

Investigations onto the Recovery of Uranium and Rare Earth Elements from High Sulfate Concentrations Leach Liquor of Abu Hamata Sandy claystone ore material via Anion-Exchange Resin process

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Abstract

Excess of free sulfate anions in a sulfate leach solution became an offending object for uranium recovery by using Amberlite IRA₄₀₀ anion exchange resin. This is because, not only uranium U loading efficiency is reduced significantly, but also the selectivity for the uranyl sulfate complex, $[\text{UO}_2(\text{SO}_4)_3]^{4-}$ over ferric sulfate complex, $[\text{FeO}(\text{SO}_4)]^-$, is reduced.

The present study concerned with the feasibility of using Amberlite IRA₄₀₀ anion exchange resin for effective recovery of uranium from highly concentrated sulfate solution of Abu Hamata sandy claystone ore sample. The latter solution was found assaying 0.45 g/L and 26 g/L of U and SO_4^{2-} , respectively, together with 0.32 g/L, 0.05 g/L, 0.008 g/L and 2.3 g/L of rare earth elements, (REEs), V, B as well as Cl^- , respectively. Such solution was treated with solid CaCO_3 at different pH values ranged from 1 upto 3 for precipitating the excess free sulfate. The obtained results indicate that, U extraction efficiency has been highly improved from 48.4% up to 94.4% at pH 1.8.

Keywords: sandy claystone ore, H_2SO_4 , agitation leaching, Recovery, Anion exchange resin, Amberlite IRA₄₀₀.

Introduction

Abu Hamata sandy claystone rock facies of Um Bogma Formation, which occurs at Abu Zeneima area, Southwestern Sinai, Egypt, is considered as one of the most important occurrences of U mineralization beside the associated economic metal values e.g. Ln, V, B, Zn, Cu,.... etc (AlShami, 2003). Several workers have studied in detail the mineralogy of different rock units

of Um Bogma Formation. The most important of them were **Dabbour and Mahdy (1988)**; **Hussein et al. (1992)**; **Amer (1993&1997)**; **Abd el Monem et al., (1997)**. They recorded various important secondary U minerals such as uranophane, beta-uranophane, carnotite, kasolite, meta-torbernite, meta-uranocircite, bassetite and clarkeite.

With respect to U recovery studies, agitation leaching with H_2SO_4 is the predominate process for U dissolution from most of Abu Zeneima ore materials. From the latter, U is effectively recovered from the sulfate leach liquors via ion exchange resins. The anion exchange resins such as Amberlite IRA₄₀₀ exhibits high selectivity for different U anionic complexes e.g. $[UO_2(SO_4)_2]^{2-}$, $[UO_2(SO_4)_3]^{4-}$ and $[UO_2(CO_3)_3]^{4-}$. The main problem affecting the extraction of U anionic species from the sulfate solutions is the presence of interfering anions especially MoO_4^- , HSO_4^- , SO_4^{2-} , $H_2PO_4^-$ and Cl^- , as well as, the cation of Fe^{3+} which compete U anionic complexes ones on the resin sites and decrease its loading capacity. The order of the relative affinity of these anions for resin of the quaternary ammonium functional group was found to be as follows: $MoO_4^- > U > HSO_4^- > SO_4^{2-} > H_2PO_4^- > Cl^-$.

Many authors studied the recovery of U from different sulfate solutions containing high concentrations of the interfering sulfate anions, e.g. **Amer (1993)**; **Ghonaim et al. (2004)**; **Ladeira and Morais (2005)**; **Rezik (2005)**; **Zaki (2007)**; **Guettaf et al.(2009)**; **Abdel Hy, (2012)**. Accordingly, they reported that the loading capacity of the used resins was decreased to limit values ranging from 43-75% of their theoretical capacities due to the competing of some anions especially sulfates. Likewise, **Gupta and Singh (2003)** reported that the amount of U adsorption is decreased when the ratio of $[SO_4^{2-}] / [U]$ is more than 2 in the leach liquor.

In this regard, according to **Morais et al. (2005)** the amounts of free sulfate anions in the sulfate solutions could be reduced via precipitation as hydrated calcium sulfate ($CaSO_4 \cdot 2H_2O$) by the addition of aqueous lime milk (CaO solution) at pH values ranging from 1 to 4. Also, **Coleman (1953)** used a solid $MgCO_3$ to raise the pH value of the sulfate solution with reduction of the excess amounts of free sulfate to improve U loading capacity of the resin.

In this context, the main target of the present work is to recover both of U and REEs, as well as, to reduce the effect of high concentrations of free sulfate interfering anions via the addition of solid $CaCO_3$. The treated solution would raise the sulfate solution pH value suitable for the extraction process when using the anion- exchange resin Amberlite IRA₄₀₀.

Experimental

Experimental procedures include not only studying in detail both of mineralogical and chemical characteristics of a representative sample of Abu Hamata sandy claystone, but also studying its chemical processing

Mineralogical Characteristics

A weighted 1kg of the studied ore material ground to a mesh size of -60 was carefully washed several times with tap water to remove slimes. After filtration, dryness and sieving by using a set of different sieves ranging from 500 to 100µm (-35 to -140 mesh size). Each of the collected fractions was subjected to heavy liquid separation procedures by using bromoform (sp.gr. 2.84). The obtained heavy fractions were investigated using binuclear microscope to pick up several different mineral grains. The latter were examined using X-ray diffraction analysis, (XRD), and scan electron microscope, SEM-EDAX, PHILIPS. Figs. (1&2) revealed the presence of both U mineral, [Renardite, $\text{Pb}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$] and Clay mineral, [Montmorillonite (K,Ca) Al_2SiO_4].

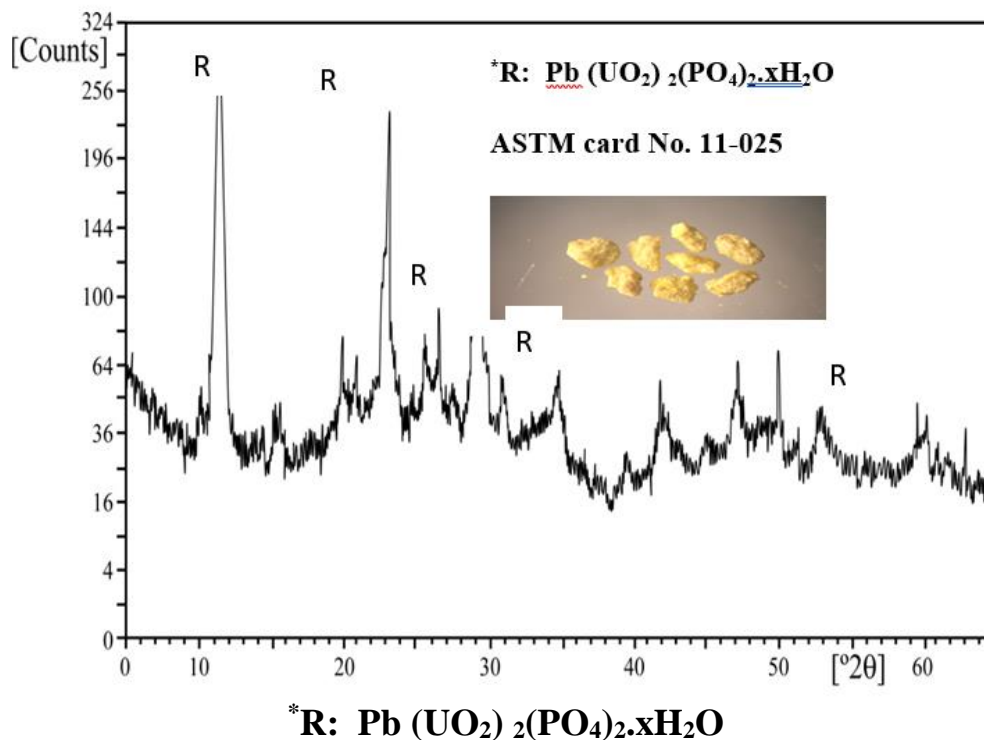


Fig. (1): XRD pattern of Renardite mineral grains

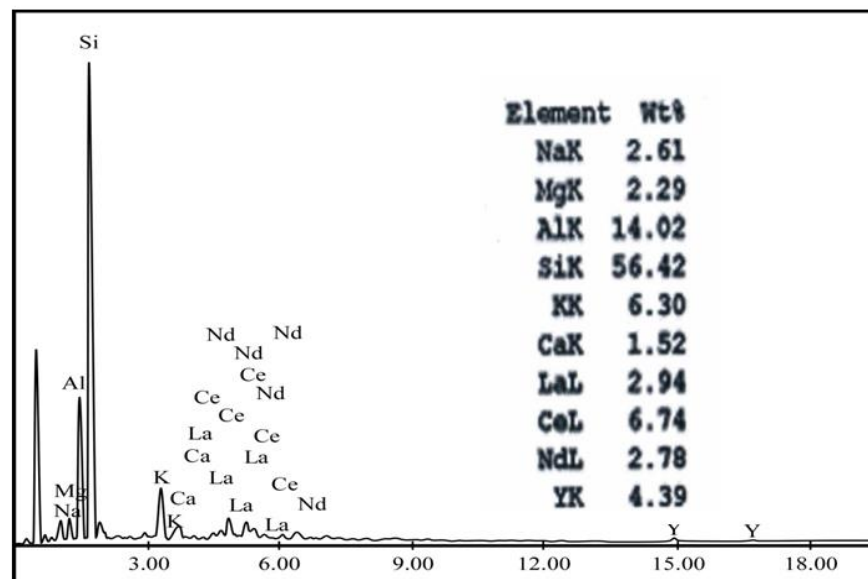


Fig. (2): SEM-EDAX chart of adsorbed REEs on montmorillonite mineral

Chemical Characteristics:

With respect to chemical composition of the working ore sample, a weighted 1g portion ground to a mesh size of -200 was digested with the acid mixture, [H₂SO₄, HCl, HNO₃ and HF] for the complete dissociation and transferred into a 100 mL measuring flask. The prepared sample solution was subjected to different analytical techniques for its chemical composition. The major oxides such as: Fe₂O₃, Al₂O₃, MgO and CaO were classically estimated by wet chemistry according to **Shapiro (1975)**. While the analysis of Si required the fusion of 0.1g of the fine ground ore sample with 1g of NaOH pellets in a nickel crucible for 20 min directly on Bunsen flame and the fused matrix is dissolved with HCl 1:1 and closed up to volume of 100 mL. The estimation of Si was carried out using UV-vis spectrophotometer (Shemadzu 610) at λ_{640} nm. On the other hand, UV-vis spectrophotometer (Shemadzu 610) was used for the quantitative analysis of the REEs using Arsenazo III at λ_{654} nm, [**Marczenko (1986)**]. Uranium is analyzed via oxidometric titration method against NH₄VO₃, [**Mathew et al., (2009)**]. While other trace elements e.g. V, B, Zn, Cu, Pb, Ni,...etc were determined by using the flame atomic absorption spectrophotometer, (FAAS), Unicam 969. Finally, the products of U and REEs were confirmed using both of XRD and SEM-EDAX analysis techniques. Table (1) represents the total chemical composition of Abu Hamata ore sample

Table (1): Chemical constituents of Abu-Hamata ore sample.

Major oxides	Conc., %	Trace elements	Conc., ppm
SiO ₂	55.6	U	1830
Al ₂ O ₃	12.7	REEs	1375
Fe ₂ O ₃	9.81	V	1750
CaO	0.68	B	1100
MgO	2.0	Cu	650
K ₂ O	3.7	Zn	345
Na ₂ O	0.92	Cr	833
TiO ₂	1.0	Pb	289
P ₂ O ₅	0.21	Ba	448
MnO	0.04	Co	46
*L.O.I	11.1	Ni	64
Cl ⁻	0.94	Ga	25
SO ₄ ²⁻	0.043		
Total	98.743		

* Loss in ignition

Chemical analysis represented in Table (1) of the studied ore sample revealed that it is mainly composed of SiO₂ (55.6%) beside Al₂O₃ (12.70%) and Fe₂O₃ (9.81%) together with of K₂O (3%). So, this ore sample is mainly sandy claystone ore material. On the other hand, the chemical composition of the ore sample indicates the presence of considerable concentrations of some adsorbed trace elements e.g. V and B.

Preparation of Sulfate Pregnant Solution

Agitation leaching experiments for preparing pregnant sulfate solution were conducted by mixing 1kg of the studied ore material (ground to a mesh size of -150) with 5% H₂SO₄ acid solution at solid / liquid (S/L) ratio of 1/2 and stirring for 1h at a leaching temperature of 75 °C. After filtration and washing, the total volume of the yielded sulfate solution became 4L and had pH 0.5. Elements of interest e.g. U and REEs beside the interfering elements e.g. V and B were estimated. The latter was subjected to different extraction processes to recover the elements of interest. The prepared sulfate leach liquor of the chemical composition given in Table (2) was then directed to sequential extraction procedures. The expected chemical reactions of the ore sample with H₂SO₄ are illustrated in the following equations:

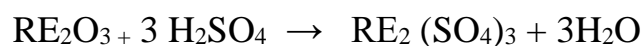


Table (2): Chemical composition of the pregnant sulfate solution

Elements ions	Conc., g/L	Leaching efficiency, %
U	0.450	98.9
REEs	0.320	93.1
SO ₄ ²⁻	26.000	Free acidity
Fe	0.876	5.1
Cl ⁻	2.340	99.6

Extraction Processes

Extraction processes involved two subsequent successive steps, the extraction of U from the sulfate solution by using the anion exchanger resin, Amberlite IRA₄₀₀ followed by direct precipitation of the total REEs.

Ion exchange extraction of U

The prepared sulfate solution was first treated with solid CaCO₃ to reduce the concentration of interfering sulfate anions from one hand and to adjust the pH of the pregnant solution to its suitable value for loading process on the other hand. However, SO₄²⁻ anions were precipitated in the form of CaSO₄.2H₂O. After filtration, the filtrate was directed to the ion exchange unit consists of a column of 50 cm height and 5 mm diameter packed with 10 mL of wet settled resin (wsr) of Amberlite IRA₄₀₀ in its sulfate form. This column was used to recover U from the mentioned treated sulfate solution at the applied flow rate of 1ml/min. U in the collected effluent solutions of bed volumes of 100 mL was analyzed and the extraction efficiency was calculated.

Before applying the elution process, the saturated loaded resin was washed with suitable volume of distilled H₂O to get rid of any impurities of the pregnant solution. The elution process was performed by using an eluant solution of 1N NaCl acidified with 0.2 N H₂SO₄ at a slow flow rate of 0.5 ml/min (equivalent to 11 min contact time). Fractions of 10 mL bed volumes were collected to determine the relative elution efficiency.

Direct precipitation of REEs

The effluent solution of pH 1.8 free from U was subjected to selective precipitation of REEs by using 10% H₂C₂O₄ acid solution. After filtration and washing, the precipitated RE₂(C₂O₄)₃ cake was then dissolved in 5% HCl solution and re-precipitated with NH₄OH solution at pH 9.5 to remove Ca impurities.

Results and discussion

Extraction of U from the Pregnant Solution.

The prepared sulfate solution given in Table (2) emphasized the presence of high concentration of both interfering SO₄²⁻ and Cl⁻ anions, (26 & 2.3 g/L, respectively). These relatively high concentrations of both SO₄²⁻ and Cl⁻

would complicate the U loading process when using Amberlite IRA₄₀₀ anion-exchange resin, [Gupta and Singh (2003); La Brooy, et al. (2009) and Ivanova, et al. (2009)]. When using this solution, the maximum U loading efficiency did not exceed 48.4%. Fig. (3) clearly shows the low amount of the loaded U. So, it was very important to reduce the concentrations of both interfering SO₄²⁻ and Cl⁻ to their minimum lower limits to overcome this problem and increasing U loading efficiency. For this purpose, the pregnant sulfate solution should be treated with the addition of solid CaCO₃. Where the excess SO₄²⁻ anions were already precipitated as CaSO₄ cake which adsorbed the chloride salts upon its outer surface. The addition of solid CaCO₃ also increased the pH value of the pregnant solution via decreasing the concentration of free SO₄²⁻ and Cl⁻ anions to be suitable for U loading by the anion exchange resin as represented by Table (3). The expected chemical reaction is illustrated in the following equation:

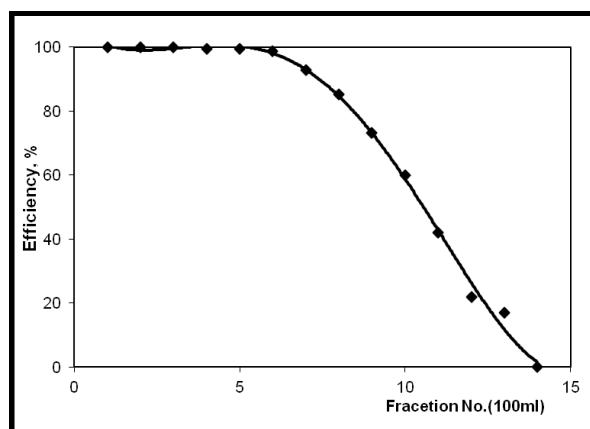


Fig. (3): Adsorption curve of U from the pregnant sulfate solution

Table (3): Effect of addition of solid CaSO₄ to sulfate solution

Added CaCO ₃ , g	SO ₄ ²⁻ Conc., g/L	Cl ⁻ Conc., g/L	pH value	Lost U, mg/L
5	21.6	2.1	1.2	0
10	16	1.9	1.3	0
15	11.6	1.5	1.5	0
20	6.1	1.1	1.8	3
22.5	3.8	0.83	2.5	55
25	2	0.56	3	120

The obtained data show that the addition of 20 g of CaCO₃ per liter of the pregnant sulfate solution leads to decrease the concentration of free SO₄²⁻

anions gradually up to 6.1g/L. Also, the concentration of the adsorbed U upon the precipitate of $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ is negligible. In the mean time, the solution pH value increased to be pH 1.8 which is very suitable for U loading process. On the other hand, further addition of CaCO_3 from 20 to 25 g increased significantly the loss of U by adsorption on the precipitate to 120 mg /L. Therefore, addition of 20 g of CaCO_3 per liter of the sulfate solution was preferred to overcome the loss of U.

Extraction of U from the Treated Sulfate Solution.

The pregnant sulfate solution of Abu Hamata sandy claystone ore sample was treated with the addition of 20 g CaCO_3 solid per liter to reduce the concentration of the interfering SO_4^{2-} anions to suitable value, (6.1 g/L) by precipitation as $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. After filtration, the filtrate solution was passed through the prepared anion exchange resin column to extract its U content. The applied flow rate was 1mL/min (equivalent to a contact time of 4min). The obtained effluent was collected every 100 mL for U analysis and the results were plotted to establish U adsorption (loading) curve, (Fig. 4). From the figure it is noticed that U loading amount was increased, and its loading efficiency was highly improved to achieve a maximum value of 94.4%.

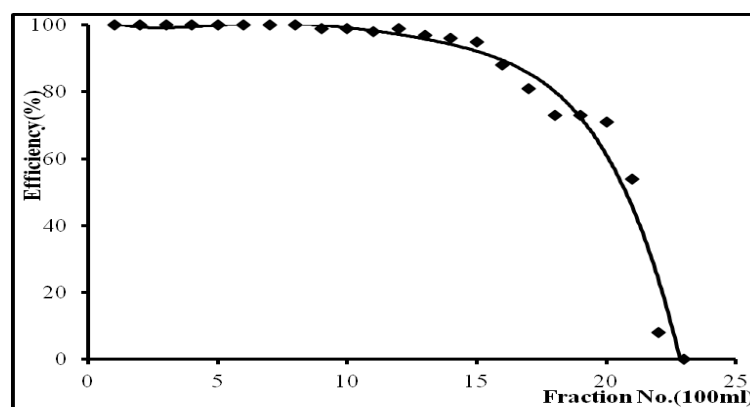


Fig. (4): Loading curve of U from the treated sulfate solution

Elution Process

The elution process is not only used to regenerate the loaded resin but also to obtain a U rich eluate solution suitable to prepare the preferred U products. After the resin bed was fully saturated with U, it was rapidly washed with suitable volume of distilled H_2O to get rid of any impurities. An eluant solution of 1N NaCl acidified with 0.2N H_2SO_4 was used to perform the U elution process. The eluant was passed through the loaded resin at a working flow rate of 0.5 mL/min (equivalent to 11min contact time). The eluant solutions of each 10mL bed volume were collected to determine their U

concentration. The corresponding elution curve was plotted as shown in Fig. (5) which represents the amount of the eluted U. This amount of the eluted U represents a maximum U elution efficiency of 98.8%.

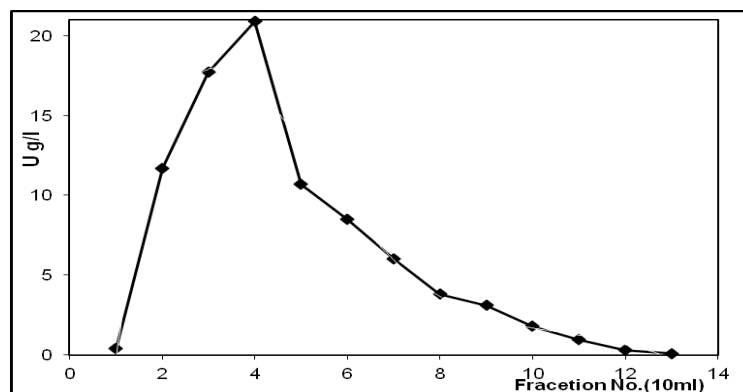


Fig. (5): Elution curve of U from the loaded Amberlite IRA₄₀₀

Precipitation of Yellow Cake

The eluted fractions rich in U were collected and then subjected to direct U precipitation via neutralization by using NaOH solution. The total eluant solution which assays 8.6 g/L of U was gradually neutralized by the addition of 20% NaOH solution. The complete precipitation of yellow cake, ($\text{Na}_2\text{U}_2\text{O}_7 \cdot 6\text{H}_2\text{O}$) was attained at pH 7.8 with continues stirring for 1h. After filtration and washing, the produced crude yellow cake was then dried at 110 °C and confirmed by SEM- EDAX analysis as shown in Fig. (6). While the chemical analysis of the prepared yellow cake revealed that it assayed 59.4% of U with total purity of 80% and had some impurities such as: 6.8% of Na, 0.23% of V, 5.34% of Fe, 0.6% of Zn, 0.16% of Si and 0.03% of Mg. The latter would be adjusted to a purification process to prepare U product in a nuclear grade.

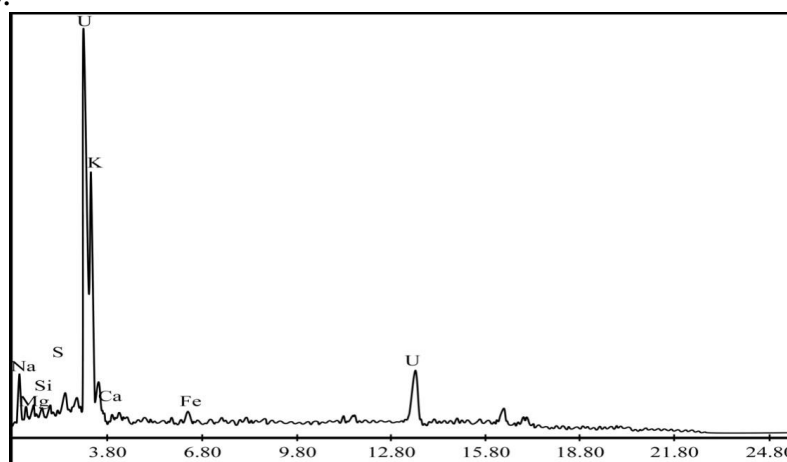


Fig. (6): EDAX chart of the precipitated crude yellow cake

Purification of Yellow Cake

Purification process of the crude yellow cake was conducted via dissolving in 3M HNO₃ and re-precipitation of uranyl nitrate U anion complex [UO₂(NO₃)₃]⁻ with gradual addition of H₂O₂ solution together with continues stirring for 3h at 20-25 °C. Not less than 98.9% of U was precipitated at pH 1 in agreement with **Bhowmik et al. (2009)**. After settling and filtration, U and the associated impurities were analyzed in the filtrate to calculate their % precipitation efficiencies. The U precipitate was washed with suitable volume of distilled H₂O to get rid of any adsorbed impurities. After drying and ignition at 850 °C for 1h, the produced U₃O₈ was confirmed by using either XRD and SEM-EDAX techniques as shown in Figs. (7& 8). The purification equations are illustrated in the following equations:

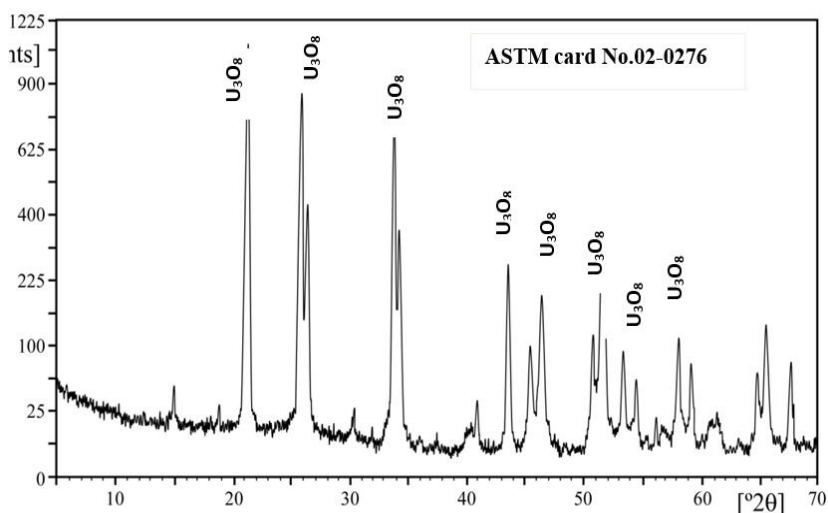
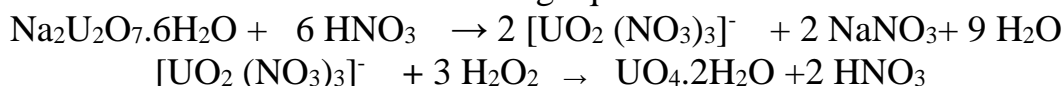


Fig. (7): XRD pattern of highly pure U₃O₈

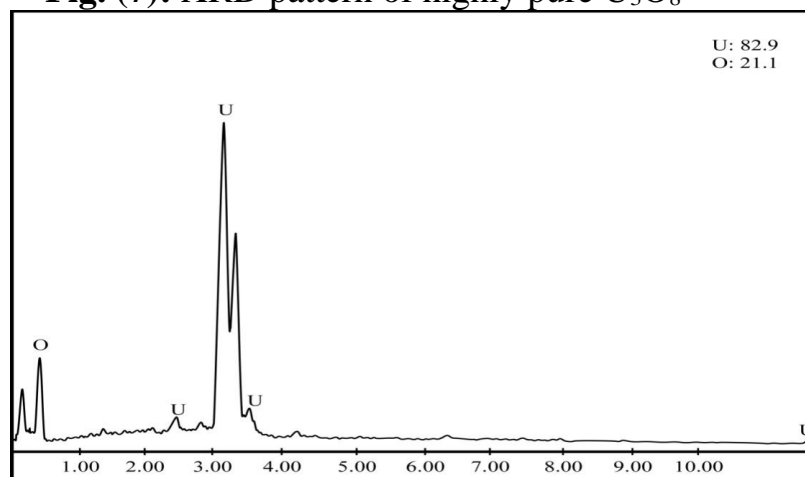
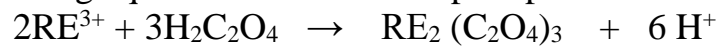


Fig. (8): SEM-EDAX chart of highly pure U₃O₈

Direct Precipitation of REEs

The total REEs in the pregnant sulfate solution free from U were selectively precipitated by the addition of 10% H₂C₂O₄ acid to form stable insoluble REEs oxalates, **Habashi (1993)**. Complete precipitation of REEs was attained at pH 1.1 after stirring for 45 min. After filtration and washing, the obtained RE-oxalate cake was dried at 110°C for 2h and then subjected to chemical analysis to identify its content of total REEs and the associated impurities. It was found that, the total REEs content in the produced cake achieved 85.2%, while the other matching impurities include 12% of Ca, 0.2% of Zn and 0.3% of Cu. The following equation illustrates the precipitation of RE oxalate cake:



On the other hand, to purify the produced RE oxalate cake, it was dissolved in 5% HCl acid solution and then re-precipitated by adding NH₄OH solution at pH 9.5 to obtain RE₂(OH)₃ cake free from Ca ions, **Agasyan and Tsyurupa (1980)**. After filtration, washing and ignition at 550°C, the pure product of RE₂O₃ was obtained which confirmed by means of SEM-EDAX technique, Fig. (9). It is clearly evident that the produced RE₂O₃ is composed of light REEs mainly Ce, La and Nd which represent about 74% of the prepared RE₂O₃. The following equations illustrate the purification process.

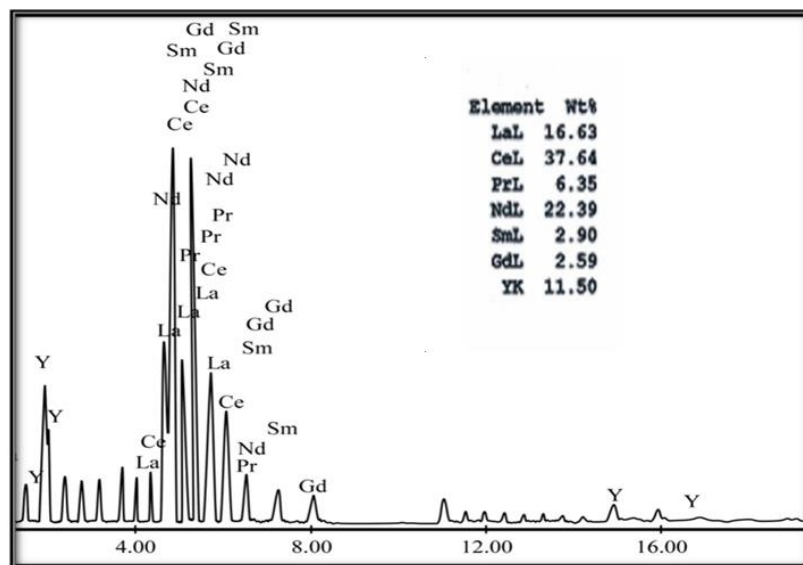
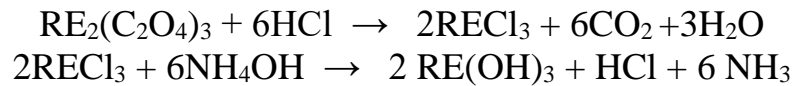


Fig. (9): SEM-EDAX chart of pure RE₂O₃

Conclusions

The mineralogical investigation into Abu Hamata sandy claystone ore sample proved the presence of both U mineral, [Renardite, $\text{Pb}(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 8\text{H}_2\text{O}$] and the REEs adsorbent clay mineral, [Montmorillonite $(\text{K},\text{Ca})\text{Al}_2\text{SiO}_4$]. Therefore, it is easily process for dissolving 98.9% of U and 93.2% of total REEs by using 5% H_2SO_4 acid solution at S/L mixing ratio of 1/2 and stirring for 1h at 75°C.

The prepared pregnant sulfate solution has high concentrations of the interfering free sulfate (26g/L). So, the U loading capacity upon the anion-exchange resin, Amberlite IRA 400, decreased to very low limit about 48.4%. Thus, a solid CaCO_3 was added to reduce these interfering anions. The loading capacity of U on the resin improved to a value more than 94%. The loaded U was eluated by using 1N NaCl solution acidified with 0.2N H_2SO_4 with the achieved elution efficiency of 98%. Up to 99% of U in the concentrated U eluate solution was directly precipitated by using 30% NaOH solution to precipitate yellow cake of Sodium diuranate of purity reached 80%. The latter was dissolved in 3M HNO_3 solution and re-precipitated by using H_2O_2 solution to prepare nuclear grade U product. The effluent solution free of U was subjected to total REEs recovery via their oxalate precipitation at pH 1.1. Finally, a working flowsheet represented in Fig. (10) was suggested to summarize the processing of Abu-Hammata ore material to produce pure U_3O_8 and RE_2O_3 .

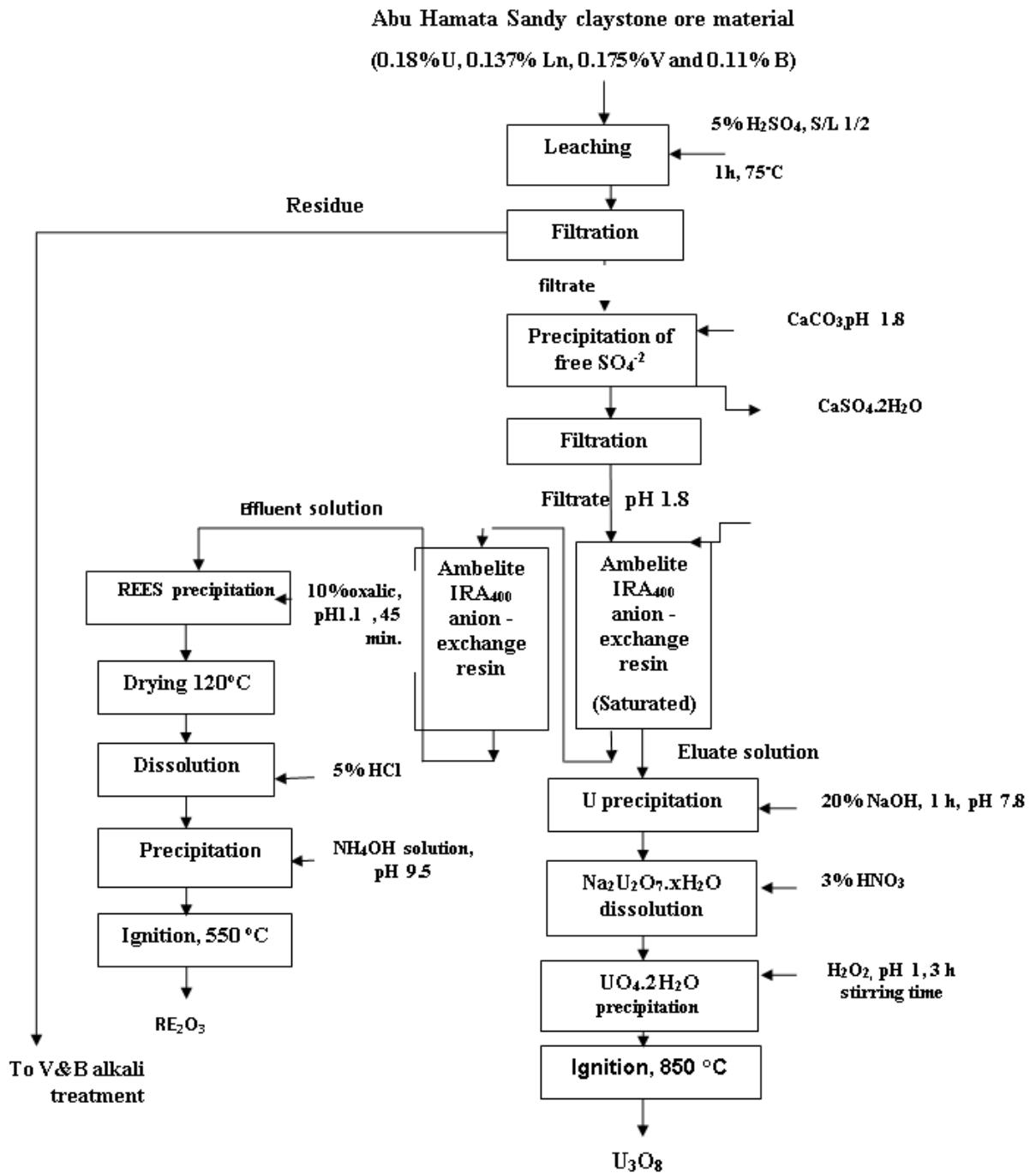


Fig. (10): Proposed technical flowsheet for the processing Abu Hamata sandy claystone ore material.

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تتبع إستخلاص اليورانيوم والعناصر الأرضية النادرة من محلول الإذابة عالي الكبريتات لخامة أبو حماسة بعملية التبادل الأنيوني

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الملخص العربى

التركيزات العالية لأنيونات الكبريتات فى المحاليل الكبريتية أصبحت مشكلة ملحة فى عملية إستخلاص اليورانيوم بطريقة التبادل الأنيوني على راتنج الأمبرليت IRA400 وذلك بسبب تقليل كفاءة تحميل أنيون متراكب اليورانيوم على الراتنج لإنتقائه من متراكبات أنيون الحديد. وهذه الدراسة أهتمت بإمكانية إستخدام عملية التبادل الأنيوني الفعال لإستخلاص اليورانيوم من محاليل الإذابة عالية الكبريتات لخامة أبو حماسة. وهذا المحلول يحتوى على 0.45 جم/لتر من اليورانيوم و 26 جم/لتر من أنيونات الكبريتات 0.32 جم /لتر من العناصر الأرضية النادرة و 0.05 جم/لتر من الفانديوم و 0.008 جم/لتر من البورون وكذلك 2.3 جم/لتر من أنيونات الكلوريد. مثل هذا المحلول يتم معالجته بإضافة كربونات الكالسيوم الصلبة عند أرقام أسية هيدروجينية مختلفة تتراوح بين 1 , 3 وذلك لترسيب أنيونات الكبريتات الزائدة. والنتائج التى تم الحصول عليها أوضحت زيادة كفاءة الراتنج لإستخلاص اليورانيوم من 48.4 % الى 94.4 % عند رقم الأس الهيدروجينى 1.8.