
Fe	0.61	2.16
Ca	2.98	–

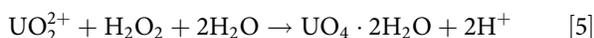
where R represents the alkyls in the Aliquat-336/kerosene structure.

3.4. Preparation of uranium product

Uranium was precipitated and the obtained precipitate was characterized by ESEM, model XL30.

3.4.1. Precipitation using hydrogen peroxide

U(VI) precipitated from acidic solutions with hydrogen peroxide as shown in the following equation:

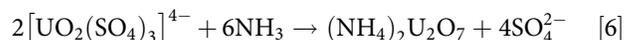


Hydrogen peroxide is added in excess of the stoichiometric ratio at a temperature of about 25°C and at pH 2. Diluted sodium hydroxide is added to maintain the desired pH value. The solution was kept under mechanical agitation and the pH was maintained to the end of the test. Precipitation was carried out for 4 hours and then the product was filtered out.^[21–24] The precipitate was dried at 110–120°C for 48 hours and

analyzed for its chemical composition; as shown in Table 1, the obtained U(VI) product attains a purity of 95.8%.

3.4.2. Precipitation using ammonium hydroxide

U(VI) was precipitated as ammonium diuranate at pH 7.5 by drop-wise addition of 25% ammonium hydroxide according to the following equation:

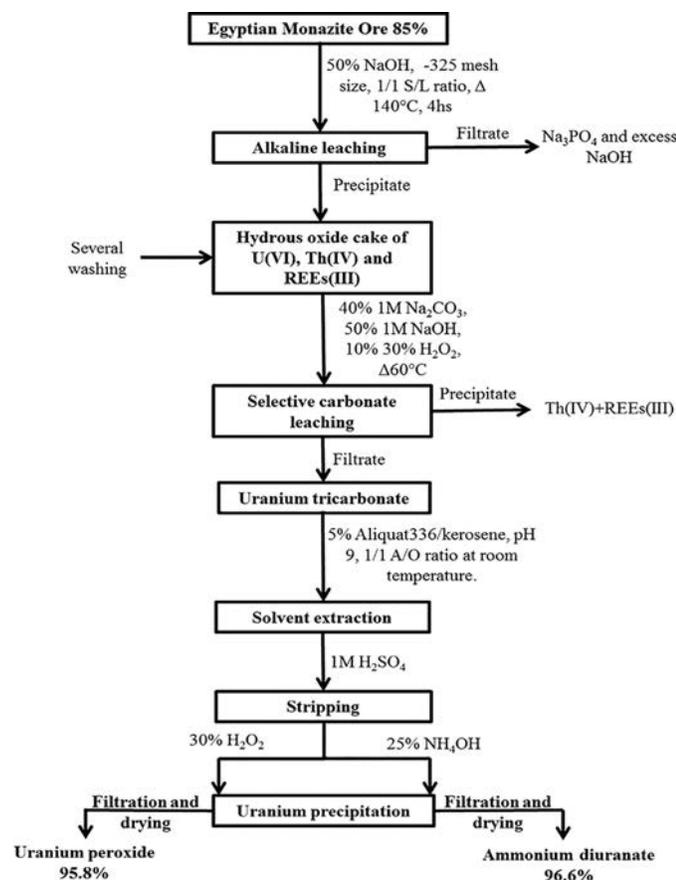


The (NH₄)₂U₂O₇ was separated by decantation and filtration, washed several times with deionized water, and dried in an oven at 105°C.^[25,26] The dried precipitate was then analyzed for U(VI), and as shown in Table 1, the obtained U(VI) product attains a purity of 96.6%.

From the above-mentioned results, it has been possible to formulate and integrate a flow sheet for the extraction of U(VI) from the hydrous oxide cake of Egyptian monazite as shown in Figure 11.

4. Conclusion

This study reveals that Aliquat-336/kerosene extractant was used for the extraction of U(VI) metal ions from selective carbonate leach liquor. The optimum conditions for the extraction of U(VI) metal ions was 5% concentration of Aliquat-336/kerosene, (1/1) A/O, 10 minutes contacting time, room temperature, and pH 9. Thermodynamic studies of the extraction of U(VI) metal ions with Aliquat-336/kerosene were also performed. The thermodynamic parameters such as ΔG, ΔH, and ΔS were calculated. The ΔH of uranium (VI) equals 26.075 KJ/mol. The positive value of ΔH indicates that the extraction of U(VI) in the investigated system is an endothermic process, with a decrease in the randomness of the system shown by the negative value of the entropy change (ΔS). The obtained results show that the extraction rate of U(VI) by Aliquat-336/kerosene from selective carbonate leach liquor is accelerated by the increase in the extractant concentration. The construction of the McCabe–Thiele diagram indicates that the number of theoretical stages required for U(VI) extraction is four stages, four stages were achieved for stripping uranium(VI) with 1.0 mol/l H₂SO₄ at an A/O phase ratio of 1 for 5 minutes. The complex formation between U(VI) metal ions and Aliquat-336/kerosene would take place by the reaction of uranium metal ions and sulfate ions of the extractant, as confirmed by FTIR analysis. Finally, it has to be indicated that the final obtained U(VI) product from the selective carbonate leach liquor of hydrous oxide cake from Egyptian monazite by Aliquat-336/kerosene has attained a purity of 95.8% and 96.6% when using H₂O₂ and NH₄OH as the precipitating agents, respectively, and a proposed flow sheet has been constructed for U(VI) extraction from hydrous oxide cake of Egyptian monazite.

**Figure 11.** Proposed flow sheet for the separation of U(VI) from hydrous oxide cake of Egyptian monazite.

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