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Research Paper

"Potential uranium recovery by percolation leaching from El Missikat mineralized silica vein, Eastern Desert, Egypt"

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ABSTRACT

El-Missikat area is one of the most important uranium occurrences in the Eastern Desert of Egypt. Uranium occurrences are indeed strongly associated with silicified and fracture shear zones. It is characterized by the presence of galena, pyrite, uranophane, kasolite, wulvenite and colombite. Agitation and column percolation leaching techniques applied upon the uranium rich mineralization (El Missikat) showed that these techniques succeeded in providing considerable results. It can be concluded that El Missikat uranium mineralized ore is feasible for leaching (column percolation) as about 140 kg ore sample assayed 1850 mgU//kg, after 75% leaching efficiency using 24 g/kg acid consumption in 40 days of work. Ion exchange technique was used to recover uranium from pregnant uranium solution, which gave 83% and 96% loading and elution efficiency, respectively.

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Key words: El Missikat, Uranium, Agitation, Percolation, Recovery.

INTRODUCTION

El-Missikat area represents one of the most important uranium occurrences in the Eastern Desert of Egypt. It lies at a distance of about three kilometers to the south of landmark Km.85 on the Qena-Safaga asphaltic road. El-Missikat pluton is an oval shaped pluton (covering an area of about 75 km^2), with its maximum length of 12.5 km trending NW (Abu Deif and El-Tahir, 2008; Omar, 2010; Arafa and Omar, 2011; Omar et al., 2012).

From the field observation, El-missikat area is occupied by older granitoid, younger granites, and dykes and veins. The younger granite in El-Missikat area is the most important rock hosting the U-mineralized silica veins (Figure 1) (Ghoneim and Abdel Gawad, 2018).

Uranium occurrences in El Missikat younger granite are indeed strongly associated with silicified and fracture shear zones which extend in the Northern peripheral parts. This shear zone is thus filled with siliceous material which belongs to three generations of silica veins (white, black and red) in El Missikat area (Abu Dief, 1992).

On the other hands, all mineralizations in El Missikat area are related to the main shear zone (Figure 2).This shear zone has three types of silica veins. The most important one is the jasper vein (red silica) which has all the mineralization in El Missikat area as compared with the other two types (white and black silica).

According to authors (Bakhit, 1978; Bakhit et al., 1985), the black silica veins are occupied by secondary uranium minerals besides fluorite, galena, pyrite, chalcopyrite and molybdenite minerals as well as jasper. Silica veins were subjected to an upgrading procedure followed by analysis of the separated fractions and revealed that the separated accessory mineral assemblage can be classified into four heavy mineral groups as follows (Mohamed, 1995):

- Radioactive minerals: uranophane, uraninite and pitchblende
- Sulphide minerals: galena and pyrite
- Transparent minerals: fluorite, zircon and monazite
- Iron and titanium oxides: magnetite, hematite, rutile and sphene

Studies on uraniferous iron grains were performed at Gabal

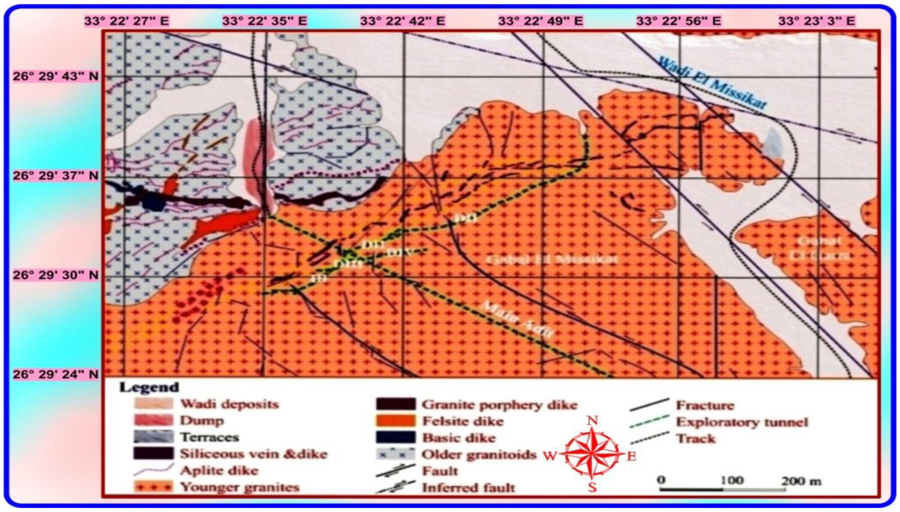

Figure 1: Geological map of El-Missikat area.

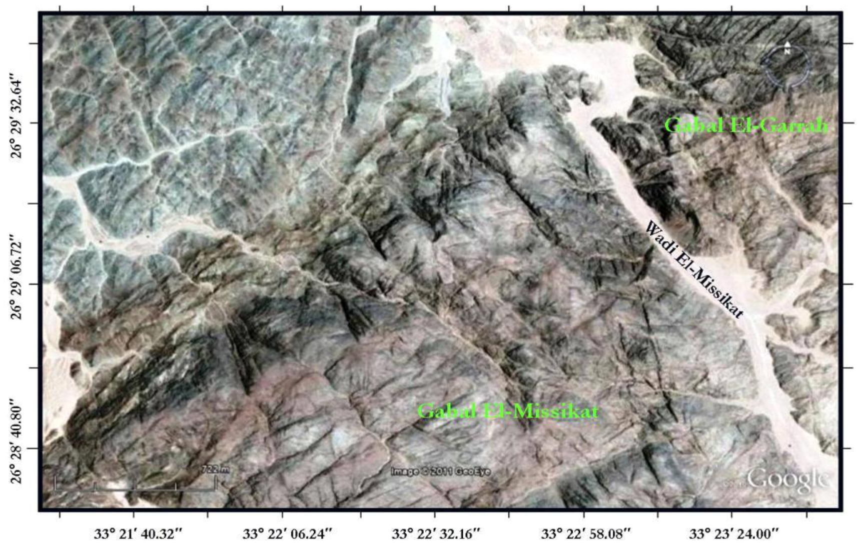

Figure 2: Shear zone with Jasperoid vein in El-Missikat area.

Gattar, El Missikat and El Erediya granites in Eastern Desert of Egypt. The results showed that their weight abundances are up to 17.50, 18.00 and 26.00% of the total accessory heavy minerals of the uranium-mineralized samples of the three plutons, respectively (Raslan, 2009).

Two alteration facies namely propylitic and advanced argillic zones which reflect the presence of hydrothermal deposits were determined. The propylitic facies contains quartz, fluorite, and Fe-oxides. It is characterized by the presence of galena, pyrite, uranophane, kasolite, wulvenite and colombite, whereas the advanced argillic facies is characterized by the presence of galena, pyrite, gold, barite, celestine, magnetite, hematite, and goethite (El-Sherif, 2010).

Acid leaching has been applied mainly in large operations (Bhargava et al., 2015). Acid leaching is highly selective, and uranium recovery range from 70 to 90% is possible. The leaching behavior of uranium from a low-grade unconformity-related uranium ore assaying 0.06% occurring in the northwestern part of the Kadapa basin in southern India was studied (Sreenivas and Padmanabhan, 2012).

In this regard, it is interesting to mention that the results of acid agitation leaching efficiency attained about 91% after 8 h of agitation from El Missikat mineralization (Mohamed, 1995). Recovery of uranium and REE from El Missikat pluton and the results indicated that uranium leaching efficiency 95% (Mousa et al., 2014). Under the optimum agitation leaching conditions, uranium leaching efficiency attained about 95% from El Missikat mineralized shear zone (Amin et al., 2017). The recovery of uranium from El-Missikat by anion exchange resin (Lewatit Mono Plus M500) was investigated and extraction condition contact time, temperature, pregnant feed pH, resin/ leach liquor ratio and agitation rate were studied (Gawad, 2015). Uranium recovery was performed using ion exchange resin technique using D263B anion exchange type. After adsorption and elution process, uranium was precipitated using sodium hydroxide solution at pH 7.5 where 0.27 gm of sodium di-uranate (Yellow Cake) was obtained (Amin et al., 2017).

Percolation leaching of uranium has been studied on Eastern Desert, Egypt mineralization, from these studies, the authors performed a column percolation leaching study to recover uranium from El-Sela mineralization using sulfuric acid in which, uranium leaching efficiency attained about 81.03% (Nagar et al., 2016). Uranium percolation leaching from both Gattar-II and Gattar-V mineralized samples was studied. From the results obtained, they notice that the particle size has a significant impact on the leaching efficiency. Regarding GII, the leaching efficiency (based on the solution) of –10 mm sample is 76.9%, but the leaching efficiency of –40 mm sample is 47.4**%** (Mahmoud et al., 2001). Percolation Leaching of uranium from Abu-Rusheid Mylonite Eastern Desert, Egypt was studied. The results indicated that the percolation leaching efficiency attained about 65% (Mahmoud et al., 2017).

Agitation leaching always leads to better solubilisation efficiencies for metals, but requires a large investment in reactor maintenance and energy for the implementation of agitation compared to percolation leaching (Ghorbani et al., 2016; Oxley et al., 2016; Staden et al., 2017). As far as percolation leaching is concerned, it has the advantages of having a low investment cost required for the implementation of the leaching operations and a relatively low operating cost. In addition, percolation leaching is typically conducted at a coarser size fraction than agitated tank leaching, thus reducing the power requirement and reagent consumption.

This study aims to evaluate the leachability and recovery of uranium from El missikat mineralized silica vein by applying agitation and column percolation leaching techniques and obtain its end product precipitate, sodium di-uranate (yellow cake). Relevant technical and economical parameters are gotten through the laboratory study. These parameters can be used to guide the design of El Missikat experimental yellow cake production unit and its practical operation.

EXPERIMENTAL

Characteristics of the study mineralized sample

The representative composite sample used in this study was obtained from Gabal El Missikat mineralized silica veins by Nuclear Material Authority (NMA), Egypt having the chemical composition of 0.0130% ΣREE, 0.185% U, 3.4%Al_2O_3, 89.97% SiO_2, 0.55% CaO, 2.95% Fe_2O_3 and 0.11% TiO_2. While some of the trace elements of the same vein sample after (Amin et al., 2017) revealed the presence of U (3030 ppm), Zr (1070 ppm), V (35 ppm), Co (10 ppm) and ΣREE (100 ppm).

Uranium leaching procedures

Granulometric analysis

This type of analysis was performed upon 1.0 Kg of the study sample where it was subjected to crushing and sieving along with a range of sizes from - 1.25 to - 0.25 mm. After that, all fractions of grain size were analyzed for uranium determination.

Agitation leaching

To study and determine the optimizing factors affecting the acid agitation leaching of uranium, different series of agitation leaching experiments were performed after selecting the appropriate. Certain weight of the uranium mineralization, ground to appropriate size, was mixed well with a suitable volume of different sulfuric acid concentrations. The studied factors are, grain size, uranium distribution, agitation time, and sulphuric acid concentration.

Percolation (column) leaching experiment

Recommended columns materials are PVC pipe with 12-inch inside diameter. The bottom of the column is equipped with a PVC base plate containing a piece of screen to prevent loss of solid sample from the column during leaching. Optionally, 2 to 4 mm of clean silica sand and gravel can be loaded into the base of the columns before sample addition. The columns charged with the mineralized samples in which average uranium content is 0.185%. The base plate is equipped with a valve to control the discharge of column effluent. A funnel and suitable 30-L receptacle were placed under the column. A 20-L feed container was placed ± 50 cm above the column. Sulphuric acid solution range from 20-70 g/L was used as a lixiviant in the experiment. Lixiviant is stored in the overhead tank, adjust the adjustable valve to control the flowrate of lixiviant, allow the lixiviant down flow through mineralized bed by a suitable flow rate of ($10L/m^2/h$), leaching liquor is collected in the solution collection basin. Take sample from leaching liquor, and analyze its uranium content, pH (free acidity) and other impurities to decide whether the effluent is recycled as intermediate leaching solutions (ILS) or

Table 1: Granulometric analysis and uranium distribution in the sample.

Size (mm)	Fraction weight, (g.)	Size distribution wt.%	Uranium distribution (%)
+1.25	140	14.0	41
-1.25 to +0.5	437	43.7	33
-0.5 to +0.25	180	18.0	9.4
-0.25	245	24.5	17
Total	1002	100.2	100.4

moved to recovery circles pregnant leaching solution (PLS). The end point of leaching mainly depends on economic indicator, if the value of extracted uranium is less than its operation cost, leaching can be stopped. Of course, if technical indicator such as leaching efficiency is uniquely concerned, leaching could continue. Reaction with hexavalent uranium, which dissolves as UO_2^{2+}cation, produces uranyl sulfate and complex uranyl sulfate anions as follows:

$$UO_2^{2+}+SO_4^{2-}=UO_2SO_4$$
$$UO_2SO_4+SO_4^{2-}=[UO_2(SO_4)_2]^{2-}$$
$$[UO_2(SO_4)_2]^{2-}+SO_4^{2-}=[UO_2(SO_4)_3]^{4-}$$

Ion exchange recovery and precipitation

Recovery of uranium was done using a PVC column of 1.0 m height and 5 cm in diameter packed with (D263B strong base anionic resin). The obtained leach liquor was poured over the packed resin after adjusting its pH at 1.8 and the PLS flow rate at 2.0 L/h. Loading of uranium upon the resin was followed through uranium analysis in the effluent samples that have been collected every 10 L. The elution process was carried out on the same adsorption column using 1.0 M NaCl acidified by 0.1M H_2SO_4. The precipitation process was performed using 20% NaOH till pH 6.5-7.5. A bright yellow precipitate (yellow cake) of sodium di-uranate ($Na_2U_2O_7$) was obtained.

Uranium control analysis

Uranium was analyzed in the corresponding low concentration of aqueous phases using Arsenazo III reagent under different conditions (Marczenko, 1976). In high concentration (≥ 10ppm) uranium was determined in the pregnant solution and the crude uranium concentrate using the oxidimetric titration procedure with a standard solution of NH_4VO_3 till the appearance of a purplish red color representing the end point (Davies and Gray, 1964).

RESULTS AND DISCUSSION

Granulometric analysis

Uranium distribution was investigated in the study sample for each grain size faction (**Table 1**) for determining the most suitable size to achieve the aim of the study.

Relevant factors of uranium acid leaching from El Missikat uranium mineralization

Effect H_2SO_4 acid concentration

Agitation Leaching experiments were carried out using a sulfuric acid concentration that ranged from 20 to 100 g/L. The applied conditions were fixed at a weighted sample of 20 g, assaying 1850 ppm U,- 0.25 mm grain size and solid/liquid ratio of 1/2 for a contact time of 240 min. From the obtained data shown in **Figure 3**, it is clear that increasing acid concentration from 20 to 40 g/L cases increased uranium leaching percentage from 81 to 83%, while at 60 g/l U leaching efficiency gave 83% indicating that a slight increase in acid concentration had no effect while at 100 g/L leaching percent reached 86%. From these data, it can be concluded that an economically 40 g/L acid concentration was the best one used.

Effect of grain size

The effect of the grain size on the leachability of uranium was achieved by studying the grain size of mineralized samples that ranged from + 1.25 to – 0.25 mm. Other leaching conditions were fixed at 40 g/L acid concentration, 1/2 solid/liquid ratio for 240 min agitation time at room temperature. The results obtained are shown in **Table 2**, and as shown uranium leaching percent has increased from 48 to 85% with decreasing crashed size from +1.25 to -0.25 mm. This can be explained by the fact that by decreasing the grain size, the surface area exposed to the reaction increases which enhances the mass transfer process of leaching. Another reason is that the solid particles were activated during grinding.

Effect of agitation time

The leaching experiments were performed using agitation time of 30 up to 240 min, while the ether conditions were fixed at optimum values of acid concentration, L/S ratio, room temperature, and grain size of -0.25 mm. The obtained results (**Figure 4**) showed that the uranium

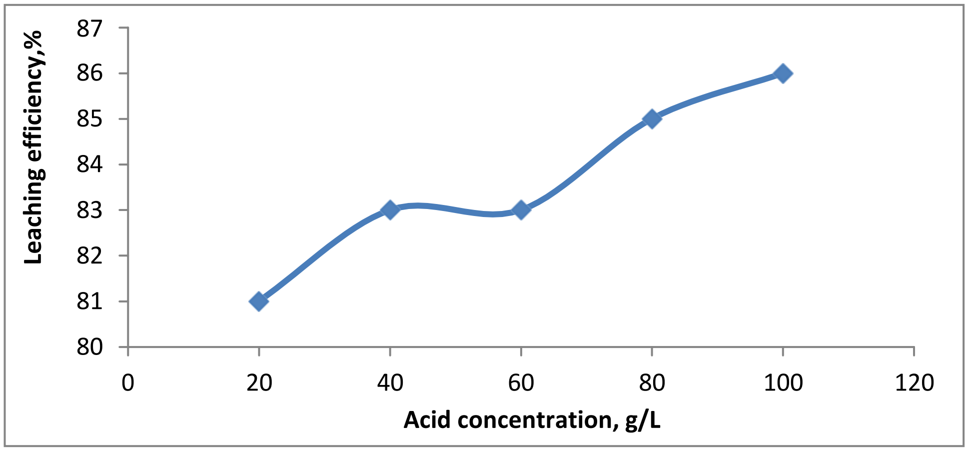

Figure 3: Effect of sulfuric acid concentration upon uranium leaching efficiency from El Missikat mineralized silica vein.

Table 2: Effect of grain size upon uranium leaching efficiency upon El Missikat mineralized silica veins.

Grain size, (mm)	U leaching efficiency, %
+ 1.25	48
-1.25 +0.5	58
-0.5 +0.25	64
- 0.25	85

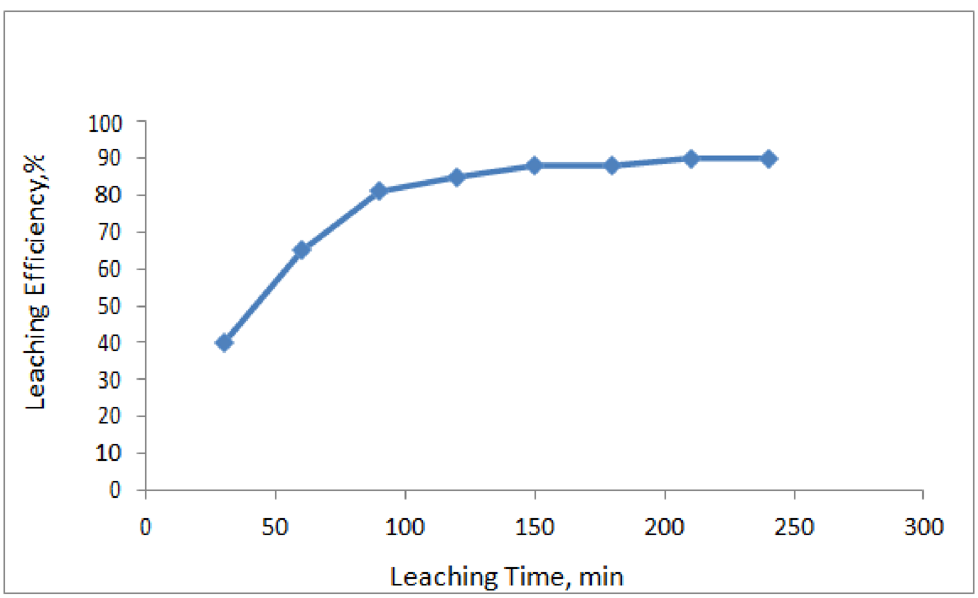

Figure 4: Effect of contact time upon uranium leaching efficiency from El Missikat mineralized silica vein.

dissolution efficiency was increased from 80 to 88% with increasing agitation time of 30 to 240 min and therefore 150 min leaching time is considered quite enough as the required contact time.

Choice of the optimum leaching conditions:

It is very important to mention here that the agitation leaching study gives the needed background before starting

Table 3: Grain size distribution of the column packed sample.

Particle size, mm	Weight, kg	Weight %
-10+5	69.0	49.2
-5+2	45	32.2
-2	26	18.5

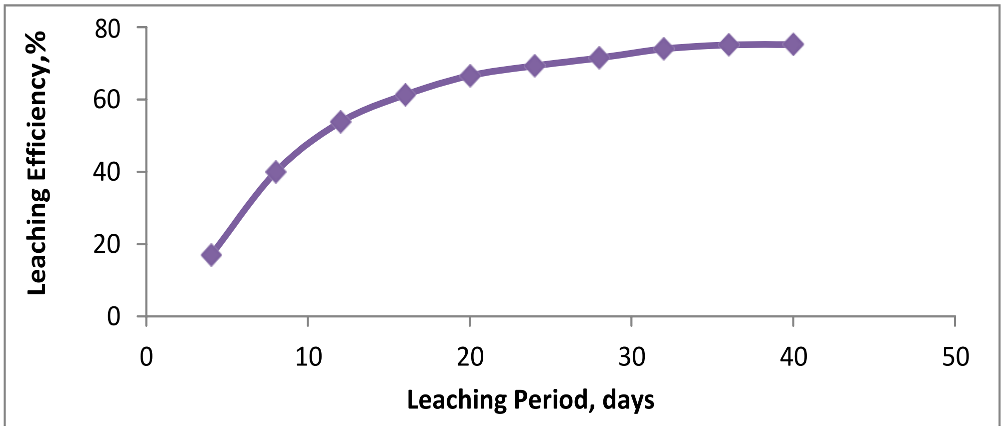

Figure 5: Percolation leaching efficiency from El Missikat mineralization.

Table 4: Applied conditions in the percolation column leaching experiments as a guide for column leaching of a future pilot plant scale.

Ore weight, (Kg)	140
Ore size, mm	≤10
Average uranium, mg/Kg	1850
Total uranium, g	259
Leaching liquor acidity	20 – 70 g/l H_2SO_4
Flow rate, L/m²/h	10
Leaching time, day	40
Consumed acid, g	(24 g/kg ore) 2.4% (w/w)
Total pregnant liquor, L	130
Total leached uranium, g	194.5
Leaching efficiency, %	75.0

percolation column leaching. It would seem economic to select the following conditions for leaching uranium from El Missikat mineralized silica vein: 40 g/L sulfuric acid concentration, 150 min leaching time, -0.25 mm grain size and reaction temperature of 25°C. Performing a leaching experiment under the selected optimum condition gave about 85% uranium leaching efficiency.

Column percolation leaching

In this type of leaching, the column was packed with the study sample of grain size ≤10mm distributed as shown in (**Table 3**) having total uranium amount of about 259 g. The leaching period was extended for 40 days at a lixiviant flow rate of 10 L/m²/h.

Figure 5 shows the results of the continuous analysis of the uranium resulting from the percolation leaching process during the columns. While the applied conditions for practical application as a guide for heap leaching can be summarized in **Table 4**.

From the obtained results in **Figure 5**, in the early stages of percolation leaching, the recovery of uranium increases gradually with increase in the acid consumption until a plateau is reached, beyond which further addition of the acid has little effect on the recoveries. The total acid consumption averaged 24 kg per tonne of ore (kg/t). The uranium leaching efficiency attained about 75.0%, when

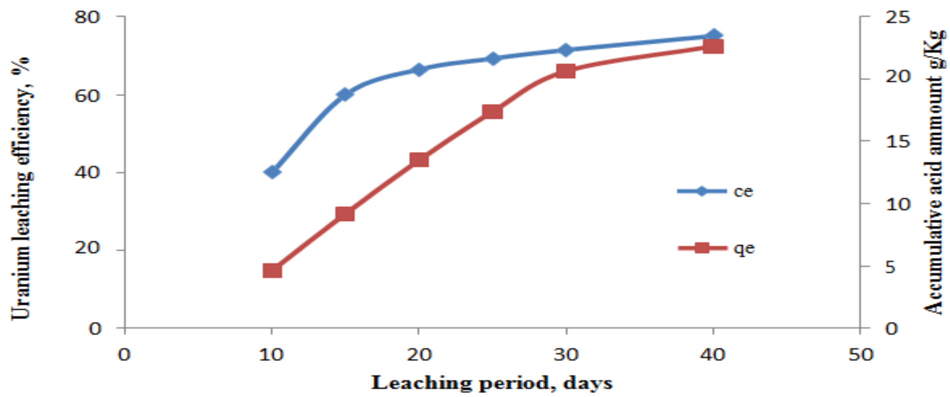

Figure 6: Relation between the leaching efficiency and acid consuming.

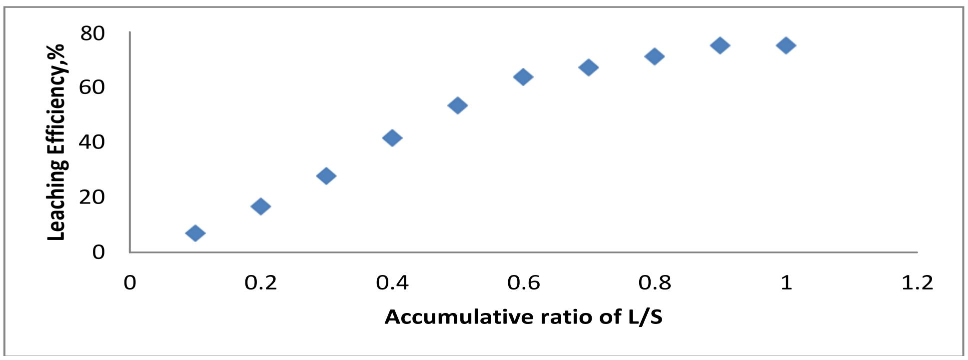

Figure 7: Relation between the leaching efficiency and L/S ratio of Column leaching.

a Lixiviant solution was passed at a flow of 10 L/m^2/h. The effluent leach liquors of the first three days were recycled to upgrade the uranium assay within the leaching solution. It's generally concluded that El Missikat mineralized sample is feasible to uranium leaching using this method and its leaching efficiency is often increased after a further period. Grade of the tested sample decreases gradually with leaching program, and uranium concentration of leaching liquor decreases accordingly. The endpoint of leaching mainly depends on an economic indicator, if the value of extracted uranium may be a smaller amount than its operation cost, leaching is often stopped. Of course, if technical indicator like leaching efficiency is uniquely concerned, leaching could continue.

Relation between the leaching efficiency and acid consumption

Acid consumption in uranium percolation leaching from El-Missikat mineralization is related to gunge mineralogy more than other factors. Silicate and iron oxide gangue minerals consume sulfuric acid during initial reactions. Silicate minerals consume acid by breakdown to a wide range of soluble solution products. K-feldspar, Na-feldspar, Ca-plagioclase and Biotite breakdown to Ortho-silicic acid, H_4SiO_4, and various metal cations such as Na^+, K^+, Ca^{2+}, Fe^{2+}, and Fe^{3+}. The results of the consumed acid in the column leaching test are presented in Figure 6. After 20 days, acid consumed was increased in column leaching of El-missikate mineralization to 75% of the total consumed acid in operation. While the remaining 25% were consumed in the last 20 days to Silicate and iron oxide gangue minerals consuming sulfuric acid during initial reactions.

Relation between the leaching efficiency and L/S ratio on column leaching

During leaching, sprinkling intensity refers to the volume of lixiviant per square meter used for leaching in one cycle. In this experiment, the sprinkling intensity is expressed in the ratio of liquid to solid. The column with low sprinkling intensity needs more leaching time, at the same time it leads to a high maximum uranium concentration and average uranium concentration of leaching liquor. From Figure 7, concerning the analysis results of the pregnant leaching solution at different accumulative L/S ratio, the

Table 5: Material balance of uranium during percolation leaching.

In put Uranium, g	Out put U, g	U in Residue, g	Total account U,g	Unaccount U,g	Unaccount U,%
259	194.5	0.28×140 =39.2	194.5+39.2=233.7	259- 233.7=25.3	9.7

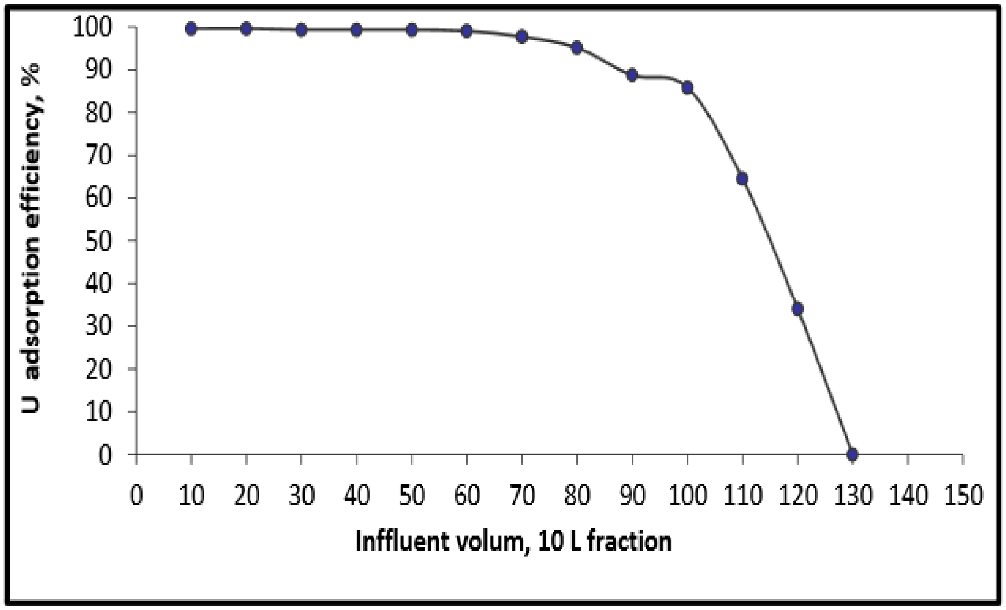

Figure 8: Adsorption curve of uranium loaded upon D263B resin.

leaching efficiency becomes stable at 75%, at the same time the liquid to slid ratio reached 0.9.

Material balance of uranium during percolation leaching

The material balance of uranium mineralization leaching was found to coincide within ±10% of uranium content in the mineralized sample as shown in **(Table 5)**. This is due to the heterogeneity of the solid samples, the lack of good manual mixing during the experiment and the inefficiency of washing the fine particles. Also the volumetric analysis method may not be accurate.

Uranium recovery using Ion exchange resin (D263B)

Uranium adsorption

El Missikat pregnant uranium solution (130 L) was adsorbed by strong anion exchange resin technique (D263B), to concentrate and purify uranium. Uranium loading results are shown in **Figure 8**. From the obtained results, the ratio of the breakthrough volume (BV=70) to the saturation volume (SV=130 L) is an important characteristic, which reflects the loading efficiency of the resin. The value (0.54) for BV/SV is considered suitable for uranium recovery operation and the (D263B), resin presents good suitability of adsorption, and has desirable loading characteristics for the feed solution. The obtained adsorption efficiency was 83.75% for El Missikat pregnant solution.

Uranium elution

The uranium elution process was achieved using 1.0 M NaCl acidified by 0.1 M H_2SO_4. This occurred after the uranium saturation of the working resin column was achieved. In this experiment, the elution efficiency reached about 96% at a down flow rate of 250 ml/h. The obtained results are shown in **Figure 9**.

Uranium precipitation

The obtained uranium eluate solution of 17 L assaying 9.3g/L was subjected to precipitation using 20% sodium hydroxide solution at pH 7. After filtration and drying, a uranium precipitate (Yellow Cake) weighing 800 g was obtained. Finally, a proposed flow sheet for the treatment

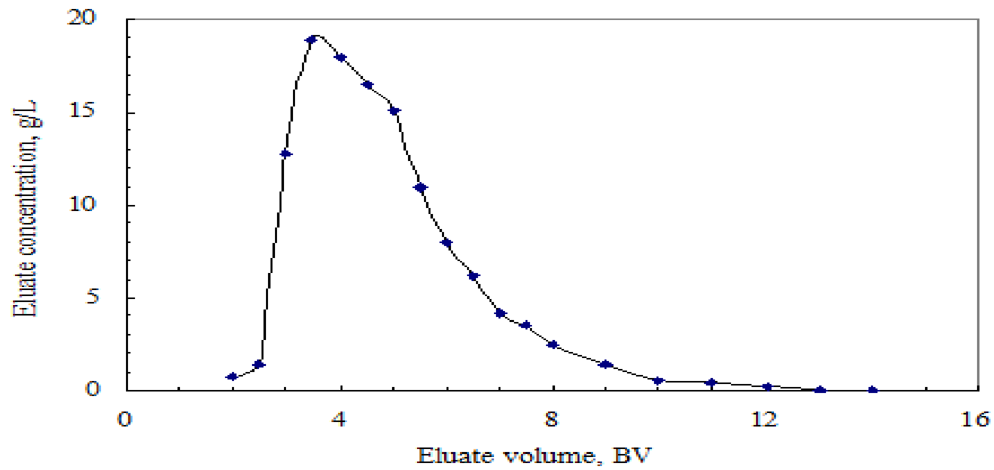

Figure 9: Elution of uranium loaded upon D263B resin.

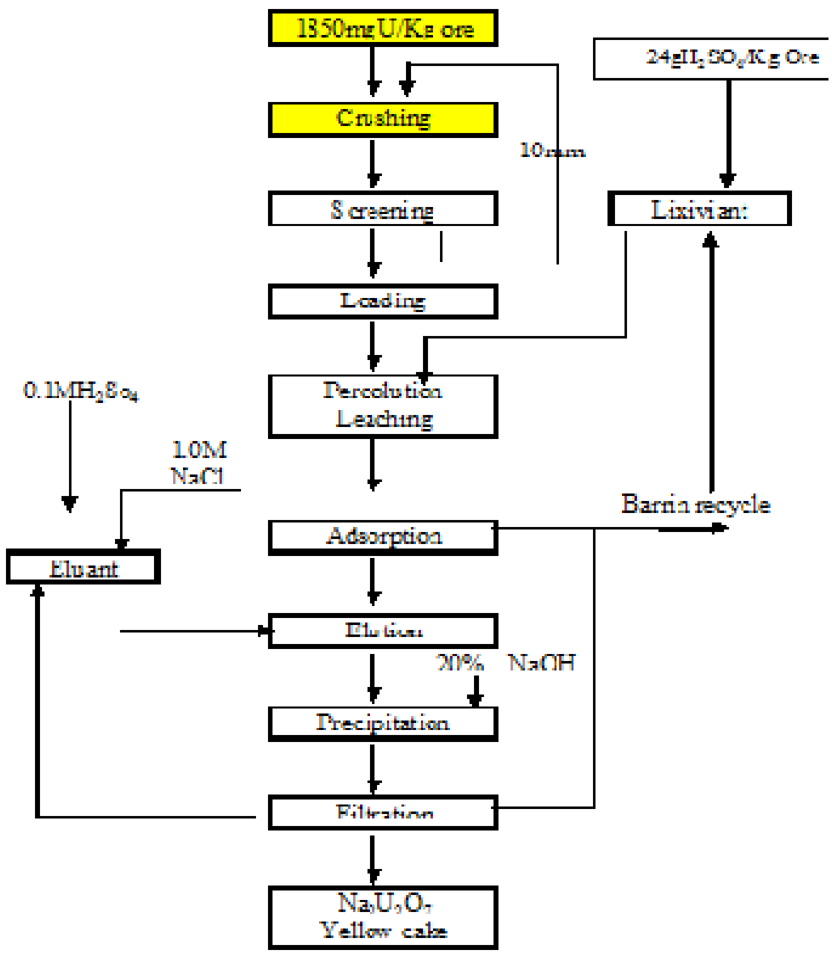

Figure 10: Proposed flow sheet for U-recovery from U-mineralization of El-Missikat area.

of uranium mineralization is shown in (**Figure 10**), starting with leaching using 24g H_2SO_4 /Kg ore and ended with uranium precipitation as sodium di-uranate ($Na_2U_2O_7$).

Conclusion

Agitation and percolation leaching techniques applied upon the uranium rich mineralization (El Missikat) showed that these technique succeeded in giving outstanding and promising results. It is concluded that El Missikat uranium mineralized ore is feasible to leaching (through percolation) as about 140 kg ore sample assaying 1850 mgU/Kg after 75% leaching efficiency, 83% loading efficiency and 96% elution efficiency gave a product of yellow cake. Relevant technical and economical parameters are gotten through the laboratory study. These parameters can be used to guide the design of experimental yellow cake production unit and its practical operation.

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