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Research Paper

Upgrading Of Uranium Content in Its Crude Concentrate (Yellow Cake) Using Anion Exchange Resin Technique

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ABSTRACT
Depending on the high uranium concentration of dry crude yellow cake (14.175%) or (141750 mgkg^{-1}) compared with the other concentrations of associated elements, the ion exchange by using the Chinese resin (D263B) was used as attempt to upgrading uranium ore concentrate. The total capacity of the activated wet settled resin (w.s.r.) was 60 mg/g. The optimum loading parameters obtained: 6000 mg/L uranium concentration, 1.5 PH, 1.5 hours Stirring time, 150 round per minute and 1/20 solid/liquid ratio. The maximum amount of loaded uranium during adsorption process was about 52.8 mg which represents about 88.1% upon the capacity of the resin. on the other hand, the maximum amount of uranium desorption was reached to 47.1 mg which represents about 89.2%. In the elution process, the optimum conditions obtained: 1.5 M NaCl as eluant solution, 1/10 solid/liquid ratio, 150 round per minute and 3 hours stirring time and 1 pH . All the experiments were carried out at room temperature. The uranium was precipitated from the eluted solution by 40% sodium hydroxide at pH 7.5 as sodium diuranite ($Na_2U_2O_7$). Uranium in the obtained end product ($Na_2U_2O_7$) reached to 61.2% while the purity reached to about 81.53%. In refining of the upgraded product by solvent extraction, such impurities and other remaining elements would be easily decreased to the permissible limits. So, It is very important to follow the production of yellow cake by this recommended upgrading process.
***KEY WORDS**: Uranium element, anion exchange, upgrading of uranium ore concentrate.*

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I. INTRODUCTION

One of the most important steps in the nuclear fuel cycle is the uranium refining and conversion which goes from the yellow cake to three different uranium products: uranium dioxide (UO_2), natural metallic uranium (U) and uranium hexafluoride (UF_6). Refining step is a process of minimizing the concentration of the harmful impurities which always defect nuclear fuel fabrication and performance. As well as , upgrading uranium content in the produced crude yellow cake through different practical procedures. Uranium precipitated from its bearing solution over a wide pH range, acid or alkaline, depending upon the solution type and precipitant used [1-4]. In the production of yellow cake by precipitation techniques, the most commonly used method was ammonium hydroxide precipitation to form ammonium diuranate [5]. Although the yellow cake produced either byNH_4OH or by MgO. The refining step is indeed a process of minimizing the concentration of the harmful impurities which always defect nuclear fuel fabrication and performance. Yellow cake is a commercial name for the uranium ore concentrate from mining and milling process [6].

There are many studies have been conducted on the extraction of uranium and producing yellow cake. The first commercial process for producing pure uranium in tonnage quantities began at1942 [7].

El- Kamash and El- Sayed [8] used the extraction chromatography to study the extraction of both U(VI) and U(lV) from nitric acid solutions using TBP solvent impregnated poly acrylic acid polymer (SM-7) as inert supporting material. Awwad [9] used TOPO to extract uranium (Vl) from aqueous nitrate medium, it was found that uranium extraction by TOPO was suitable in toluene as diluent than cyclohexane and chloroform.

The extraction and isolation of 27 metal ions from HCl and HNO3 solutions were investigated using a dithizone-modified polymer. For adsorbing and concentrating metal ions from ethanol solution, a silica gel functionalized with 3-(1-imidazolyl)-propyl groups was used. [10-13]

Amberlite-XAD resins can be chemically or physically modified with several chelators to prepare chelating resins for metal pre-concentration. Their characteristics allow multiple sorption– desorption cycles to be performed, as well as excellent mechanical stability and repeatable sorption characteristics. The chelating collectors are made by impregnating resin matrices with chelating ligands, which is a simple technique.

Amberlite XAD-2 was used as a metal ion collector, and it was filled with 1-(2-pyridylazo)-2-naphthol (PAN) [14].

In Egypt, several promising uranium mineralization have been discovered in both the Eastern and Western Deserts as well as in Sinai and which are either associated with igneous or sedimentary host rocks. In the present work, uranium concentrates crude yellow cake produced from Abu Zenima pilot plant project, Sinai, Egypt, directed by the Nuclear Materials Authority was subjected to uranium upgrading process using the anion exchange resin technique.

II. MATERIAL AND METHODS

2.1. Preparation of the study sample

The working sample of uranium crude yellow cake was obtained from Abu-Zenima uranium pilot plant and was subjected to complete chemical analysis of uranium and some other associated elements which may be found in the crude sample according to the chemical composition of leached ore sample **.**

2.2 Preparation of stock solution

A stock solution of uranium crude yellow cake was prepared by dissolving 37.5g dry sample in 200 ml (26554 mgl^{-1}) of 50 g/L sulfuric acid 98% (Table 2). A stock solution of uranium crude yellow cake(200ml) was prepared by dissolving 37.5g dry sample in 50 g/L sulfuric acid solution 98%, which represents about (26554 mgl^{-1} uranium concentration) **.** during dissolution the solid suspension impurities have been removed was then filtered and washed by distilled water to the (200 ml) of the pregnant solution . The latter has also been subjected to complete chemical analysis. In dilute sulfuric acid solution the uranyl caion, uranium sulfate,and both the divalent and tetravalent uranium sulfate complex anions will be present in the following equations:

$Na_2 U_2 O_7 + 3H_2 SO_4 \rightarrow$
$2UO_2 SO_4 + Na_2 SO_4 + 3H_2 O$
$UO2SO4 + SO_4^{2-} \rightarrow UO2 (SO4)_2^{2-}$
$UO2 (SO4)_2^{2-} + SO_4^{2-} \rightarrow UO2 (SO4)_3^{4-}$

2.3. Ion exchange resin operation

The Ion exchange Chinese (D263B) resin was the resin adopted in the Abu Zeinma pilot plant which is characterized by some physical and chemical properties, it is macro porous of strong basic styrene series, it has a maximum resistance to oxidation and reduction reactions, its functional group is $–N-(CH_3)Cl$. Accordingly, uranium in adsorption process should be converted to the mobile phase of Cl^{-1} to bisulfate HSO_4^{-1} group by soaking for 24 hours in 50 g/L H_2SO_4. All these, because of the presence of chloride ion on the resin particles may be leads to early breakthrough point and reduces the loading efficiency during the adsorption process. The reaction between the mobile ion adsorbed the resin and the uranium ions in solution(R designating the fixed ion group and x the mobile ion) as the following:

$4RX+[UO_2(SO_4)_3]^{4-} \rightarrow R_4UO_2(SO_4)_3 +4X$

2.4. Upgrading procedure

2.4.1. Uranium adsorption process

Adsorption process is the loading of the dissolved ions especially uranium on the activated resin from its pregnant solution by ion exchange. Many of agitation adsorption experiments were conducted to determine the optimum conditions giving the maximum uranium loading efficiency by stirring certain amount (1 gram) of wet settled activated resin (D263B) at different parameters such as: different concentration of uranium crude yellow cake solution, pH, solid/liquid ratio, stirring time and revolution per minute at room temperature (25 ^{o} C). The amount of uranium ions adsorped up on the used activated resin, q_e (mg/g) and the adsorption efficiency are calculated using the following equations respectively:

$$q_e = (C_o - C_e)\frac{V}{m}$$

$$\mathrm{E}\,\% = \frac{(C_0 - C_e)}{C_0} \times 100$$

where q_e is the amount of uranium adsorbed on the used resin (mg/g), C_0 and C_e are the initial and final concentrations of uranium (mg/L), V is the solution volume (L), m is the resin weight (g) and E is the adsorption efficiency.

2.4.2. Uranium elution process

Elution procedures were carried out upon the uranium loaded resin where a series of experiments were conducted by mixing certain volume of the eluant solution (1 molar sodium chloride) with different amounts of the loaded resin. As well as adjusting the solid/liquid ratio, shaking time per hour and pH .

2.4.3. Analytical procedures

Uranium concentrate was subjected to chemical analysis for uranium and many of the associated elements. The obtained solutions were subjected to uranium analysis to follow the its efficiency during both adsorption and desorption processes.

The pregnant solution of uranium crude yellow cake was analyzed using atomic absorption spectrometer (AAS, unicum 969, England) to determine the associated elements and by oxidimetric titration method against ammonium metavanadate for uranium after its reduction **[15].**

III. RESULTS AND DISCUSSION

3.1. Characteristics of the crude yellow cake sample

Chemical analysis of the study crude yellow cake sample (Table 1), the total concentration of uranium element in the dry crude yellow cake at first before dissolution was (13.75%) while it attained (14.175%) after filtration. Also, ESM analysis of the study crud yellow cake is presented in Fig. (1). While the prepared uranium crude yellow cake stock solution analysis (Table 2) showed that the total uranium concentration is (26554 mg/l).

Table (1): Chemical analysis of Abu-Zenima dry yellow cake.

Element	Concentration, %	Element	Concentration, %
U	14.175	Si	0.156
Cu	0.075	K	0.15
Fe	0.562	Zn	0.056
Pb	0.0065	Co	0.011
Ni	0.037	Mg	0.258
Ca	0.755	Mn	0.07
Cd	0.00075	Na	4.77
Al	0.085	p	0.345

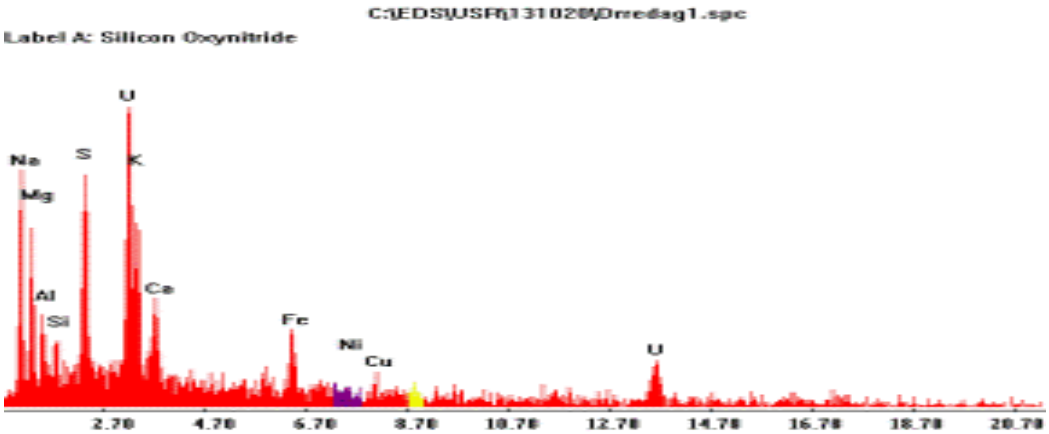

Fig. (1): ESM analysis of the study crud yellow cake

Table (2): Chemical analysis of crude yellow cake stock solution.

Element	Concentration mg/l	Element	Concentration mg/l
U	26554	Si	293.0
Cu	141.10	K	280.3
Fe	1054.2	Zn	102.75
Pb	12.31	Mg	485.06
Ni	69.20	Ca	1411.4
Mn	131.1		

3.2. Ion exchange process

3.2.1. Uranium adsorption

In order to achieve the aim of the adsorption process, different factors affecting uranium loading efficiency were studied such as: uranium concentration and its content, solid/liquid ratio, shaking time, round per minute and pH of the solution.

3.2.1.1. Effect of the uranium concentration and its content

Uranium loading efficiency on the wet settled resin (capacity 60 mg/g) depends on the effect of uranium concentration in the crude yellow cake solution and the total content of loaded uranium. Different concentrations of the pregnant solution ranging from (10.000 to 1000 mg/l) corresponding to (100 to 10 mg) uranium content was studied. The other conditions were fixed as follow: 1 pH, 1/10 solid/ liquid ratio,1 hour stirring time and 150 round per minute at room Temperature**.**

The obtained data(Table 3**)** showed that, the best uranium loading efficiency upon the total capacity of the wet settled resin (D263B) was (60.6%) that represent 36.36 mg loaded amount of uranium at 6000 mg/l while there is a slight increase in the case of 8000 and 10.000 mgL^{-1}.

Table (3): Effect of uranium concentration and content upon loading efficiency

Uranium concentrate solution		Capacity of w. s. r (mg/g)	**Loaded uranium (mg)**	Uranium Loading efficiency upon the resin capacity, (%)
Uranium conc., mg/l	uranium content, (mg)			
1000	10	60	9.06	15.1
2000	20		16.56	27.6
4000	40		28.44	47.4
6000	60		36.36	60.6
8000	80		36.66	61.1
10000	100		36.90	61.5

3.2.1.2. Effect of solid/liquid ratio

Effect of solid/liquid ratio upon uranium loading efficiency according to the total capacity of the wet settled resin and the loaded amount of uranium were studied between 1/5 and 1/30 while the fixed factors were 6000 mg/l uranium concentration and different contents in the solution, 1pH, 1hour stirring time and 150 round per minute at room temperature (Table 4).

Table (4): Effect of resin/liquid ratio upon uranium loading efficiency.

Solid/Liquid (ratio)	Uranium concentrate Solution		Capacity of w. s. r (mg/g)	loaded uranium, (mg)	Uranium Loading efficiency upon the resin capacity (%)
	uraniumConc., mg/l	Uranium content, (mg)			
1/5	6000	30	60	18.9	31.2
1/10		60		36.36	60.6
1/20		120		45.36	75.6
1/30		180		46.32	77.2

From the obtained data, it is clear that with increasing the S/L ratio the uranium loading efficiency based on the resin capacity increased from 1/5, 1/10 to 1/20 (31.2, 60.6 and 75.6%) and a slight increase at (1/30)that represent uranium77.2% loading efficiency and loaded amount (46.32 mg).The optimum solid/liquid ratio giving the maximum uranium loading efficiency was at (1/20) (75.6%).

3.2.1.3. Effect of stirring time

To study effect of stirring time upon the uranium loading efficiency, many of the working experiments were performed at a range from 1 to 3 hours under the conditions of 6000 mg/L uranium concentration, 1/20 S/L ratio, 1pH and 150 round per minutes at room temperature.

The obtained results (Table 5) showed that, the suitable stirring time is 1.5 hour where the maximum uranium loading efficiency based on the capacity of the resin was (83%) and 49.8 mg of the loaded uranium.

While there is a slight decrease of loading efficiency reached to (82.0, 81.4%) with increasing the time from 2.5 to 3.0 hours respectively.

Table (5): Effect of stirring time upon uranium loading efficiency (%)

Stirring time, (hour)	Uranium concentrate solution		Solid/ liquid ratio	Capacity of w. s .r (mg/g)	Loaded uranium, (mg)	Uranium Loading efficiency upon the resin capacity, (%)
	Conc., mg/L	Content, (mg)				
1.0	6000	120	1/20	60	45.36	75.6
1.5					49.8	83.0
2.0					50.16	83.6
2.5					49.20	82.0
3.0					48.48	81.4

3.2.1.4. Effect of round per minute

This effect was studied using a mechanical shaker at different speeds between (50 to 200 round per minute) with fixing the other conditions at 6000 mg/L uranium concentration, 1/20 S/L ratio,1.5 stirring time and 1pH at room temperature.

The obtained results **(**Table 6**)** showed that, the maximum uranium loading efficiency reached to (84.7%) and (50.82 mg uranium amount) loaded at (200 round per minute) while the minimum was about (53.2%) at (50 round per minute). The (150 round per minute) was the optimum speed reached.

Table (6): Effect of round per minute upon uranium loading efficiency (%)

Round per (minute)	Uranium concentrate solution		**Solid/Liquid (ratio)**	stirring time, (hour)	Capacity of w. s. r (mg/g)	Loaded uranium, (mg)	Uranium Loading efficiency upon the resin capacity %
	Uranium conc., mg/L	**Uranium content (mg)**					
50	6000	120	1/20	1.5	60	31.92	53.2
100						41.7	69.5
150						49.8	83.0
200						50.82	84.7

3.2.1.5. Effect of pH

The pH of the uranium pregnant solution is one of most important factors that affected its loading efficiency on the active sites of the anion exchange resin, where the solution has many of anions especially H_2SO^-, SO_4^{-2} and other anion impurities. Any of these anions will be adsorbed on the activated resin according to their relatively concentration. The high ratio of sulfate ion to uranium shifts the equilibrium between uranyle tetravalent and uranyle divalent complexes towards formation of the uranyle tetra valent complex. The uranyle tetra valent $(UO_2SO_4)^{4-}$ is considered the main chemical form adsorbed by the resin. The obtained results (Table 7) in which the range of the studied pH varying between (1 to 2), the uranium loading efficiency was increased with increasing the pH from (1 to 2) (83 to 88.9%) at the excremental conditions of (6000 mg/l) uranium concentration, (1/20) S/L ratio, 1.5 hour stirring time and 150 round per minute. According to the obtained data, the pH 1.5 is the most suitable one.

Table (7): Effect of pH upon uranium loading efficiency (%)

pH	Uranium concentrate solution		Solid/liquid (ratio)	stirring time, (hour)	Capacity of w. s. r (mg/g)	Loaded uranium, (mg)	Uranium Loading efficiency upon the resin capacity %
	Conc., mg/L	content (mg)					
1	6000	120	1/20	1.5	60	49.8	83.0
1.5						53.22	88.7
1.7						53.33	88.89
2						53.34	88.90

Batch experiment

Depending on the obtained results of uranium loading experiments, the optimum loading conditions of uranium adsorbed on the anion exchange resin (D263B) were applied giving **88.1 % or (52.8 mg)** where:

- The total uranium concentration of the crude yellow cake solution is: 6000 mg/L.
- Solid/liquid ratio : 1/20 ratio
- Stirring time : 1.5 hour
- Round per minute : 150 rpm
- pH : 1.5
- Room temperature

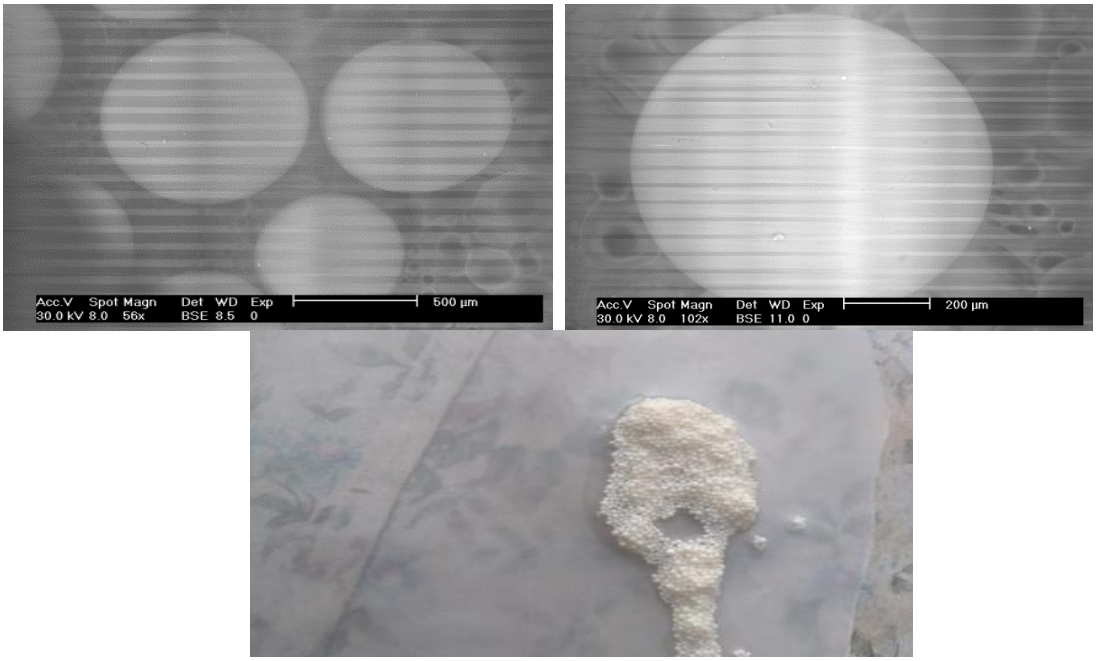

Fig. (2): A seed of the unloaded worked activated resin

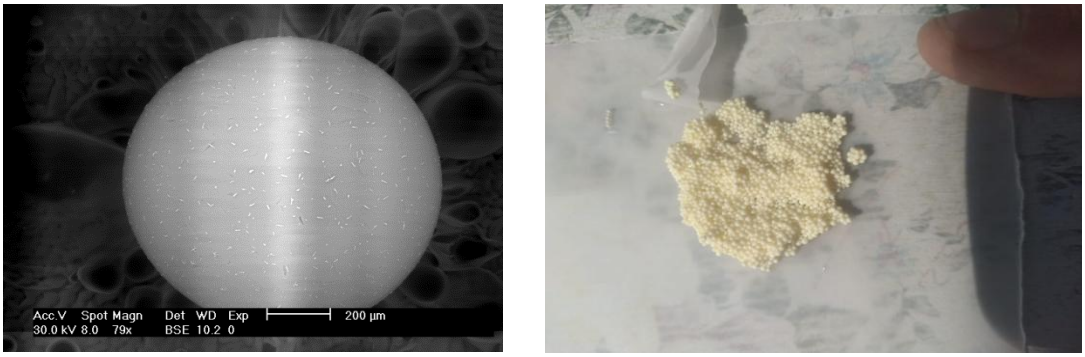

Fig. (3): A seed of the loaded worked resin surrounded by uranium ions

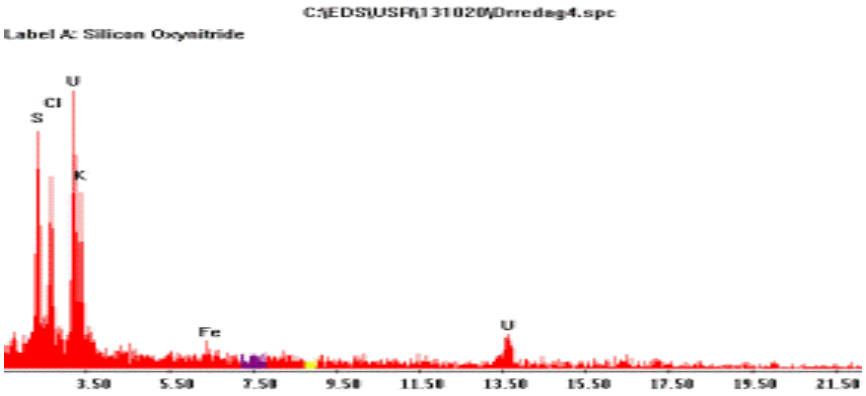

Fig. (4): EDX of the uranium loaded resin

3.2.2 Uranium elution process

3.2.2.1 Effect of eluant type

There are many types of different eluting agents in the uranium elution process, their choice should depend on the cheap price, available and must not bound with the resin to not exchange slow during the next loading of ion exchange. Solutions of H_2SO_4, HNO_3 and NaCl were studied (Table 8). This factor was performed under the following conditions: 1M eluant concentration, 1/5 S/L ratio, 1hour stirring time, 100 round per minute and the loaded resin contains about 88% about 52.8 mg uranium and 1 or 0.05 pH.

Table (8): Effect of eluant type upon uranium elution efficiency

Eluant solution	Uranium elution efficiency%
HNO_3	34.4
NaCl	32.2
H_2SO_4	28.7

The obtained results showed that, the maximum uranium elution efficiency attained 34.4 % by HNO_3 but because of its high price compared with that of NaCl, also due to the little difference in their efficiencies sodium chloride solution was chosen as the optimum eluant.

3.2.2.2 Effect of eluant concentration

For this factor, different NaCl solution concentrations ranging from 0.5 to 2 M were used under the following conditions: 1hour stirring time, 100 round per minute, 1/5 solid/liquid ratio and 1 pH at room temperature (Table 9). It is clear that, with increasing the eluant solution concentration the uranium elution efficiency increased from 23.8% at 0.5 M to 35.9 % at 2 M which is the optimum concentration economically is1 M.

Table (9): Effect of eluant concentration upon uranium elution efficiency

Concentration of NaCl, M	Uranium elution efficiency %
0.5	23.8
1.0	32.2
1.5	33.5
1.8	35.4
2.0	35.9

3.2.3. Effect of solid/liquid ratio

The solid/liquid ratio is considered as one of the important factors in the elution process where different solid/liquid ratios were studied at a range from 1/5 to 1/30. The experimental parameters were 1 M NaCl, 100 round per minute, 1 pH and 1hour stirring time. The obtained data(Table 10), indicated that, the suitable S/L ratio that gave 48.8% uranium elution efficiency and less amount of the eluant used is 1/10 **.**

Table (10): Effect of solid/liquid ratio upon uranium elution efficiency

Solid/Liquid ratio	Uranium elution efficiency %
1/5	32.1
1/10	48.8
1/20	51.4
1/30	51.7

3.2.4. Effect of stirring time

To study the effect of stirring time upon uranium elution efficiency, five experiments were carried out to determine the maximum uranium elution efficiency. This factor was studied in the time range from 0.5 to 4 hours. On the other hand, the other conditions were 1 M NaCl, 1/10 S/L ratio, 100 rpm and 1pH at room temperature. From the obtained results, the maximum uranium elution efficiency was reached to 77.41 % at 4 hours while it decreased to76.15% at three hours. The optimum time is for 3 hours especially there is a low difference in their efficiencies (Table11).

Table (11): Effect of stirring time upon uranium elution efficiency

Shaking time, hour	Uranium elution efficiency %
0.5	27.1
1	44.32
2	62.71
3	76.15
4	77.41

3.2.5. Effect of round per minute

The effect of the round per minute of uranium elution efficiency was studied at different rpm ranging from 50 to 200 rpm. The other fixed parameters 1 M NaCl, 1/10 S/L ratio and 3 hours stirring time at room temperature. From the obtained data (Table 12**),** it is noticed that, with increasing the shaking time, uranium elution efficiency increased from 39.8% to 91.64% at 50 rpm to 200 rpm respectively. Accordingly, the optimum round per minute is the 150 rpm which gave 89.1% uranium elution efficiency.

Table (12): Effect of round per minute upon uranium elution efficiency

Round per minute	Uranium elution efficiency, %
50	39.8
100	76.15
150	89.1
200	91.64

Depending on the obtained optimum factors of uranium elution process, a batch experiment was performed upon the loaded resin (D263B) where the obtained uranium elution efficiency attained about 89.1 % (47.1 mg) at the following parameters:

- The total amount of the eluted uranium is: 52.8 mg
- Solid/Liquid ratio : 1/10 ratio.
- Stirring time : 3 an hour .
- Round per minute : 150 rpm .
- pH : 1

- Room temperature

IV. URANIUM PRECIPITATION

The eluted solution obtained from the loaded resin was subjected to neutralization by adding 40% caustic soda at pH from 7 to 8. Uranium precipitation efficiency reached to about (97.2 %). After filtration,

uranium precipitate was left to about half an hour then washing the precipitate for three times by distilled water to remove the existed amount of chloride. Finally, the product was dried at 105-$^{+}5^{0}$C (Fig. 5).

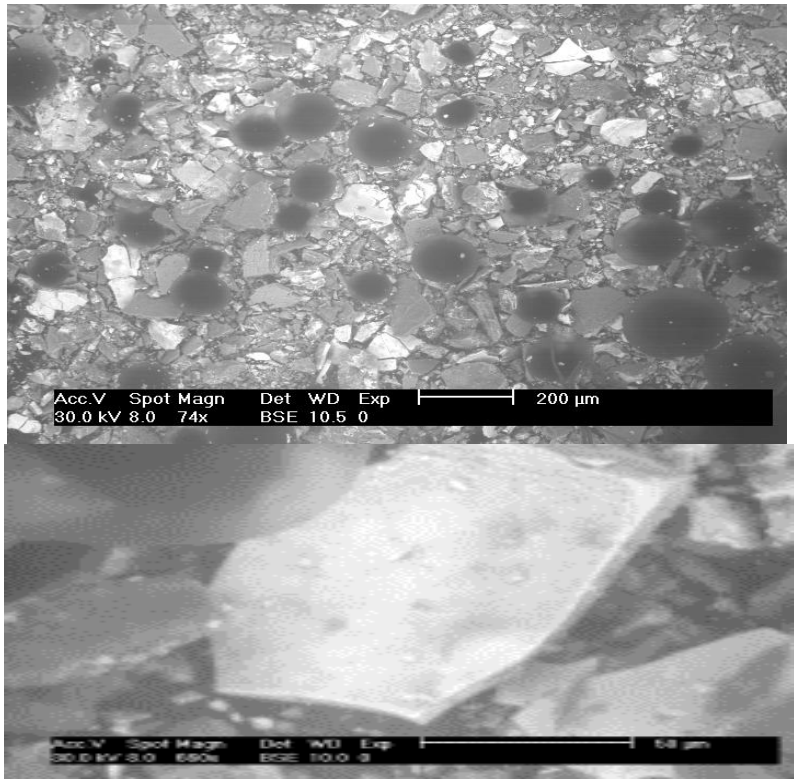

Fig. (5): the precipitated yellow cake

V. CONCLUSION

The ion exchange Chinese resin (D263B) was applied for upgrading uranium in the crude yellow cake of produced from Abu Zienma pilot plant project at Sinai, Egypt by both adsorption and elution processes. The obtained optimum conditions for the loading operation were: 6000 mg/l uranium concentration, 1.5 pH, 150 rpm, 1.5 stirring time and 1/20 solid/liquid ratio at room temperature. The obtained uranium loading efficiency depending on the total capacity of the D263B resin (60 mg/g) reached to 88.1%. On the other hand, the obtained optimum conditions of the elution process were: 1.5 M of NaCl as eluant agent solution, 1/10 S/L ratio, 3 hours stirring time and 150 round per minute at room temperature. The obtained uranium elution efficiency depending on total amount of the loaded uranium reached to 89.1% .

The eulted solution was subjected to a precipitation process by adding 40% NaOH at pH 3 to precipitate iron and raised the pH to 7.5 to precipitate uranium as sodium diuranate ($Na_2U_2O_7$). The obtained precipitate was filtered, washed for three times by distilled water to remove the chloride ions then dried at 105-$^{+}$ ^{0}C. It is worthy to mention here that, through the ion exchange resin technique and agitation with uranium solution in the present work the grade of the study crude yellow cake was increased from 14.17% to 61.2% while the purity reached to (81.54%).%). It is very important to follow the production of yellow cake by this recommended upgrading process.

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