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Processing of Cracked Mud Raw Material with Thiourea Based Reagent for the Recovery of Uranium and Copper, Talet Selem, Southwestern Sinai, Egypt

Elham Awny Gamil[*]

Nuclear Materials Authority, P.O. Box 530 El-Maadi, Cairo, Egypt.

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Abstract: Abnormal leaching reagent, thiourea has been studied for leaching both of uranium (U) and copper (Cu) content from mud crack ore material, Talet Selem area, Sinai, Egypt. Several leaching parameters have been investigated e.g. Thiourea concentration, mixed solid / liquid ratio, leaching time and finally leaching temperature to optimize the leaching process. The prepared leaching solution rich in U species and Cu ions was adjusted to direct precipitation process using Na_2S for separating Cu ions and directed to ion exchange process (batch technique) using Amberlite IRA_{400} (Cl^- form) to recover U species. The recovered product ($Na_4U_2O_7.xH_2O$) and CuS where confirmed using SEM- EDAX unit.

Keywords: Talet Selem area; Thiourea Reagent; Southwestern Sinai; Cracked Mud.

1 Introduction

The studied mud crack sample was collected from the ground of along trench located in the western part of Talet Selem locality directly on the cliff of the wadi Baba as represented in fig. (1) [1]. The wall of this trench is represented by a section of middle Um Bogma formation which represented by black shale, siltstone and thin marly interlayer. The uranium concentration of the sediments in this trench reached to about 250 ppm. The famous Uranium minerals considered as the sources of nuclear fuels and the most important minerals of uranium are pitchblende (U_3O_8), uraninite (UO_2), carnotite (potassium uranium vanadate), autunite (Cu-U phosphate), and torbernite (Cu-U phosphate) constitutes in Earth's crust is about 2ppm [2].

Under acidic and alkaline oxidizing conditions, uranium is extremely mobile forming the uranyl ion $(UO_2)^{2+}$ in which uranium occurs as its hexavalent state which could combine with a number of anions to form soluble complexes in acid and alkaline solutions. Uranyl ion is characteristic with its linear structure $[O = U = O]^{2+}$, where it can coordinate with atoms on the equatorial plane perpendicular to its linear structure forming a configuration of quadrilateral, pentagonal or hexagonal bipyramids [4].

With respect to Cu is one of the most important metals used in a lot of industries due to its electrical, thermal, optical and catalytic properties [5]. In addition it is quite biologically active, due to its tendency to form strong complexes in water or soil with organic ligands containing nitrogen or sulfur. It also forms complexes with some large macromolecules (proteins, nucleotides, etc.).

It is found mainly in nature in the form of sulfide and oxide minerals such as malachite [$Cu_2(OH)_2CO_3$], azurite [$Cu_3(OH)_2(CO_3)_2$], tenorite (CuO), chrysocolla, bornite, brochantite, enargite, chalcopyrite, chalcocite, or covellite [5-7]. Uranium and copper leaching from their ores using acidic and alkaline reagents has been reported by many authors [8-14]. Several authors studied the leaching behavior of copper oxides and sulfides ores using different lixiviant including mineral acids [15-17]; organic acids such as citric, adipic and lactic acid [18-20]; ammonium hydroxide or ammonium salts [21-24]; in addition to glycine solution [25-31].

Thiourea, (NH_2CSNH_2), (TU) is an organosulfur compound, it is relatively stable in acidic solution and rapidly decomposed in alkaline solution [32]. It can form stable cationic complexes when it reacts with some transition metal ions [33-34]. TU used as lixiviant for precious metals where it is less toxic and more eco-friendly, and it can achieve a high leaching rate about 99% for Au [35-36]. TU can coordinate with metals through nitrogen atom or sulfur atom as amonodentate ligand or by the two atoms as bidentate. Thiourea is very unstable especially at pH > 4.3, and easily oxidized and decomposed

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*Corresponding author e-mail: Elhamawny1976@yahoo.com

[37]. Acidic thiourea leaching is normally conducted at pH of 1–2, where in acidic environment, base metals such as Fe, Cu, Ni, Zn and Pb contained in WPCBs may also be leached out with precious metals Au and Ag resulting in additional consumption of thiourea [38]. At low concentrations of thiourea it is used as an additive in the electrode position of metals such as copper from acid solutions, whereas at high concentrations [6], it is considered as a possible leachant in hydrometallurgy for the recovery of gold and silver from minerals and slimes [39]. TU can absorb upon the metal surface which block active sites of it [7], for that TU can used as an inhibitor for metals corrosion [40-41]. On the other hand, the reaction behaviors of different concentrations of TU on Cu surface in the presence of 0.5 mol/L H_2SO_4 using electrochemical methods and single crystallogram, it illustrated that at low concentration of TU it is first oxidized to formamidine disulfide (FDS) and adsorbed on copper surface which lead to inhibition of copper electro dissolution, whereas at high concentration of TU it will bonded to Cu via its sulphur atom to form complex ion and hence promotes corrosion reaction on Cu surface [42].

The cuprous species is coordinated with four thiourea molecules. They found the stability constant for formation of the tetra thiourea copper (I) ion to be 2.44 x 10^{15}, which indicates that the complex is very stable [8]. Luis [44] has studied the effect of thiourea as additives in acidic ferric sulfate leaching solutions which show significant increases in the leaching of copper from chalcopyrite, covellite, and chalcocite. After physical and magnetic separation of Au, Ag and Cu from scrap integrated circuits, thiourea was able to recover the above three metals 100% in the presence of sulfuric acid and ferric sulfate [45].

The complex salts of Cu(I)–TU have been crystallized from aqueous solutions from either the anodization of copper in TU-containing 0.5 M sulphuric acid solutions or when mixing different amounts of dissolved TU and aqueous acid $CuSO_4$ and determined by X-ray diffractometry, it was found that complexes $[Cu_2(TU)_5]^{2+}$ and $[Cu_2(TU)_6]^{2+}$ are soluble and easy to recrystallise from solutions having a Cu(I):TU molar ratio lower than 4:7 and Insoluble $[Cu_4(TU)7]^{4+}$ complex is formed as a polymer like solid for a Cu(II):TU molar ratio close to 4:7[46]. Beside the ability of TU to leach precious and transition metals, it can leach uranium from its ores. Salem and Kassab studied the leaching characterization of uranium from El-Sella mineralized Granite which attained 89.12 % at 2.24 mol/L concentration of TU at L/S ratio of 1/5 for 180 min at 298 K leaching temperature and -0.25 mm grain size [4].

The essential goal of the present study is to investigate the optimum leaching conditions for U and Cu using TU as leaching reagent. In addition, applying the achieved optimum leaching conditions in preparing a suitable volume of leach liquor using in the recovery of U and Cu as the elements of interest.

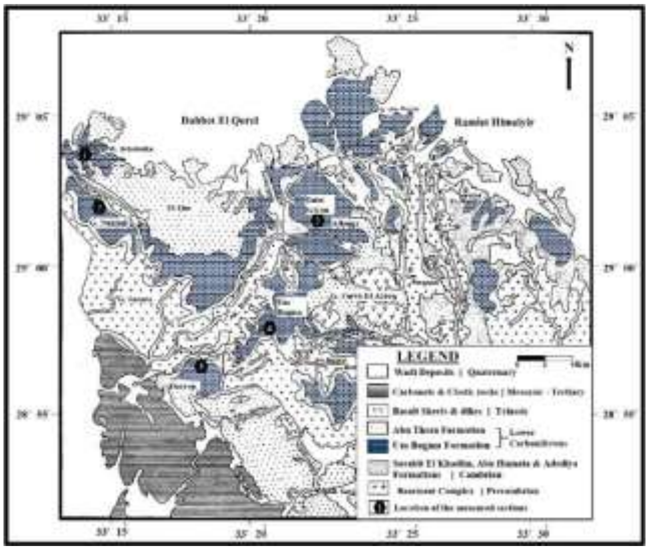


Fig.1. Modified Geological map of Um Bogma area in Southwestern Sinai showing the lateral distribution of the Paleozoic rocks and location of the measured lithostratigraphic sections (after [1]).

2 Experimental

2.1 Instrumentation

X-ray fluorescence (XRF) was used for trace elements analyses, using Philips Unique II unit fitted with an automatic sample changer PW 1510 (30 position), which connected to a computer system using X-40 program for spectrometry, in the Nuclear Materials Authority Laboratories (NMA Lab). Flame atomic absorption (FAAS), Unicam 969 of NMA Lab was used for copper analyses during experiments. Environmental Scanning Electron Microscope (ESEM) EXL 130 supported by EDAX unit for a semi-quantitative analysis of the accessory and secondary minerals used to identify the obtained heavy minerals after their picking and the obtained products.

2.2. Mineralogical and Chemical composition

To identify the minerals constituents, a ground representative sample, (-1.0 mm size) was adequate to liberate the accessory minerals. It was washed several times with tap water where the slimes were removed by decantation. The dry fraction was then sieved into the -0.5+0.063 mm. The light and heavy fractions were separated by bromoform (sp. gr. 2.8), different mineral grains were picked up, collected under binocular microscope and finally identified using SEM-EDAX unit.

With respect to the chemical composition of the studied sample, a representative sample was properly crushed, around to -75 μm grain size and subjected to the major elements oxide analysis according to Shapiro and Barnnock [47], special sample portions was used for the determination of loss of ignition (obtained at 1000 °C) which is corresponding to humidity, combined water, CO_2 as well as possible organic matter. Uranium was analyzed using an oxidimetric titration method against ammonium metavanadate after reduction of U (VI) with ferrous sulfate in the presence of diphenyl amine-4-sulforic acid sodium salt as indicator [48]. While the other trace elements e.g. Cu, Fe, Mn, Mg….etc were determined using FAAS.

2.3. Leaching and recovery procedure

Several experiments were performed using 10 g of the studied sample via agitation leaching with thiourea solution. The variables that can influence the leaching rate of both U and Cu was already studied, including thiourea (TU) concentration; solid / liquid (S/L) ratio; agitation time and temperature. The obtained optimum leaching conditions were applied upon500 g of the working sample to prepare a pregnant leach liquor rich in U and Cu species which directed to the different extraction processes to recover the elements of interest.

3 Results and Discussion

3.1 Mineralogical characterization

The obtained data of SEM-EDAX analysis revealed the presence of different valuable Cu and REEs minerals including monazite, malachite, sphalerite, chromite and some iron minerals e.g. ilmenite, hematite, goethite, and magnetite. It was also observed that no real U minerals has been identified, thus it might be concluded that U element might be existed as adsorbed on the presence iron minerals [49-51]. With respect to the chemical composition, Table (1), it can be concluded that the studied sample mainly consists of major elements oxide e.g. SiO_2, Al_2O_3, Fe_2O_3, CaO and MgO. It also contains some trace elements such as U, Cu, Zn, Cr …etc.

Table (1): The chemical analysis of the mud crack sample.

Major Oxides	Conc. (%)	Trace Elements	Conc. (ppm)
SiO_2	47.9	**U**	700
TiO_2	0.51	**Cu**	1500
Al_2O_3	16.5	**Zn**	897
Fe_2O_3	6.3	**REE**	2000
CaO	4.8	**Ni**	171
MgO	2.1	**V**	343
MnO	0.3	**Co**	111
Na_2O	2.9	**Cr**	354
K_2O	1.8	**Pb**	386
P_2O_5	0.4	**Cd**	27
SO_4^{--}	3.4	**Zr**	1118
L.O.I*	11.47		
Total	**98.38**		

3.2 Leaching process of uranium and copper

From both chemical and mineralogical studies, abnormal leaching reagent Thiourea ($H_2N\text{-}CS\text{-}NH_2$) was used for trying to have a selective leaching for both U and Cu content from the present mud crack raw material sample. The chemical reactions of leaching process, (Equations 1-4) was explained as the complexation reaction between TU and uranyl cation to form quadrilateral, pentagonal or hexagonal bi-pyramids configurations. In case of Cu when present as Cu^{2+} possible reactions can be occurred as following [52]. Firstly TU will oxidized by Cu^{2+} yielding form amidine disulfide (FDS) and $[Cu (TU)]^+$ complex equation (2). The result complex ion from equation (2) would react further with TU as shown in equation (3). As shown in equation (2) that hydrogen ions take part in the reaction which prefers pH increases to make reaction directed to the right. On the other hand further increase in pH value, the leaching efficiency for both metals generally decreases which may be attributed to that by decreasing the acidity for the solution, the FDS begin to decompose to TU, cyanamide and elemental S as following equation (4) [55]. The resulted elemental Scan precipitate Cu^{2+} as CuS hence decrease the Cu leaching efficiency. Choice pH= 2 is best condition.

$$aUO_2^{2+} + b\,TU \rightarrow [\,(UO_2)_a(TU)_b]^{a+} \text{.................................}(1)$$

$$2Cu^{2+} + 4TU \rightarrow FDS + 2[Cu\,(TU)]^{+} + 2\,H^{+} \text{...................}(2)$$

$$x\,[Cu\,(TU)]^{+} + y\,TU \rightarrow [\,Cu_{x}(TU)_{x+y}]^{x+} \text{........................}(3)$$

$$[CS\,(NH)\,(NH_2)]_2 \rightarrow CS(NH_2)_2 + NH_2\,CN + S \text{………..}\ (4)$$

3.2.1 Effect of Thiourea concentration

The concentration of Thiourea (TU) solution at pH 2 when changed from 25 to100 g/L leaving the other conditions fixed at S/L ratio of 1/ 2 and stirring for 3 h at room temperature, the leaching efficiencies of U and Cu increased up to 42% and 67 %, respectively as shown in Fig. (2) in agreement with [4,53]. Further increase in TU concentration up to125 g/L decrease the leaching efficiencies of U and Cu up to 23.4% and 45.5%, respectively.

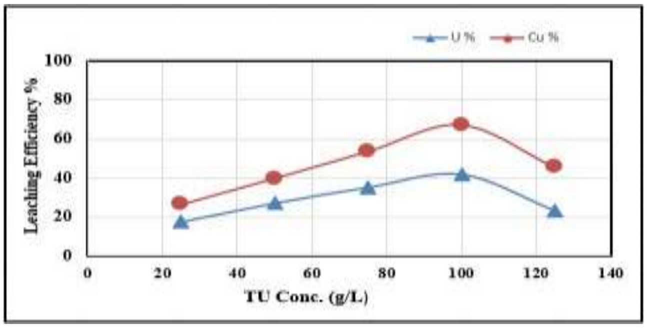


Fig. 2: Effect of Thiourea concentration upon the dissolution efficiency, % **.**

3.2.2 Effect of Ph

According to equation (2) the liberation of H^+ ion during the reaction of TU with the ore sample thus the effect of pH upon U and Cu leaching efficiencies is one of the most important parameter. Several experiments were applied suing TU solution of different pH values varying from pH 0.5 to pH 4 using 100 g/L TU at S/L ratio of 2/1 and stirring for 3h at room temperature. The obtained results, fig. (3) emphasized the best U and Cu leaching efficiencies (42 % and 67%) were achieved at pH 2

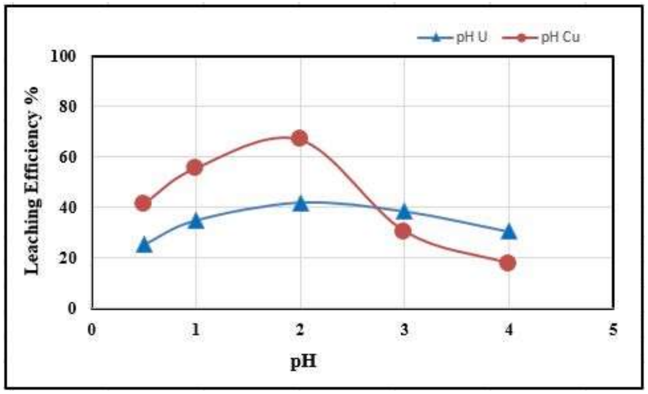


Fig. 3: The effect of pH upon the dissolution efficiency, %

3.2.3 Effect of Solid / Liquid, (S/L) Ratio

The effect of S/L ratio upon U and Cu leaching efficiencies from the present mud crack raw material was studied from using T U solution of 100 g/L at pH2 solution and stirring for 3h at room temperature. Data, fig (4) indicate that as S/L ratio decreased from 1/1 to 1/4, the leaching efficiencies of U and Cu increased up to 75 % and 98%, respectively. However further decrease S/L ratio up to 1/5 has no effect upon U leaching efficiency but decrease the Cu leaching efficiency to 79.4 % , due to the high content of TU in leaching solution as S/L ratio increase which lead to its decompose to elemental S which precipitate Cu as CuS, [55] equation (4).

3.2.4 Effect of contact time

Both of U and Cu leaching efficiencies was investigated in the time intervals from 1h to 5 h, where the other conditions were fixed at 100 g/L TU concentration, pH 2, S/ L ratio of 1/4 and room temperature. The obtained results, fig. (5) revile that the leaching efficiencies of both U and Cu increase as time increase. Almost dissolution of Cu attained at 3h while attained 75 % for U, further increase in leaching time has no effect.

3.2.5 Effect of temperature

To determine the effect of temperature upon U and Cu leaching efficiencies, several experiments were carried out from room temperature to 55 ºC using 100g/L of TU at pH2, 3h at S/L of 1/4. Results, fig. (6) refer to as temperature increase from 25 ºC to 35 ºC the leaching efficiencies of U and Cu increase up to 81.5 % and 98% respectively.

Increasing temperature above 35 °C lead to decrease the leaching efficiencies of both U and Cu which attained 52 % and 41.2 %, respectively at 55 ºC. This decrease related to rapid decomposition of TU with the formation of colloidal sulfur when the temperature was increased over 40°C [55]. In addition to decomposition which decreases TU concentration, the resulted elemental S precipitate Cu^{2+} as CuS. Choice of 35 is preferred as best leaching conditions.

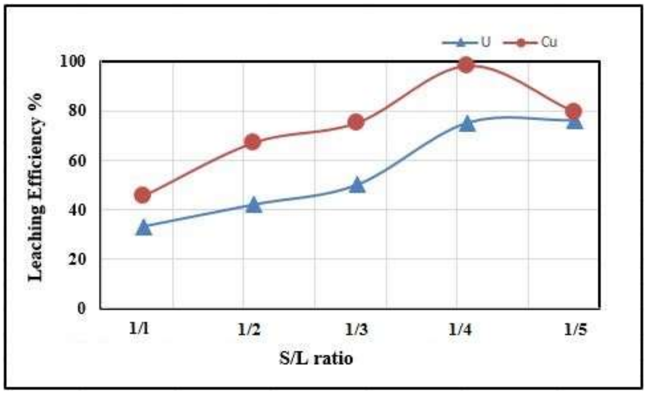


Fig.4. The effect of S /L upon the leaching efficiency, %.

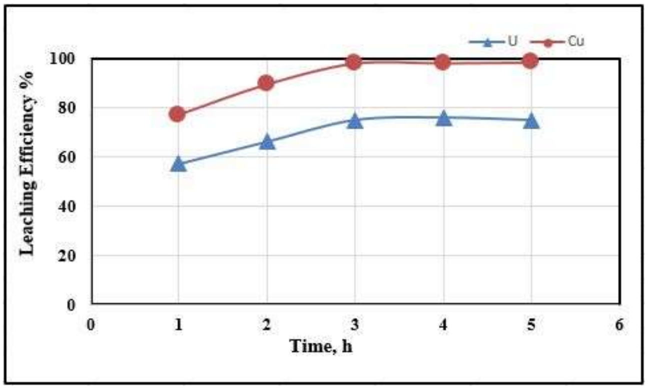


Fig. 5. Effect of time upon the leaching efficiency, % **.**

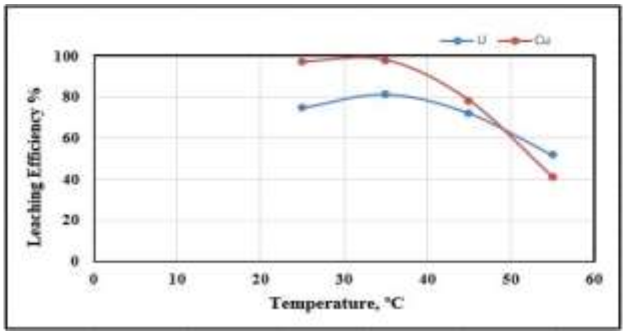


Fig. 6. Effect of temperature upon the dissolution efficiency, %.

Dissolution kinetic analysis

In a fluid–solid reaction system, the reaction rate is generally controlled by one of the following steps: diffusion through the fluid film, diffusion through the ash (or solid product) layer on the particle surface or the chemical reaction at the surface of the core of reacted particles. There are three controlling models for the rate of reaction: chemical reaction at the particle surface, diffusion through the product layer and a combination of both. The rate of the process is controlled by the slowest of these sequential steps.

The most important model suggested for derivation of the expression of the fluid-solid reaction is the shrinking core model (SCM) which is thought that the reaction takes place in the outer surface of the solid and this surface shrinks towards the center of the solid as the reaction proceeds leaving behind an inert solid layer, called ash layer, around the unreacted shrinking core.

Considering that, a solid particle M is immersed and reacts with a fluid N as the following equation:

N (fluid) + b M (solid) → products …(5)

If the reaction rate of the particle is controlled by diffusion of the fluid N through the ash layer, the time t required for a spherical solid to react can be calculated by the following equation:

$$1-3(1-x)^{2/3}+2(1-x)=\frac{6bDC_{o}}{C_{B}r_{o}^{2}}t=K_{1}t \quad ..(6)$$

where X is the uranium or copper fraction dissolved, t is the dissolution time (min), D is the diffusivity of uranium or copper ions through the ash layer (m^2/s), C_o is the concentration of the fluid outside the particle (mol/L), C_B is the apparent concentration of the solid reactant (mol/L), r_o is the initial outside radius of the particle (m), and K_1 and K_2 are the apparent rate constants.

If the reaction rate is controlled by chemical reaction, the integrated rate equation is expressed by the following equation:

$$1-(1-x)^{1/3}=\frac{bk_{d}C_{o}}{C_{B}r_{o}}t=K_{2}t \quad ..(7)$$

Where K_d is the chemical reaction rate constants. To explore the effect of temperature upon the reaction kinetics of uranium and copper, a plot of $1-(1-x)^{1/3}$ and $1-3(1-x)^{2/3}+2(1-x)$ against dissolution time at different dissolution temperatures is established and shown in Fig. (7).

From the two shrinking core models examined, only Eq (7) has been found to give a perfect straight line with a good correlation coefficient. Hence, all resulted data were found to fit the shrinking core model with chemical reaction model as the rate determining step.

The apparent rate constants K_1 and K_2 of uranium and copper for the two shrinking core models examined at different temperature were calculated from the slopes of the straight lines obtained from Fig. (8).The values of K_1 and K_2 and their corresponding correlation coefficients are summarized in both Tables (2 & 3).

Table (2): Apparent rate constants (K_1, K_2) and their correlation coefficients for uranium dissolution.

Temperature, C°	**Apparent rate constants**		**Correlation coefficients (R^2)**	
	K_1 (min^{-1})	**K_2 (min^{-1})**	**R^2_1**	**R^2_2**
25	0.0004	0.0008	0.7922	0.9849
35	0.0005	0.0009	0.8482	0.992
45	0.0007	0.0011	0.88	0.9914
55	0.0009	0.0013	0.9111	0.9926

Table (3): Apparent rate constants (K_1, K_2) and their correlation coefficients for copper dissolution.

Temperature, C°	**Apparent rate constants**		**Correlation coefficients (R^2)**	
	K_1 (min^{-1})	**K_2 (min^{-1})**	**R^2_1**	**R^2_2**
25	0.0009	0.0013	0.868	0.9968
35	0.0012	0.0015	0.8746	0.9944
45	0.0017	0.0019	0.9192	0.9948
55	0.0026	0.0025	0.9518	0.9943

The apparent rate constant values of K_2 were used to obtain the activation energy of the dissolution reaction (E_a) from the Arrhenius equation:

$$\ln K = \frac{-E_a}{R}\left(\frac{1}{T}\right) + \ln A \quad .....(8)$$

Where K is the apparent rate constant, E_a is the activation energy in kJ/mol, R is the molar gas constant in J/mol K and A is the Arrhenius constant. The plot of ln K against 1/T in Fig. (9) gives a slope

that represents the activation energies 13.33 kJ/mol for uranium and 17.68 kJ/mol for copper. Hence, all resulted data are found to fit the shrinking core model with chemical reaction as a rate determining step. Also, shrinking core model (SCM) with a mixed chemical and diffusion control mechanisms was suggested in the dissolution kinetics of many ores.

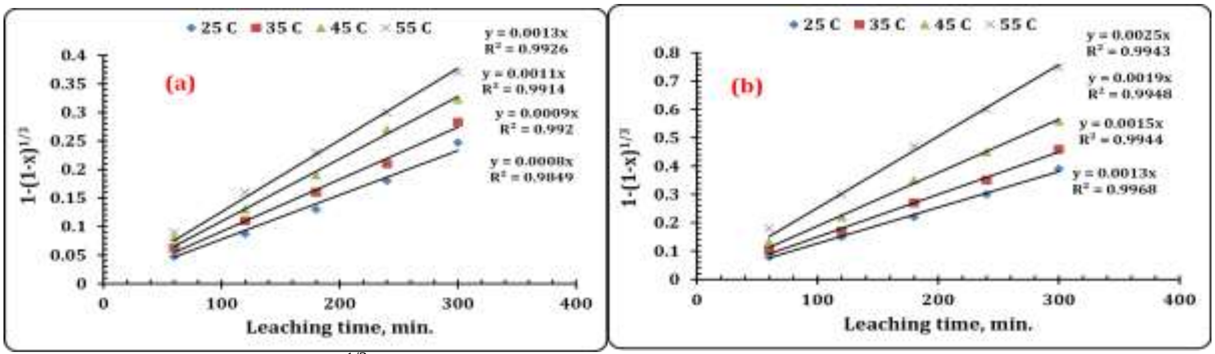


Fig.7. A Plot of $1-(1-x)^{1/3}$ against dissolution time at different temperature for (a) uranium (b) copper.

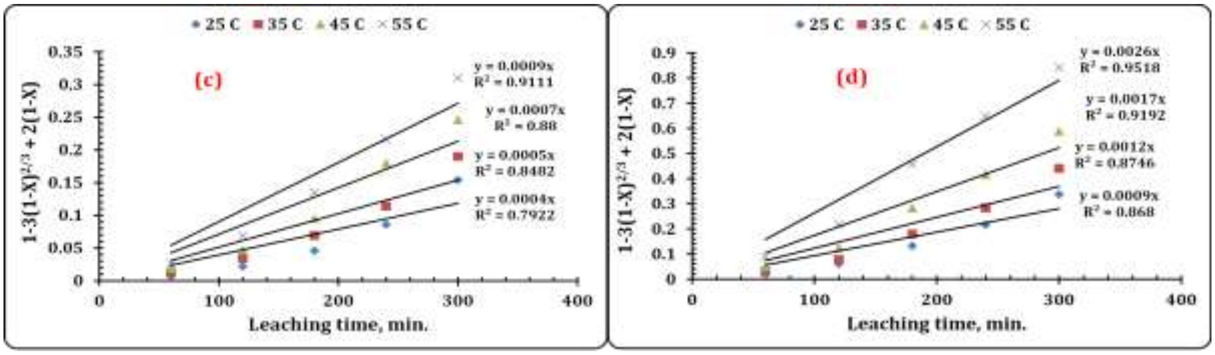


Fig.8. A Plot of 1-3(1-x)2/3+2(1-x) against dissolution time at different temperature for (c) uranium (d) copper.

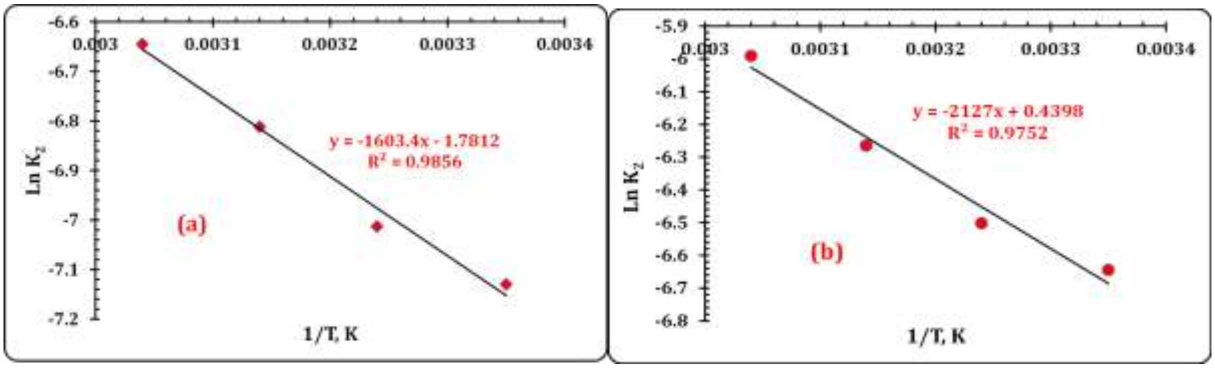


Fig. 9. Arrhenius plot of (a) uranium (b) copper kinetic analysis.

3.3. Preparation of leaching working solution

From the previous leaching studies it can be concluded that the optimum leaching conditions for leaching 81.5 and 98% of U and Cu were attained using 100g/L of TU concentration at pH 2 at S/L ratio of 1/4 and agitation leaching for 3h at 35 ºC leaching temperature.

Applying the optimum condition upon 100g of the studied sample led to prepare a pregnant leaching solutions assaying 140 mg /L of Cu and 60 mg/L of U as the elements of interest. The prepared solution was directed to different extracted methods including direct precipitation for Cu ions and ion exchange resin for U anion complex.

3.4. Subsequent Recovery of Cu and U compounds

Firstly, the prepared solution was adjusted for separating Cu^{2+} ions via direct precipitation by adding 0.29 solid of Na_2S (90 % purity) with contentious stirring for 30 min due to the lower value of CuS solubility product (Ksp 2.4 x 10^{-36}) it is very easily to precipitate almost Cu^{2+} ions as black CuS ppt. (**Alexeyev, (1973), Abd El Wahab, (2008), and Abd El Wahab et al (2012)** (Some reference of Elhame).

After thickening and filtration, the collected CuS product was washed and dried. Small portion was confirmed using SEM analysis Fig. (10). In the meantime another weight portion was dissolved and analyzed its Cu^{2+} content using FAAS which reflects that its purity achieved 98.5%. The filtrate TU solution free from almost Cu ions was subjected to the ion exchange application to recover its U anion complex content. This filtrate solution containing 60 mg/L of reduced U ions due to the addition of S^{--} species. Thus it was heated at 50 C^o with the addition of H_2O_2 solution to oxidize it U^{4+} ions to its hexavalent U^{6+} and adjusted to pH1.7 which is more suitable for the applied ion exchange batch method using the anionic ion exchanger resin, (Amberlite IRA_{400}).

Experimentally, 1 mL of Amberlite IRA_{400} (Cl^- form) was refreshed by stirring 10 mL of 0.1 M H_2SO_4 solution to change it to its suitable sulfate form and then stirred with filtrate TU solution for 30 min, **(Merritt, 1971; Preuss and Kurrin, 1965)** New references more useful. After complete loading of U species, the loaded resin was washed with distilled H_2O to get rid of the solution impurities and then directed to the regeneration process to bring out its loaded U species to the aqueous solution for recovering the suitable U compound. Thus the loaded resin was stirred with 30 mL of 1 M NaCl solution acidified with 0.2 M of H_2SO_4 to regenerate its U content to the aqueous solution. The eluate solution rich in U ions was treated with NaOH solution (pH 6) to precipitate U as a yellow cake ($Na_2U_2O_7$. xH_2O). The recovered Y C was washed with distilled H_2O and dried at 110 oC for 2 h and confirmed by SEM-EDAX analysis technique as shown in Fig. (11). Equation (5) illustrates the mechanism of both precipitation process of $Cu^{2=}$ ion and the subsequent reduction of U ions with addition of S^{--}**, Hua et al. (2006), Lirong et al. (2007)**]. (106,107).

$$U^{6+}_{aq} + Cu^{2+}_{aq} + S^{--}_{aq} \rightarrow CuS\ (\text{Black ppt}) + U^{4+}_{aq} \quad (9)$$

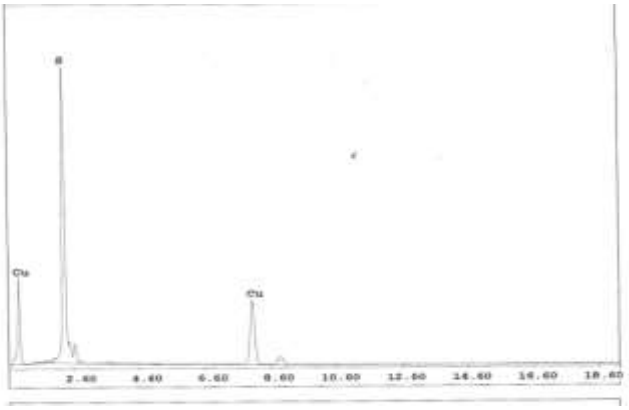
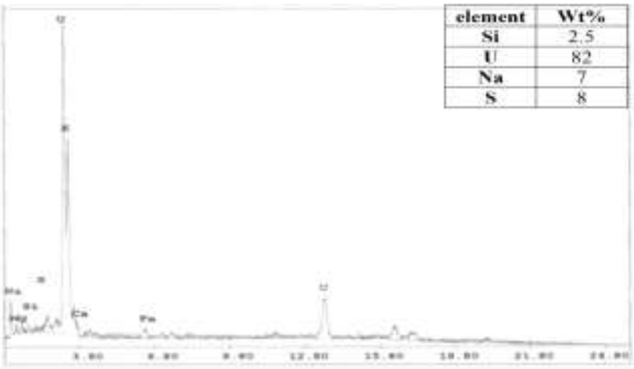


Fig. 10: EDAX chart for confirming CuS product.



element	Wt%
Si	2.5
U	82
Na	7
S	8

Fig. 11: EDAX chart for confirming U product.

4 Conclusions

Abnormal leaching reagent, thiourea has been succeeded in leaching both of U and Cu content from mud crack ore material, Talet Selem area, Sinai, Egypt. Several leaching parameters have been studied e.g. Thiourea concentration, mixed solid / liquid ratio, leaching time and finally leaching temperature to optimize the leaching process. The prepared leaching solution rich in U species and Cu ions was adjusted to direct precipitation process using Na_2S for separating Cu ions and directed to ion exchange process (batch technique) using Amberlite IRA_{400} to recover U species. The recovered product ($Na_4U_2O_7.xH_2O$) and CuS were confirmed using SEM- EDAX unit. A technical flowchart was designed for illustrating the all processing process of the present mud crack raw material.

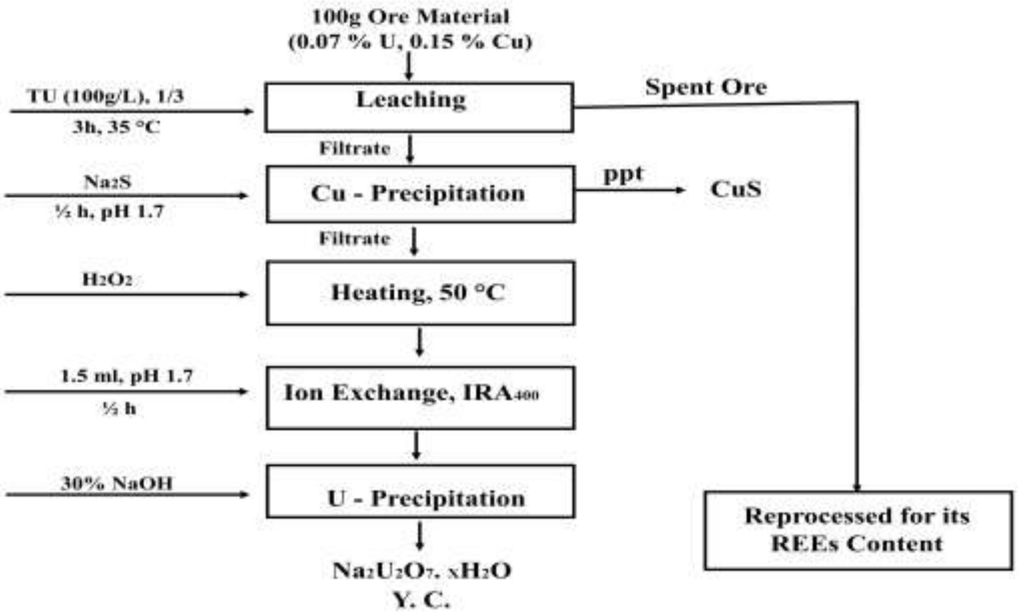


Fig.12: A technical flowsheet for chemical processing of cracked mud raw material.

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