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Extraction of uranium from el-Sella mineralization south eastern desert, Egypt by tri-Octylamine

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ABSTRACT

A uranyl sulfate leach liquor obtained by uranium leaching of El-Sella sample deposits ore was subjected to uranium extraction using the liquid–liquid technique. Uranium was effectively extracted from sulfate leach liquor by [(10 %) tri-n-octylamine (TOA)] dissolved in benzene as a diluent. The extraction efficiency was markedly enhanced as the concentration of TOA increases from 1 to 10 %. The relevant factors controlling the extraction process of uranium using tri-n-octylamine were studied. These factors include the effect of TOA concentration, contact time, and phase ratio (O/A) v/v. The optimum extraction conditions were chosen. More than 98 % of uranium was extracted by 10 % TOA, at contact time 15 min, phase ratio (V_O/V_A) 1/1 and at room temperature. The feasibility of using the TOA for preconcentration-separation of uranium was assessed by stripping studies. The loaded uranium onto TOA has been stripped by 100 % when using 5 % Na_2CO_3 as an efficient stripping agent at 15 min contact time, and phase ratio (O/A) 1/2. Uranium also stripped using 15% (v/v) sulfuric acid, 15 min contact time and phase ratio (O/A) 2/1.

INTRODUCTION

Uranium needs are going very fast in new millennium life styles. Uranium is considered as the main source to generate the atomic power as cheap and more quantity of the electricity can be generated to full fill the country demands. For recovery of the precious and rare metals through hydrometallurgical treatments, there is the possibility to develop environmental friendly methods and the vast amount of ongoing research worldwide underline the enormity of interest in hydrometallurgy. The hydrometallurgical operations involve many techniques, one of the main and important of them was solvent extraction processing. The technique of solvent extraction (also called liquid-liquid extraction) has played most important role in analytical, separation science as well as environmental sciences and has been used since long time (since 1842). For the extraction and separation of metals from various sources, this technique is the very simplest, easily handled and economically cheapest technique when compared with other analytical and separation techniques.

Amides/amines are the main classes of nitrogen based compounds used for uranium extraction and separation technology. A great number of amides[1-6] and amines[7-12] was used for the uranium extraction from various sources. The distribution ratios for the extraction of uranium and thorium show second- and third-order dependences, respectively, on the extractant concentration for both the N-alkyl and N,N-dialkyl amides.5 The results of extraction study suggested the formation of the 1:2:1 uranyl(II) ion, nitrate ion and N,N,N,N-tetrabutylsuccinylamide complex as extracted species.6 Extraction of uranium and separation of thorium/fission products from hydrochloric acid solutions using tri (iso-octyl)amine as extractant were investigated.7 The extraction of uranium(IV) from aqueous sulfuric acid media by tri-octylamine (TOA) in ben-

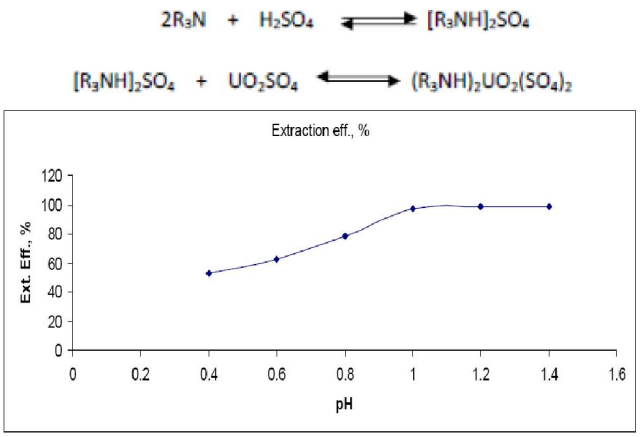

Figure 1 : Effect of pH on uranium extraction efficiency from the leach liquor of El-Sella composite sample by 0.22M TOA (in benzene)

zene was studied as a function of various experimental parameters.8 The recovery of uranium from acid heap leach liquor using tertiary amines (tri-n-octylamine) chosen as extractants because of their high selectivity and efficiency was investigated.9

A tertiary amine, trioctylamine (TOA), for prior uranium extraction from properly prepared acidic leach liquor (agitation leaching) of El Sella mineralization composite sample. Uranium would thus be extracted as its neutral (or anionic) complex while the REEs left behind in the leach liquor would subsequently recovered by using the di (2-ethylhexyl) phosphoric acid (DEHPA) solvent.

Accordingly, a stock leach liquor of the study El Sella mineralization composite sample was prepared by applying the optimum leaching conditions previously studied These included 5% v/v sulfuric acid, 1/2 solid/liquid ratio, and the leaching was performed for 4 h agitation time at a temperature 90^0 C. Analysis of the obtained leach liquor (pH 1) revealed an assay of 160 ppm for uranium.

EXPERIMENTAL

Chemicals and reagents

All the chemicals used in this work were of analytical grade (A.R.),

Control analysis

Uranium analysis was spectro-photometrically determined using Arezenazo-III at 655 nm.[10] In the mean time, this analysis was confirmed by the oxidemetric volumetric determination of uranium using ammonium metavanadate. This procedure is based on the titration U^{+4} with ammonium metavanadate NH_4VO_3; namely

$U^{+4} + 2NH_4VO_3 + 4H^+ = UO_2^{+2} + 2VO^{+2} + 2NH_4^+ + 2H_2O$ Thus, uranium in solution should first be transformed into U^{+4} and for this purpose ferrous sulfate is used

Experimental procedure

In the present work, the extraction of uranium from El-Sella leach liquor that has been leached by 5% (v/v) sulfuric acid at 90^o c for 4 hrs with solid liquid ratio 1:2 and assay 160 ppm uranium has been studied through solvent extraction by tri-octylamine

The chemical composition of El-sella prepared leach liquor is shown in TABLE 1

Uranium extraction using TOA

All the extraction experiments was performed by mixing equal volumes of the leach liquor and the solvent (at room temperature) then the two phases separated from each other using separating funnel after settled down for few min. Then uranium was analyzed in the aqueous phase to calculate the extraction efficiency while that in organic phase was calculated by difference. The extraction factors studied involved the TOA concentration, the shaking time and the A/O phase ratio.

TABLE 1: chemical composition of El-Sella leach liquor

Constituent	g/l
UO_2^{+2}	0.160
RE_2O_3	0.3
SO_4^{-2}	0.09
Fe_2O_3	7.9
CaO	0.76
MgO	0.38
P_2O_5	0.43

Uranium stripping

Uranium was stripped from the obtained loaded amine (TOA) solvent using sulfuric acid and sodium carbonate as stripping agents. Uranium concentration was determined in the aqueous phase after each contact, and the corresponding concentration in the organic phase was calculated by difference. The stripping factors studied involved the sulfuric acid and sodium carbonate concentrations, the shaking time and the A/O phase ratio.

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RESULTS AND DISCUSSION

Results of Uranium Extraction Parameters by TOA

Effect of pH Value upon Uranium Extraction

In order to study the effect of pH value of the working El–Sella acidic leach liquor upon the extraction efficiency of uranium with TOA, a series of extraction experiments was performed using different pH values ranging from 0.4 up to 1.3. For this purpose, different aliquots of the working acid leach liquor were either acidified with H_2SO_4 or treated with 10 % NaOH solution to the required pH values. The other extraction conditions were fixed at an O/A ratio of 1/1 and using 0.22 M % TOA concentration for 15 min. shaking time at room temperature. The obtained results are summarized in TABLE (1) and plotted in Figure (2).

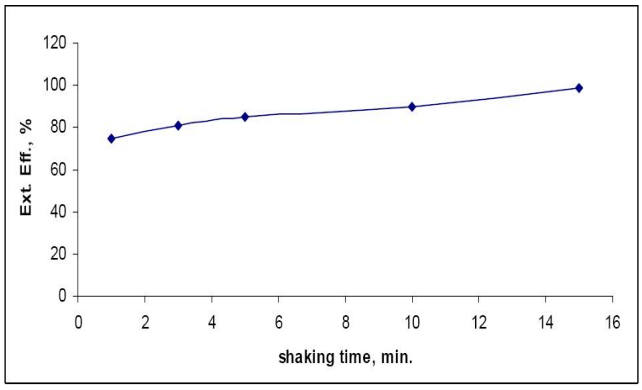

Figure 2 : Effect of shaking time upon uranium extraction efficiency from the leach liquor of El Sella composite sample

From the obtained results, it is clearly obvious that decreasing the acidity of the original leach liquor resulted in increasing the extraction efficiency of uranium. Thus, in the first experiment of the high acidity (pH 0.4), the extraction efficiency was 53 % while the highest uranium extraction efficiency (98.75 %) was obtained at the lowest studied acidity of pH 1.4. It can thus be concluded that the uranium extraction efficiency of 98.75% obtained at the pH value of 1.0 would be considered as the optimum value.

composite sample by 0.22M TOA (in benzene)

Effect of Shaking Time upon Uranium Extraction

The effect of the shaking time upon uranium extraction efficiency from the acid leach liquor of El Sella mineralization composite sample by TOA (in benzene) was studied by performing a series of extraction experiments using different shaking times ranging from 5 up to 15 min. In these experiments, the other extraction conditions were fixed at 0.22 M (10%) TOA concentration (in benzene) and an O/A phase ratio of 1/1 and the experiments were performed at room temperature.

From the obtained results given in TABLE (2), it can be observed that increasing the shaking time from 1 to 3 min. resulted in a slight increase in the extraction efficiency of uranium from 75% to 81.2%. Using 15 min. shaking time resulted in an extraction efficiency of more than 98%

Effect of TOA Concentration upon Uranium Extraction

TABLE 2 : Effect of pH upon uranium extraction efficiency from the leach liquor of El-Sella composite sample by 0.22 M TOA (in benzene)

pH	Conc, ppm		Distribution Coefficient (D_a^o)	Extraction eff., %
	Aq.	Org.		
0.4	75.2	84.4	1.1	53
0.6	60	100	1.7	62.5
0.8	35	125	3.6	78.12
1.0	4	156	39	98.75
1.2	2	158	79	98.75
1.4	2	158	79	98.75

In order to study the effect of TOA concentration upon uranium extraction efficiency from the study leach liquor, a series of extraction experiments was performed using TOA (in benzene) in various concentrations varying from 0.022 up to 0.22 M (1 up to 10 %). The other extraction conditions were fixed at 1.0 pH of the leach liquor, an O/A phase ratio of 1/1 and 15 min shaking time at room temperature. The obtained results are shown in TABLE (3) and plotted in Figure (3).

From the obtained results, it is clearly obvious that at 0.22 M TOA (10%) concentration, 98.75 % of uranium present in the study leach liquor was extracted corresponding to a distribution coefficient of 79.

Effect of O/A Phase Ratio upon Uranium Extraction -Construction of McCabe Thiele Extraction Diagram

In solvent extraction, equilibrium conditions would not allow the extraction of most of the metal species in one contact except if the O/A volume ratio is adequately high. In other words, multiple contacts would be necessary or rather a countercurrent flow of the organic and aqueous phases. The latter would indeed lead to a

TABLE 3 : Effect of shaking time upon uranium extraction efficiency from the leach liquor of El Sella composite sample by 0.22 M TOA in benzene

Shaking Time, min	Conc, ppm		Distribution Coefficient (D_a^o)	Extraction eff., %
	Aq.	Org.		
1	40	120	3	75
3	20	130	6.5	81.2
5	24	136	5.6	85
10	16	144	9	90
15	1.5	158.5	105.5	98.75

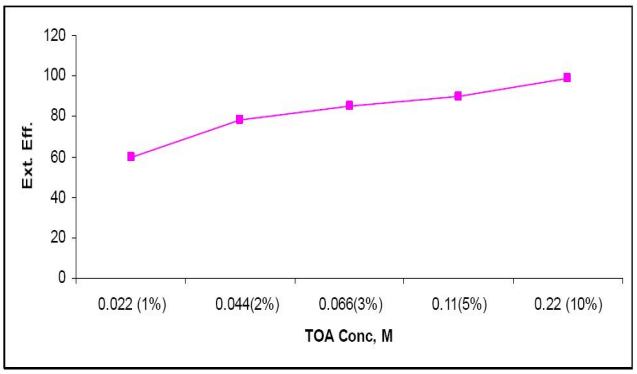

Figure 3 : Effect of TOA concentration (M) upon uranium extraction from the Leach liquor of El Sella mineralization composite sample

maximum possible loading of the organic phase at one end and a maximum possible depletion or exhaustion of the aqueous phase at the other end. The required number of theoretical stages that would realize this requirement is generally obtained by construction of the corresponding McCabe-Thiele diagram (Ritcy and Ashbrook)[14]. In this diagram, the equilibrium curve is first constructed and to which a suitable operating line (whose slope represents the suitable O/A phase volume ratio) would be fitted. To realize this objective, it is necessary to obtain extraction equilibrium data at various concentration levels. Therefore, a series of equilibrium experiments were performed using different O/A phase ratios varying from 1/10 up to 2/1 between the working TOA solvent (in benzene) and the working El -Sella acid leach liquor. These extraction experiments were performed under fixed conditions of 0.22 M TOA/ K concentration, 1.0 pH value and using 15 min shaking time at room temperature. The obtained equilibrium data are shown in TABLE (4) and plotted in Figure (4) in the form of an equilibrium isotherm. To construct the McCabe-Thiele extraction diagram, an operating line having a slope equivalent to a flow rate A/O ratio was properly fitted to the obtained equilibrium isotherm. Accordingly, in a continuous countercurrent extraction system, 3 theoretical stages would be required to almost completely extract the uranium from the acid leach liquor feed of El Sella mineralization composite sample. The maximum obtained uranium saturation level of about 1.05 g/l in the organic phase matches indeed with the following mechanism shown previously; namely

From the Figure it is clear that a phase ratio of 5/1 A/O give an extraction efficiency of more than 92% which considered as optimum

Results of uranium stripping parameters

In order to study the stripping parameters of uranium from the working TOA solvent, sulfuric acid and sodium carbonate was chosen as a suitable stripping agent. Therefore, a loaded solvent sample was first prepared by repeated contacts of a suitable volume of the working TOA/benzene (0.22M) with fresh acid leach liquor samples till almost saturation using an A/O phase ratio of 1/1 and 15 min shaking time at room temperature. Uranium concentration in the prepared loaded TOA/ benzene solvent sample attained about 1.2 g/l.

TABLE 4 : Effect of TOA concentration (M) upon uranium extraction from the Leach liquor of El Sella mineralization composite sample

T.O.A conc., M	Conc, ppm		Distribution Coefficient (D_a^o)	Extraction eff., %
	Aq.	Org.		
0.022(1%)	64	96	1.5	60
0.044(2%)	35	125	3.57	78
0.066(3%)	8	152	19	85
0.11(5%)	16	144	9	90
0.22(10%)	2	158	79	98.75

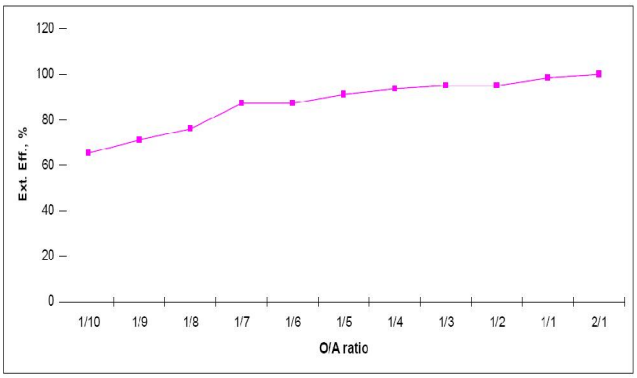

Figure 4 : Effect of O/A phase ratio upon uranium extraction efficiency of uranium by 0.22M TOA in benzene from the loaded liquor of El Sella mineralization composite sample

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Uranium stripping using sulfuric acid

Effect of sulfuric acid concentration upon uranium stripping

In order to study the effect of sulfuric acid concentration upon uranium stripping from the prepared loaded TOA/K solvent sample (1.2 g U/l) prepared form the acid leach liquor of El Sella mineralization composite sample, a series of experiments was performed using sulfuric acid with different concentrations varying from 1% to 20% v/v at an A/O phase ratio of 2/1 for 20 min shaking time at room temperature. The obtained data of uranium stripping efficiency and the corresponding distribution coefficient values are shown in TABLE 5 and the obtained stripping efficiencies are plotted in Figure 5.

From the obtained data, it could be observed that at sulfuric acid concentration of 5% by volume, about 79% of uranium was stripped. As the concentration of the former was increased, the uranium stripping efficiency progressively increased to exceed 95% at 15% acid concentration.

TABLE 5 : Effect of O/A phase ratio upon uranium extraction efficiency of uranium by 0.22M TOA in benzene from the loaded liquor of El Sella mineralization composite sample

O / A Ratio	Conc, g/l		Distribution Coefficient (D_a^o)	Extraction Eff., %
	Aq.	Org.		
1/10	55	1050	19.1	65.6
1/9	45	1030	22.9	71
1/8	37	980	26.5	76
1/7	30	910	30.3	87.5
1/6	20	840	42.0	87.5
1/5	14	730	52.1	91.25
1/4	10	600	60.0	93.75
1/3	8	456	57.0	95
1/2	6	308	51.3	95
1/1	2	158	79.0	98.75
2/1	0	80		100

Effect of shaking time

The effect of the shaking time upon uranium stripping efficiency from the loaded TOA solvent was studied by performing another series of stripping experiments using different shaking times ranging from 1 up to 20 min. In these experiments, the other stripping conditions were fixed at 15 % (v/v) sulfuric acid concentration) and an O/A phase ratio of 1/1 and the experiments were done at room temperature.

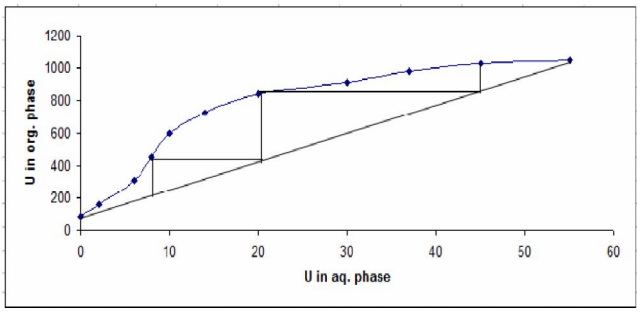

Figure 5: McCabe-Thiele diagram for uranium extraction by 10% TOA v/v from the acid leach liquor of El Sella composite sample

From the obtained results given in TABLE (6), it can be observed that increasing the shaking time from 1 to 5 min. resulted in a increase in the extraction efficiency of uranium from 70.8% to 83.4%. Using 20 min. shaking time resulted in an extraction efficiency of 98.3%

Effect of A/O phase ratio upon uranium stripping

Construction of mccabe-thiele stripping diagram

The effect of aqueous/organic (A/O) phase ratio upon uranium stripping from the loaded TOA solvent sample prepared from the acid leach liquor of El Sella sediment composite sample by sulfuric acid was studied in the range from 1/1 down to1/4. In these experiments, the other stripping conditions were fixed at 15% sulfuric acid concentration, 20 min contact time at room temperature. The obtained results are given in TABLE 7 and plotted in Figure 7 as an equilibrium isotherm.

Uranium stripping using sodium carbonate solution

Effect of sodium carbonate concentration upon

TABLE 6 : Effect of sulfuric acid concentration upon uranium stripping efficiency for the loaded 0.22 M TOA/K solvent sample (1.2 g/l) at an A/O phase ratio of 2/1 prepared from the acid leach liquor El Sella mineralization composite sample

H_2SO_4 conc., % (v/v)	Conc, ppm		Distribution Coefficient S_o^a	Stripping eff., %
	Aq.	Org.		
5%	475	250	3.8	79
10%	510	180	5.6	85
15%	590	20	23	98.3
20%	600	nil		100

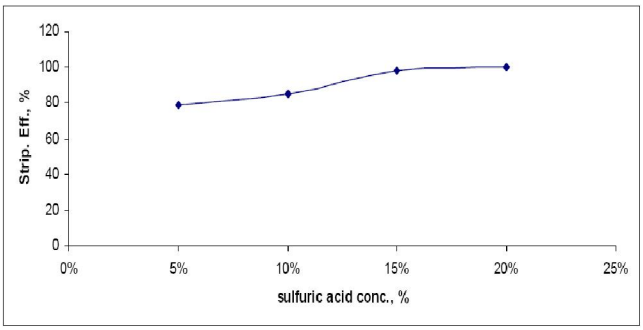

Figure 6 : Effect of sulfuric acid concentration upon uranium stripping efficiency the loaded 0.22M TOA/benzene solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

uranium stripping

In order to study the effect of sodium carbonate concentration upon uranium stripping from the prepared loaded TOA/K solvent sample (1.2 g U/l) prepared form the acid leach liquor of El Sella mineralization composite sample, a series of experiments was performed using sodium carbonate solution with different concentrations varying from 1% to 20% at an A/O phase ratio of 1/1 for 20 min shaking time at room temperature. The obtained data of uranium stripping efficiency and the corresponding distribution coefficient values are shown in TABLE 8 and the obtained stripping efficiencies are plotted in Figure (8).

From the obtained data, it could be observed that at sodium carbonate concentration of 1% by, about 40% of uranium was stripped. As the concentration of the former was increased, the uranium stripping efficiency progressively increased to be 95% at 5% sodium carbonate concentration.

TABLE 7 : Effect of shaking time upon uranium stripping efficiency by 15% H_2SO_4 from the loaded 0.11M TOA/K solvent sample with phase ratio 2/1 aq. /org. (U= 1.2g/l prepared from the acid leach liquor of El Sella composite sample

Shaking time, min	Conc,mg/l		Distribution Coefficient S_o[a]	Stripping eff., %
	Aq.	Org.		
1	425	350	1.2	70.8
5	500	200	2.5	83.4
10	525	150	3	87.5
15	550	100	5.5	91.7
20	590	20	29.5	98.3

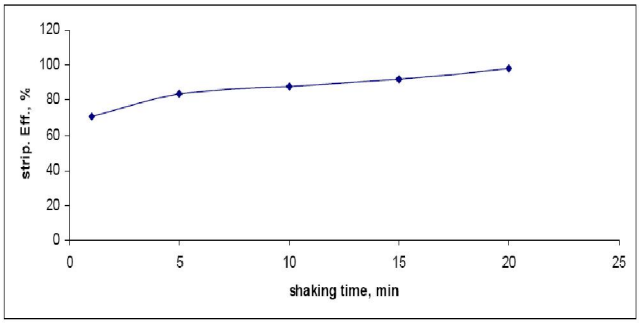

Figure 7 : Effect of shaking time upon uranium stripping efficiency by 15% H_2SO_4 from the loaded 0.22M TOA/K solvent sample with phase ratio 1/2 org /aq. (U= 1.2 g/l prepared from the acid leach liquor of El Sella composite sample

Effect of shaking time

The effect of the shaking time upon uranium stripping efficiency from the loaded TOA solvent was studied by performing another series of stripping experiments using different shaking times ranging from 1 up to 20 min. In these experiments, the other stripping conditions were fixed at 5% Sodium carbonate concentration) and an O/A phase ratio of 1/1 and the experiments were done at room temperature.

From the obtained data, it could be observed that at shaking time of 1 min. about 33% of uranium was

TABLE 8 : Effect of A/O phase ratio upon uranium stripping efficiency by 15% H_2SO_4 from the loaded 0.22M TOA/K solvent sample (U= 1.2 g/l prepared from the acid leach liquor of El Sella composite sample

A/ O Ratio	Conc, mgl		Distribution Coefficient S_o[a]	Stripping Eff., %
	Aq.	Org.		
3/1	398	6	66.3	99.5
2/1	590	20	29.5	98.3
1/1	1080	120	9.0	90
1/2	1968	360	5.4	82
1/3	2340	420	5.6	65
1/4	2640	540	4.9	55

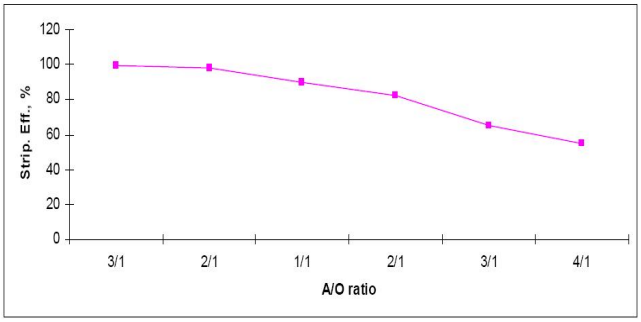

Figure 8 : Effect of A/O phase ratio upon uranium stripping efficiency by 15% H_2SO_4 v/v from the loaded 0.22M TOA/K solvent sample (U= 1.2 g/l prepared from the acid leach liquor of El Sella composite sample

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TABLE 9 : Effect of sod. Carbonate conc. upon uranium stripping efficiency for the loaded 0.11 M TOA/benzene solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

Na_2CO_3 conc., %	Conc, g/l		Distribution Coefficient S_o[a]	Stripping eff., %
	Aq.	Org.		
1	1	1.4	0.7	41
2	1.2	1.2	1.0	50
3	1.96	0.43	4.6	82
5	2.28	0.12	19	95
10	2.35	0.05	47	97.9
15	2.4	0		100

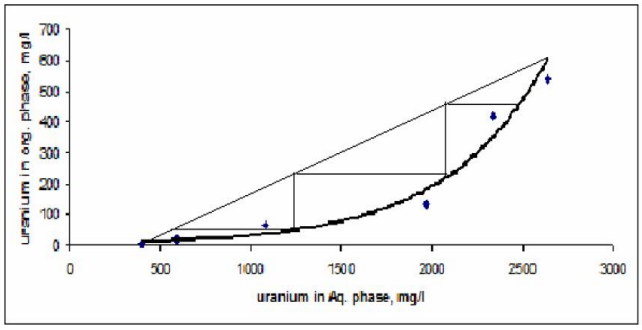

Figure 9: McCabe-Thiele diagram for uranium stripping by 15% H_2SO_4 v/v from the loaded 0.22M TOA/K solvent sample (U= 1.2 g/l prepared from the acid leach liquor of El Sella composite sample

stripped. As the time of the former was increased, the uranium stripping efficiency progressively increased to be 95% at 20 min. shaking time

Effect of A/O phase ratio upon uranium stripping by sodium carbonate

Construction of mccabe-thiele stripping diagram

The effect of aqueous/organic (A/O) phase ratio upon uranium stripping from the loaded TOA solvent sample prepared from the acid leach liquor of El Sella sediment composite sample by sodium carbonate was studied in the range from 1/1 down to1/3. In these experiments, the other stripping conditions were fixed at 5% sodium carbonate concentration, 20 min contact time at room temperature. The obtained results are given in TABLE 10 and plotted in Figure 10 as an equilibrium isotherm that is indicated that the stripping can be proceed via 4 stages

From the figure it seems that a phase ratio of 1/1 A/O is optimum as it attained 95% stripping effeciency

TABLE 10 : Effect of shaking time upon uranium stripping efficiency for the loaded 0.22 M TOA/benzene solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

Shaking time, min	Conc, g/l		Distribution Coefficient S_o[a]	Stripping eff., %
	Aq.	Org.		
1	0.8	1.4	0.6	33.3
5	1.4	1	1.4	58.3
10	1.94	0.46	4.2	80.83
15	2.25	0.15	15.0	85
20	2.35	0.05	47.0	95

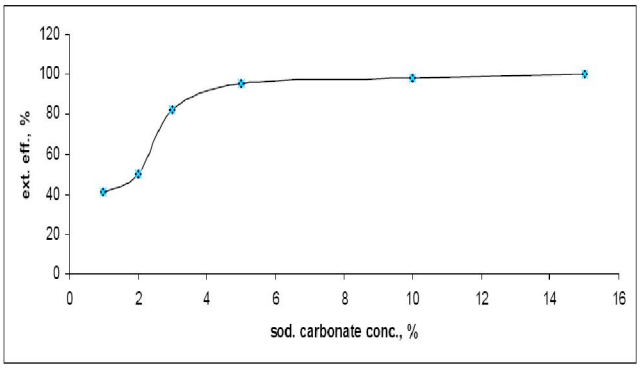

Figure 10 : Effect of sod. Carbonate conc. upon uranium stripping efficiency for the loaded 0.11 M TOA/K solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

TABLE 11 : Effect of phase ratio upon uranium stripping efficiency by 5% sod. carbonate for the loaded 0.22 M TOA/ benzene solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

O / A Ratio	Conc., mg/l		Distribution Coefficient S_o[a]	Stripping Eff., %
	Aq.	Org.		
1/3	0.4	0		100
1/2	0.595	0.01	59.5	99.16
1/1	1.140	0.06	19	95
2/1	2.208	0.192	11.5	92
3/1	2.592	0.336	7.7	72
4/1	3.024	0.444	6.81	63

CONCLUSION

From the obtained results it can be concluded that For the separate recovery of uranium from the prepared leach liquor of El-Sella composite sample, it was applied using trioctylamine (TOA) for prior uranium extraction. The corresponding relevant extraction and stripping factors have been studied. Accordingly, from

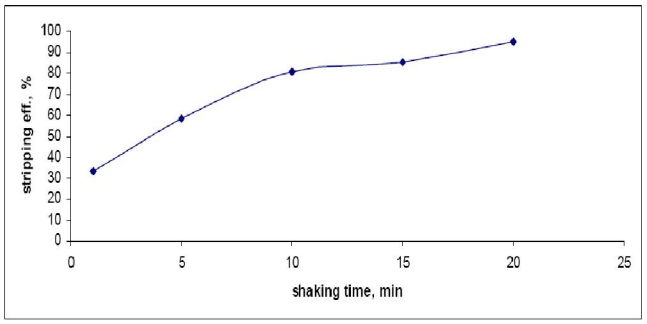

Figure 11 : Effect of shaking time upon uranium stripping efficiency for the loaded 0.22 M TOA/benzene solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

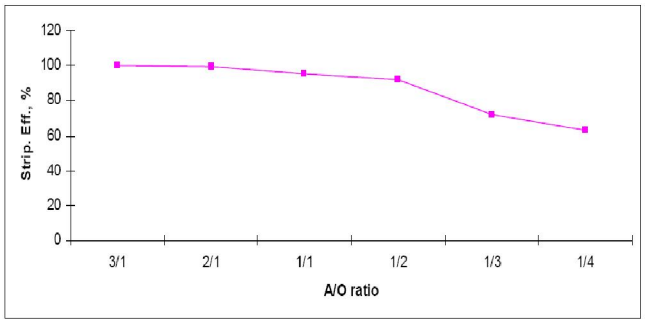

Figure 12 : Effect of phase ratio upon uranium stripping efficiency by 5% sod. carbonate for the loaded 0.22 M TOA/ benzene solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

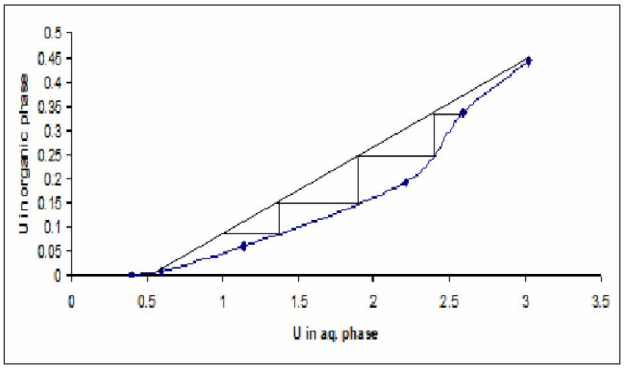

Figure 13 : McCabe-Thiele diagram for uranium stripping by 5% Na_2CO_3 from the loaded 0.22M TOA/benzene solvent sample (1.2 g/l) prepared from the acid leach liquor El Sella mineralization composite sample

the obtained extraction data it was found that about 100% of uranium could be extracted by using 0.22M TOA for 15min shaking time at pH of 1.0 with an O/A phase ratio of 2/1. From the corresponding McCabe-Thiele extraction diagram, it was found that three theoretical stages would be required in a counter current system. On the other hand, the studied stripping factors indicated that it would be possible to strip about 98% of the loaded uranium by 15%(v/v) sulfuric acid for 20 min shaking time and 2/1 O/A phase ratio. The corresponding McCabe-Thiele stripping diagram was reveled that 3 theoretical stages would be required in a counter current system. On the other hand sodium carbonate was also used as strip solution and about 99% of the loaded uranium was stripped using 5% Na_2CO_3 with 2/1 A/O phase ratio at 20 min shaking time. The corresponding McCabe-Thiele stripping diagram was reveled that 3 theoretical stages would be required in a counter current system

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