



ISSN NO. 2320-5407

Journal homepage: <http://www.journalijar.com>

INTERNATIONAL JOURNAL  
OF ADVANCED RESEARCH

## RESEARCH ARTICLE

### RECOVERY OF URANIUM MINERALIZATION, SOUTH EASTERN DESERT, EGYPT BY SOLVENT-IN-PULP TECHNIQUE

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#### Manuscript Info

##### Manuscript History:

Received: 12 July 2014

Final Accepted: 29 August 2014

Published Online: September 2014

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#### Abstract

Extraction of uranium from El Sella composite sample by solvent-in-pulp technique (SIP) were carried out solvent (tri-ethyl amine) mixed with the leaching slurry without prior filtration. About 90% uranium extraction efficiency was attained by the application of the extraction (solvent-in-pulp) optimum conditions. Also, about 92% of the loaded uranium could be stripped by using Sodium Carbonate as an effective stripping agent.

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## 1. Introduction

Occurrences of uranium have been discovered in various localities in the Egyptian terrains. El Sella area at Halaib environ is located in the southern Eastern Desert of Egypt, between Latitudes of 22° 17' 50" N - 22° 18' 6" N and Longitudes of 36° 13' 36" E and 36° 14' 22" E, at a distance of about 30 km south west of Abu-Ramaad city. The field observations proved that are highly weathered, cavernous and exposed with moderate separate hills, which form the outcropping remnants of the ore pluton. The influences of the ENE-WSW tectonic trend (the main shear zone in El Sella ore) in the successive tectonic events make its evolutionary history a good guide to describe the geologic and tectonic history of El Sella area [1]. The area is characterized by its low to moderate relief with some high peaks represented by G. El Sella (about 557 m). It is mainly affected by several wadis sloping towards the Red Sea. The climatic condition is arid proper with rare rainfall less than 50 mm/a, with a wider variation in temperature that sometimes exceeds 50 °C in the summer [2]. In the mineralogy of El Sella and geochemistry of the fracture filling mineralization and associated clayey fraction have allowed us to suggest that the transition redox facies was the prominent geochemical environment. This redox zone was characterized by the coexistence of the primary uranium as well as numerous secondary mineral phases as a result of the physico-chemical and biological changes in the environment. Also, we currently find obvious reducing conditions which form and stabilize pitchblende and coffinite.

There are major techniques can be applied to recover uranium from its leach liquors; namely, ion exchange resin [3-6], solvent extraction [7-11], Liquid Membrane Methods [12-17], direct precipitation [18-20], resin-in-pulp [21] and solvent-in-pulp [22]. The utilization of ion exchange or solvent extraction technique for the recovery of soluble metals from leach slurries has received much attention. Resin-in-pulp (RIP) process have been used in the united state, but it would appear that the RIP process has no real advantage over ion exchange process from clear liquors. Solvent-in-pulp (SIP) process appears more attractive because they do not suffer the same problems, such as resin attrition, that affect the RIP process.

The SIP processes described here deal mainly with the recovery of uranium, although limited work has been done on the recovery of other metals, including copper. The solvent-in-pulp (SIP) processes have been described by several

works for the recovery of uranium, using different types of contactors. Early work in Canada and U.S. on mixer-settlers proved unsatisfactory because the excessive agitation caused stable emulsion and crud formation with amine [23]. The solvent-in-pulp (SIP) was also developed to eliminate the high cost solid/liquid separation stage, where the solvent passed through the slurry directly. [24].

The main objective of the present investigation is studying uranium recovery from El Sella by SIP technique. Accordingly proper conventional agitation leaching of El Sella working sample was performed by sulfuric acid. Based on the previous studied leaching conditions a stock leaching slurry solution was prepared by applying the optimum condition of 40% sulfuric acid concentration, 3 hr leaching time, 1/4 solid/liquid ratio and reaction temperature of 25 °C. Under the optimum conditions about 92% uranium leaching efficiency was attained.

## 2. EXPERIMENTAL

### 2.1. EXPERIMENTAL PROCEDURE

The influence of various factors affecting uranium extraction by solvent-in-pulp technique from El Sella leaching slurry namely, solvent concentration, slurry dilution, shaking time, solid/liquid phase ratio, diluents type and solvent consumption (surfactant type and surfactant/slurry ratio) were studied.

### 2.2. Solutions and Reagents

Tri-Ethylamine (TEA) (Riedel-deHaen), kerosene (Misr.Co.), uranyl acetate ( $\text{UO}_2(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ , BDH Chemicals Ltd. Poole, England), 5-Sulphosalicylic acid 99% (S.D. Fine-Chem. LTD.), Diphenylamine sulphonic acid sod. Salt. (Riedel-deHaen), Glue (Misr.Co.), and Sodium Carbonate (Riedel-deHaen) 97% were chemical reagent. El Sella composite sample was obtained by Nuclear Material Authority.

The representative El Sella used in this study was obtained by Nuclear Material Authority (NMA), Egypt having the chemical composition of 0.190 % U, 0.090 % REE., 2.34 %  $\text{SO}_4^{2-}$ , 1.81 %  $\text{PO}_4^{3-}$ , 67.61 %  $\text{SiO}_2$ , 1.01 % CaO, 7.37 %  $\text{Fe}_2\text{O}_3$  and 1.44 %  $\text{TiO}_2$ .

### 2.3. Analytical Method

Uranium was analyzed in the corresponding aqueous phases using Arsenazo III reagent under different conditions [25]. For this purpose, a Lambda UV/VIS spectrophotometer (Perkin-Elmer, USA) was used. In addition, uranium was also analyzed by an oxidimetric titration method against ammonium metavanadate in the presence of diphenylamine sulfonate indicator prior to titration; proper reduction of uranium was performed using ferrous sulfate [26].

## 3. Results and Discussion

### 3.1. Relevant factors of uranium extraction from El Sella using solvent-in-pulp method.

#### 3.1.1. Effect of solvent concentration

To study the effect of solvent tri-ethylamine concentration upon uranium recovery from El Sella composite sample leaching slurry, series of extraction experiments were performed by contact slurry with the solvent with different concentrations from 1 to 20% in benzene. Other conditions were fixed namely, dilution factor of 130 g/l, shaking time of 15 min and organic/aqueous phase ratio of 1/1. Uranium was determined in the aqueous phase where the extraction efficiency was calculated. The obtained results were plotted in Fig. 1. From the obtained results, it was clearly obvious that at 20% TEA concentration about 83% of the present uranium was extracted. On the other hand, it's clearly that the loss of solvent ratio did not show significant change during the experiments. Accordingly, solvent concentration of 20% TEA will be preferred from the economic point of view.

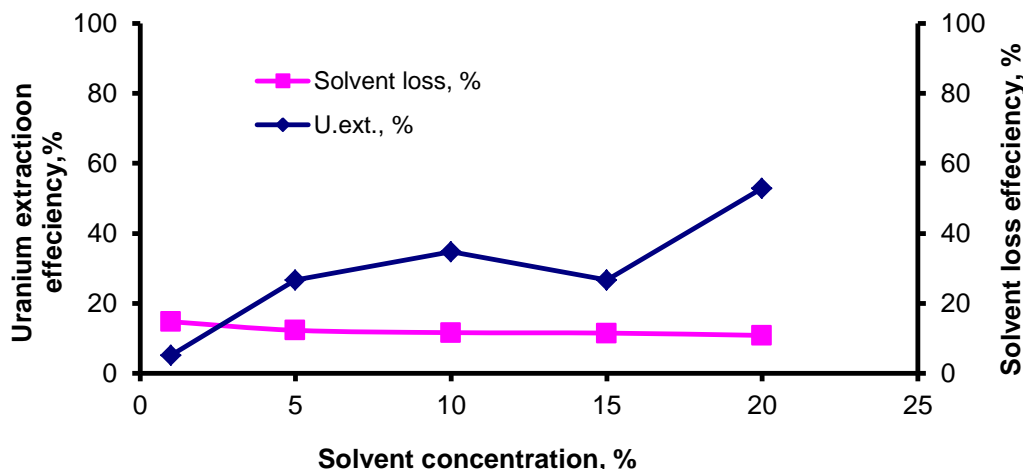


Fig. (1): Effect of solvent concentration upon uranium extraction efficiency from El Sella composite sample

### 3.1.2. Effect of Dilution

In order to study the effect of slurry dilution factor upon uranium extraction solvent leaching efficiency from El Sella composite sample leaching slurry, a series of experiments were performed by contacting slurry with different solid contents ranging from 42.5 up to 130 g/l by adding water and uranium standards to original leaching slurry. The other conditions were fixed namely, solvent concentration of 20%, shaking time of 15 min and solvent/liquid phase ratio of 1/1. The obtained results were plotted in Fig. 2. From the figure, it was clearly evident that uranium extraction efficiency showed an observed decreased from about 89 to 83% by the increase the solid content from 42.5 to 130 g/l respectively, and the solvent loss increased by increasing the solid content from about 8 to 11 % of solvent weight. On the other hand, the solvent loss is happened by absorbing the solvent molecules on the surface of the thick gangue minerals particles. Accordingly, 42.5 g/l is the optimum dilution factor for uranium extraction.

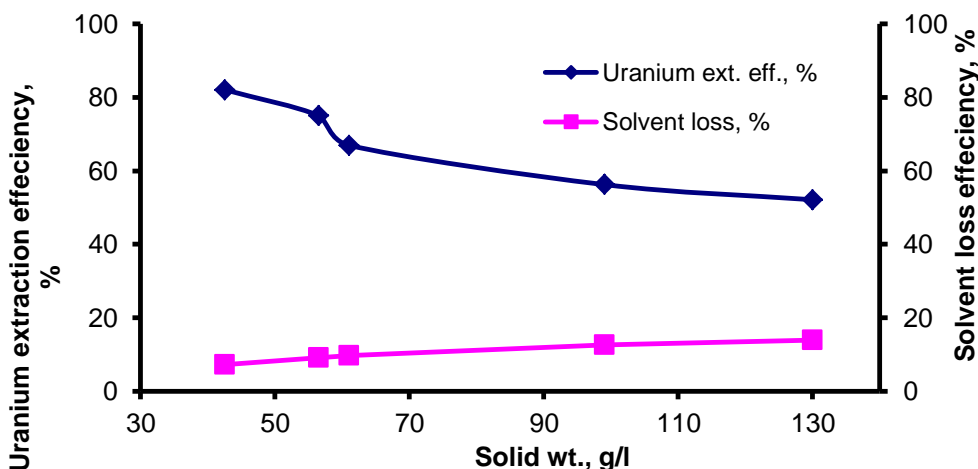


Fig. (2): Effect of dilution upon uranium extraction efficiency from El Sella composite sample

### 3.1.3. Effect of Shaking Time

In order to study the effect of shaking time upon uranium recovery efficiency from El Sella composite sample leaching slurry, a series of extraction experiments were performed by contacting slurry with the solvent with time ranging from 1 up to 120 min. The other working conditions were fixed namely; dilution factor of 42.5 g/l, organic/aqueous phase ratio of 1/1 and TEA concentration of 20%. The obtained data were plotted in Fig. 3. From the obtained data, it was clearly evident that the uranium extraction efficiency increases from about 34 to 94% as the time increases from 1 up to 120 min respectively. From the economic point of view (taking the solvent loss about 11% into our consideration) it was decided to choice 60 minute as optimum shaking time.

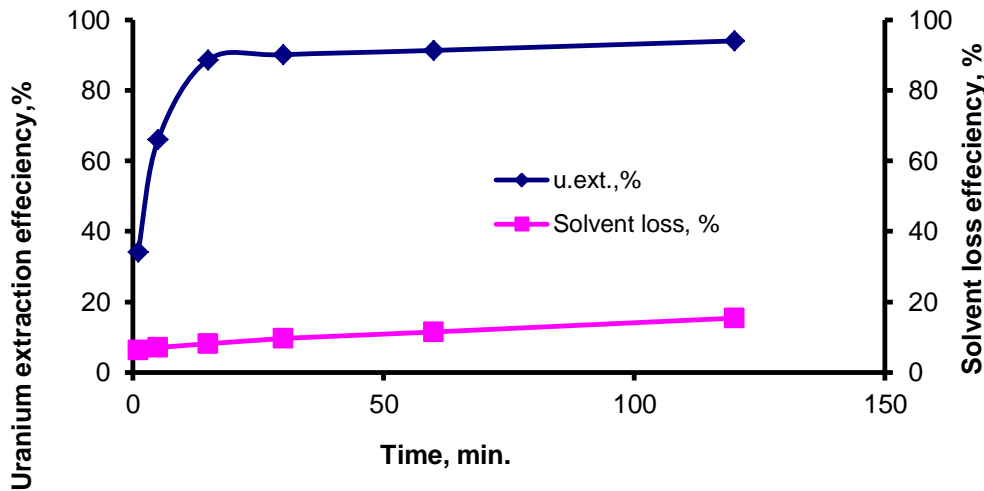


Fig. (3): Effect of shaking time upon uranium extraction efficiency from El Sella composite sample

### 3.1.4. Factors Affecting Solvent loss (Consumption)

Successful solvent-in-pulp extraction of uranium from the obtained leach slurry will be dictated by the efficiency of the solvent in extraction as well as by the lost amount by slurry raffinate. Accordingly, surfactant type and surfactant amount are studied as the main factors affecting the degree of solvent loss on the gangue mineral surfaces.

#### 3.1.4.1. Surfactant Type

To study the effect surfactant types upon extraction uranium, various types were tested mainly; molasses, glue, crud sugar and vegetable oil. All experiments were performed by mixing 0.5g (25g/l) of the surfactants with the slurries portions (20ml) for 0.5 hr prior to contact them with the solvent. The solvent loss calculated by the different weight before and after the contact. Other factors were fixed at 20% TEA concentration, dilution factor (solid content) of 42.5 g/l for 60 min contact time and solvent  $V_{org}/V_{diluted}$  slurry ratio of 1/1. From the obtained data it was clearly that, uranium extraction efficiency of about 80.44, 86.22, 62.34 and 55.65% with solvent loss of about 10.56, 8.77, 12.09 and 12.10% in molasses, glue, crud sugar and vegetable oil respectively. Accordingly, we suggested using glue as surfactant for further experiments which nearly the highest efficiency of uranium extraction and lowest efficiency of solvent loss.

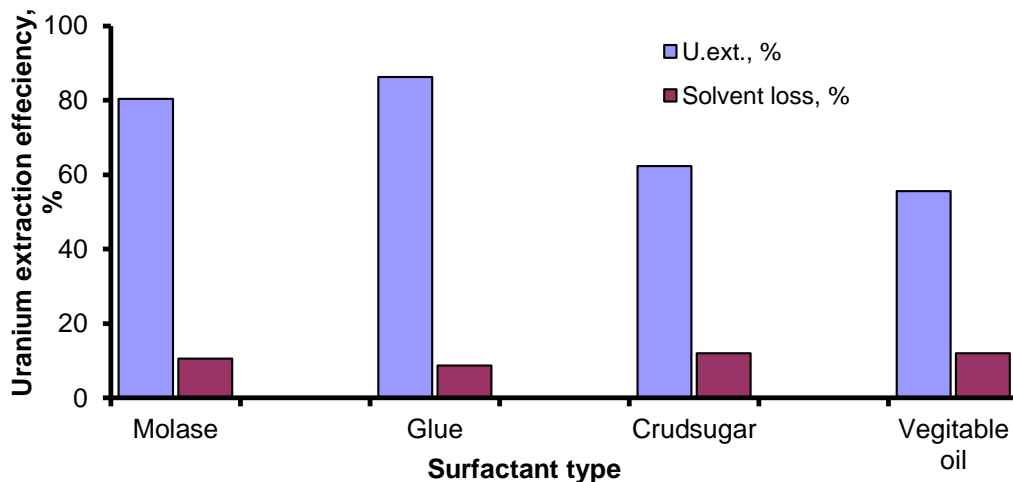


Fig.( 4): Effect of surfactant type upon uranium extraction efficiency and solvent loss .

#### 3.1.4.2. Effect of Surfactant Amount

To study the effect of surfactant amount, series of extraction experiments were performed by contacting fixed slurry portions (20ml each). Proper mixing with deferent amount of glue ranging from 25 up to 200 g/l was performed. Other factors were fixed at solvent concentration of 20 % TEA, contact time of 60 min, dilution factor (solid amount) of 42.5 g/l and glue as surfactant. The obtained data were plotted in Fig.5. From the obtained results it is clearly obvious that

the changing in surfactant amount shows a good influence on decreasing the solvent loss amount. Accordingly, the solvent loss decrease from about 8% down to about 2% at surfactant amount of 25 and 200 g/l and uranium extraction efficiency increasing from 86 to about 90% respectively. Therefore, for economic considerations, it could thus be suggested that the amount of 200 g/l as preferred surfactant amount.

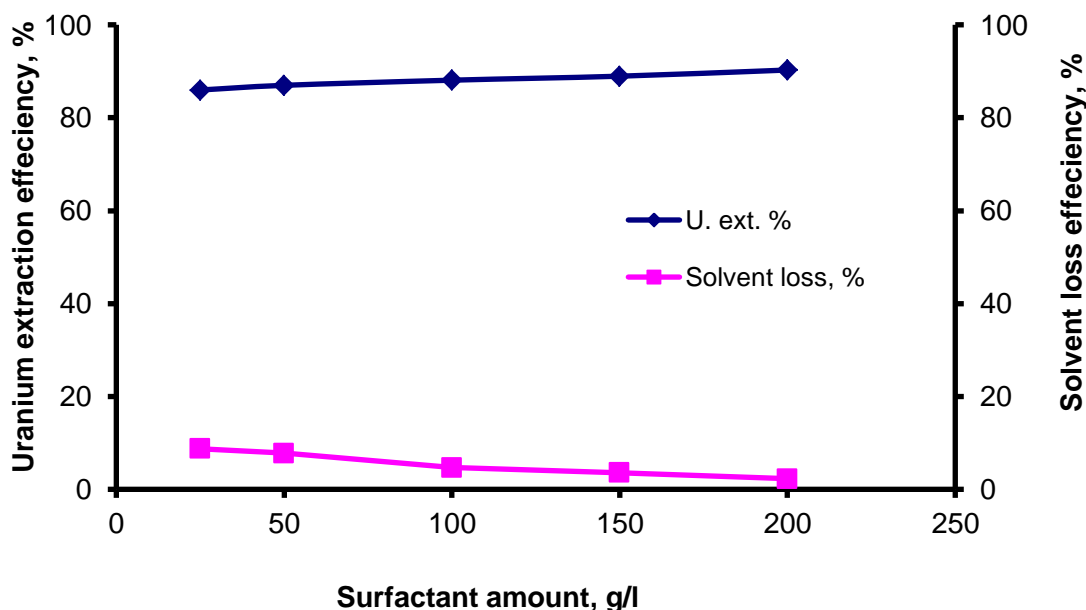


Fig. (5): Effect of surfactant amount upon uranium extraction efficiency and solvent loss.

### 3.1.5. Effect of aqueous/organic phase ratio

In order to identify the effect of aqueous (diluted slurry)/organic phase ratio upon uranium recovery from El Sella composite sample, a series of extraction experiments were performed using contacting diluted slurry with TEA solution in benzene with different ratios ranging from 0.5/1 to 4/1. These experiments were performed under the fixed conditions at solvent concentration of 20% (TEA) for 60 min contact time, dilution factor (solid content) of 42.5 g/l and 200 g/l of glue. The obtained results were graphically represented in Fig. 6. From latter figure; it is clearly evident that the uranium extraction efficiency increases with the increases of the phase ratios (about 93.5% for the last experiment). On the other hand solvent loss (consumption) decreased by increasing aqueous/organic phase ratios to minimum loss amount at the last experiment (2.3%).

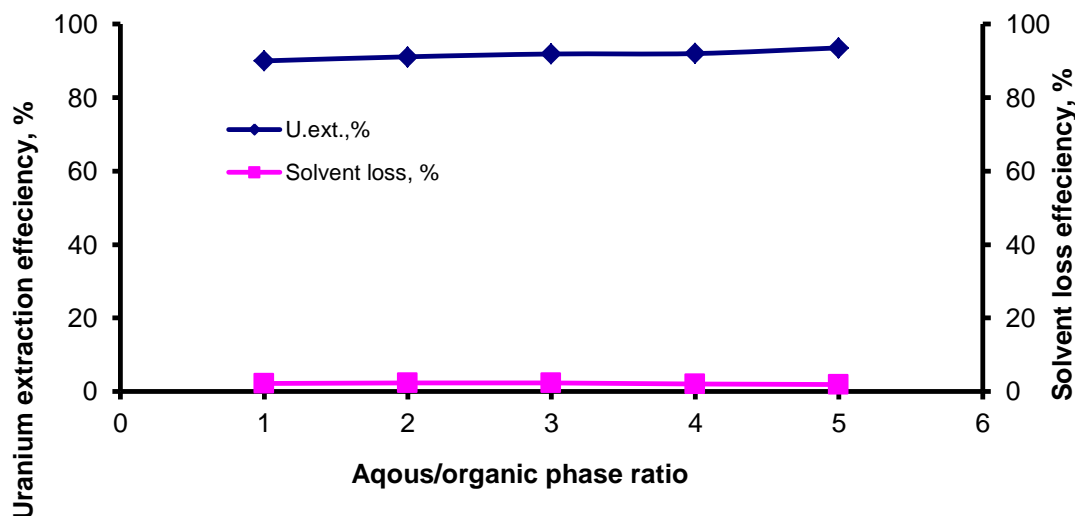


Fig : (6): Effect of aqueous/organic phase ratio upon uranium extraction efficiency and solvent loss.

### 3.1.6. Effect of Diluent Type

In order to identify the effect of the diluent type on uranium recovery from El Sella composite sample, different diluents were tested namely; benzene, toluene, cyclohexane and kerosene. Other factors were fixed at solvent concentration of 20% (TEA) for 60 min contact time, dilution factor (solid content) of 42.5 g/l and 200 g/l of glue and 4/1 aqueous (diluted slurry)/organic phase ratio. The obtained data were plotted in Fig. 7. From this figure, it is clearly evident that kerosene gave maximum uranium extraction efficiency (about 94%) and minimum solvent loss (about 2.23%).

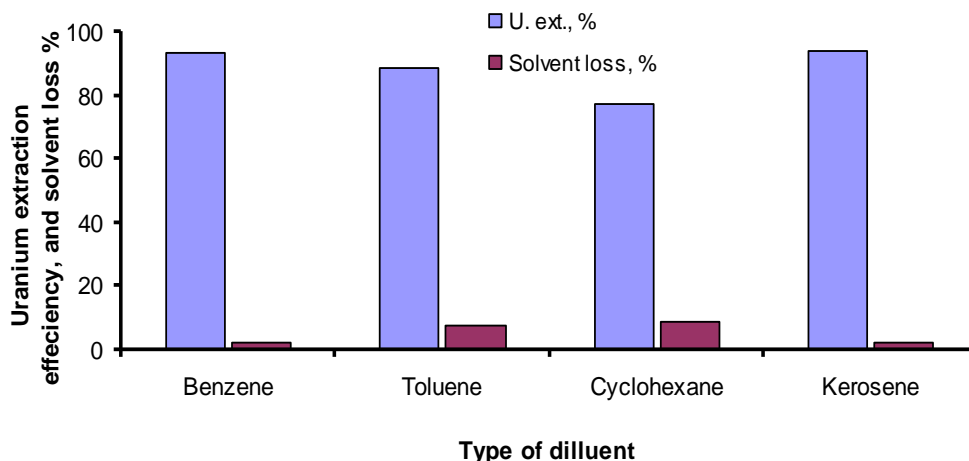


Fig (7): Effect of diluent type upon uranium extraction efficiency from El Sella composite sample

### 3.1.7. Choice of the Optimum Conditions of Uranium Extraction from El Sella composite sample

From the previously mentioned results of the relevant factors of extraction of uranium process, it is clearly obvious that it would be possible to obtain comparable extraction of uranium process by different combinations of the relevant factors of leaching ore conditions. Careful selection of the optimum values of the obtained results would depend primarily on economic considerations. In the light of the studied factors, it would seem economically to select the following conditions extraction of uranium process as optimum conditions: Solvent Concentration: 20 %, Extraction Time: 60 min, Solid content (slurry dilution): 42.5 g/l, Surfactant type: glue, Surfactant amount: 200 g/l, Aqueous (diluted slurry volume)/organic: 4/1 And Diluents Type: kerosene.

## 4. Uranium Stripping

Uranium stripping from the loaded working solvent, obtained after applying the above mentioned optimum conditions (assay about 877 mg U/l) was studied by studying the effects of the following factors, namely, stripping agent, stripping agent concentration, shaking time and organic/aqueous phase ratios.

### 4.1. Effect of Stripping Agent

In order to study the effect of strip agent (1M each) were tested namely;  $H_2SO_4$ ,  $Na_2CO_3$ ,  $HCl$ ,  $HNO_3$  and  $H_2O$ . Other factors were fixed at shaking time at 30min and aqueous/organic phase ratio of 2/1. The obtained results are plotted in Fig. 8. From this figure it is clearly obvious that sodium Carbonate could be choiced as a suitable stripping agent.

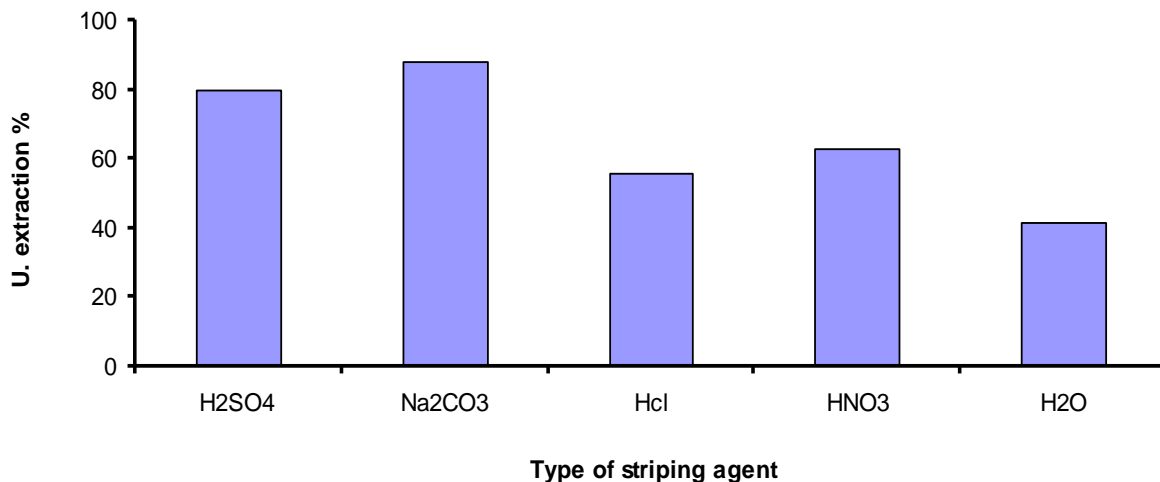


Fig. (8): Effect of some stripping agents upon uranium stripping efficiency from the loaded solvent.

#### 4.2. Effect of Stripping Agent Concentration

To investigate the effect of strip agent concentration upon uranium stripping from the loaded solvent sample prepared by solvent-in-pulp of El Sella composite sample, different sodium carbonate concentrations were applied ranging from 1 up to 4 M. The other stripping conditions were fixed at 30 min shaking time and aqueous\organic phase ratio of 2\1. The results were plotted in Fig. 9. From the obtained results it is clearly obvious that, stripping efficiency increases from 88 to 92% by increasing sodium carbonate concentration from 1 to 4M. Consequently 4M is sufficient to strip more than 90% of loaded uranium.

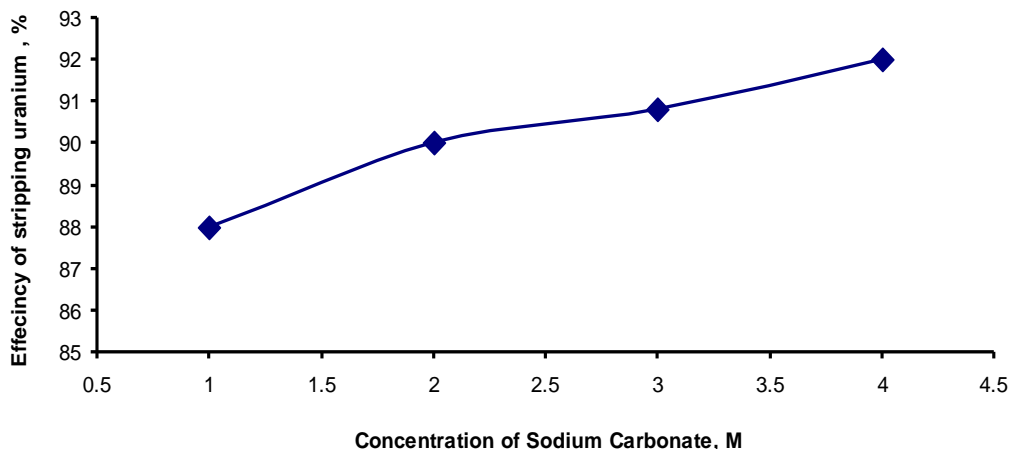
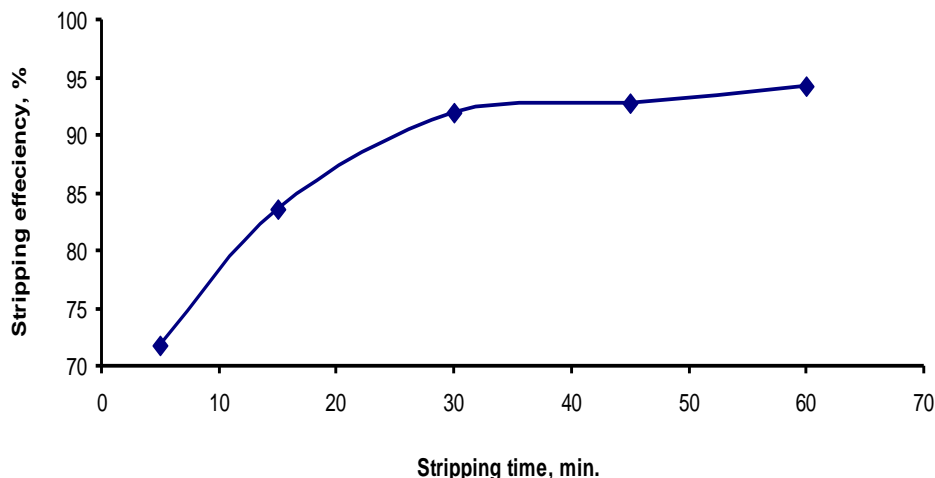


Fig (9): Effect of sodium carbonate concentration upon uranium stripping from the loaded solvent

#### 4.3. Effect of Shaking Time upon Uranium Stripping Efficiency

For studying the effect of contact time upon uranium stripping from the uranium-loaded solvent of El Sella composite sample diluted slurry, a series of experiments was performed using time intervals varying from 5 up to 60 minutes. The other conditions were fixed at 4M sodium carbonate concentration and aqueous\organic phase ratio of 2\1 at room temperature (~ 25 C°). The obtained results are plotted in Fig. 10. From the obtained results, the stripping efficiency is increasing significantly from about 71 to 92% by increasing stripping time from 5 to 30min respectively. Then there are no significant improvement has been observed from 30 up to 60min. Accordingly, 30 minutes shaking time would be quite sufficient for stripping about (92%).



**Fig (10): Effect of shaking time upon uranium stripping from the loaded solvent**

#### 4.4. Effect of O/A Phase Ratio upon Uranium Stripping Efficiency and Construction of McCabe Thiele Stripping Diagram

In order to study the effect of the aqueous/organic phase ratio upon uranium stripping efficiency from the uranium-loaded solvent sample of El Sella composite sample diluted slurry sample and calculate the theoretical stages required for complete stripping of uranium from the loaded solvent sample (877 mg U/l) prepared from El Sella composite sample diluted slurry. A series of stripping experiments were performed using O/A ratios ranging from 4/1 down to 1/4. In these experiments, the other stripping conditions applied involved at 4 M sodium carbonate concentration was used and the time was fixed at 30 min contact time. The obtained data given in Table (1) and were used to construct the equilibrium isotherm in the McCabe Thiele diagram [27]. (Fig.11). From the constructed diagram, it is clearly obvious that an almost two Theoretical stages are required for the removal of almost all uranium from the loaded solvent sample. It would be possible to deplete uranium from the loaded solvent down to 24 mg/l on one hand and to enrich the stripping sodium carbonate solution to about 719 mg/l.

**Table (1): Effect of O/A phase ratio upon uranium stripping efficiency from the uranium loaded solvent of El Sella diluted slurry sample.**

O/A Ratio	U Conc., mg/l		S <sup>a</sup> <sub>o</sub>	Stripping eff., %
	Org.	Aq.		
4/1	697.10	719.58	1.03	20.51
3/1	644.48	697.10	1.08	26.51
2/1	562.17	629.64	1.12	35.89
1/1	483.47	393.52	0.81	44.87
1/2	73.44	402.17	3.73	91.67
1/3	63.41	271.19	4.27	92.76
1/4	24.28	212.50	8.75	97.23

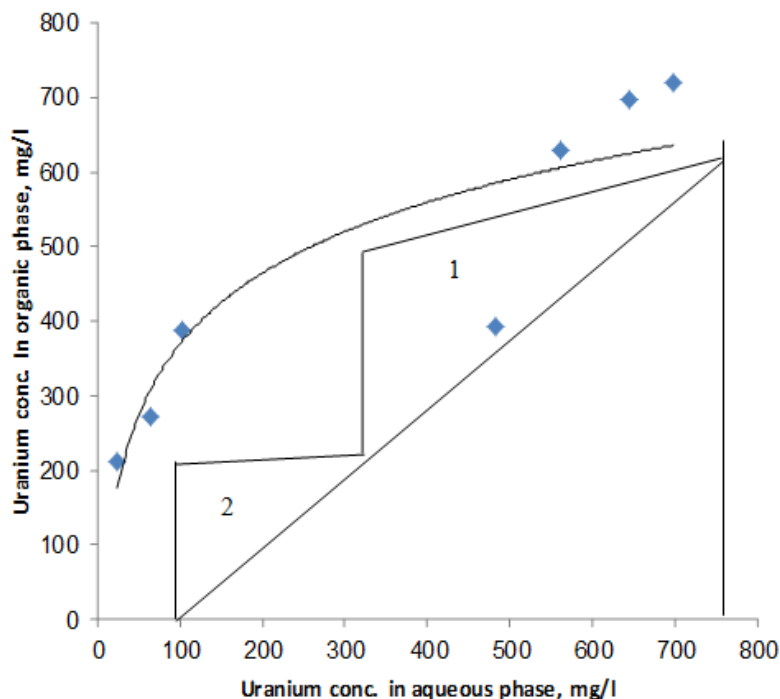


Fig (11): Mc-Cbe Thiele Diagrame of uranium stripping from the loaded solvent

## Conclusion

About 90% from uranium extraction efficiency was attained (and a minimum solvent loss of about 2.3%) by the application of the extraction optimum conditions of El Sella diluted slurry sample (by applying the solvent-in-pulp method). In addition, about 92% of the loaded uranium could be stripped using 4 M Sodium Carbonate as an effective stripping agent.

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