EXTRACTION OF URANIUM USING ALAMINE$_{336}$ IN KEROSENE FROM SULFATE SOLUTION OF ABU-HAMATA SANDY CLAYSTONE ORE SAMPLE, SOUTHWESTERN SINAI, EGYPT

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Abstract

The present study is concerned with the feasibility of using organic solvent, Alamine$_{336}$ in kerosene to extract U from sulfate leach liquor of Abu-Hamata sandy clay stone ore sample. The prepared sulfate leach liquor assays 0.45g/L of U with achieved leaching efficiency of 98.9%. The obtained results indicate that U extraction efficiency depends mainly on Alamine 336 concentration, strongly influenced by pH value, contact time and the organic / aqueous (O/A) ratio.

Uranium in the organic phase was stripped using a solution of 1M NaCl acidified with 0.2M H$_2$SO$_4$ with an achieved stripping efficiency of 93.9% at A/O ratio of 1/1 and shaking time of 5min. Not less than 99% of U in the obtained U rich strip solution was directly precipitated using H$_2$O$_2$ solution to produce the final pure product of U$_3$O$_8$.

Key words: Uranium, Extraction, Alamine$_{336}$, Sandy Claystone.

Introduction

The global U demand (the main source to generate the atomic power) is expected to grow up in the next years as cheap and more quantity of the electricity to full fill the country demands. Sandstone deposits are considered as one of seven conventional U resources with the highest reserve in the world, **Dahlkamp, (1980)**. Abu Hamata clay stone rock fancies of Um Bogma formation which occurs at Abu Zeneima area, Southwestern Sinai, Egypt is considered indeed one of the most important occurrence of U beside some associated economic metal values e.g. REEs, V, B, Zn, Cu,…. etc. **Al Shami, (2003)** recorded some trace elements in the claystone rocks such as Ba and U at Abu Hamata area. Field study observation revealed the presence of minerals of U, Cu as well as Fe-Mn at the area under study.

In addition, various ore minerals have actually been identified in the various rock units of Abu Zeneima as has been reported by **Dabbour and Mahdy (1988); Hussein et al. (1992); Amer (1993 &1997); Abdel Momen et al. (1997)**. The main identified secondary U minerals include uranophane, beta-uranophane, carnotite, kasolite, meta-torbernite, meta-uranocircite and bassetite**.**

Sulfuric acid is the predominate agent used for U dissolution because of its relatively low cost, wide availability and shorter leaching time**.** U is effectively recovered from the sulfate leach liquor through two mainly techniques namely; ion exchange and solvent extraction, **Ritcy and Ashrock (1982)**. Moreover **Amer (1997)** and **Ghonaim et al., (2004)** have studied H_2SO_4 acid leaching of Abu Zeneima gibbsite ore materials for recovering Al, Zn, U and Cu.

In recent years organic solvents are the most popular choice for U extraction and purification circuits. The phosphorous based extractants namely; tri-butyl-phosphate (TBP), di- (2-ethylhexyl) phosphoric acid (D2EHPA), 2-ethylhexyl phosphonic acid, mono-2-ethylhexyl ester (PC 88A), tri-n-octyl-phosphine oxide (TOPO or Cyanex 923) and bis (2, 4, 4-trimethylpentyl) phosphonic acid (Cyanex 272) were used for U(VI) extraction from various acidic solutions**.** Tertiary amines of the general formula R_3N, where R (alkyl group containing eight or ten carbon atoms) are commonly used in acid leach circuits**.**

Kumar, et al. (2010) studied the recovery of U and its separation from V in synthetic sulfate leach liquor. While NH_4Cl solution was used as stripping agent. In addition, selective separation of U from the associated REEs and Th in monazite sulfate leach liquor was attained by **Amaral, et al., (2010).** The extraction of 95% of U from diluted sulfate leach liquor that contains high concentrations of REEs was conducted by using 2% (v/v) Alamine 336. A stripping efficiency of 98% from the loaded U was attained by 1NaCl acidified with 0.2M H_2SO_4**, Ramadevi, et al. (2012)**.

The present paper is concerned with the concentration of U from Abu-Hamata sandy claystone sulfate leach liquor rich with REEs via organic solvent extraction technique. Alamine 336 was used for this purpose, where pure U product was attained from the concentrated U strip solution via direct precipitation using H_2O_2.

Experimental

Experimental work requires the investigation of both mineralogical and chemical constituents of the representative technological sample as well as the processing procedures (leaching & extraction) of the metal values**.**

Investigation of mineralogical and chemical constituents

To identify the mineralogical composition of Abu Hamata sandy clay stone ore sample, a 1kg weight of the working bulk sample ground to a mesh size of -60 was carefully washed with tap water several times to remove the slimes. After dryness and using a set of sieves ranging from 500 to 100 µm, different portions of different volume size ranging from 35 to 140 mesh were obtained. The latter under heavy liquid separation using bromoform (sp.gr. 2.84), were separated to different light and heavy fractions. Each fraction was isodynamically separated at (0.2, 0.5, 1.0 and 1.5 Amp.) using Frantz isodynamic separator. Several different grains from each fraction were picked up by using binuclear microscope. The latter were investigated using X-ray diffraction, (XRD), analysis technique to identify the present minerals.

For chemical composition, a weight of 1g portion ground to a mesh size of -200 from the working sample was digested with the acid mixture, (H_2SO_4, HCl, HNO_3 and HF) for the complete dissociation and closed up to a measuring volume of 100mL. The latter was subjected to different analytical techniques to show its chemical composition. However, the major oxides such as: Fe_2O_3, Al_2O_3, MgO and CaO are classically estimated by wet chemistry, **Shapiro and Brannock (1975)**, while the trace elements e.g. V, B, Zn, Cu, Pb, Ni, Co, etc. were estimated by using the flam atomic absorption spectrophotometer (FAAS) Unicam 969.

On the other hand, UV-vis spectrophotometer (Shemadzu 610) is used for the quantitative analysis of the total REEs using Arsenazo III at λ_{654} nm, **Marczencko (2000)**. U is analyzed via oxidometric titration method against NH_4VO_3, **Mathew, et al., (2009).** For Si analysis a small portion weighted 0.1g of the fine ground ore sample is fused with 1g portion of NaOH pellets in a nickel crucible for 20 min directly on Bunsen flame. The fused matrix is dissolved with HCl 1:1 and closed up to a measuring volume of 250 mL where Si estimated by using UV-vis spectrophotometer (Shemadzu 610) at λ_{640} nm**, Marczencko (2000)**. In the mean time, the final product of U is identified by using both of XRD and SEM-EDAX analysis techniques. While the pH values of all solutions are measured with a DM-21pH meter (Digimed, S. Paulo, Brazil).

Preparation of working sulfate solution.

The working sulfate solution of the studied ore sample was prepared via acidic agitation leaching of 1kg weight by using 5% H_2SO_4 at a solid/ liquid, (S/L) ratio of 1/2 and stirring time of 1h, while the leaching temperature was kept at 75°C. After filtration and washing with distilled H_2O, the final volume of the prepared sulfate leach liquor was 4 liters and its pH value was found to

attain pH 0.5. The latter was directed to solvent extraction unit to recover its U content.

Optimization of Alamine$_{336}$ effective extraction parameters

Uranium in the prepared sulfate leach liquor was extracted by using Alamine$_{336}$ in Kerosene. The diluted neutral Alamine 336 was pretreated with 0.2M H$_2$SO$_4$ for 10 min at an O/A ratio of 1/1 to convert to its suitable extractant sulfate form. The relevant effective extraction parameters such as: extractant concentration, pH value, contact time and phases volume ratios (O/A) were investigated to optimize the loading U process. The type and concentration of the stripping solution and phases ratios (O/A) were studied to optimize the U stripping process.

Preparation of U pure product

The strip U concentrated solution was adjusted to direct precipitation process by using H$_2$O$_2$ solution at pH 2. After filtration and washing, the precipitated U cake was ignited at 850°C for 1h to be crystallized. The produced U$_3$O$_8$ was washed, dried and then identified using XRD analysis technique and chemically analyzed to estimate its final purity, **Bhowmik et al. (2009)**.

Results and discussion

Mineralogical and chemical composition of working ore sample

XRD analysis data represented by Fig. (1) emphasized the presence of Renardite U mineral [Pb(UO$_2$)$_2$(PO$_4$)$_2$.8H$_2$O]. The latter has a wide distribution in 1.5 Amp. of non magnetic fraction with lesser extent in 1.0A magnetic fractions. U in such secondary U minerals was characteristic by its easier dissolution, thus the leachability of U was very easy and applicable. Renardite, is a yellow mineral consists of hydrous basic Pb uranyl phosphate with chemical composition as UO$_2$ of 64.62wt.%, P$_2$O$_5$ of 8.49 wt. %, H$_2$O of 9.7 wt.% and PbO of 13.35 wt.%. It is a rare secondary uranyl mineral formed by alteration of uraninte (UO$_2$) or earlier-formed secondary uranium minerals and it is characterized by the ease of its dissolution.

Finally, it was quite important to mention herein that, the mineralogical study proved that, the working sandy clay stone ore material has no identified mineral grains for either REEs, B or V although their relative high content in the study ore sample. This is probably due to their adsorption upon the clay mineral, [montmorillonite]. Complete chemical analysis of the studied sandy clay stone ore sample, Table (1) revealed that it is mainly composed of SiO$_2$ which represents the main constituent (55.6%) beside Al$_2$O$_3$ (12.70%) and Fe$_2$O$_3$ (9.81%). K$_2$O as well as MgO represent the much lesser constituents of

the study ore material. Moreover, the sample was found to contain trace amounts of TiO_2, Na_2O, CaO and P_2O_5.
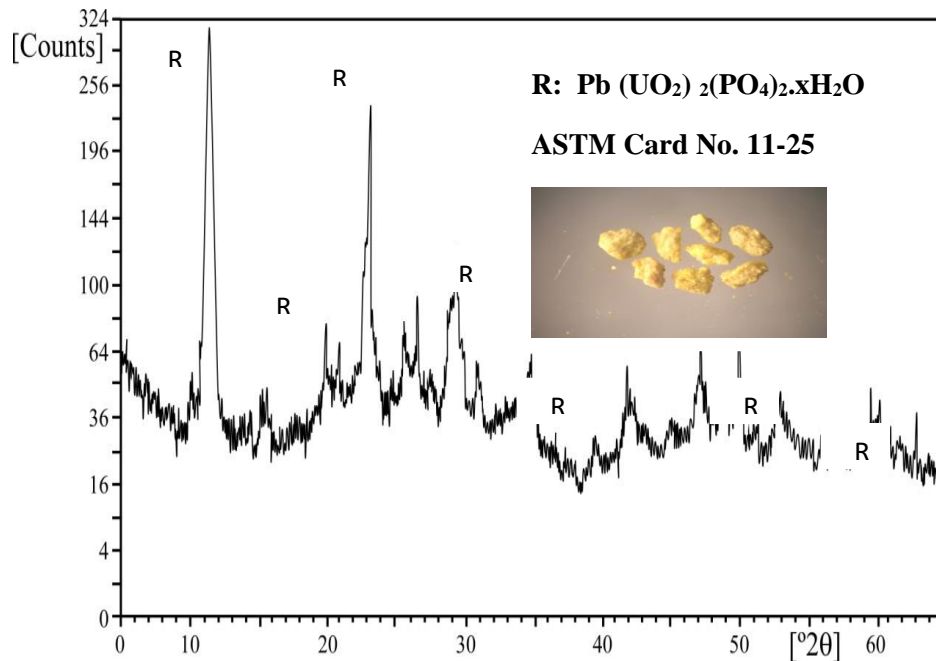
Fig. (1): XRD pattern of Renardite mineral.

Table (1): Chemical composition of Abu-Hamata sandy claystone sample.

Major oxides	Wt., %	Trace elements	Conc., ppm
SiO_2	55.6	U	1830
Al_2O_3	12.7	REEs	1375
Fe_2O_3	9.81	V	1750
K_2O	3.7	B	1100
MgO	2.0	Cr	833
TiO_2	1.0	Cu	650
Na_2O	0.92	Ba	448
CaO	0.68	Zn	345
P_2O_5	0.21	Co	46
MnO	0.04	Ni	64
*L.O.I	11.1	Ga	25
Cl^-	0.7		
SO_4^{2-}	0.043		
Total	98.503		

* **Loss of ignition.**

It is worthy to mention herein that the high contents of SiO_2, Al_2O_3 together with loss of ignition (11.1%) and K_2O reflect the sandy clay nature of the studied ore material. The loss was found to be distributed between humidity (4.6%), water of crystallization (3.2%) as well as organic matter and CO_2

(3.3%). The high moisture as well as the high crystalline H_2O content are attributed to the presence of montmorillonite mineral which has the tendency to absorb H_2O and able to swell. On the other hand, the chemical composition of the studied ore material shows the presence of considerable concentrations of some valuable trace elements e.g. U, V, REEs and B.
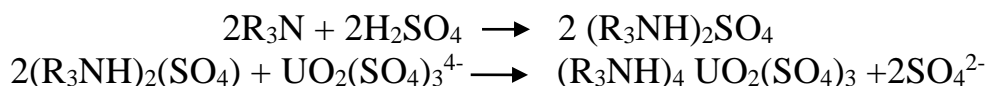
Working sulfate solution.
Table (2) shows the chemical composition of the prepared working sulfate solution. This table reveals that there are several interfering metal ions with high concentrations especially SO_4^{2-} and Cl^-. These metal ions inhibited extraction process of uranyl sulfate complex, $[UO_2(SO_4)_3^{4-}]$ when ion exchange method is applied, **Merrit (1971), Ritcy, et al., (1993) and Mackenzie (1997).** Thus, the suitable choice for U extraction from the prepared sulfate solution will be done using organic solvents. For this purpose, Alamine 336 in kerosene would be used to recover U.

Table (2): Chemical constituents of the working sulfate solution.

Metal ion	Conc., g/L
SO_4^{2-}	26
Cl^-	2.34
REES	0.32
U	0.45
Fe	0.88
V	0.05
Ca	0.04

Mechanism of loading process
The mechanism of the loading process of the present uranyl sulfate complex $[UO_2(SO_4)_3]^{4-}$ using the activated form of Alamine 336 is illustrated in equations:

$$2R_3N + 2H_2SO_4 \longrightarrow 2\,(R_3NH)_2SO_4$$
$$2(R_3NH)_2(SO_4) + UO_2(SO_4)_3^{4-} \longrightarrow (R_3NH)_4\,UO_2(SO_4)_3 + 2SO_4^{2-}$$

Optimization of loading step
Loading of the uranyl sulfate ion complex, $[UO_2(SO_4)_3]^{4-}$, from aqueous phase of sulfate solution to organic phase of Alamine 336 required the investigation of several effective loading parameters as follow:
Effect of Alamine 336 concentration
Fig. (2) Shows the effect of $Alamine_{336}$ concentration upon U extraction efficiency from the working sulfate solution of pH 0.5. Four equilibrium experiments were conducted by using different Alamine 336 concentrations

ranging from 1 to 5% in kerosene at O/A volume ratio of 1/1 and contact time of 5 min. After separation, U was analyzed in the raffinate aqueous solution to calculate its extraction efficiency. The obtained data emphasized that Alamine 336 concentration has a great influence on U extraction efficiency which is progressively improved from 64.8 to 92.2% as the Alamine concentration increases from 1-2%. It is also observed that further increase in the solvent concentration, more than 2%, shows limit increase in U extraction efficiency.
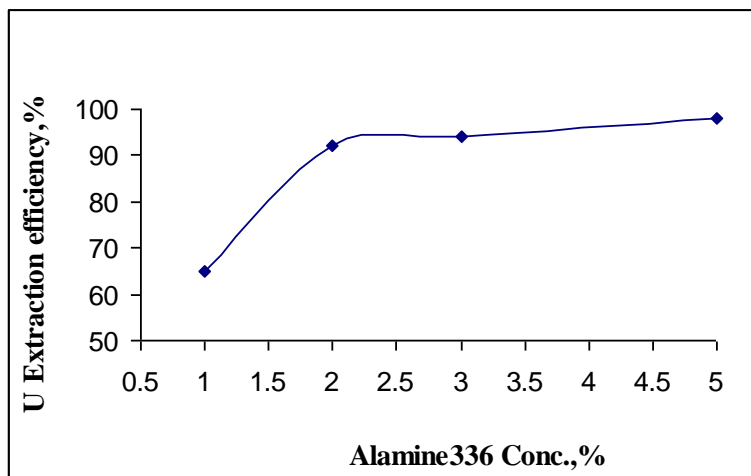
Fig. (2): Effect of Alamine$_{336}$ conc., % upon U extraction efficiency, %

Effect of pH values

The effect of pH values of the prepared sulfate solution upon U extraction efficiency, % was studied in the range from 0.5 to 1.5. The other parameters were kept constant at Alamine$_{336}$ concentration of 2% (v/v) in kerosene, O/A volume ratio of 1/1 and contact time of 5min. Data shown in Fig. (3) revealed that the percentage of U extraction efficiency increased from 92.2% to 97.6% with increasing the pH value of the sulfate leach liquor from 0.5 to 0.75. While further increase in the pH values to 1 and 1.5 has an opposite effect, where U extraction efficiency deceased up to 73.3%. This decrease in efficiency is due to the decreasing in the acidity of the working sulfate which inhibits quaternary liquid amines for U uploading.

Effect of contact time

Figure (4) indicates the effect of contact time (shaking time) upon U extraction efficiency from the working sulfate solution of pH 0.75. The latter was mixed with 2% (v/v) Alamine 336 in kerosene and shacked at different time periods ranging from 2 to 10 min at O/A volume ratio of 1/1. The resulted data clearly indicate that the % extraction efficiency of U increases to its maximum value (97.6%) at the contact time of 7min. It shows also that the % extraction efficiency of U did not affected by further contact time increasing up to 10 min. This may be explained by achieving the organic solvent to its equilibrium capacity.
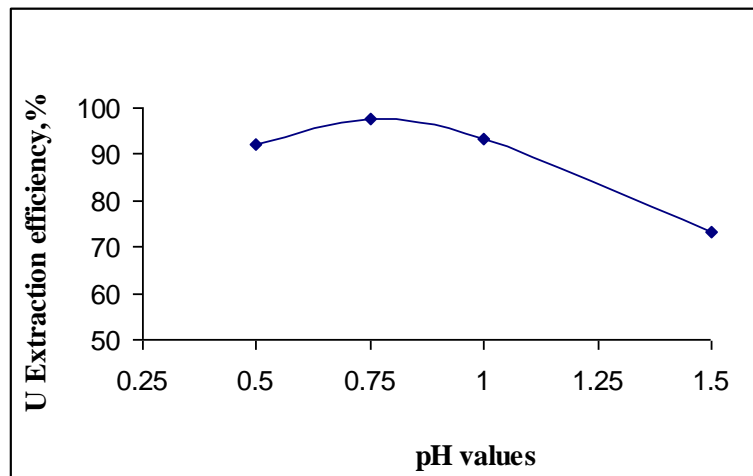
Fig. (3): Effect of pH values solution upon U extraction efficiency, %
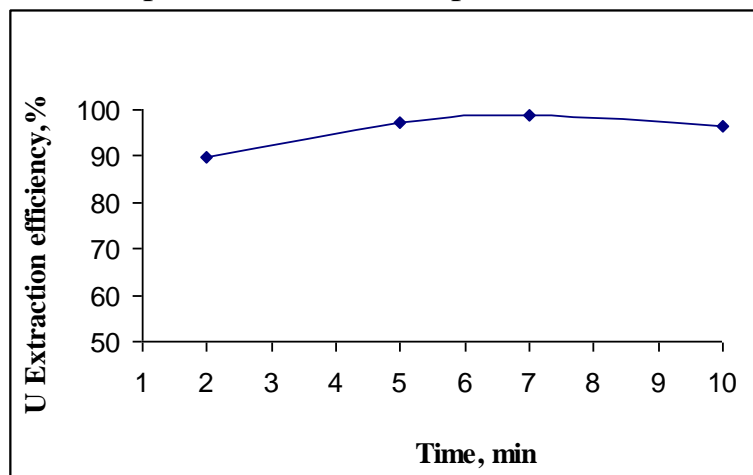

Fig. (4): Effect of contact time upon U extraction efficiency, %

Effect of (O/A) ratio, (McCabe Thiele diagram)

The effect of different O/A ratios upon U extraction efficiency was studied by contacting the present sulfate solution adjusted to pH0.75 with 2% (v/v) Alamine₃₃₆ in kerosene at different O/A ratios ranging from 2/1…. 1/1…… 1/2 for a contact time of 7min. U in the raffinate aqueous solution was analyzed after separation and its % extraction efficiency was calculated and tabulated as given in Table (5). The obtained data indicated that although the U extraction efficiency at O/A ratios of 2/1, 2/1.5 were 99 and 100%, respectively and their distribution coefficients D_A^O were 33.3 and ∞, respectively are higher than that of O/A ratio of 1/1 (98.7%) and D_A^O 44, but these ratios are not applied because they give a diluted loaded U in the organic phase.

On the other hand, the U extraction efficiencies at O/A ratios of 1/2 and 1/1.5 which were 84 and 92%, respectively and their D_A^O were 12.2 and 5.25, respectively. These O/A ratios were also not applied because they are lower

than that of 1/1. Indeed, the applied A/O ratio was 1/1which was also economic to save the organic solvent. McCabe Thiele diagram, Fig. (5) shows that there are only two extraction stages required for almost extraction of U from the prepared solution

Table (5): Effect of O/A volume ratio upon U extraction efficiency, %

O/A ratio	Aqueous phase conc., ppm	Organic phase conc., ppm	D_A^O	U Extraction efficiency, %
1/2	216	1134	5.25	84
1/1.5	68	828	12.2	92
1/1	10	440	44	97.6
1.5/1	4.5	223	33.3	99
2/1	0	150	∞	100

From the above study, it could be concluded that the maximum U extraction efficiency of 98% from Abu-Hamata sandy claystone sulfate solution was achieved by using 2% v/v of Alamine 336 at pH 0.75 and contact time of 7 min at O/A ratio of 1/1. By applying these conditions upon 500 mL of the working sulfate solution, it was found that 2% Alamine 336 in kerosene could uptake about 2000 mg of U with achieved U extraction efficiency of 89%. This could be attributed to the presence of interfering anions in the prepared sulfate solution e.g. SO_4^{2-}, HSO_4^- and Cl^- **[Merrit (1971), Ritcy, et al., (1993) and Mackenzie (1997)].** The loaded solvent was then directed to the stripping step for recovering its loaded U.
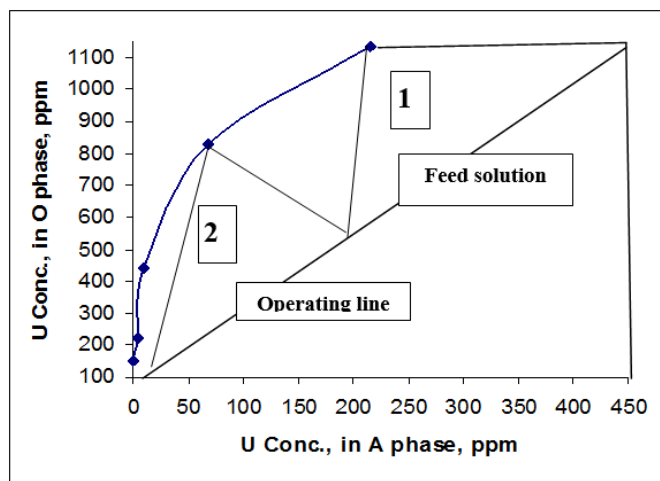

Fig. (5): McCabe Thiele diagram for extraction

Optimization of the stripping process
The stripping process is not only to regenerate the loaded organic solvent but also to obtain a U rich solution suitable to produce the preferred products. The

stripping process of the loaded U represented in equation. Different effective stripping parameters such as: type and concentration of stripping reagent, contact time and O/A volume ratios were studied to attain the maximum U stripping efficiency.

$$(R_3NH)_4 \, UO_2(SO_4)_3 + 4NaCl \longrightarrow 4(R_3NHCl) + Na_4UO_2(SO_4)_3$$
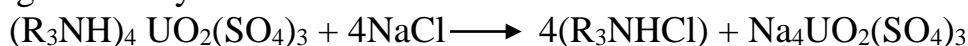
Effect of stripping reagent types.
The loaded Alamine 336 was stripped with different stripping reagents such as: 5% (H_2SO_4, HCl, HNO_3, NaCl and NaCl acidified in 0.2M H_2SO_4) at O/A volume ratio of 1/1 and contact time of 5 min. After separation, U in the strip solution was analyzed and its % stripping efficiency was calculated. The obtained data revealed that the maximum U stripping efficiency (93.9%) was given by using the acidified NaCl in 0.2M H_2SO_4 solution as the stripping solution. While the other reagents gave lower values as shown in Fig. (6).

Effect of strip solution concentrations
The of changing strip solution concentrations upon U stripping efficiency was conducted by mixing the loaded solvent with NaCl acidified in 0.2M H_2SO_4 in different concentrations such as: 2.5, 5, 7.5 and 10%. The other stripping parameters were kept constant at O/A volume ratio of 1/1 and contact time of 5 min. Results represented by Fig. (7) clearly revealed that U stripping efficiency showed great improvement from 78 to 93.9% as the strip solution concentration increased from 2.5 to 5%. On the other hand, further increase in strip solution concentration to 7.5 and 10% show slightly improvement in the U stripping efficiency which increased to 95.2 and 96.8%, respectively. Indeed, a concentration of 5% of the strip solution was already chosen and improved with other parameters.
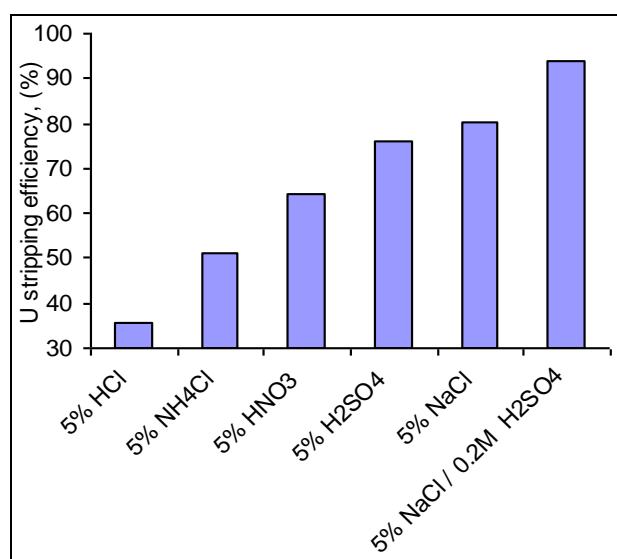

Fig. (6): Effect of reagents type upon U stripping efficiency, %.
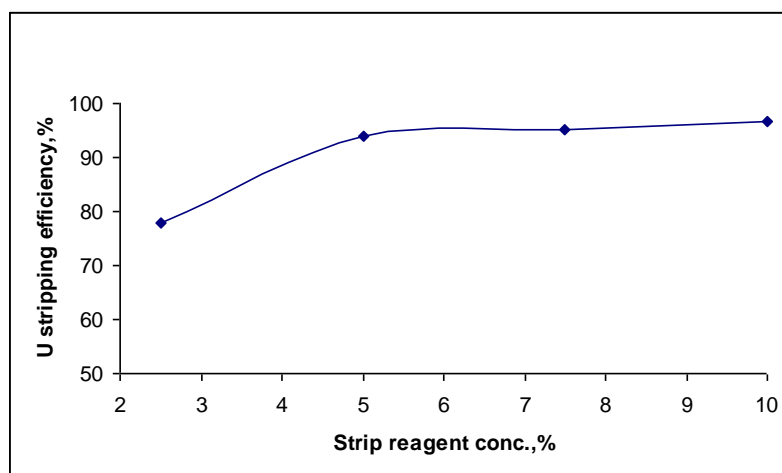
Fig. (7): Effect of reagent concentration on U stripping efficiency, %

Effect of contact time.

The effect of contact time was performed by contacting the loaded solvent with the strip solution of 5% NaCl acidified with 0.2M H_2SO_4 at different contact period times varying from 2 to 7 min at O/A volume ratio of 1/1. It was actually noticed that, U stripping efficiency increased from 76.2 to 93.9% by increasing the reaction time from 2 to 5min. Further increasing in the reaction time to 7 min decrease U stripping efficiency to 78%, Fig (8). The obtained data may be attributed to the transition of some U sulfate complex again to the regenerated solvent by increasing the striping time.
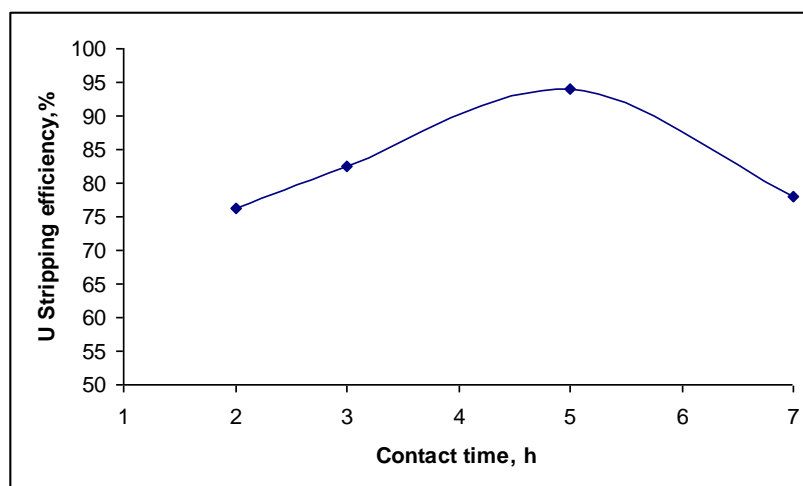
**Fig. (8):** Effect contact time upon U stripping efficiency, %

Effect of aqueous / organic (A/O) ratio.

The stripping efficiency of U from the saturated loaded Alamine 336 using 5% NaCl acidified with 0.2 M H_2SO_4 strip solution was investigated at different O/A volume ratios ranged from 1/2….1/1….1/2 by contacting for

5min. After separation and analysis of U in the strip aqueous solution, it was clearly observed that the almost loaded U was stripped with achieved stripping efficiency of 100% at O/A ratio of 1/2 and reached 96% at O/A of 1/15. These ratios were not applied because they yield U diluted solutions. On the other side, U stripping efficiency decreased to 63% and 72.2% at O/A ratios of 2/1 and 1.5/1, respectively, (Table 10 & Fig. 9). These ratios were also not applied because they are lower than the U stripping efficiency at O/A ratio of 1/1, (93.9%) which was the ideal for application.

Table (10): Effect of O/A volume ratio upon U stripping efficiency, %

O/A ratio	Conc., (O), ppm	Conc., (A), ppm	D_O^A	U Stripping efficiency, %
2/1	800	4800	6	63
1.5/1	720	4380	6.08	72.2
1/1	268	3932	14.67	93.9
1/1.5	160	2960	18.5	96
1/2	0	2000	∞	100
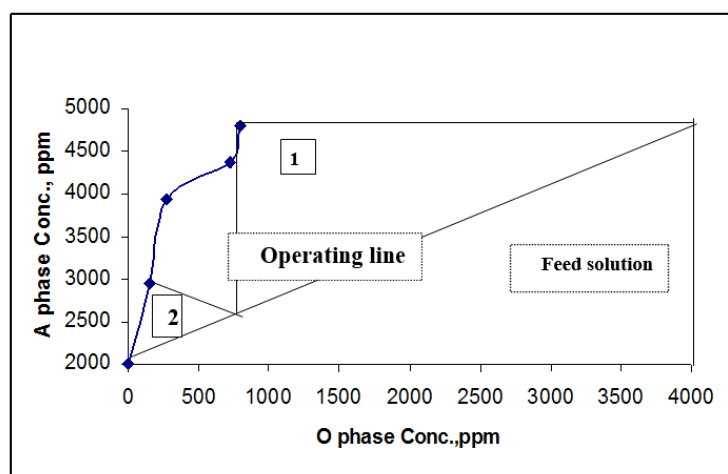
Fig. (9): McCabe Thiele diagram for stripping.

From the forgoing study, U was almost transfered to the aqueous soltion with an achieved U stripping efficiency of 93.9% by using 5% NaCl solution acidified with 0.2M H_2SO_4 and stripping time of 5 min at O/A ratio of 1/1.

Preparation of pure U_3O_8

The obtained U rich strip solution which assays 4.1 g/L of U was then treated with H_2O_2 solution for precipitating its U content as uranyl hydroxide, $UO_2(OH)_2$ with an achieved precipitation efficiency of 99%. The optimum precipitation conditions are pH 1, stirring time period of 3h at a temperature ranged from 20 to 25°C, (**Merritt, 1971**; **Shabbir and Tame, 1974; Gupta et**

al., 2004). After filtration and washing, the precipitated U hydroxide cake was ignited at 850°C for 1h to be crystallized. The produced U_3O_8 was washed, dried and directed to identification by using XRD analysis technique as shown in Fig (10). The chemical analysis of the final product of U_3O_8 showed that it had a purity of 98.5% with impurities such as: 0.2% of Cl^-, 0.13 of SO_4^{2-}, 0.1% of Fe, and 0.06% of V. The precipitation process is illustrated in equations:
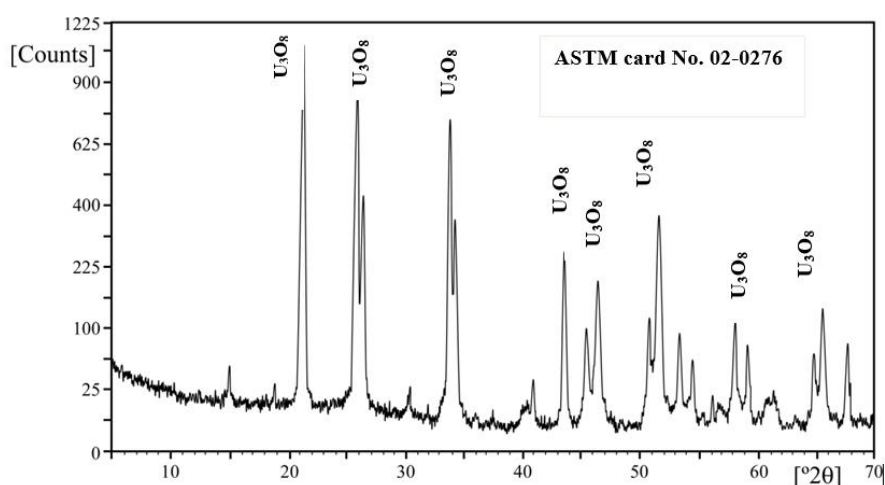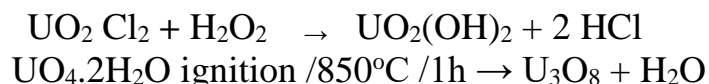
$$UO_2 Cl_2 + H_2O_2 \quad \rightarrow \quad UO_2(OH)_2 + 2\ HCl$$
$$UO_4.2H_2O\ \text{ignition}\ /850^{\circ}C\ /1h \rightarrow U_3O_8 + H_2O$$

Fig. (10): XRD pattern for the identification of pure U_3O_8

Conclusions

The potentiality of preparing highly pure U_3O_8 from sulfate solution of Abu Hamata sandy clay stone ore material assaying 0.45g/L of U has been attained via solvent extraction process. Alamine 336 was used for extracting 97% this purpose at O/A volume ratio of 1/1, contact time of 7.5mim and pH1. More than 93% of the loaded U was already stripped and transferred to the aqueous solution by using 5% NaCl solution acidified with 0.2M H_2SO_4 at A/O volume ratio of 1/1and stripping time of 5min.

Not less than 99% of U in the obtained strip solution was precipitated as $UO_2(OH)_2$ by using H_2O_2 solution at pH1, stirring time of 3h at a temperature ranged from 20 to 25°C. The obtained uranyl hydroxide cake was ignited at $850\,^{0}$C to produce U_3O_8 of achieved purity of 98.5%.

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استخلاص اليورانيوم باستخدام اللامين 336 فى الكيروسين من محلول الكبريتات الخاص بخام الطين الرملى منطقة ابوحماطة ـ جنوب غرب سيناء ـ مصر

1 جلال محمود عبدالوهاب و1 وليد محمود عبداللاه 2 محمد فتحى الشحات

1 هيئة المواد النووية
2 كلية العلوم جامعة عين شمس

هذه الدراسة تهتم بامكانية استخدام المذيب العضوى باستخدام الالامين 336 فى الكيروسين فى استخلاص اليورانيوم من محلول الاذابة الكبريتى الخاص بخام الطين الرملى منطقة ابوحماطة جنوب غرب سيناء هذا المحلول يحتوى على 0.45جم/لتر من اليورانيوم المذاب بنسبة 98.8% واثبتت النتائج ان عملية الاستخلاص تعتمد على عدة عوامل منها تركيز الامين 336 و الاس الهيدروجينى و زمن الخلط و نسبة الخلط المذيب والمحلول .

اليورانيوم المحمل فى المذيب يتم استرجاعه بواسطة محلول 1 مولر كلوريد الصوديوم المحمض بحامض الكبريتيك و كانت نسبة الاسترجاع 93.9% عند نسبة الخلط المذيب والمحلول 1/1 و زمن الخلط 5 دقائق . تم ترسيب 99% من اليورانيوم المسترجع بواسطة محلول فوق اكسيد الهيدروجين لإنتاج اكسيد اليورانيوم.