

Sequential extraction studies of Cu, Cd and U compounds from Gibbsite – bearing shale material, Abu Thor area, Southwestern Sinai, Egypt.

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Abstract

Gibbsite-bearing shale material was recorded within intra-Carboniferous paleokarst profile of Um-Bogma Formation at Abu Thor area. The present paper is concerned with subsequent separation of Cu, Cd and U compounds from the sulfate solution of the present ore material. Abu Thor representative sample assays 0.5% of Cu, 0.014% of Cd and 0.04% of U as the elements of interest. The effective leaching parameters such as: curing temperature, H₂SO₄ concentrations, solid / liquid addition ratio, leaching time and leaching temperature were planned to investigate. The obtained sulfate solution under the resulting optimum leaching conditions was found assaying 2.24 g/L of Cu, 0.066 g/L of Cd and 0.19 g/L of U with maximum leaching efficiencies of 92%, 96% and 95% , respectively. In addition 0.008% of V with maximum leaching efficiency of 8.6% The prepared sulfate solution was treated with 0.5% Na₂S solution at pH 0.35 for selective separation of CuS where CdS was subsequently selectively separated at pH 0.5. The present U in the solution free from both Cu and Cd was oxidized with H₂O₂ solution and recovered with anion exchange resin Amberlite IRA₄₀₀.

1-Introduction

Uranium mineralization of Um-Bogma Formation southwestern Sinai, Egypt was studied by many authors, eg: **(El Reedy et al. 1988), abbour and Mahdy (1988), El-Sharkawi et al (1990), Hussein et al (1992), Aita (1996) and Abd El Moneim et al (1997).** Mira and Aita, **(2009)** postulated that, the Carboniferous stratabound Cu and U in the form of copper carbonate, chloride, silicate, sulfate and phosphate minerals of supergene origin concentrated by pedogenic processes and latosol formation.

Such Cu-U mineralized ores were already leached with sulfuric acid as reported with several researchers, eg : **Amer (1993 &1997)** and **Abd El Fatah (2003).**

Cadmium metal and salts has gained great importance due to its wide applications in civil and many purposes as well as in nuclear field. It is characterized by its ability to absorb thermal neutrons during the chain reaction inside the nuclear reactors. It is also used in the manufacture of sheets which directed the chain reactions, **El Hazek, (1991).** Cd is comparatively rare in almost Zn ores which are already distributed in all Sinai ores and Pb-Zn deposits which are already distributed along Red Sea coast. It was estimated that Zn is 200 times as abundant as Cd.

According to **Abd El Wahab (2008)** 0.1% of Cd was carried by the Zn mineral hemimorphite (15.3% Zn) in Egypt. Also **Bodes (1996)** reported that about 2.1% of Cd was found in the same Zn mineral in Thailand. Also, **Onal et al. (2005)** found 164g/t Cd carried by Pb–Zn deposits ore sample containing 0.17 % Pb and 10.98 % Zn from Aladag in Turkey.

Several studies were mentioned upon the leaching of Cd carried in both Pb-Zn deposits and Zn ores using sulfuric acid: according to **M1ahdy et al. (2009)** about 90% of Cd was leached by using 40% H₂SO₄ solution from Um-Gheig Pb-Zn deposits Red Sea Coast, Egypt. Also, **El Sheikh and Abd El Wahab (2009)** dissolved about 96% of Cd from Ni-Cd scrap rechargeable 6batteries by using 5M H₂SO₄ solution. Moreover, **Collins (1990)**, **Ashman (1990)**, **Bodas (1996)** and **Nathsarama (2006)** used 15% H₂SO₄ solution for leaching Cd from different ores. In addition **Abd El Wahab (2016)** has been dissolved 100% of Cd from the residual fly ash sample of Egyptian power stations by using 25% H₂SO₄ solution.

In fact, it is possible to separate Cd²⁺ from the acidic solutions containing Cu²⁺ by direct precipitation using sulfide solution depending upon the large difference in their solubility products ($K_{sp} \text{ CuS} = 2.4 \times 10^{-36}$ and $K_{sp} \text{ of CdS} = 7.9 \times 10^{-27}$) **Alexeyev (1973)** ; **Abd El Wahab et al (2012)**. In addition, by pH adjustment the complete separation between the two elements by varying the sulfide ion concentration could be achieved, **Posypaiko and Vasina(1979)**.

2-Experimental

2.1. Sample preparation and characterization.

A representative sample weighted 50Kg was collected from the upper organic-rich gibbsite-bearing shale mudstone sub horizon of the latosol horizon of Abu Thor area (Fig.1). A small portion of the representative quartered sample was sent to *AcmeLabs* in Canada for complete chemical composition, except SiO₂.

A sample weighted 2Kg of the representative quartered sample was roasted on oven at 250°C for 2h to eliminate both moisture and crystalline water to be more concentrated. For Si estimation a small portion weighted of 0.1g of the fine ground ore sample is fused with 1g of NaOH pellets in a Nickel crucible for 20min directly on Bunsen flame and the fused matrix is dissolved with 1:1 HCl solution and closed up to a measuring volume of 100mL. By using UV-vis spectrophotometer (Shemadzu 610) at $\lambda_{640\text{nm}}$ Si content could be analyzed, **Marczencko (2000)**. In addition, the elements of interest Cu, Cd, V as well as other elements e.g. Zn, Ni, Co,...etc

were determined during experiments using the flame atomic absorption, (FAAS), Unicam 969. On the other hand, U was analyzed by oximetric titration against NH_4VO_3 , (Daveis and Gray, 1964). While the elemental relative concentration of the final products was identified by (SEM-EDX & XRD), PHILIPS. The pH values of the aqueous solutions were adjusted with a pH meter was calibrated by using standard solution of pH buffer value 1.04, 7.00 and 10.00. (Digimed DM-21).

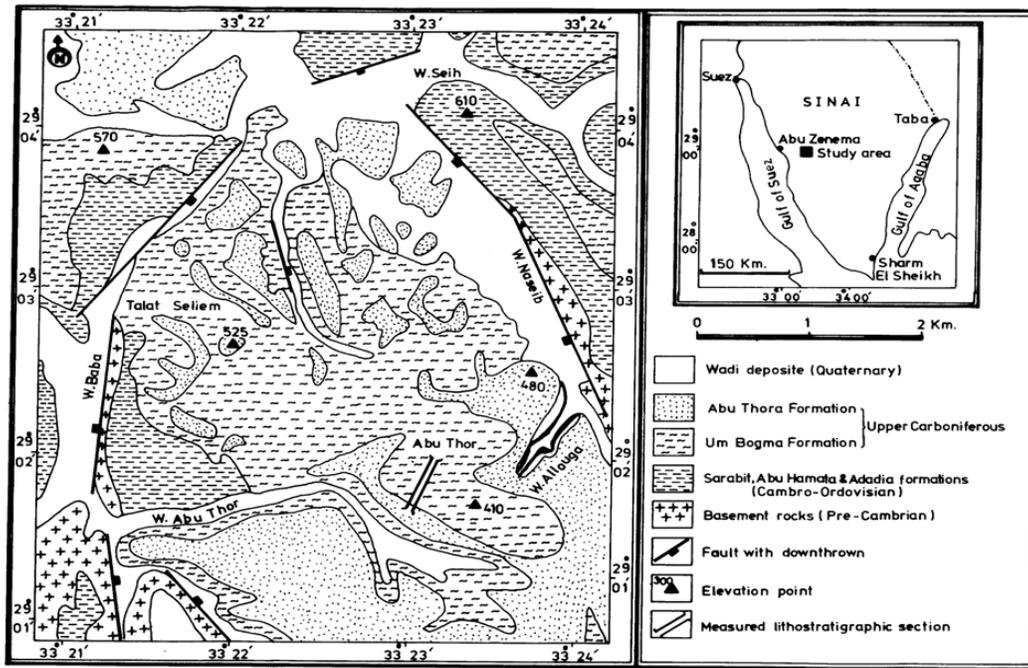


Fig. (1): Geologic Map of Abu Thor Locality. (Mira and Aita, 2009)

2.2. Optimization of H_2SO_4 leaching parameters.

All the agitation leaching procedures were performed by mixing 10g of the fine ground prepared ore sample (-200 mesh) with H_2SO_4 acid. Different effective leaching parameters such as: H_2SO_4 acid concentrations, solid/liquid (S/L) mixed ratios leaching time and the leaching temperature were investigated. The elements of interest, Cu, Cd and U were analyzed spontaneously in all agitation leaching experiments to calculate their leaching efficiencies. On the other side, the pregnant sulfate leach liquor was prepared via leaching a weight of 500 g of the present ore sample under the obtained optimum leaching conditions.

2.3. Sequential separation of Cu, Cd and U salts

Copper ions in the prepared sulfate solution could be easily separated by addition excess of 1%Na₂S solution at pH ≤ 0.35 at a volume ratio of 2/1 (reagent) and stirring for 30 min at room temperature. After filtration and washing , the sulfate solution free from Cu²⁺ ions was treated with 0.25%Na₂S solution at pH 0.5 at a volume ratio of 2/1 (reagent) and stirring for 30 min at room temperature to separate CdS salt. On the other hand, the sulfate solution pretreated with Na₂S solution and free from both Cu and Cd was concentrated by heating and oxidized by using solution of H₂O₂ to convert the reduced U⁴⁺ to U⁶⁺ and adjusted to pH1.7. The latter was then passed through a column packed with 5 mL of wet settled resin, (wsr) anion exchanger resin, Amberlite IRA₄₀₀ at flow rate of 1mL / min to elute the preconcentrated uranyl sulfate anion complex, [UO₂(SO₄)₂]²⁻. The loaded resin was then washed with distilled water to get rid of the adsorbed impurities and treated with acidified 1N NaCl solution to elute U. The elute solution rich in UO₂⁺² was treated with NaOH solution to precipitate yellow cake of Na₂U₂O₇. xH₂O. It is very important to mention here in that, the all forgoing subsequent separation processes would be performed upon the remained solution volume.

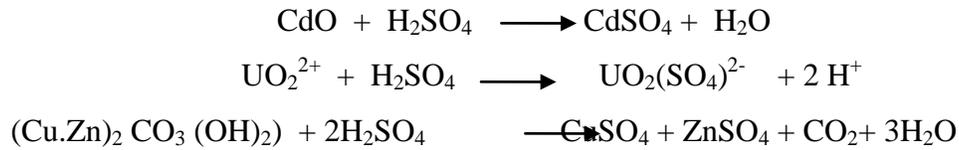
3-RESULTS and DISCUSSIONS

3.1. Mineralogical and chemical composition

As has been reported by **Mira and Aita (2009)** the mineralogical investigations of the collected sample from Abu Thor area revealed the presence of different Cu and U minerals such as: zippeite, (K₄(UO₂)₄(SO₄)₄(OH)₁₀.4H₂O) [*Card No.,8-138*], atacamite (Cu₂Cl(OH)₃) [*Card No.,23-948*], paratacamite (Cu₂(OH)₃Cl) [*card No.,23-947*] rosasite (Cu.Zn)₂ CO₃ (OH)₂), [*Card No.,17-216*], calcite, (CaCO₃) [*Card No., 5-586*], and dolomite, (CaMg(CO₃)₂). [*Card No., 11-78*]. On the other hand, the complete chemical analyses of the studied ore sample given in table (1) revealed the presence of 0.04% of U, 0.5% of Cu, 0.014% of Cd as the elements of interest. As well as 40% of SiO₂ and 27% of Al₂O₃ which indicates that ore sample could be classified as gibbsite - bearing shale.

3.2. Optimization of H₂SO₄ agitation leaching parameters

Not only the adsorbed valuable element oxides Cd and U on the working gibbsite bearing shale ore sample are easily leached via agitation leaching by using H₂SO₄ but also the mineralized Cu is easily dissolved. The expected chemical reactions are illustrated in the following equilibrium equations:



Different effective leaching parameters were studied to prepare suitable leach liquor containing maximum concentration of the elements of interest as follow.

Table (1): Chemical composition of Abu Thor gibbsite bearing shale material, AcmeLabs, VAN13004169.1.

Major oxides	Conc., (%)	Trace elements	Conc., ppm
SiO ₂	40	Co	2700
Al ₂ O ₃	27	Ni	1000
Fe ₂ O ₃	6.87	V	500
MnO	1.3	U	400
ZnO	0.68	∑REEs	356
CuO	0.62	Cd	140
K ₂ O	0.62		
CaO	0.45		
MgO	0.4		
PbO	0.36		
Na ₂ O	0.24		
*T.L.I.	20		
Total	98.54		

* Total loss by ignition

3.2.1. Effect of H₂SO₄ acid concentrations

Different concentrations of H₂SO₄ ranged from 10% to 40% were used to investigate the effect of acid concentrations upon Cd, Cu and U leaching efficiencies from the present ore sample. The other leaching parameters were kept constant at S/L ratio of 1/2 and agitation time of 30 min at room temperature. After washing, filtration and analysis, the obtained data, (Table 2),

clarify that, the leaching efficiencies of these elements increased gradually with increasing the acid concentration up to 25%. On the other hand, further increase in acid concentration resulted in decreasing the leaching efficiencies of Cu, Cd and U. This may be attributed to the dissolution of some other elements such as: Ni , Fe, Zn and Al elements.

Table (2): Effect of H₂SO₄ concentration upon Cd, U and Cu leaching efficiencies.

H ₂ SO ₄ Conc., (%)	Leaching efficiency, %		
	U	Cu	Cd
10	40	56	47
20	46	64	53
25	62	78	59
30	58	79	53
40	55	74	45

3.2.2. Effect of solid/ liquid (S/L) ratio

This effect was studied by mixing 25% H₂SO₄ solution with the working sample at different S/L ratios ranged from 1/2 to 1/5 and stirring for 30 min at room temperature. Data represented by (Table 3) indicate that, decreasing S/L ratio from 1/2 to 1/3 increased leaching efficiencies of Cu, Cd and U up to 87.5%, 64% and 69%, respectively. On the other side, further decrease in S/L ratio more than 1/3 leads to decrease the leaching efficiencies of Cu, Cd and U Cd up to 78%, 56% and 61%, respectively due to the dissolution of interfering elements compound e.g. Al, Fe and Zn.

Table (3): Effect of S/L ratio upon Cd, U and Cu leaching efficiencies.

S/L ratio	Leaching efficiency, %		
	U	Cu	Cd

1/2	62	78	59
1/3	69	87.5	64
1/4	65	83	57.8
1/5	61	78	56

3.2.3. Effect of leaching time

This effect was studied by mixing the studied ore sample with 25% H₂SO₄ solution at S/L ratio of 1/3 with stirring for different time periods ranged from 30 to 120 min at room temperature. Results, (Table 4), revealed that, Cu, Cd and U leaching efficiencies increased up to 92%, 75.8% and 76.4%, respectively by increasing the leaching time from 30 min to 90min where further time increasing to 120 min.

3.2.4. Effect of leaching temperature

(Table 5) shows the effect of changing leaching temperature from 25°C up to 100°C upon Cu, Cd and U leaching efficiencies from the working ore sample. The other leaching conditions were kept constant at acid concentration of 25% H₂SO₄, S/L ratio of 1/3 and leaching time of 90min. The obtained data shows remarkable improvement in Cu, Cd and U leaching efficiencies which achieved 96.5%, 99.3% and 95%, respectively at 80 °C . Further increase in temperature to 100°C decreased Cu, Cd and U leaching efficiencies to 93%, 90.4% and 89%, respectively due to the high dissolution of interfering elements especially Al, V, Zn and Fe.

Table (4): Effect of leaching time upon Cd, U and Cu leaching efficiencies.

Time, min	Leaching efficiency, %		
	U	Cu	Cd
30	69	87.5	64
60	71.4	90.3	69.2
90	76.4	92	75.8
120	70	89	72.4

Table (5): Effect of leaching temperature upon Cd, U and Cu leaching efficiencies.

Temperature, °C	Leaching efficiency, %		
	U	Cu	Cd
25	76.4	92	75.8
60	88.5	94.6	89.6
80	95	96.5	99.3
100	89%	93	90.4

From the foregoing leaching study, it can be concluded that, the effective leaching conditions for dissolving about 96.5% of Cu, 99.3% of Cd and 95.6% of U content from Abu Thor gibbsite-bearing shale ore sample are summarized as following:

H₂SO₄ concentration : 25%
Solid/Liquid mixed ratio (S/L) : 1/3
Leaching time : 90min
Leaching temperature : 80 °C

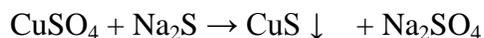
Applying the above mentioned effective leaching conditions upon 500g of the working sample, washing and then filtration yields 2L of sulfate leach liquor (pH 0.22). The resulted sulfate solution was found assaying 1.12g/L of Cu, 0.033g/L of Cd, and 0.096 g/L of U and 0.008g/L of V with leaching maximum efficiencies of 92%, 96%, 95% and 8.6%, respectively. The latter was used for subsequent separation of Cu, Cd and U salts as the target of this study.

3.3. Subsequent separation of Cu, Cd and U

Copper ions, Cu²⁺, in the prepared sulfate solution could be effectively separated via direct precipitation with the addition of sulfide anions at pH ≤ 0.35 due to its lower value of solubility product (K_{sp} CuS 2.4×10^{-36}) compared to (K_{sp} of CdS = 7.9×10^{-27} and K_{sp} of ZnS = 7.9×10^{-22}) which never ever precipitates with sulfide anions at pH ≤ 0.4 and pH ≤ 0.9, respectively, Alexeyev (1973), Mahdy et al (2008) and Abd El Wahab et al (2012). On the other hand, U⁶⁺

and Fe^{3+} could be reduced by S^{2-} anion to U^{4+} and Fe^{2+} , **Hua et al (2006)** and **Lirong et al (2007)**. The following equation illustrates the separation of Cu ions with sulfide anions:

pH 0.35



Black ppt

3.3.1. Separation of Cu salts

A volume of 500 mL of the prepared sulfate solution (pH0.22) which assays 1.12 g/L of Cu, 0.033 g/L of Cd beside 0.096 g/L of U was adjusted to pH0.3 with 5% NaOH. The latter was treated with the same volume of 0.5% Na_2S solution and stirred for 30 min at room temperature where Cu^{2+} ions were directly precipitated as CuS. The complete precipitation of CuS was achieved at pH0.35, after filtration, washing and drying a weight 0.82g of CuS was recovered with precipitation efficiency achieved 99.3%. The product was identified by using SEM-EDAX technique, Fig. (2). the purity of CuS achieved 97.9% with impurities of 0.9% Zn, 0.3% Pb, 0.5% Mn and 0.4%Na.

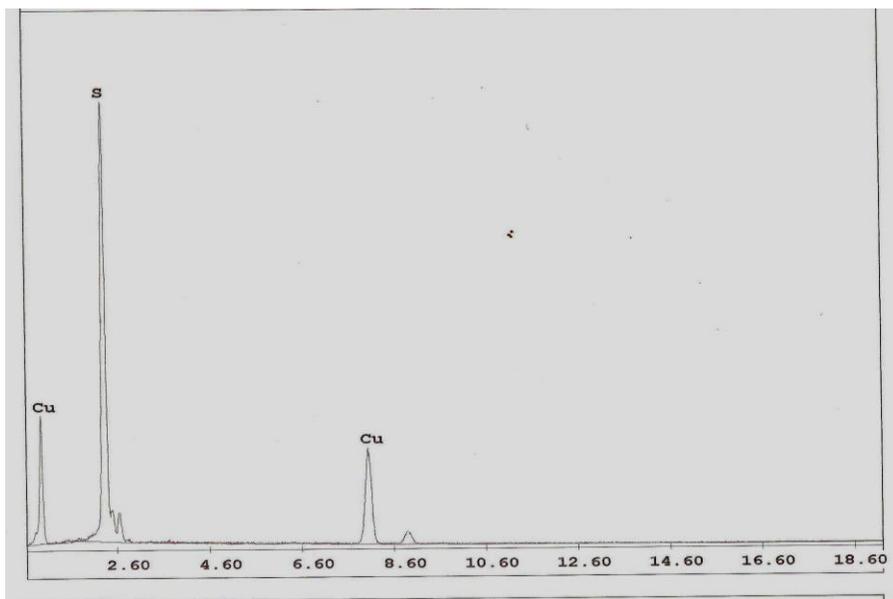
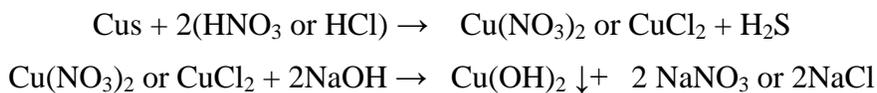


Fig. (2): EDAX chart for the identification of pure CuS.

On the other hand, the produced CuS ppt was dissolved in 5% solution of HNO_3 or HCl to prepare $\text{Cu}(\text{NO}_3)_2$ or CuCl_2 solutions. The latter was treated with 10% NaOH solution to precipitate $\text{Cu}(\text{OH})_2$ at pH8.5. The latter was washed, dried and identified with SEM-EDAX analysis technique, Fig.(3). the following equations illustrate these reactions:



Blue ppt

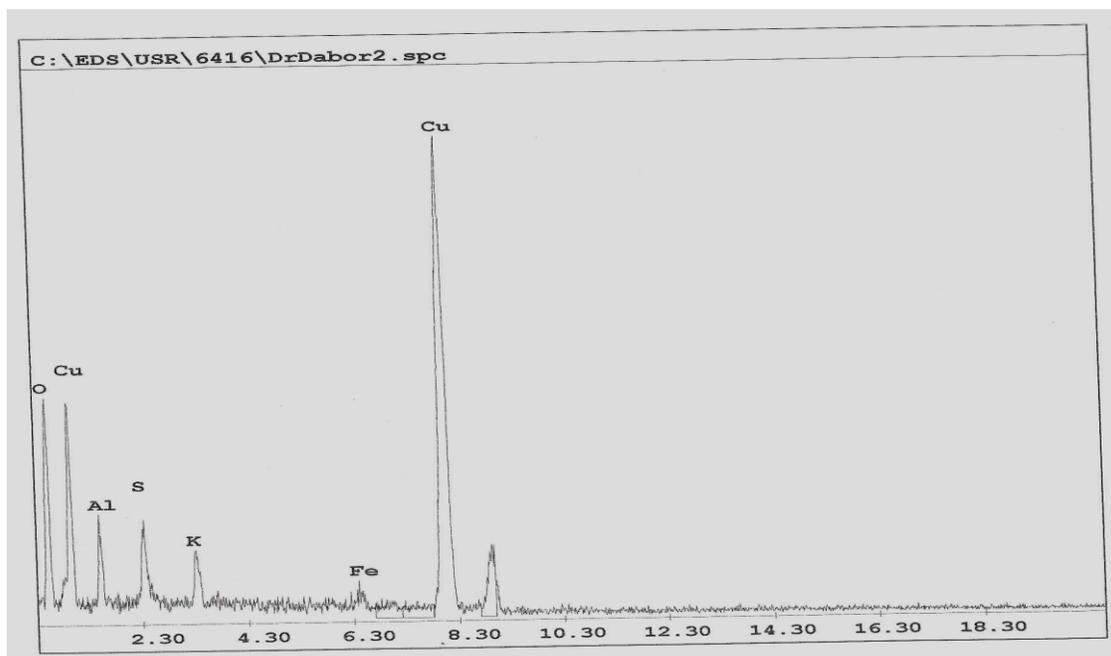


Fig. (3): EDAX chart for the identification of pure $\text{Cu}(\text{OH})_2$.

3.3.2. Separation of Cd salts

The prepared sulfate solution preprocessed with Na_2S solution and free from Cu^{2+} ions (very low concentration) its volume became 800 mL and thus it assays 0.0216 g/L of Cd and 0.043 g/L of U and its pH value rose up to pH0.38 . The latter was treated with 5% NaOH solution to adjust its pH value to pH 0.5 and then treated with 0.25% Na_2S in a volume ratio of 1/1. After stirring for 30 min at room temperature, about 99.5% of Cd^{2+} ions were selectively precipitated as a mixed cake of $(\text{CdS}+\text{S metal})$. The following equation illustrates the precipitation process, **Mahdy et al (2008) and Abd El Wahab (2016)**.



The precipitated $(\text{CdS} + \text{S})$ cake was then dissolved in 100mL of 5% HNO_3 solution where CdS was completely dissolved at room temperature leaving behind elemental S. On the other

hand, the prepared $\text{Cd}(\text{NO}_3)_2$ solution was then treated with 5% NaOH solution and stirring for 1h at room temperature and left to precipitate $\text{Cd}(\text{OH})_2$. Complete precipitation of Cd^{+2} ions takes place at pH8.5. The produced $\text{Cd}(\text{OH})_2$ cake was then carefully washed with distilled H_2O to get rid of any adsorbed impurities and then ignited at 750°C for 1h to produce CdO . The latter was washed, dried and then directed to XRD analysis for identification, Fig. (4). Chemical analysis of the produced CdO revealed that, it assays 85% of Cd with purity of 97.5%. Some impurities of concentrations of about 0.5% of Na, 0.3% of Cu were determined. The following equations illustrate these reactions.

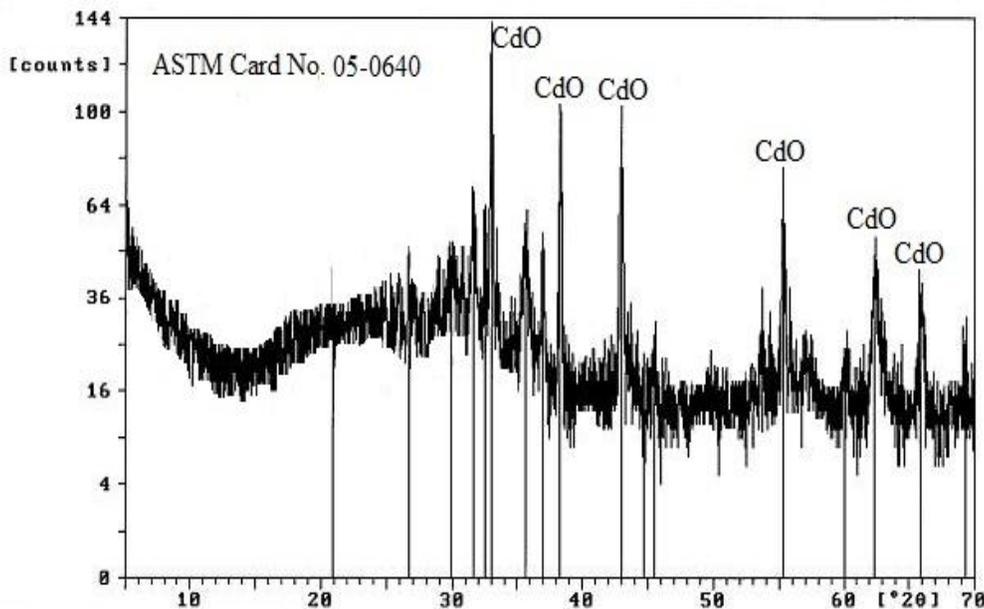
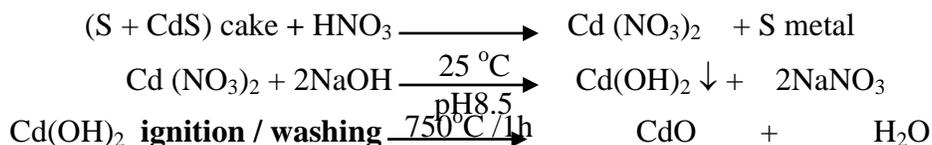


Fig. (4): XRD pattern of the highly pure CdO .

3.3.2. Separation of U salts

Separation of U was carried out via anionic exchange extraction process in two subsequent processes, loading and elution, while U salt, (yellow cake) was prepared from the U eluate solution via precipitation with NaOH solution

3.3.2.1. Loading process

The volume of the working sulfate solution pretreated with Na_2S solution and free from both Cu and Cd has closed up to 1300 mL and assays 0.036g/L. It was concentrated by heating with addition of H_2O_2 solution to oxidize the reduced U^{4+} to U^{6+} and adjusted to pH1.7. The latter was then passed through a column packed with 2 mL of wet settled resin,(wsr) anion exchanger resin, Amberlite IRA₄₀₀ at flow rate of 1mL/min for extracting the present uranyl sulfate anion complex, $[\text{UO}_2(\text{SO}_4)]$. Bed volumes of 50 mL of effluent solution were collected for U analysis where its extraction efficiency was calculated and plotted against number of bed volumes. Results, (Fig.5), clarify that the amount of the loaded U was 0.036g with the total loading efficiency of 79%. After full saturation, the loaded resin was washed with distilled H_2O to get rid of the adsorbed impurities before applying the elution process.

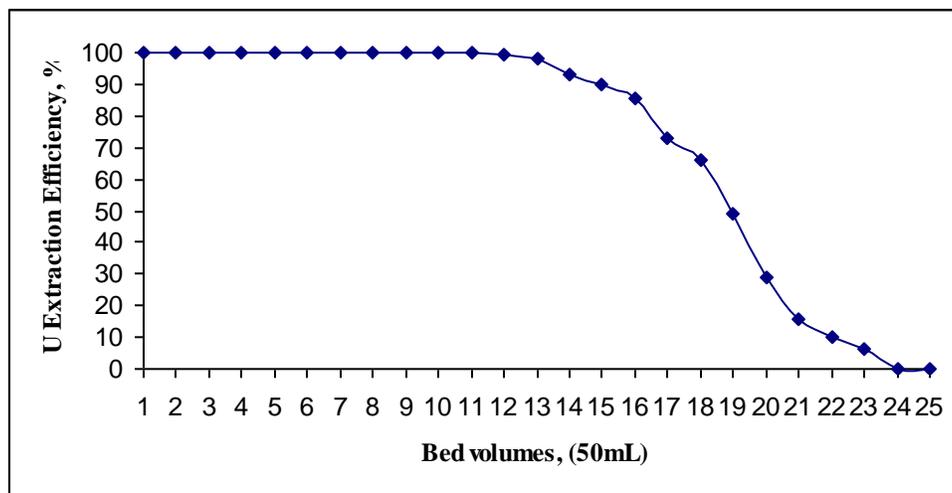


Fig.(5): The loading curve of U from sulfate solution of Abu Thor gibbsite bearing shale sample

3.3.2.2. Elution process

Elution process is not only used to regenerate the loaded resin but also to prepare a concentrated U solution suitable for the subsequent precipitation process to prepare the final U salt. The loaded Amberlite IRA₄₀₀ should be rapidly washed with distilled water to get rid of the adsorbed impurities. The up taken urinate species, was easily eluated by treating with 100 mL of 1% NaCl solution acidified with 0.1M H_2SO_4 . By plotting the amount of the eluated U against the number of the picked up bed volumes, (Fig. 6), it could be easily determined elution efficiency which achieved 97.25% and also easily chosen the eluate U rich solution which used for preparing U products.

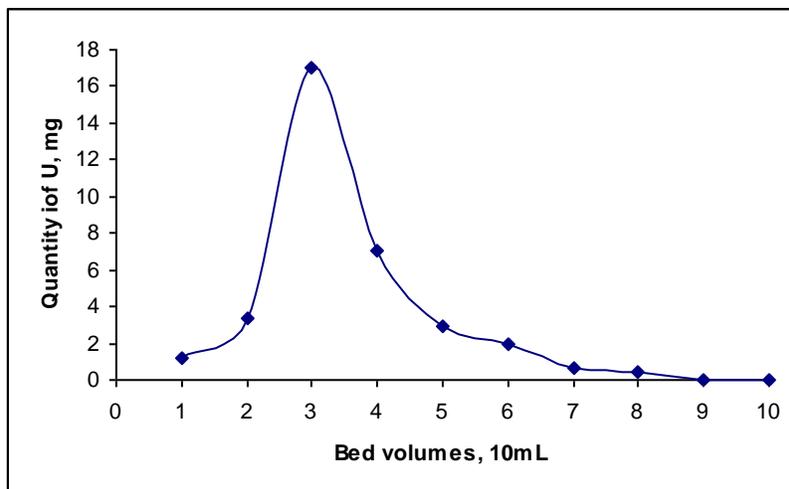


Fig.(6): Elution curve of the loaded U

3.3.2.3. Preparation of Yellow cake (U salt)

A volume of 60 mL, (V_1 to V_6) of the eluate solution rich in U and containing 0.034 mg was treated with NaOH solution to precipitate yellow cake of $\text{Na}_2\text{U}_2\text{O}_7 \cdot 4\text{H}_2\text{O}$ at pH6.7. After filtration, washing and drying a weight of 50 mg of impure yellow cake was recovered. A small portion of the latter was identified with SEM-EDAX, Fig. (7) while a weight of 2mg was completely dissolved in 10mL of 10% HNO_3 solution and its U was analyzed to qualify its purity. The obtained analysis results revealed that U assays 52% with purity achieved $52/67 * 100 = 77\%$. By performing the forgoing extraction processes up on the remaining sulfate solution, (1500 mL) The author could be recovered about 200 mg of yellow cake with recovering efficiency of $200/290 * 100 = 69\%$.

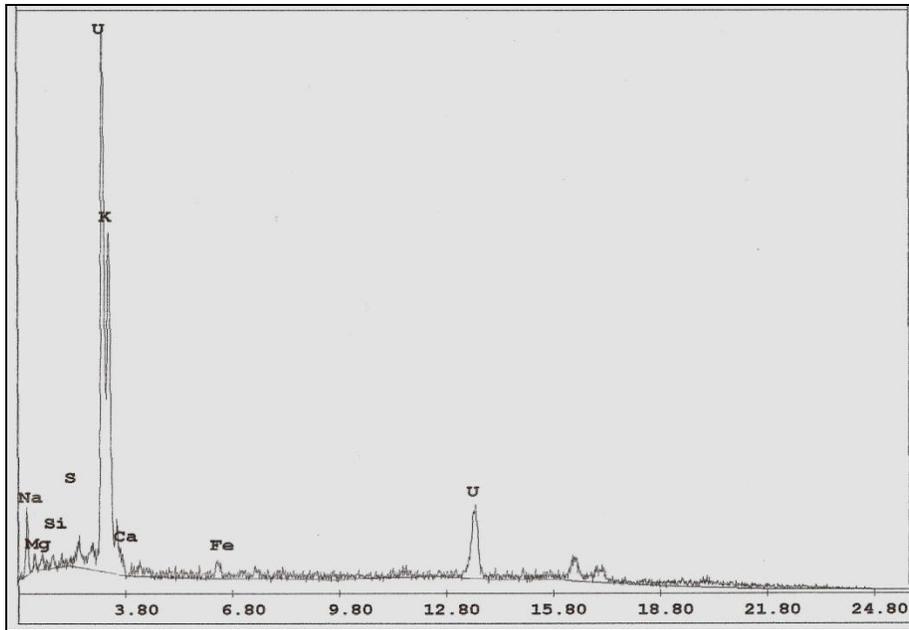


Fig. (7): EDAX chart for the identification of impure yellow cake.

4-CONCLUSIONS

It could be concluded the following:

Agitation leaching process with 25% H_2SO_4 solution at S/L ratio of 1/3 and stirring for 90min and temperature of $80^\circ C$ was successfully for leaching Cu, Cd and U from the representative sample of Abu Thor area West Sinai, Egypt. The resulted sulfate solution was found assaying 1.12g/L of Cu, 0.033g/L of Cd, and 0.096 g/L of U and 0.008g/L of V with leaching maximum efficiencies of 92%, 96%, 95% and 8.6%, respectively. The latter was used for subsequent separation of Cu, Cd and U salts as the target of this study.

A worked follow-sheet represented in Fig. (8) for the processing of Abu Thor representative ore sample to produce Cu, Cd, and yellow cake.

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الملخص العربى

الاستخلاص المرحلى لكل من النحاس والكاديوم واليورانيوم من خامات الطمى الحاملة للجيسيت، منطقة أبو ثور، جنوب غرب سيناء، مصر

خامات الطمى الحاملة للجيسيت تم تسجيلها ضمن رواسب العصر الكربونى بطبقة أم بجمة بمنطقة أبو ثور. هذا العمل مهتم بالاستخلاص المرحلى لكل من عناصر النحاس والكاديوم واليورانيوم من محلول الكبريتات الخاص بهذه الخامات.

العينة الممثلة تحتوى على 0.5% من النحاس و 0.014% من الكاديوم و 0.04% من اليورانيوم. وكانت عوامل الاذابة المؤثرة مثل حرارة التحميص وتركيز الحمض و نسبة اضافة الحمض الى الخام ووقت الاذابة ودرجة حرارة الاذابة قد تم دراستها.

محلول الكبريتات الذى تم تحضيره بالظروف المثلى للاذابة يحتوى على 2.4 جم/لتر من النحاس و 0.066 جم/لتر من الكاديوم و 0.19 جم/لتر من اليورانيوم بنسبة اذابة وصلت الى 92% و 96% و 95% على الترتيب بالاضافة لتواجد 0.008 جم/لتر من الفانديوم بنسبة تركيز 8.6%.

محلول الكبريتات المجهز تم معالجته بمحلول 0.5% من كبريتيد الصوديوم عند الرقم الهيدروجينى 0.35 للفصل التتابعى لكبريتيد النحاس وتم ترسيب كبريتيد الكاديوم عند الرقم الهيدروجينى 0.5

أما بالنسبة لليورانيوم فقد تم استخلاصه من المحلول الخالى من النحاس والكاديوم بعد اكسدته بمحلول فوق اكسيد الهيدروجين واستخلاصه باستخدام التبادل الايونى على راتنج امبرليت IRA₄₀₀.