Column leaching of uranium of uraniferous calcareous shale

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Abstract—This study aims to investigate in some details, the leaching of U from its local ore uraniferous calcareous shale using column leaching technique. The results of the different factors affecting the leaching process of uranium value from the working ore material. This study deals with two sets of columns have been conducted namely; small and medium. The work then shifted to recovery of uranium from leach liquor by ion exchange technique. It was found that, the final concentrates of yellow cake (uranium oxide) were purified and has about 84% U.

Index Terms—uraniferous shale ore; Sulfuric acid leaching; column leaching

1 Introduction

Abu Zeneima area is situated at Southwestern Sinai between longitudes 33º 20` to 33º 25` E and latitudes 29º 00` to 29º 05 N and represents Um-Bogma Formation which belongs to the Carboniferous age (El Assy et al., 2008). This area revealed the presence of various metal values including Al, Zn, Mn, Co, Ni, Cu, REEs and U. Several leaching studies have been performed upon comparable with different constituents and grades of the economic metal values. These studies include those of Mahdy et al. (1988); Mahdy (1989); Amer (1997); Amer et al. (2000); Abdel Fattah (2003); Abdel Moneim (2005). Mahdy et al. (1988) and Mahdy (1989) have studied the leaching characteristics of both U and Cu from a siltstone ore from the Abu Zeneima area. They concluded that both metal values could be almost completely leached by mineral acid under mild agitation or percolation leaching conditions. This work aims to investigate Abu Zarab ore material and properly discussed.

Development of the ion exchange process for uranium recovery has actually been based upon the discovery that hexavalent uranium can exist in sulphate or carbonate leach liquors as an anionic complex $[UO_2\ (SO_4)^{n2-2}$ and $UO_2(CO_3)]^{4-}$, and that quaternary ammonium anion exchange resins exhibit a high selectivity for these anionic species (Lutz, 1956),(Ahriand, 1951). After uranium adsorption from its leach liquors, its elution is normally carried out by 1M ammonium nitrate or 1 M sodium chloride solutions with free acidity of 0.1 to 0.3 MH^+. Nitrate system is indeed efficient eluting agent but more expensive than the chloride system. The major disadvantages of the chloride elution system include its slow exchange rate and would therefore require more eluant volumes three times than nitrate elation resulting in dilute eluates beside necessary more expensive alloys, such as Hastelloy, to withstand its corrosive properties.

2 EXPERIMENTAL TECHNIQUES

2.1 Materials

The experimental work is conducted on a collected uraniferous shale bulk sample. This bulk sample was collected from Abu Zarab locality, southwestern Sinai. Leaching tests and subsequent operations were carried out on very low-grade uraniferous shale ore, using sulphuric acid. The analysis evident that, Abu-Zarab samples are mainly composed of SiO2 (43.80), Al2O3 (6.50) and, Fe2O3 (10.34) and to a much lesser extent of TiO2, MgO, CaO, Na2O, K2O and P2O5. The main trace elements in this calcareous clay stone are; Zn (1050ppm), V (560ppm), Cu (327ppm) and U (122ppm).

2.2 Methodology

2.2.1 Column leaching

To achieve the chemical characterization of Abu Zarab a number of chemical studies were done including the major oxides as well as some interesting trace. The first set of column is composed of three PVC as in fig. (1).small columns with the dimensions (40cm height and 2.5cm diameter). The second set of columns is composed of three Plexiglas medium columns with the dimensions (60cm height and 10cm diameter) as in fig. (2).

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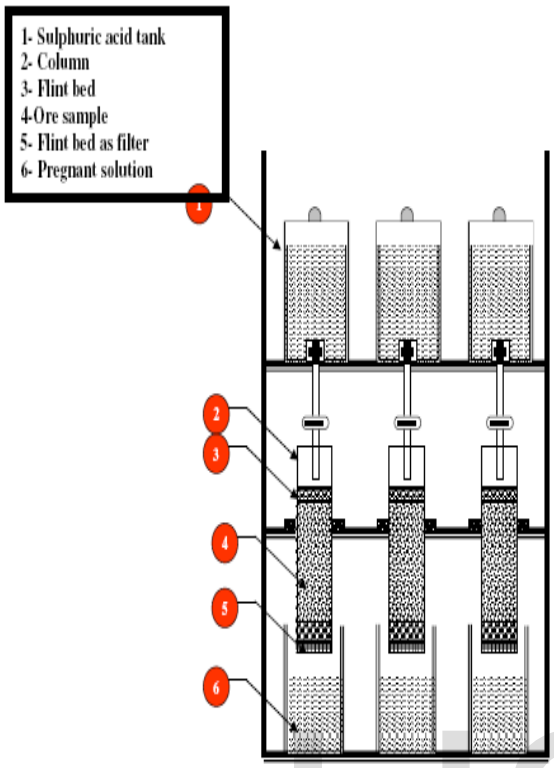

Fig.1. Experimental setup for small column method (after Wael, (2010)

2.2.2 Recovery of uranium

The leach liquor prepared according to main suitable acid condition of column leaching process was then subjected to uranium recovery.

2.2.2.1 Uranium adsorption

In order to adsorb uranium alone, without interference of other elements, especially iron oxide, the latter must be removed. The filtrate leach liquor was then adjusted to pH 1.7-1.8 before it is becoming in contact with the I.R.A. Amberlite 400 ion exchange, for the adsorption of uranium leaving the other elements in solution. The I.R.A. Amberlite 400 ion exchange was packed over a suitable amount of glass wool in Pyrex columns. Accurate 1.6 ml wet settled resin (w.s.r) was packed and then resin was treated with 15% sulfuric acid.

Adsorption experiments were conducted by passing the leach liquor at pH 1.7 through the I.R.A. Amberlite 400 ion exchange with a flow rate of (0.33ml/min.). Periodical 150 ml samples were collected through the adsorption steps until the representative resin was completely saturated (influent concentration = effluent concentration). Uranium analysis was achieved by oxidimetric titration method

2.2.2.2 Uranium elution

The previously loaded uranium values were removed or eluted from I.R.A Amberlite 400 ion exchange, using a suitable eluting solution. The eluant in the present study is 1M NaCl solution acidified by 0.25M sulfuric acid. The contact time was (1.6 ml/min.).

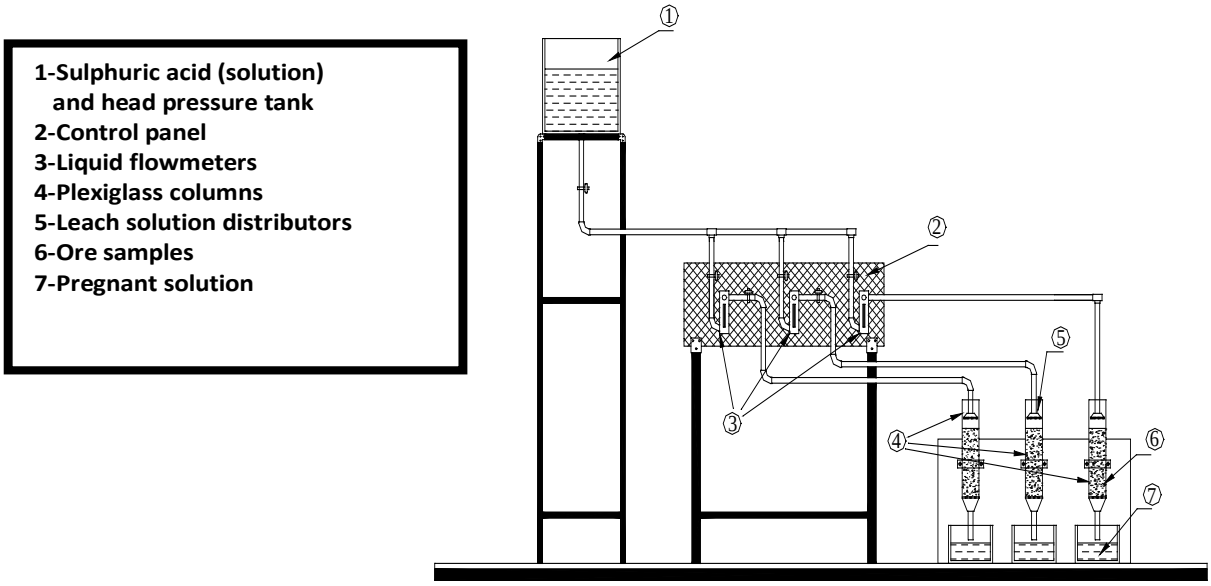

Fig.2. Experimental setup for medium column testing (after Wael, (2010)

Constant rate samples were collected for each 20 ml, followed

by determination of uranium, by using titration technique.

2.2.2.3 Uranium precipitation

Subsequently, the uranium-loaded was subjected to uranium precipitating at pH 7. This pH was adjusted by adding 1M NaCl and 1M H2SO4 under the studied optimum stripping factors. The produced uranium is contaminated with iron. Refining of U/Fe precipitate was taken place through dissolve it in carbonate solution as a selective leaching of uranium followed by precipitated the pure uranium at pH 12-13, followed by calcinations to produce U3O8. The obtained precipitate has been confirmed using EDAX analysis.

2.2.2.4 Chemical analysis

Chemical analysis was carried out of the major oxides using the rapid silicate analysis method of Shapiro and Brannock (1962). On the other hand uranium measured by gravimetry and an oxidimetric titration method against ammonium metavanadate was used for U analysis in the presence of diphenylamine sulfonate indicator. Prior to titration, proper reduction of U was performed using ammonium ferrous sulfate (Mathew, 2009). The obtained precipitate has been confirmed using EDAX analysis

3 RESULTS AND DISCUSSION

3.1 Leaching

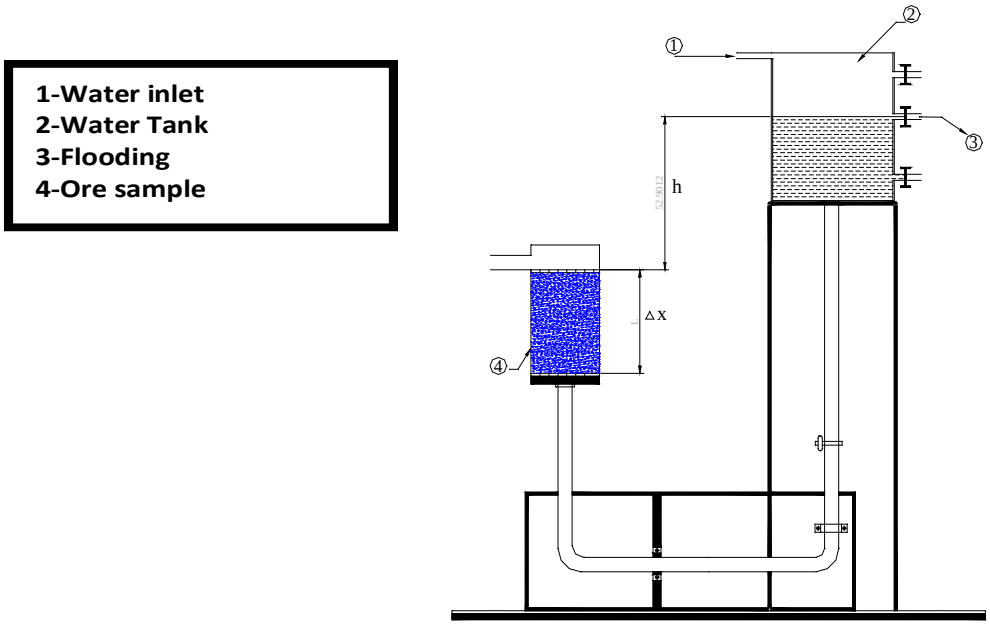

Fig.3. Constant head permeamter (after Wael, 2010)

In the case of column leaching, experimental 'blocks" are constructed with the same number of columns as there are separate test conditions. The predictive nature of leaching are treated identically throughout the test, with differences between columns within each block being the variables of interest. To achieve this, two sets of columns have been conducted; the first set is composed of three PVC. Small columns with the dimensions (40cm height and 2.5cm diameter). The second set of columns is composed of three Plexiglas medium columns with the dimensions (60cm height and 10cm diameter). Proper design for experimental work leads to scaled – up columns with heights close to planned height in the ore heap. Each column is intended to simulate a vertical section through the eventual pre- heap from top to bottom under different conditions.

3.1.1 Small column (2.5 cm wide) leaching tests

The work has started using the small set of columns. For all small columns tests, sulphuric acid is used for leaching the ore. The acid is continuously percolated through the cylindrical perforated feeder across the top cross sectional area of the ore column until it reaches to the bottom and passes through the filter and is received as a pregnant solution. Small column leaching tests include all the factors that may affect leaching operation when fully applied to the medium columns. Different operating parameters are investigated in this section. The results obtained are presented, discussed, and interpreted in the following.

3.1.1.1 Measurements of intrinsic permeability and porosity for multi-sized particles

Prior to the investigation of the operating parameters affecting the leaching of the uraniferous shale in columns, the

intrinsic permeability for different multi-sized particles available for such tests is measured. The hydraulic conductivity can be measured in an ore column set up as a constant head permeameter (constructed at the laboratory and its design is adopted from (Barlett, 1998) which is illustrated in (Fig.3) and then calculated by using the following equation: $u = Q / A = K h / \Delta x$ Where, u is the superficial velocity, Q is the volumetric flow rate, A is the cross section area of ore, K is the hydraulic conductivity, h is the hydrostatic head and ΔX is the ore column length. Then the intrinsic permeability can be deduced from the following equation (Barlett, 1998): $K = Ki\, g\, \rho / \mu$ Where, g is the gravity acceleration constant, ρ is the fluid density, μ is the dynamic viscosity of fluid, Ki is the intrinsic permeability. A known weight(w1) of dry sample (dried at 100° C for 48 hours) is submerged in water at room temperature for 48 hours, then the sample is filtered, weighed (wet) (W2) .

The volume of the sample is measured using graduated cylinder (V). The volume of micro- pores is given by (W2-W1). The porosity is calculated from the formula (Barlett, 1998): Porosity = W2-W1 / V * 100 %

The permeability and porosity for each multi–sized sample used in each column testing have been carried out before applying leaching. The results obtained are shown in (Fig. 4).

sized particles, the open size (permeability) is proportional to the squared particle size (d2), hence ki α d2 (Barlett, 1998). Therefore the permeability (open size) is increased as the squared particle size increases and vice–versa, from the (Fig. 4) it can be seen that the permeability is inversely proportional to the porosity for all the studied particle sizes.

3.1.1.2 Effect of particle size on U leachability

Decreasing the grain size is expected to increase the leaching efficiency as more surface area would be subjected to the acid and in turn, the leaching rate increases. This test is well suited to cases where the material is porous (sandy), and is inapplicable to material which tend to pack into impervious masses. Regularity in the size of the particles rather than their natural size distance is the chief factor governing good percolation. The idea is that where the particles of unequal size, the small one is pack in the openings between the larger ones, thereby clogging the channels. Extraction becomes slow, and channeling of solutions through the bed takes place. The method is therefore unsatisfactory if much slime is present. In this work, the effect of the particle size upon the leachability of uranium from uraniferous shale was studied after crushing the ore to different particle sizes. Used Sample was screened into five size fractions, (-2, -4, -6.7, -9.5 and -12.5 mm.) for roasted and un-roasted ore material.

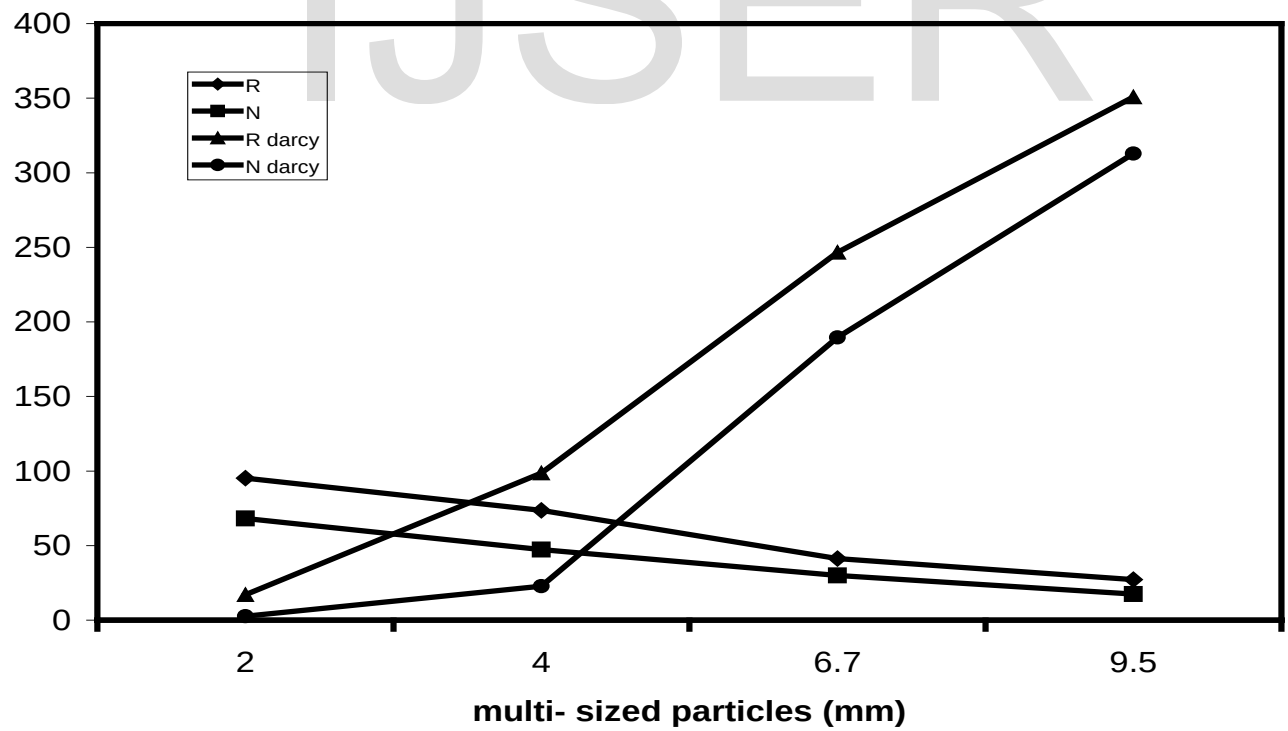

Fig.4. Effect of multi-sized particles on permeability and porosity

increases by increasing the particle size and increases for roasted (at 600°C) for removing organic material and roasted ore more than unroasted (untreated) ore material. For multi–

They are subjected firstly to permeability tests. Each size was packed in column with fixed leaching conditions solid/ liquid ratio 1/3, using sulfuric acid 12% and height / diameter ratio

8 at same solution volume. The leaching processes were performed at flow rates 1, 0.5 and 0.25 ml/min.

The results are shown plotted in (Fig. 5). From these figures, it is obvious that the less the relatively coarse particles size range, the less is the permeability, and hence the residence time for leachant in the column is longer. As a result, the contact time between the leachant and the ore particle surface will increase.

From the results, it is clear that the best uranium leachability is obtained with the particle size range of -2mm (low in the relatively coarse particle size range). This may be due to the more residence time of leachant in the column, contained the uraniferous shale ore, at the particle size range of -2mm. Therefore, the parameters that may affect leaching operation, namely, flow rate of leachant through the column as well as the leaching time, were investigated at fixed range of -2mm. The figures show also that the low flow rates are the high leachability. In addition, from these results, it is clear that the uranium percent was found to increase by decreasing the leachant flow rate and the optimum flow rate was found to be 0.25 ml/min. and it is found that the rates over1.0 ml/min are not convenient for unroasted ore due to the existence of floods of the leachant. The flow rate of 0.25ml/min. is found to be suitable and it has been used through over all the experiments. The existence of floods may be due to the lower permeability of the particles used.

Different concentrations of sulphuric acid solution are tried in order to get the optimum concentration for two fractions, roasted and un-roasted. These acid solutions concentrations are; 8%, 10%, 12% 15%, 20%, 25%, 30% and 35% by vol. / vol. The results are plotted in (Fig.6). A set of experiments under constant operating conditions (flow rate = 1.0 ml/min, height / diameter: 8, solid / liquid ratio 1/3, particle size =-2 mm) was carried out. The obtained results indicated that, by gradual increasing acid concentration from 8% to 20%, the uranium leaching efficiency increased from 36 to 68 % and from 15.4 to 29.8% for roasted and un-roasted samples respectively. The significant increase of uranium leachability percentage with gradual increasing of acid concentration up to 12% may be due to the ease dissolution of uranium in the available acid. The slight increase of uranium leachability beyond the 12% of acid concentration may be attributed to the consumption of a great part of acid in side reactions other than uranium. Therefore, 12 % of acid was the suitable concentration for uranium leaching investigations.

3.1.1.4 Effect of height/ diameter ratio on the U leachability

To study the effect of increasing height/ diameter ratio of the small column on the uranium leachability, a set of experiments was carried out at the same operating conditions (acid conc. =12% V/V., flow rate = 0.25 ml/min, solid / liquid ratio:

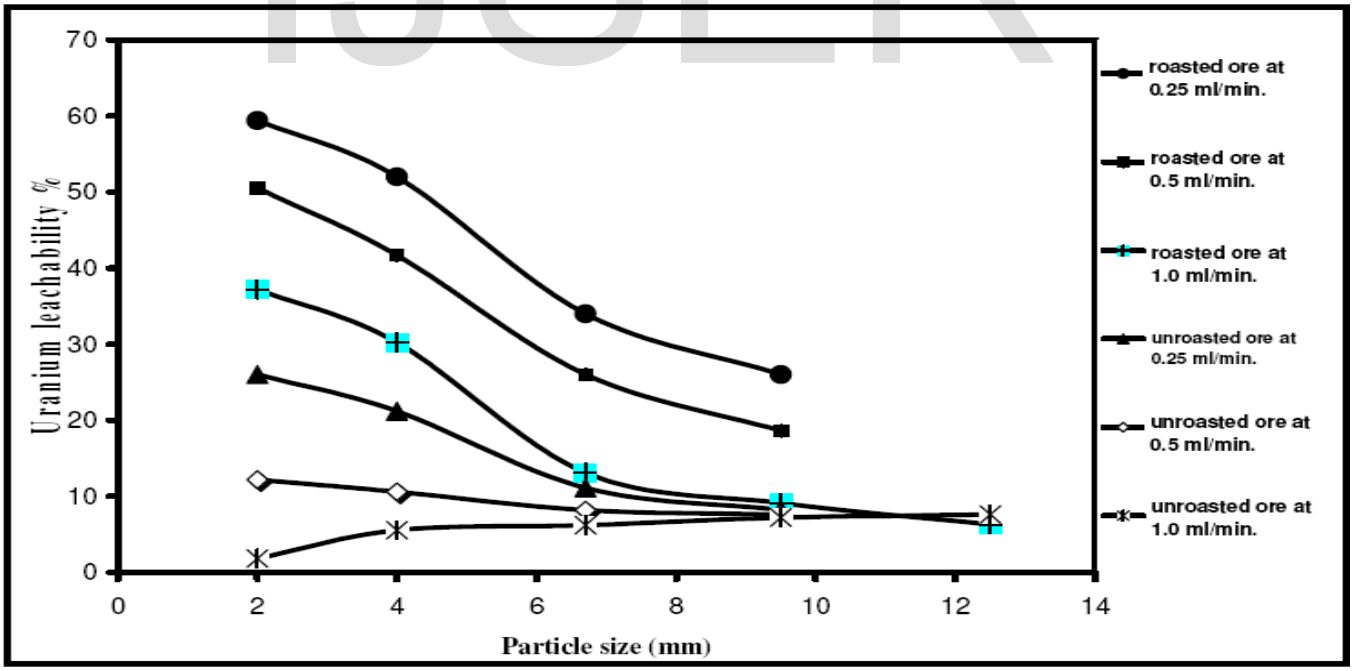

Fig.5. Effect of particles size on the U leachability

3.1.1.3 Effect of sulphuric acid concentrations on the U leachability

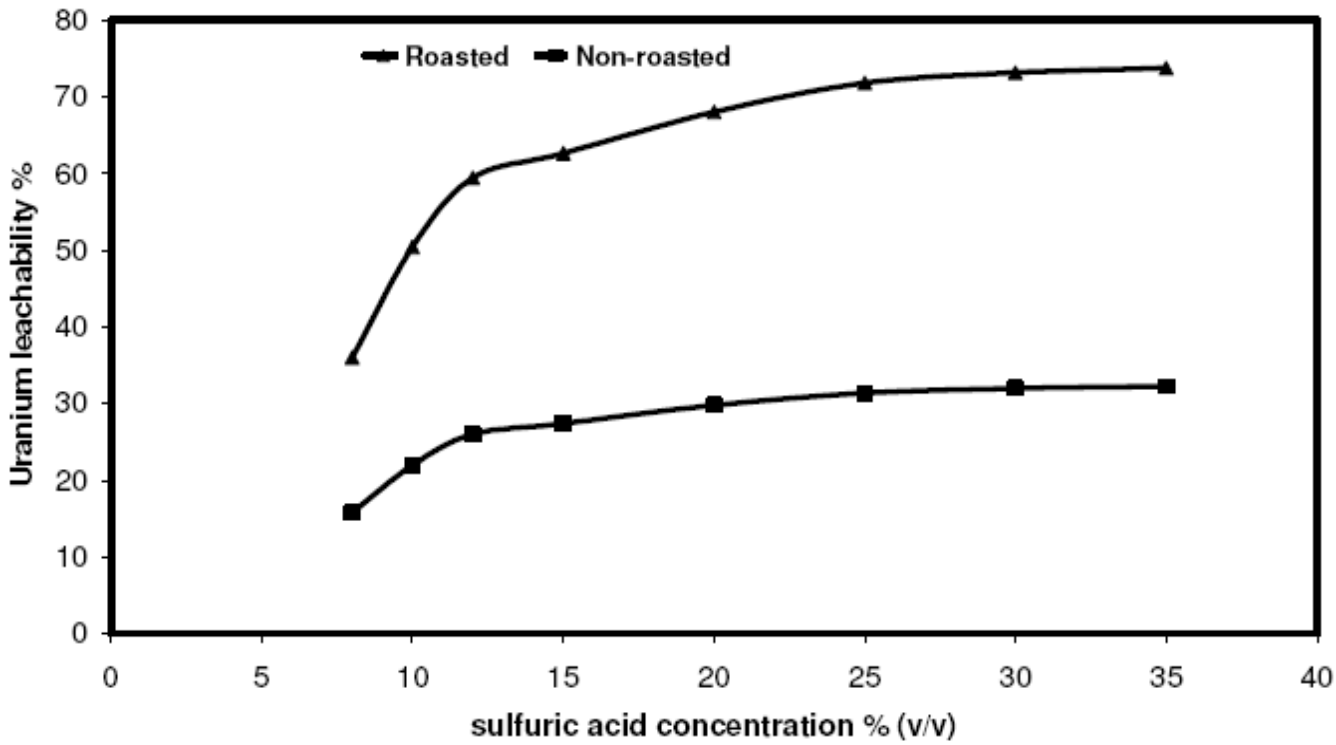

Fig.6. Effect of sulphuric acid concentrations on the U leachability

By gradually increasing the height of uraniferous shale ore in the column to the diameter of the column from the ratio 4 to the ratio 12 the uranium leachability was found to increase for both roasted ore and unroasted ore because the residence time for leachant in the column is longer. So the optimum height /diameter ratio is selected to be 12.

3.1.1.5 Effect of leaching time on the U leachability in small Column

Leaching time (measured at the inception of leacheant droplets from the top to the bottom of ore bed) is an important variable parameter in column leaching, so this variable is investigated through this work.

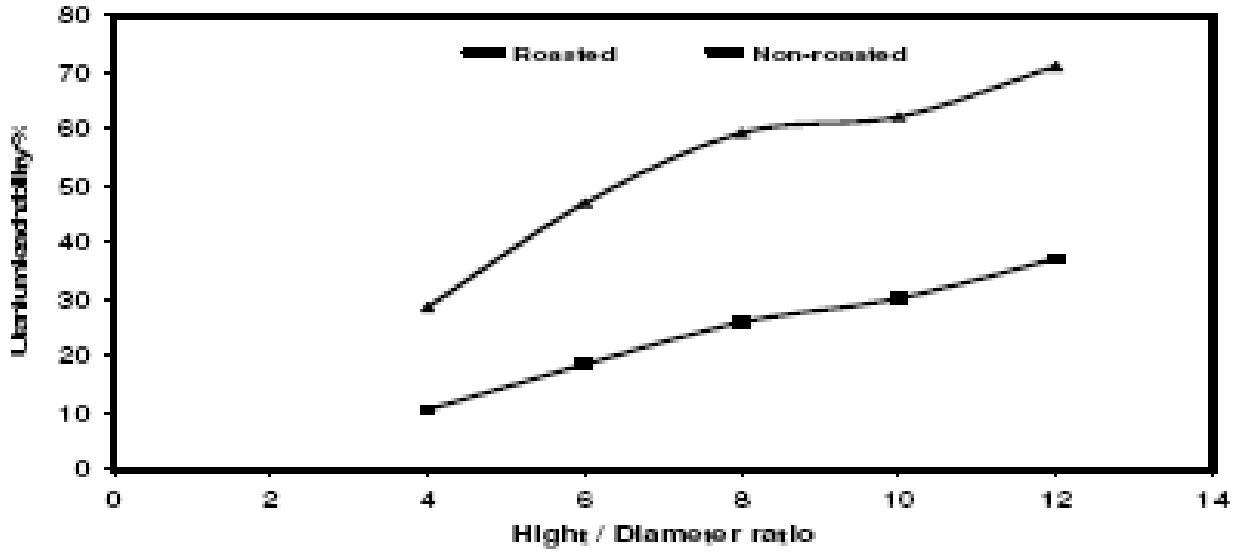

Fig.7.Effect of height/ diameter ratio on the U leachability

Its effect on uranium ore leachability is indicated and presented in (Fig.8) From this figure, it is obvious that the uranium leachability percentage increases by increasing the leaching time. By gradual increase of the leaching time from 10 hours to 40 hours, the uranium leachability percentage is found to increase drastically, while very slightly from 50 hours to 140 hours .This may be attributed to the more contact time between the leachant solution and the ore particles.

3.1.2 Medium column (10cm wide) leaching tests

The medium column (60cm height and 10cm diameter) is illustrated in (Fig. 2). Before charging the ore into the column, a bed of washed graduated gravel (size range from 30mm to 5mm) was introduced. Over the gravel bed at the bottom of column, the ore is introduced into the column followed by another layer of washed gravel with thickness of 2cm at the top of the ore.

Medium column leaching tests include all the factors that may affect leaching operation, namely, the particle size, acid concentration, leachant flow rate, column height diameter ratio and leaching time. Different operating parameters are investigated in this section on the roasted samples. The results obtained are presented, discussed, and interpreted in the following paragraphs.

3.1.2.1 Effect of particles size on the U leachability

A sample was screened into three size fractions, (- 2mm,-4mm and -9.5mm). They are subjected firstly to permeability tests and then each size was packed in the column with solid /liquid ratio 1/3 using sulfuric acid 12% concentration and height to diameter ratio 3.

range, the less is the permeability and hence the more is the residence time for leachant in the column. As a result, the contact time between the leachant and the ore particle surface will increase, and hence, the U yield is increased.

Consequently, this size range is selected to investigate the all factors that may affect leaching operation such as the flow rate of leachant through the ore column and the leaching time

3.1.2.2 Effect of Leachant Flow rate on U leachability

The effect of leachant flow rate on uranium recovery was investigated at different values of flow rates and constant operating parameters (acid conc. =12% V/V., solid / liquid ratio: 1/3, particle size=-2mm, Height / Diameter: 3) and the results are illustrated in (Fig. 10). Three different flow rates were selected (2 ml/min, 1.5 ml/min and 0.75ml/min). It is clear that the uranium leachability was found to increase by decreasing the leachant flow rate due to more contact time between leachant and ore in the column and the optimum rate was found to be 1.5ml/min., which achieve the minimum cost and consumption of acid.

3.1.2.3 Effect of height/ diameter ratio on the U leachability

To study the effect of increasing the height / diameter ratio of the medium column on the U leachability, a set of experiments was carried out with the same operating conditions (acid conc. =12% V/V., flow rate = 1.5 ml/min, solid / liquid ratio: 1/3, particle size =-2mm) in the case of column of 60 cm height; four different ratios have been studied, which are (2, 3, 4 and 5).

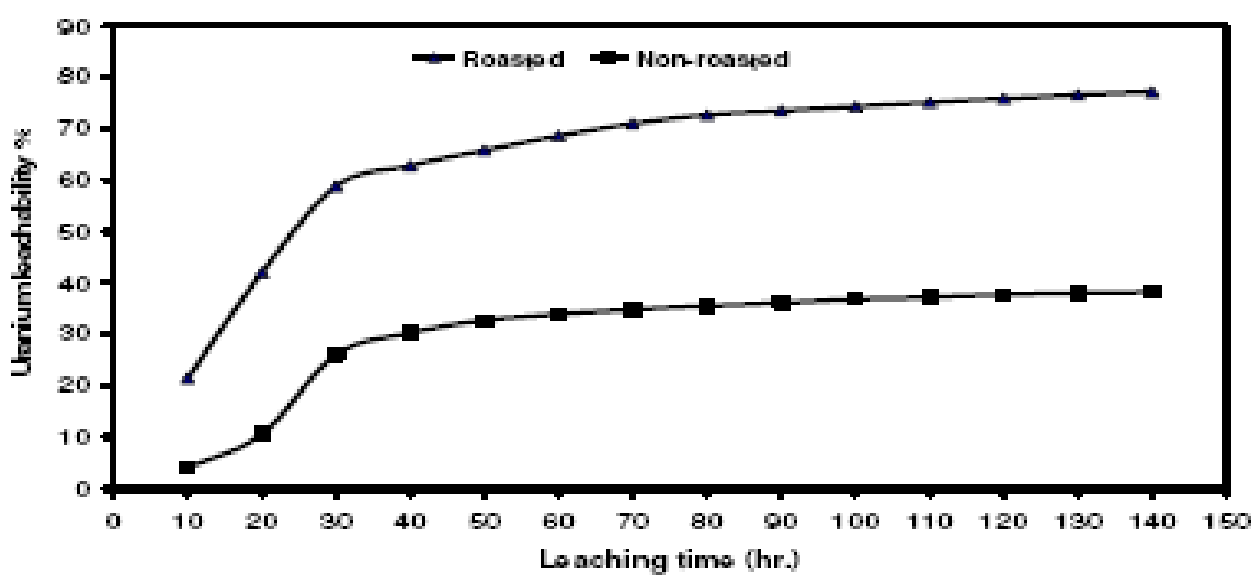

Fig.8. Effect of leaching time on the U leachability

is obvious that the less the relatively coarse particles size

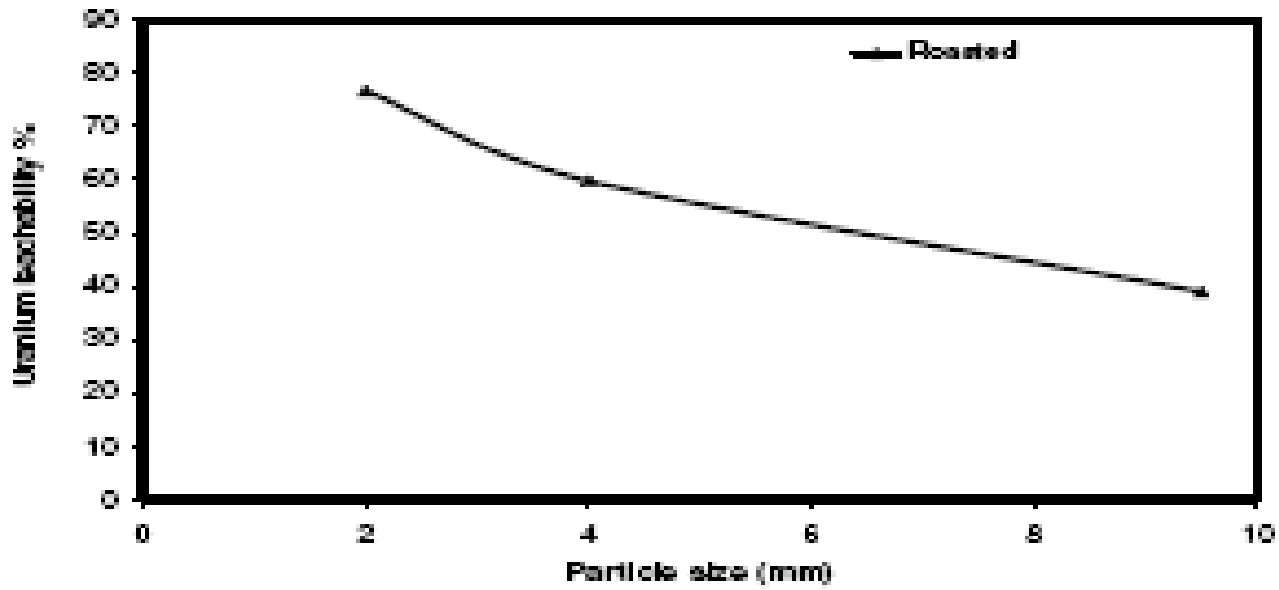

Fig.9. Effect of particles size on the U leachability

It is clear that the uranium leachability increases by increasing the leaching time (Fig. 11). By gradual increasing the height of uraniferous shale ore in the column to the diameter of the column, the leachability increases from 67.7 % to 81.3 % respectively.

3.1.2.4 Effect of leaching time

Leaching time is an important variable parameter in heap leaching, sothis variable is investigated through this work. Its effect on uranium ore leachability is indicated and the results were presented in (Fig.12). From this figure, it is obvious that the uranium leachability percentage increases by increasing the leaching time and the maximum leachability is attained at 3 days leaching time beyond which the leachability seems to be constant with the time. This may be attributed to the more contact time between the leachant solution and the ore particles.

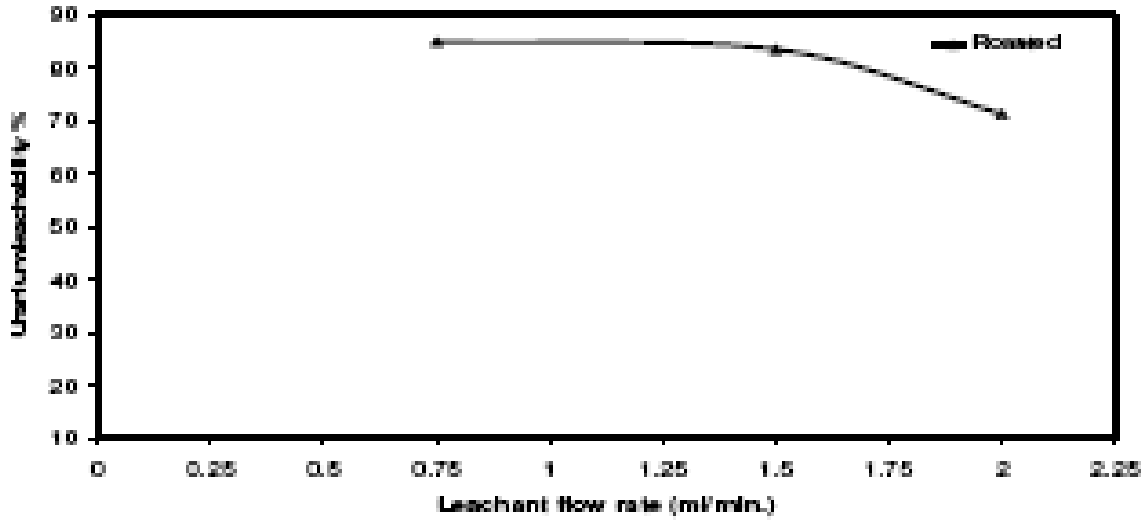

Fig.10. Effect of Leachant Flow rate on U leachability

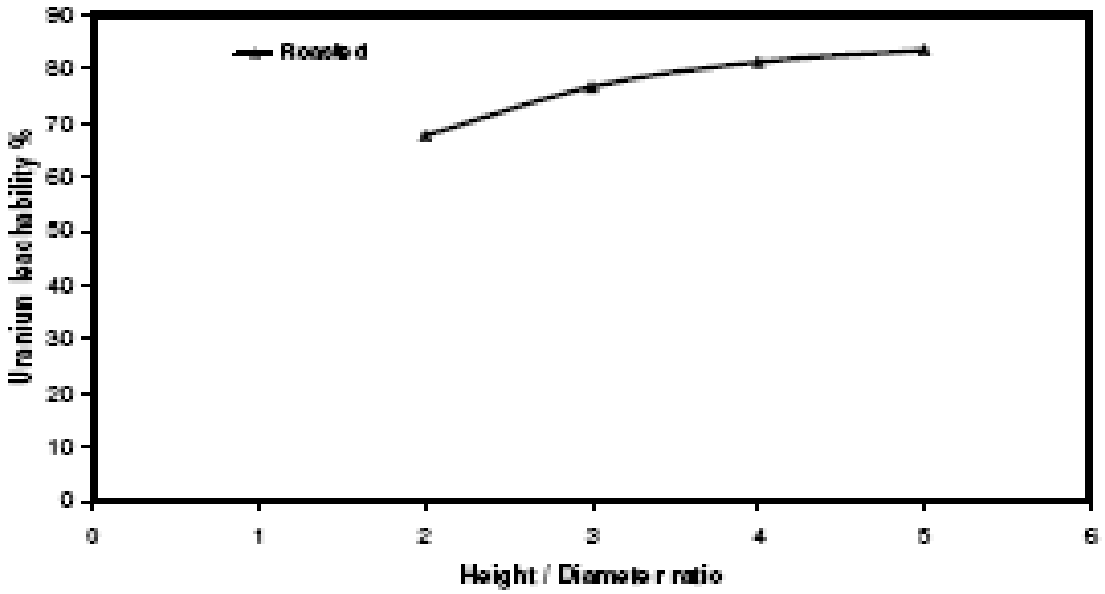

Fig.11. Effect of height/ diameter ratio on the U leachability

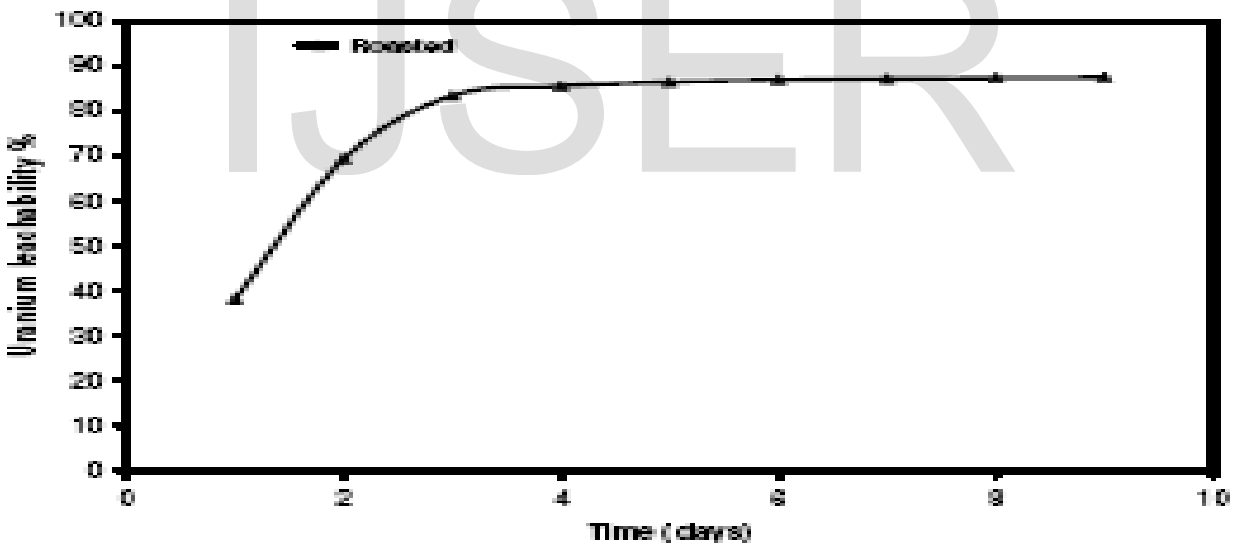

Fig.12. Effect of leaching time on the U leachability

3.2 Recovery of uranium from sulfate leach liquor

3.2.1 Uranium adsorption

After the leaching process has been completed, a resin sample of Amberlite IRA 400 measuring 2.6 ml wet settled resin (w s r) was packed over a glass wool plug in a Pyrex glass column of (0.5 cm radius). The prepared 3000 ml sulfate was then subjected to adsorption by passing through the prepared resin bed allowing contact time of 3.3 min i.e. flow rate of about 0.33ml / min. The concentration of influent solution was 80 ppm. The effluent was collected every 150 ml and analyzed for uranium and the obtained results are shown in Table (1) & Fig (1).

3.2.2 Uranium elution

Under the working conditions, it was found that the obtained uranium saturation level amounted to 79.25 g/l wsr, while represents about 86.14 % of the theoretical capacity of the working resin. Before uranium elution, the resin column was first washed with distilled water to displace of the pregnant solution. This was followed by passing the eluant solution composed of 1N NaCl acidified to 0.1M sulfuric acid using a contact time of 1.6 ml/min. and collecting the obtained eluate every 20 ml for uranium analysis. From the obtained elution results tabulated in table (2) and plotted in fig (2), it was found that the total eluted uranium amount reached has 192.8 mg indicating an elution efficiency of about 93.5 %.

Uranium from the collected eluate was precipitated at a PH of about 5.5 using 40 % NH_4OH solution in the form of ammonium diuranate. However the latter was found to be greatly contaminated with some impurities and for this reason it was exposed to alkali leaching using mixed solution of 15% a Na_2CO_3 and 5% in a S/l ratio of ¼ for 15 minute at 80°C where uranium would be selectively leached Eq.(4). The obtained uranyl concentrate filtrate was adjusted to a pH value of about 5.5 using 1molar H_2SO_4 solution and the resultant precipitated was calcined at 800 °C to U_3O_8 product. The latter was then analyzed by the electron microscope ESEM-EDX analysis and the obtained results indicate that U assay has increased up to about 84% Fig. (3).

UO3+3Na2CO3+H2O ⟶a4UO2 (CO3)3+2NaOH… (4)

TABLE 1
Analytical results of uranium in the leach effluent sulfate liquor of Abu Zarab mineralized ore material using Amberlite IRA 400 resin

Effluent sample No.(150 ml)	Effluent uranium assay, ppm	Uranium adsorption efficiency %	Adsorbed uranium ,mg
1	Nil	100	12
2	Nil	100	12
3	Nil	100	12
4	Nil	100	12
5	Nil	100	12
6	Nil	100	12
7	Nil	100	12
8	Nil	100	12
9	Nil	100	12
10	Nil	100	12
11	Nil	100	12
12	Nil	100	12
13	Nil	100	12
14	Nil	100	12
15	Nil	100	12
16	12	85.00	10.2
17	23	71.25	8.55
18	48	40.0	4.8
19	63	21.25	2.5
20	80	0	0
	Sum		206.05

3.2.3 Uranium precipitation and purification

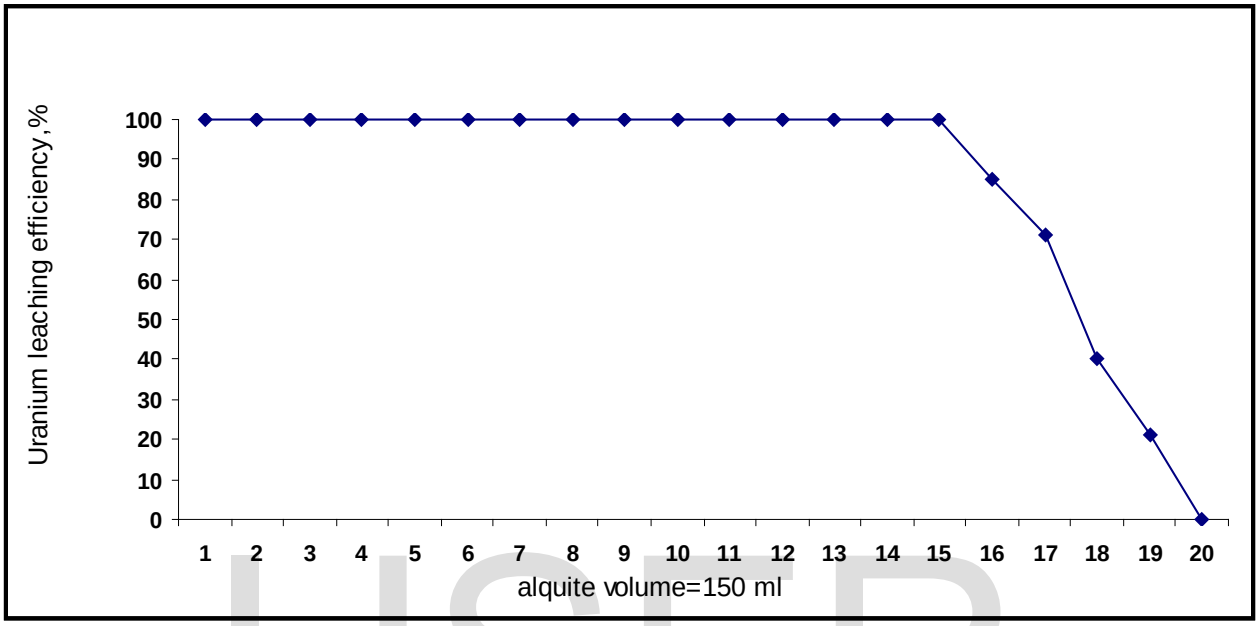

Fig.13. Analytical results of uranium in the leach effluent sulfate liquor of Abu Zarab mineralized ore material using Amberlite IRA 400 resin

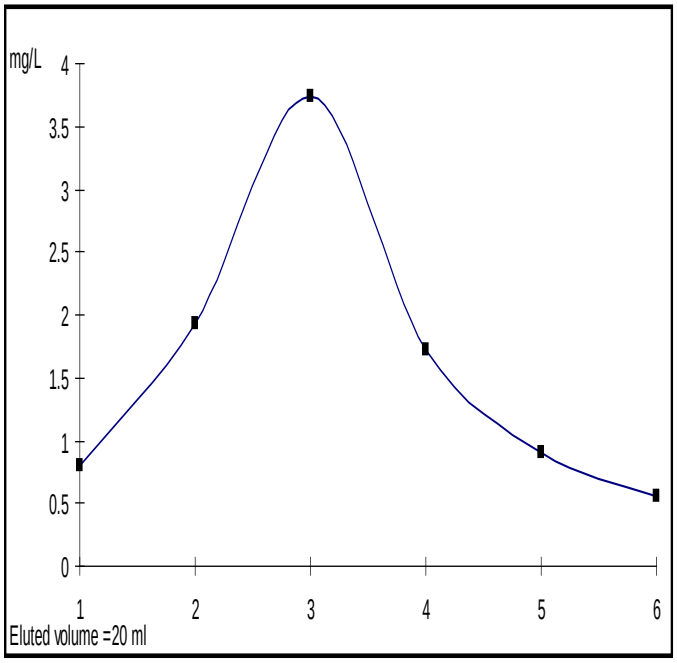

Fig.14. Analytical results of uranium in the eluate samples from the working Amberlite IRA 400 saturated resin column

TABLE 2

Analytical results of uranium in the eluate samples from the working Amberlite IRA 400 saturated resin column

Eluate sample No.(20 ml)	Uranium assay g/l
1	0.795
2	1.935
3	3.74
4	1.72
5	0.9
6	0.55

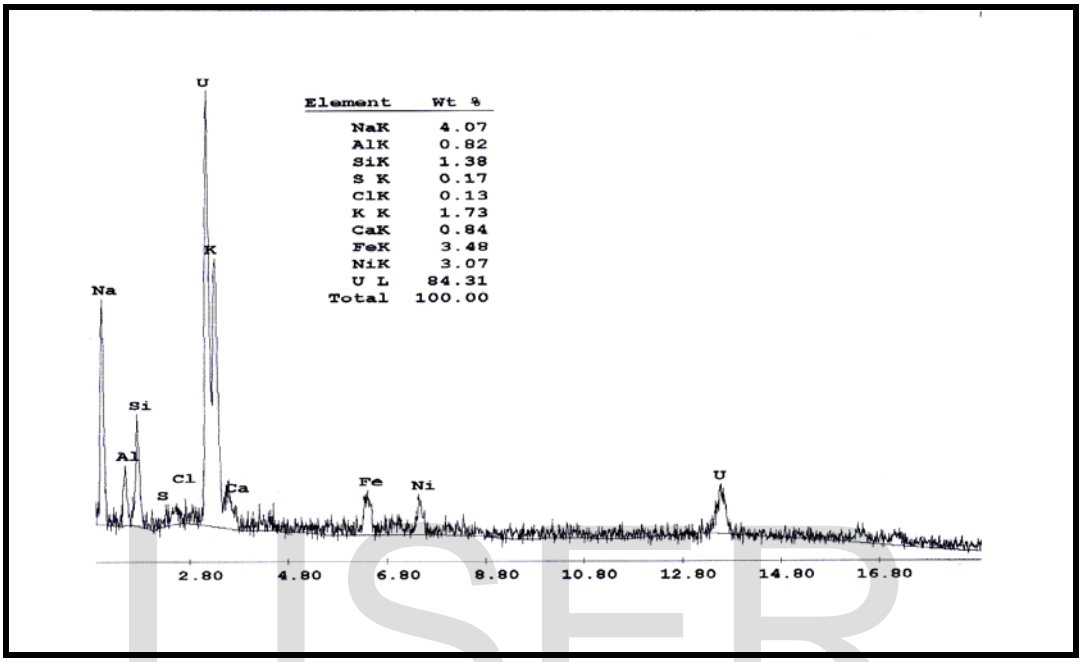

Fig.15. ESM – EDEX of uranium concentrate

CONCLUSION

The results indicate that, the factors controlling the leaching process-using column are best at 12% V/V, Flow rate = 0.25 ml/min, Height / Diameter 8, Particle Size =-2mm. for the small column, while for medium column were Acid conc. =12% V/V., Flow rate = 1.5 ml/min, Particle size =-2(mm). Height / Diameter:5.

From the above studies of the leaching parameters and techniques, it can be concluded that The results indicate that, The prepared 3000 ml sulfate leach liquor was then subjected to adsorption by passing through the prepared resin bed allowing contact time of 3.3 min i.e. flow rate of about 0.33ml / min. The concentration of influent solution was 80 ppm. The effluent was collected every 150 ml and analyzed for uranium. Under the working conditions, it was found that the obtained uranium saturation level amounted to 79.25 g/l wsr, while represents about 86.14 % of the theoretical capacity of the working resin. Before uranium elution, the resin column was first washed with distilled water to displace of the pregnant solution.

This was followed by passing the eluant solution composed of 1N NaCl acidified to 0.1M sulfuric acid using a contact time of 1.6 /min. and collecting the obtained eluate every 20 ml for uranium analysis. it was found that the total eluted uranium amount reached has192.8 mg indicating an elution efficiency of about 93.5 %.

Uranium from the collected eluate was precipitated at a PH of about 5.5 using 40% NH_4OH solution in the form of ammonium diuranate. However the latter was found to be greatly contaminated with some impurities and for this reason it was exposed to alkali leaching using mixed solution of 15% Na_2CO_3 and 5% in a S/l ratio of ¼ for 15 minute at 80C° where uranium would be selectively leached. The obtained uranyl concentrate filtrate was adjusted to a pH value of about 5.5 using 1molar H_2SO_4 solution and the resultant precipitated was calcined at 800 C° to U_3O_8 product. The latter was then analyzed by the electron microscope ESEM-EDX analysis and the obtained results indicate that U assay has increased up to about 84%.

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