Separation of Uranium from Rare Earths using Tertiary Amine from Sulfate Solutions

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Abstract

A sulfate leach liquor of 0.78 g/l uranium and 0.57 g/L rare earths produced from Abu-Zeniema wastes was subjected to different extraction experiments using tri-octyl amine as a solvent. Complete extraction of uranium from rare earths sulfate solutions was performed using a sulfate leach liqueur of pH 1.25, 10% TOA in kerosene, 1/20 organic to aqueous phase ratio and stirring for three minutes at room temperature. The saturation capacity of 14.0 g U/L was performed after three contacts between sulfate leach liquor and TOA organic solvent. The optimum uranium stripping conditions from the saturated TOA solvent were performed using sodium carbonate as a stripping agent with 0.75M molarity, 3 minutes contact time and 1/4 aqueous to organic ratio. Three stages of contacts counter currently were required for uranium stripping from the loaded organic using the optimum stripping conditions. A strip solution of 47.6 g U/L was obtained by two contacts between a fresh saturated solvent and the same carbonate strip solution. A fine grained uranium peroxide cake with 65.1% U was attained from the stripping solution by hydrogen peroxide precipitation at pH 2.

Keywords: Uranium; Rare earths; Tri-octyl amine; Extraction; Stripping.

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1. Introduction

Recently, the demand for uranium and rare earths has increased significantly, driving the development of new processes to recover and recycling uranium and rare earths from industrial wastes and un-convent resources [1-5]. Strenuous efforts were performed upon the mineralizations of Abu-Zeniema area, Sinai, Egypt, to recover its uranium contents [6-8]. A carbonate treated wastes were collected from several hydrometallurgical processing batches applied on Abu-Zeniema mineralizations. These wastes were subjected to sulfate leaching processes in order to dissolve uranium and rare earths content in one step (The paper is being published by the same authors), the sulfate leach liquor was considered the primal solutions to apply the uranium extraction processes.

The separation of U (VI) using amine compounds from rare earths in sulphate solutions was succeeded and studied extensively [9]. All amine compounds including primary, secondary and tertiary amines and quaternary amine salts have been widely studied. However, tertiary amine has very high selectivity for the extraction of U(VI) over REEs(III), it was still commonly recommended in the uranium industry. Alamine 336 (tri-octyl/dodecyl amine), Alamine 304 (tri-dodecyl amine), Alamine 308 (tri isooctyl amine) and Adogen 364 (similar to Alamine 336) were commercial tertiary amine regents. Several studies have been carried out and a number of new processes have been developed using tertiary amines and primary amines to separate uranium from rare earths in acidic sulphate leach solutions [10, 11]. Secondary amine compounds may also extract and separate uranium from rare earths [12]. The selectivity of uranium was significantly poorer with secondary amines than using tertiary amines. There are other disadvantages such as poor performance resulted from strong association with phosphate and significant adverse effect of alcohol modifier. Therefore, secondary amines have not gained high interest for this purpose. As rare earths are also extracted by primary amine from sulphuric acid solutions [13, 14], tertiary amines are the most significant extracting amines for uranium from rare earths sulfate solutions [15].

The present work deals with the recovery of uranium from U-REEs solutions obtained from sulfate leaching of Abu-Zeniema wastes while the total rare earths was still in the raffinate solutions. The recovery of uranium was occurred by applying a conventional extracting process using tri-octyl amine. The extraction and stripping processes of uranium were herein studied in details, and an acceptable yellow cake was also produced from the stripping solutions.

Accordingly, three points were achieved namely; increasing the process environmental impact, recovering of its uranium content and leaving a raffinate solution containing the total rare earths.

2. Experimental

2.1. Feed solution preparation

The feed solution was prepared by leaching a representative sample of the Abu-zeniema wastes using 3M sulfuric acid concentration; 15 min contact time; 1/10 solid/liquid ratio (w/v) and 500 rpm agitation speed at 90°C. Using the optimum leaching conditions, complete dissolution for uranium and about 97.3% dissolution efficiency for REE were obtained. The using of highly acid concentration and ratio were due to the alkaline

nature of the uranium wastes, where these solid wastes were collected after several uranium processing batches using carbonate leaching technique. This leach liquor was used as the feed solution for the subsequent solvent extraction study.

2.2. The Recovery procedure

- The organic phase was prepared by dissolving tri-n-octyl amine (TOA) in the relevant diluent and pre-equilibrated by shaking three times with sulphuric acid of a concentration similar to that present in the leach liquor to convert the amine to the hydrosulphate form.

- 2-ethyl-1-hexanol was used as a modifier and added to the solvent with 10 % fixed amount , in order to avoid the production of a third liquid phase, which may often occur when tertiary amines are contacted with mineral acids [16], due to the formation of salts insoluble in the organic phase during extraction.

- Kerosene was used as diluents because it is produced locally in Egypt, making it accessible for all and with good price.

- In each extraction experiment, the organic phase was contacted with the aqueous phase in 250 ml glass beaker where they were mixed together for the desired time using a hot plat magnetic stirrer (Fisher Scientific model) and all experiments were performed in duplicate. The hydrogen ion concentrations and the temperature of the feed solutions were adjusted using pH of HAANA instrument.

- The two phases were then allowed to settle, separate and an aliquot sample of the aqueous phase was analyzed for its remaining metal content in order to calculate the uranium and rare earths extraction efficiencies.

$$\text{Metal Extraction efficiency, \%} = \frac{\text{Extracted Metal concentration}}{\text{Original Metal concentration}} \times 100$$

- The relevant solvent extraction factors (Table 1); namely the pH of the feed solution, Contact time, solvent concentration, organic/aqueous ratio (O/A) and extraction temperature were studied. In addition, number of contacts between aqueous and organic using fresh aqueous was carried out to calculate the saturation capacity of the solvent.

- On the other hand, a loaded solvent sample was prepared for studying the stripping behavior of uranium using sodium carbonate solution as a stripping solution. The relevant stripping factors including molarity of the sodium carbonate, contact time and A/O ratio were studied.

2.3. Analytical procedure

- Total rare earths were analyzed by Arsenazo III where the absorbance of their complex was measured at the wavelength 650nmusing UV-spectrophotometer "single beam multi-cells-positions model SP-8001, Metretech Inc., version 1.02, with glass cell of 10 mm length [17].

- Uranium was determined by titration against ammonium meta vanadate [18].

- The feed solution elemental analysis was accomplished using inductively coupled plasma optical emission spectrometer (Prodigy Axial high dispersion ICP-OES – USA).

Finally, complete uranium precipitation in peroxide form (as uranium peroxide cake $UO_4.2H_2O$) was carried out from the strip solutions using hydrogen peroxide. After that, a rare earths oxalate cake was attained by selective precipitation from the effluent solutions produced through the uranium extraction step.

Table 1: The studied extraction and stripping factors

	Factor	Variation range	Fixed conditions
Extraction process	pH of the feed solution	0.5, 0.75, 1.0, 1.25, 1.5	O/A =1/1, 10% TOA, 5 min., 25 ^{o}C,500 rpm
	Contact time, min.	1, 2, 3, 4, 5	O/A =1/1, 10% TOA, pH 1.25, 25 ^{o}C,500 rpm
	solvent concentration TOA, %	2.5, 5, 7.5, 10, 15	O/A =1/1, pH 1.25, 3 min., 25 ^{o}C,500 rpm
	organic/aqueous ratio (O/A)	1/1, 1/2, 1/5, 1/10, 1/15, 1/20, 1/30	pH 1.25, 3 min., 10% TOA, 25 ^{o}C, 500 rpm
	Temperature, ^{o}C	25, 30, 40, 50, 60	pH 1.25, O/A =1/1, 10% TOA, 3 min., 500 rpm
Stripping process	Sod. carbonate molarity, M	0.125, 0.25, 0.5, 0.75, 1.0, 1.25, 1.5	3 min, A/O = 1/1, 25 ^{o}C, 500 rpm
	Contact time, min.	0.25, 0.5, 1, 2, 3, 4, 5	0.75 M, A/O = 1/1, 25 ^{o}C, 500 rpm
	Aqueous/Organic ratio, v/v	2/1, 1/1, 1/2, 1/4, 1/8	0.75 M, 3 min., 25 ^{o}C, 500 rpm

3. Results and Discussion

3.1. Preparations of a sulfate leach liquor

Sulfate leach liquor was prepared from Abu-Zeniema wastes using the preferable leaching conditions of 3M

H2SO4; 15 min., 1/10 solid to liquid ratio and 500 rpm at 90°C. Complete dissolution for uranium and about 97.3% dissolution efficiency were carried out using the above favorable leaching conditions. Therefore, the produced feed solution of pH 1.5 assayed 0.78 g/L U and 0.57 g/L REEs as shown in Table 2.

3.2. Uranium extraction

According to the selectivity of tertiary amines compounds to extract uranium from rare earths sulfate solutions, the extraction of uranium by TOA in kerosene from U/REE sulfate leach liquor of Abu-Zenima waste and recovery of total rare earths from the effluent solutions which represented the aim of this study were herein studied.

Table 2: ICP-Elemental analysis for the sulfate feed solution

Element	U*	REE	Fe	Si	Cu	Cr	Mg	Ca	K	Na	Al
Concentration, g/L	0.78	0.57	58.2	7.09	1.14	1.19	1.56	1.26	3.21	16.9	3.99

* Uranium was analyzed using titration method

3.2.1. Effect of the leach liquor pH

To study the effect of feed pH on the extraction efficiency of uranium and rare earths, several experiments with ranged pHs of feed solutions from 0.5 to 1.5 were carried out using solvent of 10% TOA and 10% 2-ethyl-1-hexanol as a modifier dissolved in kerosene as a diluent. The extraction results for U and REE were graphically given in Figure 1.
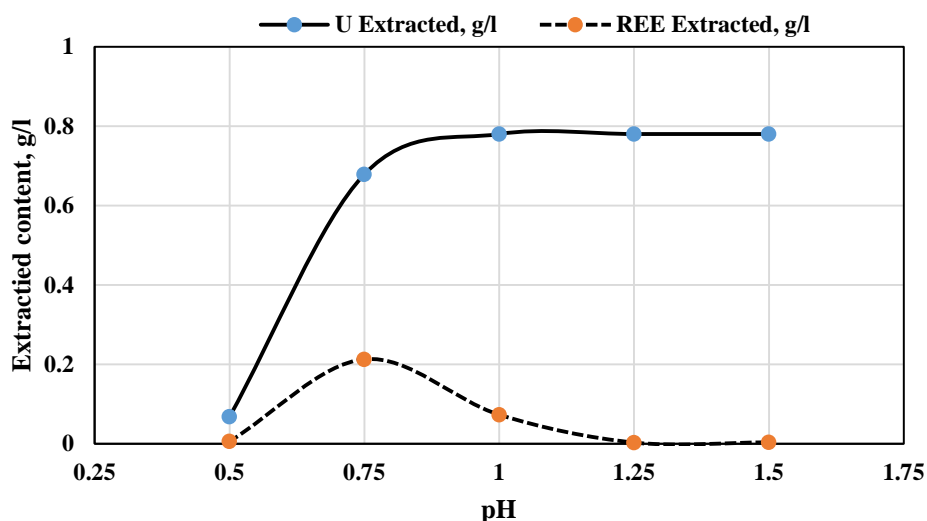

Figure 1: Effect of the feed solution pH on the extractability of U and REE

(A/O=1/1, 10% TOA, 5 min., 25 $^{\circ}$C, 500 rpm)

From the obtained results, the uranium extraction was sharply increased by increasing the pH from 0.5 to 1.0. Complete extraction for uranium was achieved at pH ranged from 1.0 to 1.5. However, it is clearly evident that the aqueous phase pH value of 1.25 was the preferable pH value which achieved maximum extraction for uranium and the total rare earths remained in the effluent solution.

3.2.2. Effect of contact time

The contact times ranged from 1 to 8 minutes and their effect on the extraction of U and REEs from the solid waste sulfate solution were studied to determine the equilibrium time at which maximum uranium recovery occurred with the least amount extracted from rare earths. The extraction results were given in Figure 2.
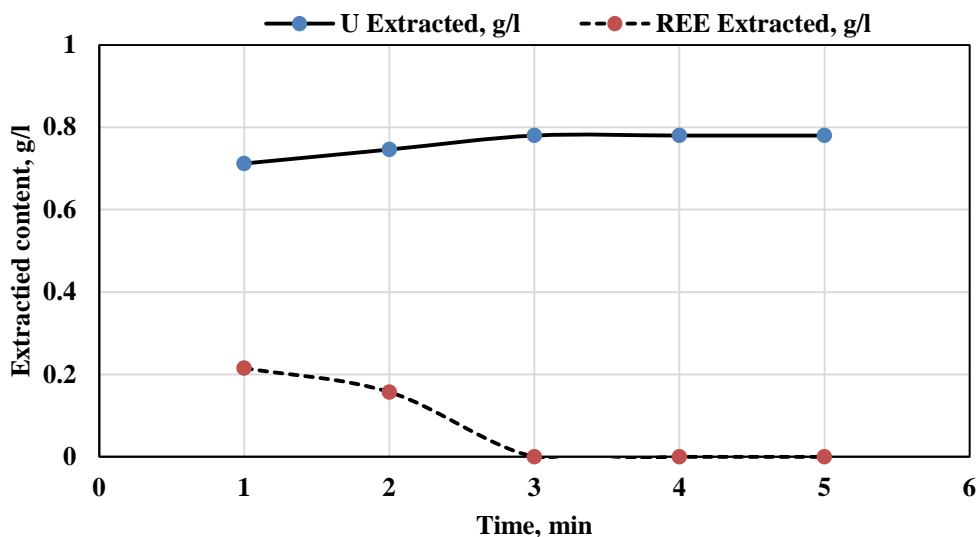
Figure 2: Effect of contact time on the extractability of U and REE

(1/1, 10% solvent, pH 1.25, 25 ^{o}C, 500 rpm)

By prolonging the contact time from 1 to 3 minutes, the extraction of uranium and rare earths were enhanced from 0.71 to 0.78 g/L and decreased from 0.2 to 0.0 g/L respectively. However, no enhancement in the extraction of uranium obtained by increasing the contact time over 3 minutes. So, three minutes was the more convenient time at which all uranium content shifted from sulfate phase to the organic phase and leaving rare earths in the aqueous effluent solutions.

3.2.3. Effect of solvent concentration

The effect of TOA concentration on extraction of U and REE from the sulfate solution was studied in the range from 2.5 to 15 %TOA dissolved in kerosene. The results of U and REE extraction efficiencies were given in Figure 3.
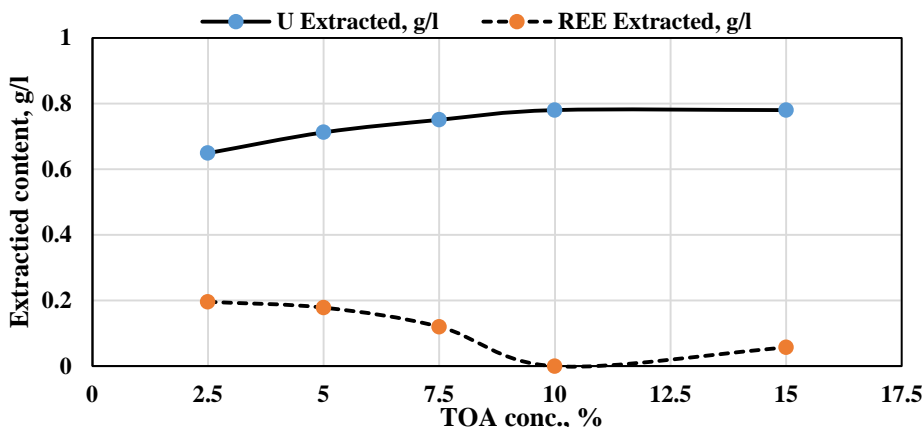
Figure 3: Effect of TOA concentration on the extractability of U and REE

(pH 1.25, O/A = 1/1, 3 min., 25 oC, 500 rpm)

From these results, it is obvious that uranium extraction enhanced from 0.65 to 0.78g/L and REE extraction decreased from 0.2 to 0.0g/L by increasing the TOA concentration from 5 to 10%. However, the augmentation in the solvent concentration over 10% resulted in withdrawing the rare earths through the organic phase which caused complications in the uranium separation process later.

3.2.4. Effect of organic to aqueous phase ratio

To study the organic to aqueous phase ratio (O/A) effect, a series of experiments were carried out with O/A ranged from 1/1 to 1/30 under conditions of pH 1.25, 10% TOA in kerosene and 3 minutes contact time at ambient temperature. The extraction results were given in Figure 4. The results of O/A ratio effect revealed that the uranium extracted by the solvent increased gradually from 0.78 to 6 g U/l by changing the ratio from 1/1 to 1/10 which considered the inversion point with the highest distribution coefficient. On the other hand, the extractability of rare earths was enhanced over zero by using different O/A ratios of 1/5 and 1/10. So, high ratios (1/15, 1/20, 1/30) were considered the more convenient ratios, especially the 1/20 ratio which achieved the maximum extractability of uranium (7.6gU/L) without any rare earths.
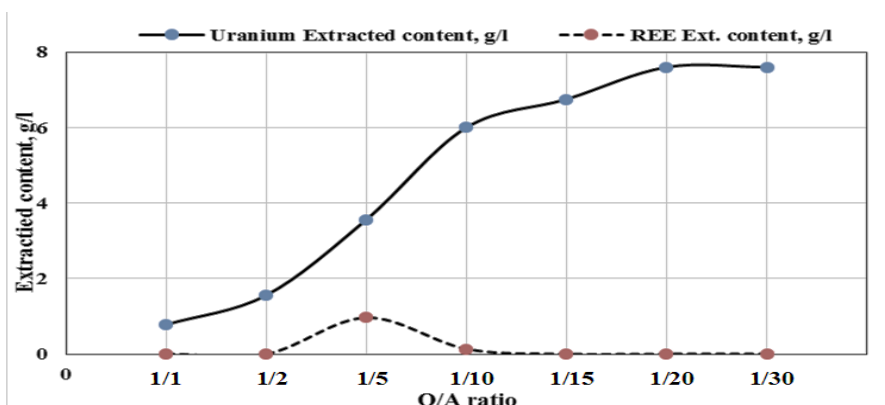

Figure 4: Effect of organic to aqueous on the extractability of U and REE

(pH 1.25, 3 min., 10% solvent, 25 ^{o}C, 500 rpm)

3.2.5. Effect of temperature

The effect of the reaction temperature on the extraction of uranium and rare earths was studied in the range between 25 to 60 ^{o}C. The extraction results of U and REE were given in Figure 5.

From the obtained data, it was observed that a sharp decrease in the uranium extraction from 7.6 to 2.1gU/L when the temperature increased from 25 to 60 ^{o}C. So, increasing the reaction temperature was considered an inhibiting factor for the uranium extraction process.

There was no significant difference in extraction of rare earths by raising the extraction temperature. Therefore, it was better to perform the extraction at room temperature.
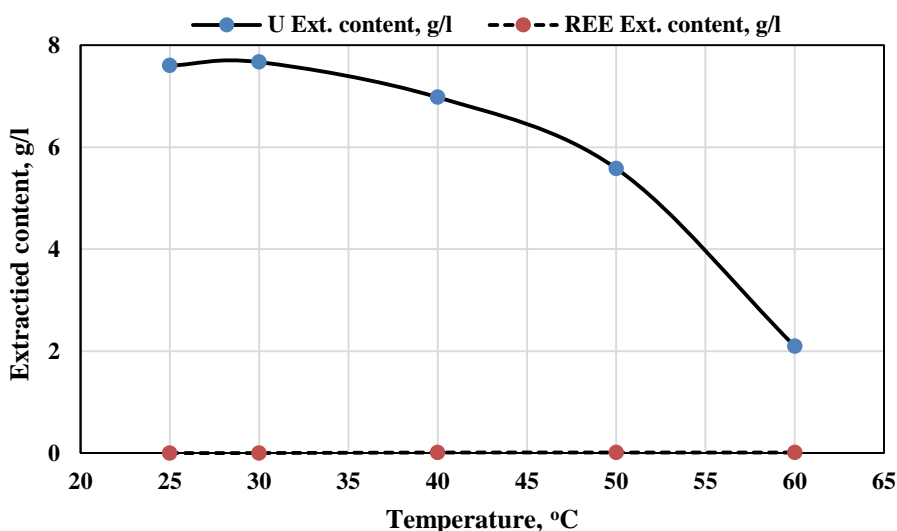

Figure 5: Effect of temperature on the extractability of U

(pH 1.25, O/A=1/20, 3 min., 10% solvent, 500 rpm)

3.2.6. Saturation capacity of the TOA solvent

Several contacts between organic solvent and sulfate leach liquor were carried out to determine the saturation capacity of the organic solvent; this was performed using the resulted optimum conditions of O/A ratio of 1/20, pH 1.25, 10% TOA in kerosene with 3 minutes contact time at ambient temperature. These successive tests were performed between fresh aqueous leach liquor and the same organic solvent in the all contacts.

From the results which graphically plotted in Figure 6, it was found that the saturation capacity of the solvent reached to 14.0gU/L, this was performed after 3 contacts under the previously mentioned operating conditions.
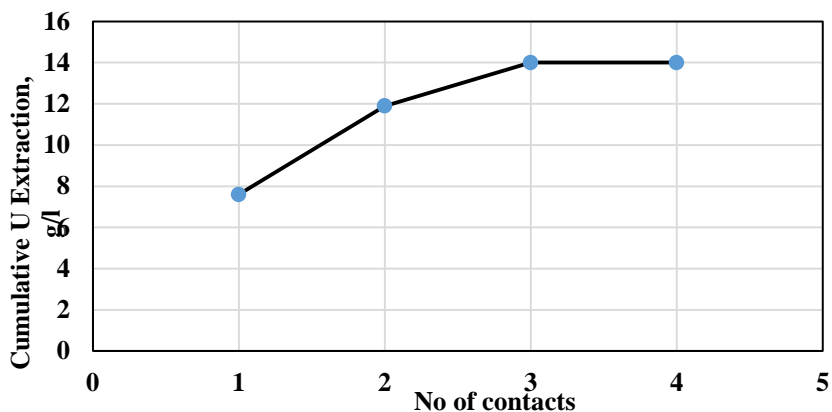
Figure 6: Saturation capacity of the TOA solvent

(pH 1.25, 3 min., 10%TOA, 1:20 O/A ratio at 500 rpm)

From these results and constructing the McCabe-Thiele diagram it was predicted that if uranium extraction process carried out counter currently on a pilot scale this require 3 stages under the operating conditions of contacting the aqueous phase of concentration of 780 ppm U with O/A ratio of 1/20, pH 1.25, 10% TOA in kerosene with 3 minutes contact time at ambient temperature. This would produce a raffinate of 5 ppm as illustrated in Figure 7.
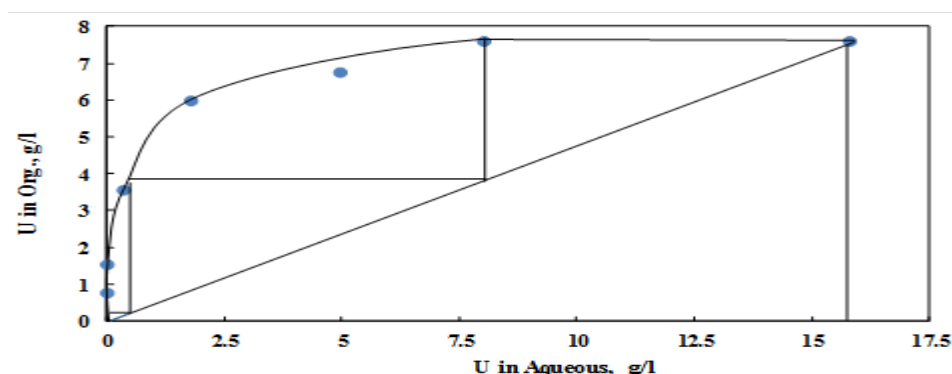

Figure 7: The equilibrium isotherm for the U extraction from the sulfate leach liqueur

(pH 1.25, 3 min., 10%TOA, 1:20 O/A ratio at 500 rpm)

Finally, the effluent solution which obtained after the uranium extraction processes, were riches with the total rare earths content without any uranium content. These effluents were collected and would be subjected to further later studies on the rare earths recovery.

3.3. Uranium Stripping

A saturated TOA solvent sample of 14gU/L was prepared by applying the preferable studied conditions as previously mentioned, several stripping tests for uranium from the saturated TOA solvent were carried out using

sodium carbonate solution as a stripping solution.

3.3.1. Effect of sodium carbonate molarity on uranium stripping efficiency

Different sodium carbonate molarities (from 0.125 to 1.5M) were tested to determine their effect on the uranium stripping. The results of uranium stripping efficiency were plotted in Figure 8.
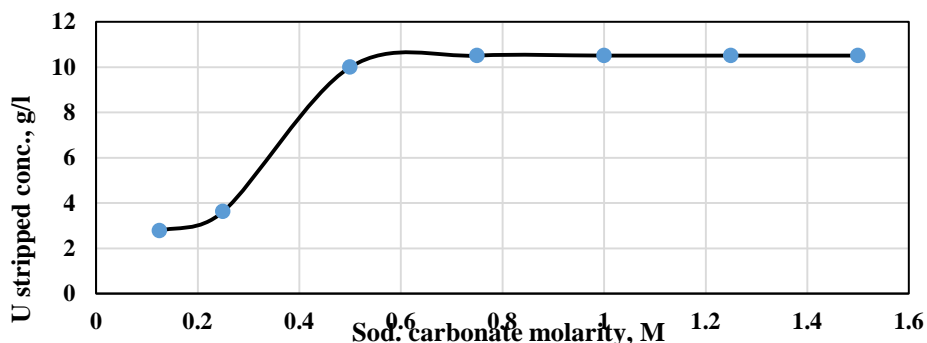

Figure 8: Effect of concentration of Na_2CO_3 on U stripping efficiency

(3 min, A/O = 1/1, 25 ^{o}C, 500 rpm)

By increasing the sodium carbonate molarity from 0.125 to 0.75M the uranium stripping efficiency was increased from 2.8 to 10.5gU/L, and there wasn't any enhancement in the stripping efficiency over 0.75M Na_2CO_3.

3.3.2. Effect of contact time on uranium stripping efficiency

The contact time between aqueous and organic phases for the stripping process was studied from 0.25 to 5.0 minutes to determine the required time to attain the equilibrium. Results of contact time effect were graphically represented in Figure 9. The results revealed that uranium stripping efficiency was rabidly increased to reach 10.5gU/L using 3 minutes and there weren't any enhancement in stripping efficiency using 4 or 5 minutes. So, three minutes contact time was considered the most efficient time.
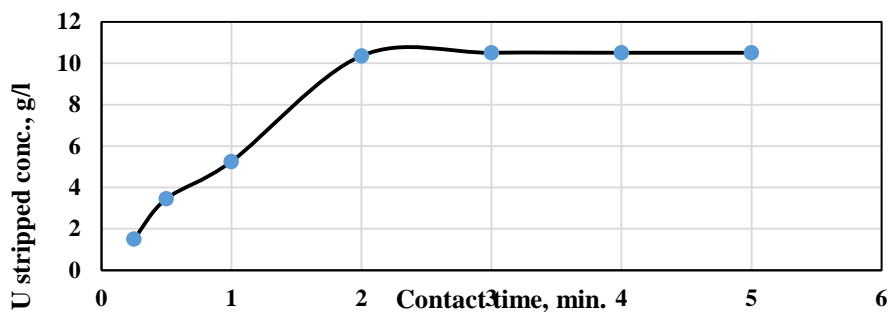

Figure 9: Effect of contact time on uranium stripping efficiency

95

(0.75 M, A/O = 1/1, 25 ^{o}C, 500 rpm)

3.3.3. Effect of aqueous to organic phase ratio

The effect of aqueous to organic A/O phase ratio on uranium stripping efficiency was studied by using ranged ratios from 2/1 to 1/8 under the conditions of 0.75M Na_2CO_3 and agitated the two phases for 3 minutes at ambient temperature. The results of uranium stripping efficiency were plotted in Figure 10.

It noticed that the uranium stripping was increased by increasing the A/O ratio from 2/1 to 1/4 to reach to 39.2gU/L in the aqueous phase and there wasn't any development in the uranium stripped concentration by increasing the phase ratio above 1/4.

3.3.3. Equilibrium isotherm for the U stripping process

Equilibrium isotherm for uranium stripping from the loaded TOA solvent was studied to evaluate the stripping process. Several contacts between loaded organic solvent and the aqueous phase using different A/O ratios (2/1, 1/1, 1/2, 1/4 and 1/8) were carried out under the conditions of 0.75M Na_2CO_3 and agitated the two phases for 3 minutes at ambient temperature. The results were represented in Figure 11.
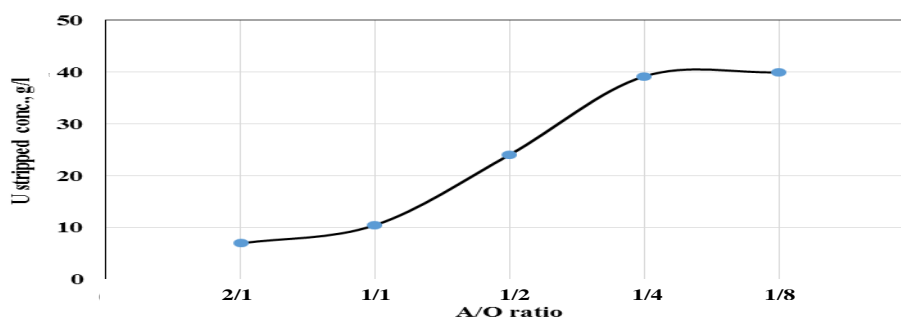

Figure 10: Effect of aqueous to organic ratio on uranium stripping efficiency

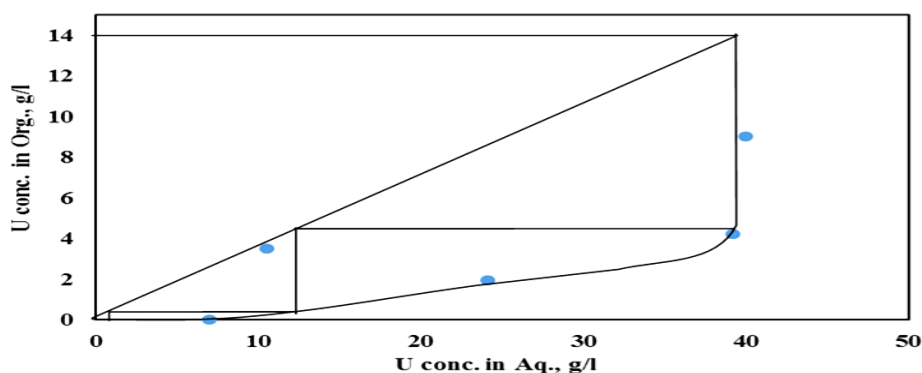(0.75 M, 3 min., 25 ^{o}C, 500 rpm)

Figure 11: Equilibrium Isotherm of uranium stripping from TOA loaded solvent

(0.75 M, 3 min., 25 ^{o}C, 500 rpm)

From the results and the construction of McCabe-Thiele diagram it is clear that if uranium stripping process was performed counter currently, then three stages were sufficient to recovery the uranium from the loaded solvent. On the other side, using fresh saturated organic solvent in all contacts with the same aqueous stripping solution and the optimum stripping conditions, two contacts were enough to reach maximum concentration of uranium in the aqueous phase (47.6 gU/L) and there wasn't any enhancement in uranium concentration after the second contact as shown in Figure 12.
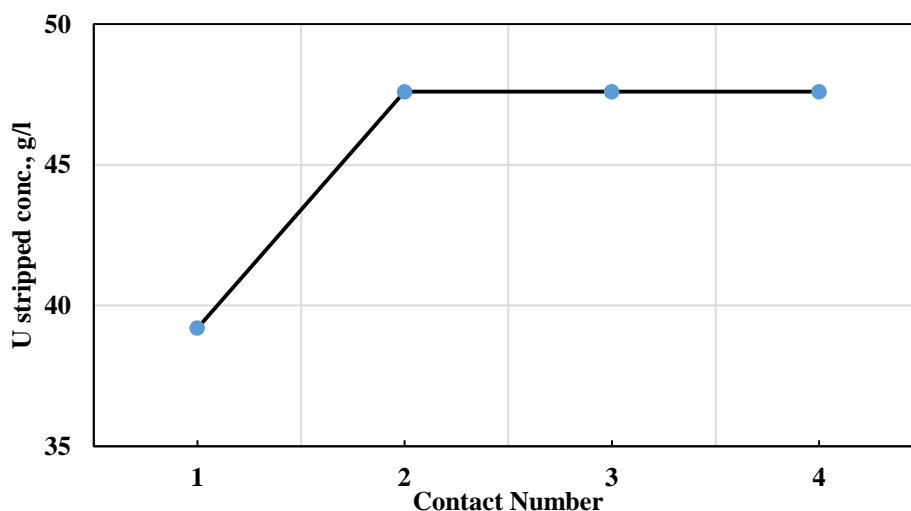

Figure 12: Effect of contact number on U stripping efficiency

(0.75 M, 3 min., A/O ratio 1/4, 25 ^{o}C, 500 rpm and fresh loaded organic)

3.4. Uranium Precipitation from Strip Solution

The carbonate stripping solutions were collected and the pH was reduced from 10 to 2 using diluted sulfuric acid. These solutions were subjected to complete uranium precipitation using hydrogen peroxide. A fine grained lemon yellow cake concentrate was obtained from the hydrogen peroxide precipitation of the strip solution at pH 2.0. The obtained uranium concentrate, produced from Abu-Zeniema wastes using TOA solvent, was chemically analyzed using titration method and X-ray fluorescence analytical instrument at Analytical Lab in Egyptian Atomic Power Agency to determine and confirm its constituents. From Figure 13 and Table 3, it is clear that the prepared uranium peroxide concentrates has been accepted specifications from the nuclear point of view according to the international limits. Finally, a fine grained lemon yellow cake concentrate ($UO_4.nH_2O$) of about 65.1% U was attained from the hydrogen peroxide precipitation of the strip solution.

4. Conclusion

A sulfate leach liquor assaying 0.78 g/L U and 0.57 g/L REEs was subjected to several experiments in order to selective extraction of the uranium content from rare earths sulfate leach liquors. Tri-octyl amine was used as a

solvent in the uranium recovery process.

Using the optimum conditions of 1/20 O/A ratio, pH 1.25, 10% TOA in kerosene with 3 minutes contact time at ambient temperature, the saturation capacity of the solvent reached 14.0 gU/L. Three contacts were also needed to complete extraction of the uranium content from the aqueous leach liquor using a counter current manner under the optimum conditions
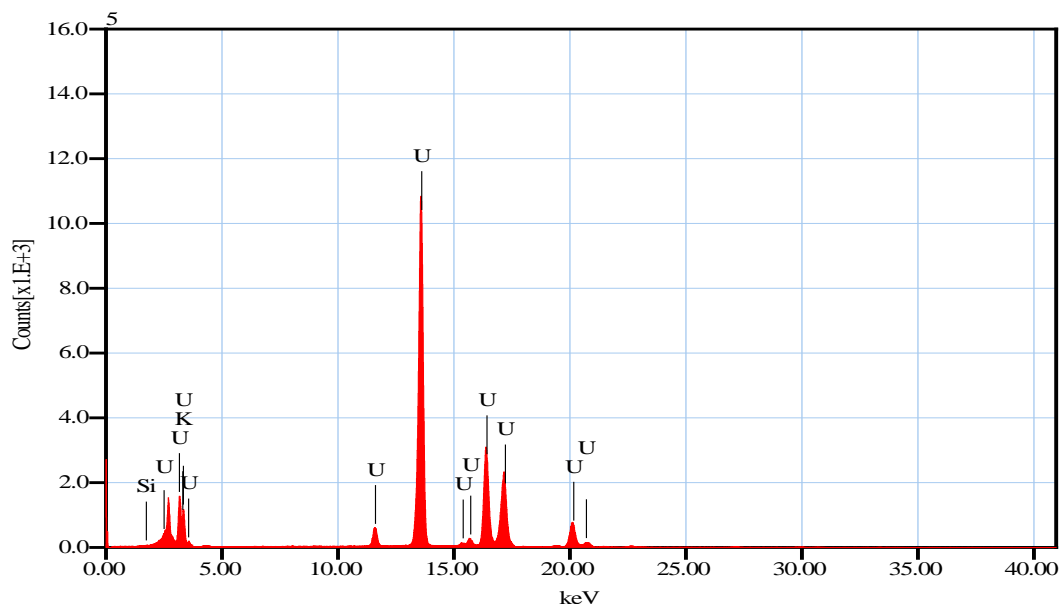

Figure 13: XRF-chart for uranium peroxide end product

Table 3: XRF- Elemental analysis for the uranium peroxide end product

Elemental oxide	ms%
U_3O_8	99.37
K_2O	0.08
SiO_2	0.55

A saturated TOA solvent sample of 14 gU/L was contacted with sodium carbonate as a stripping agent under the optimum stripping conditions of 0.75 M Na2CO3 molarity, 3 minutes contact time, 1/4 O/A ratio, 25 oC and 500 rpm. Three stages of contacts in a counter current were required for complete uranium stripping process from the loaded organic solvent using the optimum stripping conditions. Two contacts were enough to reach maximum concentration of uranium in the aqueous phase (47.6 gU/L) using a fresh saturated solvent in all contacts and the same carbonate aqueous solution.

Accepted fine grained lemon yellow cake concentrate of about 65.1% U was attained from the hydrogen

peroxide precipitation of the strip solution at pH 2.

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