

Leachability of uranium from low grade uraniferous Granites, Eastern Desert, Egypt

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Abstract- A highly altered uraniferous granite prospect namely Gattar II in the north eastern Desert, Egypt has been studied for the recovery of uranium by the agitation leaching technique. The relevant factors of acidic leaching of a technological sample of this rock material assaying uranium of 150 ppm were studied. Under the optimum conditions, it was possible to realize a dissolution efficiency of about 94 % for uranium. The kinetics of reaction was found fit to the diffusion reaction model. The activation energy was calculated 11.914 KJ/mol.

Key Words: Kinetics, Acid leaching, Uraniferous granites, Uranium, Gabal gattar.

1. INTRODUCTION

Uranium is the basic element of the peaceful nuclear power industry. It is a common element in nature that has for centuries been used as a coloring agent in decorative glass and ceramics. The initiation of the Egyptian nuclear program to produce electrical energy requires big reserve of radioactive raw materials, especially uranium minerals. The younger Granites of G.Gattar at the northern Eastern Desert was found to be favorable for uranium mineralizations and notably show higher level of radioactivity [1-2].

Extraction of uranium is indeed a hydrometallurgical operation in which uranium is directly leached first by suitable acid or alkaline reagents [5-6]. In general, acid leaching is more widely used than the alkaline one because of relatively coarse preparatory grinding, comparatively mild reagent concentration, shorter leaching times, applied under an ambient temperature and atmospheric pressure, the highest extraction efficiency, convenient for subsequent recovery processes [7].

Sulphuric acid is the most common acidic reagent used in uranium leaching because of its availability and low cost. In addition, sulphuric acid may be generated autogenously by treating uranium ores that contain sulphide minerals by air or oxygen under pressure or by bacterial action [4].

Several acidic leaching techniques have been applied to achieve better efficiencies of uranium dissolution on industrial or at least on pilot scales. Agitation leaching at atmospheric pressure is most commonly used and recommended over a conventional leach because of high leachability, better reaction rate, relatively shorter leaching time and can be used for treating low grade of uranium.

Some studies have also succeeded to leach U and associated elements from Gabal Gattar. The leaching of uranium and molybdenum from G-Gattar mineralization using acid and alkaline agitation leaching was studied. Acid leaching has indicated that complete leaching of U/MO by using 50g/l H₂SO₄ and solid/liquid ratio of 1/2 at room temperature for an agitation time of 12 hr. About 95.1% of uranium leaching efficiency was obtained at 60 °C for 8 hr by using 50g/l Na₂CO₃ or NaHCO₃ in case of alkaline leaching [3].

Uranium leaching from both G-II and G-V mineralized samples was studied. The latter was leached with 99% leaching efficiency using H₂SO₄ at room temperature during 24 hr at 0.2 liquid/ solid ratios by using only 30 and 40 kg/ t sulfuric acid respectively [8].

Studied the leaching characteristics of uranium from Wadi Belih Hammamat sediments by agitation leaching using acid, alkaline and salt as lixivants. They found that the leachability of uranium was 95%, 91% and 88% respectively [4].

In this paper, the sulfuric acid agitation leaching of G. Gattar II low grade uraniferous granite as well as the parameters affecting on the leachability which including acid concentration, particle size, solid liquid ratio, agitation speed, temperature and time of leaching. The kinetics and mechanism of dissolution process were also studied.

2. MATERIAL AND METHODS

2.1 Raw material

The uraniferous granite sample weighing about 200 kg (-25mm size) was obtained from Gabal Gattar GII in the north eastern Desert of Egypt. This sample was mixed thoroughly then crushed to (-4mm). A representative

sample weighing about 200 gm. This representative sample was used for performing complete physicochemical analysis using X-Ray Fluorescence Spectrometry (XRF) and mineralogical analysis using X-Ray Diffraction (XRD) technique. A nest of sieves namely; 4, 2, 1.4, 1, 0.71, 0.5 and 0.355, 0.250 and 0.18, 0.1 and 0.063 mm. was used to fractionate the sample. A laboratory grade of sulphuric acid (specific gravity and concentration of 1.84 g/ml and 98%, respectively is prepared and used as leaching agent in different stages of the experimental work.

2.2 Methods

2.2.1 Agitation leaching

In the leaching method, each leaching experiment was performed by agitating with magnetic stirrer of a weighed amount of the ground sample 50 gm with certain volume of the acid of a specific concentration at a certain solid/liquid ratio for certain period of time at the required temperature. The obtained slurry was cooled, filtered, and washed with slightly warm distilled water and made up to volume. The obtained leach liquors were then analyzed for the metal values to calculate their dissolution efficiency percent.

The main relevant acid leaching conditions for uranium recovery include acid concentration, particle size, solid/liquid ratio, agitation speed, temperature, and leaching time.

2.2.2 Methods of analysis

Control analysis of uranium in the different aqueous stream solutions has been determined by the oxidimetric titration after its reduction using a standard solution of ammonium metavanadate [13]. This has been possible after a prior uranium reduction step using ammonium ferrous sulfate. In this procedure, di-phenyl sulphonate has been used as indicator where upon its color would change to a slightly violet red color. The percent recovery of uranium was then calculated using the formula $(R = Cc/Ff \times 100)$ where R is the percent recovery of uranium, C is the weight of uranium in the pregnant solution, c is the assay of uranium in the pregnant solution, F is the weight of uranium in the ore sample and f is the assay of uranium in the ore sample.

Each size fraction was then chemically analyzed by oxidimetric titration method to determine its uranium content. On the other hand, to determine uranium content accurately on the head sample and pregnant solution the Inductive Coupled Plasma Mass Spectrometer (ICP-MS) was also used.

3. RESULTS AND DISCUSSION

3.1 Physicochemical analyses of Gabal Gattar uraniferous granite

The mineralogical analysis of the concentrate sample using XRD is shown in Fig.1. The results obtained from this figure revealed that the sample is composed mainly of the following minerals as shown in Table 1.

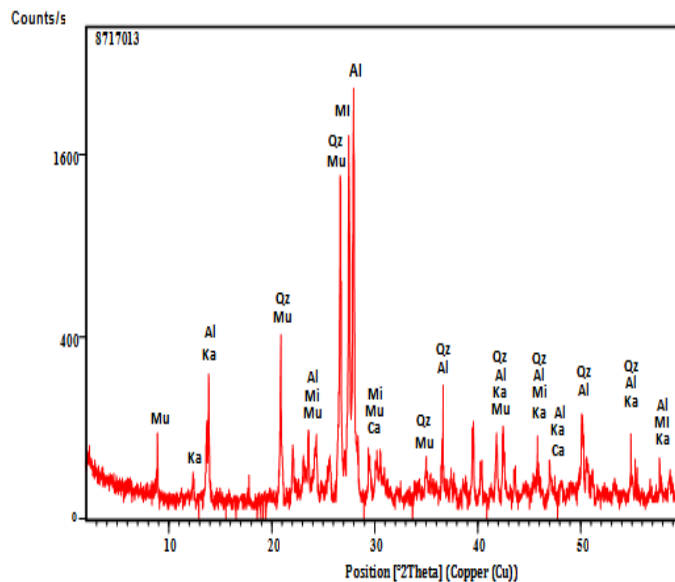


Fig-1: The XRD of head sample

Table -1: Mineralogical composition of Gattar uraniferous altered granite ore.

Major constituent	Trace constituent
Quartz, Microcline, Albite	Kaolinite, Muscovite, Calcite

To obtain pure mineral grains hand picking was carried out by hand picking using binocular Leica - microscope. Scanning electron microscope (SEM-EDX) analyses was done to identify and describe the separated grains. The results indicate that the uranophane is the main radioactive mineral responsible for the radioactivity, in which their grains are characterized by their softness to crush by pressing with picking needle. Also, they are present as massive granular particles or acicular or hair-like crystals, distinguished by their bright colors (Canary to lemon yellow) as shown in Fig.2.A. SEM-EDX analyses shows that the major elements in uranophane are U, Si, and Ca and have little traces of Al, and k as shown in Fig.3. Some grains of iron oxides are usually associated with uranium mineralization which found as tiny inclusions of uranophane on its surface Fig.2.B. The obtained data indicated that the major elements were U, Fe, Si and Ca as shown in Fig.4. These results are consistent with the previous observations of [11] who demonstrated that iron oxide is always associated with the uranium mineralization.

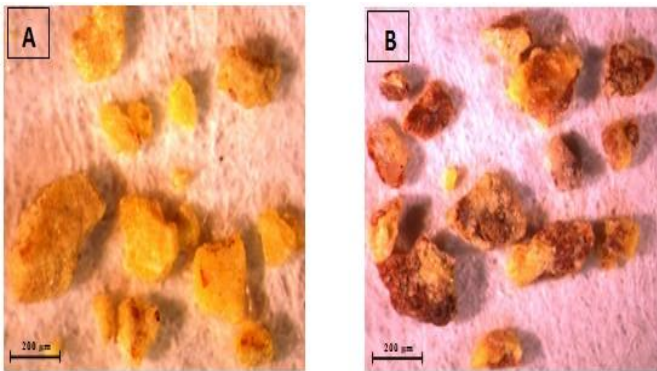


Fig -2: General view of separated grains under binocular microscope; A) uranophane, B) Iron-oxides completely stained with uranophane

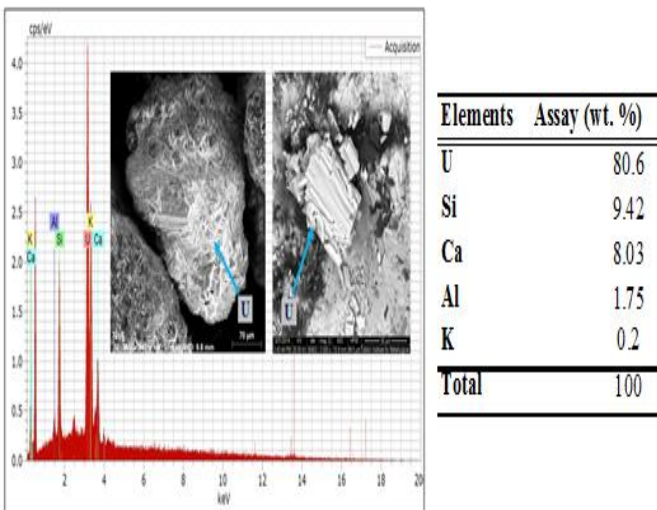


Fig-3: SEM-EDEX for separated grains of uranophane

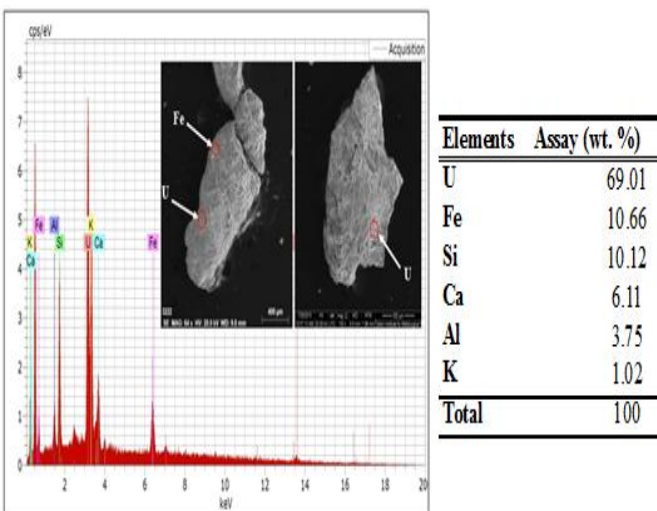


Fig -4: SEM-EDEX for separated grains of iron-oxide stained with uranophane.

The chemical analysis of the head sample using (XRF) revealed that the sample composed of the following major and trace elements as shown in Table 2.

Table -2: The chemical analysis Gattar II uraniferous altered granite samples

Oxides	Wt (%)	Trace	(ppm)
SiO ₂	76.15	Rb	242
Al ₂ O ₃	11.01	Sr	12
TiO ₂	0.11	Ba	168
Fe ₂ O ₃	3.68	Ga	15
CaO	0.65	Mn	60
MgO	0.01	V	8
Na ₂ O	2.76	Ni	25
K ₂ O	4.31	Cu	8
P ₂ O ₅	< 0.01	Zn	125
L.O.I	0.95	Pb	58
Total %	99.63	Zr	346
		Nb	61
		Y	161
		Th	40.1
		U	150

From the foregoing mineralogical investigation and microscopic examination in one hand, and, the presence of secondary uranium minerals which are distributed with highly concentration in all size fractions as shown in Table 3 on other hand it could be stated that a physical upgrading process shouldn't be applied before chemical processing. Thus the technological sample provided for uranium recovery has been used without any physical beneficiation with 100µm size.

Table -3: Size distribution, chemical analysis of the crushed sample

Size fraction (µm)	Wt. Ret., %	Assay of U (ppm)
+4000	16	50
-4000+2000	10	84
-2000+1400	8.4	108
-1400+1000	8	117
-1000+710	6	133
-710+500	8	137
-500+355	6.4	132
-355+250	7	148
-250+180	5.2	144
-180+100	8	147
-100+63	6	154
-63	11	340
Average assay = 0.0154%		

3.2 Factors affecting on the leachability of uranium

3.2.1 Effect of H₂SO₄ concentration

The effect of sulfuric acid concentration on leaching of uranium was investigated by using different acid concentrations 5, 10, 15, 20, 25 and 30 % v/v. The typical operating parameters were conducted as follows 120 min. agitation time, 55°C temperature, 1:3 solid/liquid ratio, -100 µm ore particle size and 400 r.p.m. agitation speed. The results obtained are shown in Fig. 5.

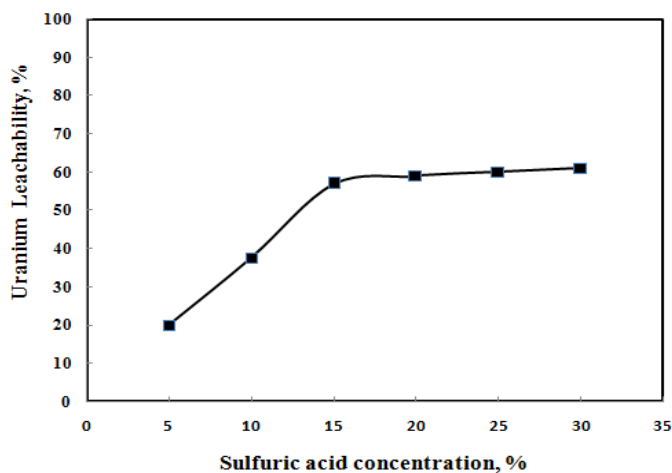


Fig -5: Effect of H₂SO₄ concentrations on the uranium leachability

[Test condition comprised: particle size -100 µm; S/L ratio 1:3; agitation speed 400; temperature 55 °C; agitation time 120 min.]

These results indicated that the ore sample is easily amenable to sulphuric acid leaching. Hence, by gradual increasing acid concentration from 5% to 15%, the leachability of uranium percent increased almost linearly until it reaches to 57% at 15% acid concentration. By increasing the acid concentration beyond 15%, uranium leachability percent was found to slightly increase until it reaches to 61 %. This may be attributed to the consumption of a great part of acid in side reactions as calcite other than uranium. Therefore, the concentration of 15 % was chosen as the optimal concentration leach of uranium.

3.2.2 Effect of particle size

The effect of particle size on the leaching process was investigated by using different size fractions; +4, -4+2, -2+1.4, -1.4+1, -1+0.71, -0.71+0.5, -0.5+0.3, -0.3+0.255, -0.255+0.18, -0.18+0.1, -0.1+63 and -0.063 mm. The uranium content of each size fraction was considered for the uranium recovery calculation. Fixed leaching conditions involved 15% acid con., 1:3 solid/liquid ratio, 120 min. agitation time, 55°C temperature and 400 r.p.m.

agitation speed. The obtained data are listed in table 4 and represented in Fig. 6.

Table -4: Effect of particle size on the leaching of uranium and the weight loss

Particle size µm	Uranium leachability (%)	Weight loss %
+4000	6	0.28
- 4000 + 2000	8.09	0.64
- 2000 + 1400	8.33	0.78
- 1400 + 1000	8.8	0.94
- 1000 + 710	10.15	1.1
- 710 + 500	12.11	1.214
- 500 + 355	18.9	1.318
- 355 + 250	23.6	1.4
- 250 + 180	31.25	2.1
- 180 + 100	40.13	2.16
-100 + 63	60.06	2.32
-63	62.05	2.4

Decreasing particle size, the velocity of dissolution and weight loss will increase. From these data, the best leachability was obtained at the finer particle size. This may be attributed to the larger surface area which increases the exposure of uranium particles to the leaching solution leading to increase the leachability. The uranium recovery was 62.05 % for -63 µm fraction, while it was 6% for +4 mm fraction.

The size fraction of -180+63µm was selected to avoid the overgrinding problems with low in the relatively fine particle of -63 µm fraction.

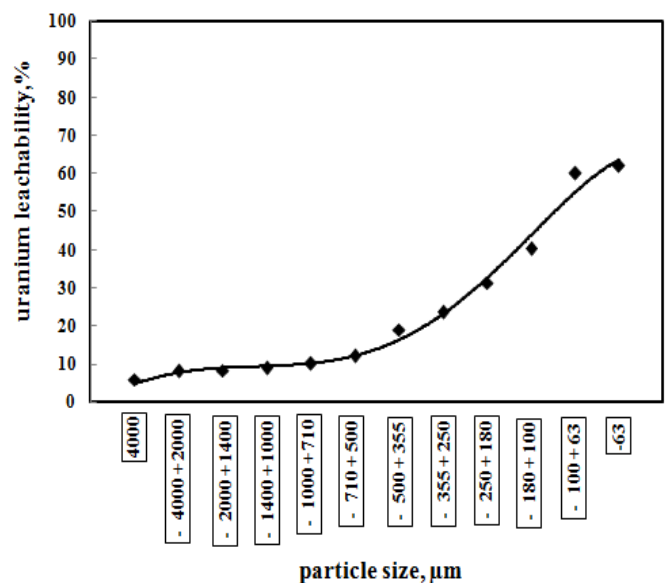


Fig -6: Effect of particle size distribution on the Uranium leachability.

[Test conditions comprised: acid concentration 15%; S/L ratio 1:3; agitation speed 400; temperature 55 °C; agitation time 120 min.]

3.2.3 Effect of solid/liquid ratio

The effect of solid/liquid ratio on the dissolution of uranium was studied using 1:2, 1:3, 1:4 and 1:5 solid/liquid ratios, while the other testing parameters comprised at 15% sulfuric acid conc., 120 min. agitation time, 55°C temperature, using -180 +63 μm ore particle size and 400 r.p.m. agitation speed. The results are shown in Fig.7.

Liquid volume was kept constant, and the amount of solid was changed to obtain the desired solid/liquid ratios. From these data, it was found that beyond 1/4 S/L ratio, only slight steady increase in the leaching efficiencies of uranium has been achieved. Accordingly, a solid ratio of 1/4 would be considered as optimal ratio at which the leachability of uranium attained 60%. Improving the latter could be realized by extending the leaching time or temperature. The obtained results are in harmony with that detected by [8].

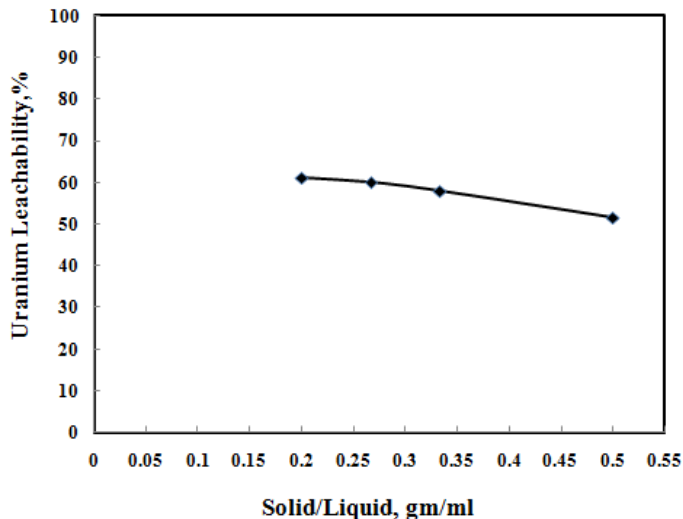


Fig -7: Effect of solid-liquid on the Uranium leachability

[Test conditions comprised: acid concentration 15%; particle size (-180+36μm); agitation speed 400; temperature 55 °C; agitation time 120 min.]

3.2.4 Effect of agitation speed

The effect of agitation speed on the leaching of uranium was studied using -180 +63 μm ore particle size. The agitation speed was varied from 100 to 800 rpm, keeping the other testing parameters fixed at 15 % sulfuric acid conc., 1:4 solid / liquid ratio, reaction temperature 55 °C and at 120 min agitation time.

Though the increase in agitation speed enhances the leachability of uranium there is no significant effect on uranium leachability after 600 r.p.m. The results are shown in Fig.8. From this figure, it was found that the variation in the leachability at different agitation rates in the entire range of 200–600 r.p.m. investigated was 54 % and 69 % respectively.

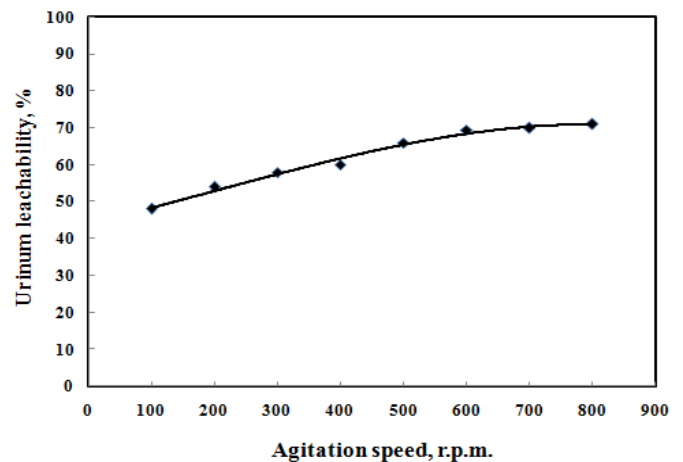


Fig -8: Effect agitation speed on the Uranium leachability

[Test conditions comprised: acid concentration 15%; particle size (-180+36μm); S /L ratio 1:4; temperature 55 °C; agitation time 120 min.]

Agitation ensures adequate suspension of the particles in the pulp and induces decrease in the thickness of the mass transfer boundary layer on the surface of the particle aiding improved kinetics. By increasing the agitation rate, therefore, the diffusion of liquor to the surface of the particles increases. However, the small differences noticed in the leachability at different agitation rates indicate that the dissolution process is not controlled by mass transfer through the liquid boundary layer [15].

3.2.5 Effect of leaching temperature

The influence of temperature on the dissolution of uranium has been studied in the temperature range from ambient (about 35°C) up to 95 °C. The other leaching conditions were fixed at 15% sulfuric acid conc., -180 +63 μm ore particle size, 1/4 solid/liquid ratio, 120 min agitation time and 600 r.p.m. agitation speed .

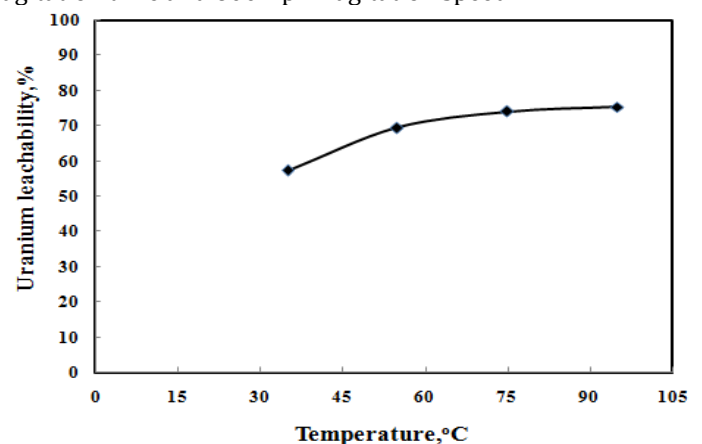


Fig -9: Effect of temperature on the Uranium leachability

[Test condition comprised: acid concentration 15%; particle size (-180+36μm); S /L ratio 1:4; agitation speed 600; agitation time 120 min.]

The results shown in Fig.9 revealed that actually the importance of temperature to obtain reasonable leaching efficiency of uranium. Working at room temperature under the above conditions did not leach more than 57.2 % only. Increasing the leaching temperature to 55°C increases the leachability to 69.4%. Further increasing of temperature to 75 and 95°C increases the leachability up to 74 and 75.41% respectively. It can be mentioned that while acid concentration is important controlling factor, the leaching temperature is quite important for uranium leaching. The 75°C was found to be optimal temperature for saving energy consumption. These results agree well with the results obtained by [11] and [12] who indicated that rise of temperature to 60°C gives a significant increase in uranium leachability.

3.2.6 Effect of agitation time

The effect of agitation time on uranium leaching was investigated to determine the necessary time required for uranium to achieve maximum solubility as possible. So, different periods of time ranged from 30 to 300 min. were investigated under testing parameters as follows 15% sulfuric acid conc., -180 +63 μm ore particle size, 1:4 solid/liquid ratio, 75 °C reaction temperature and agitation speed of 600 rpm. The results are shown in Fig.10.

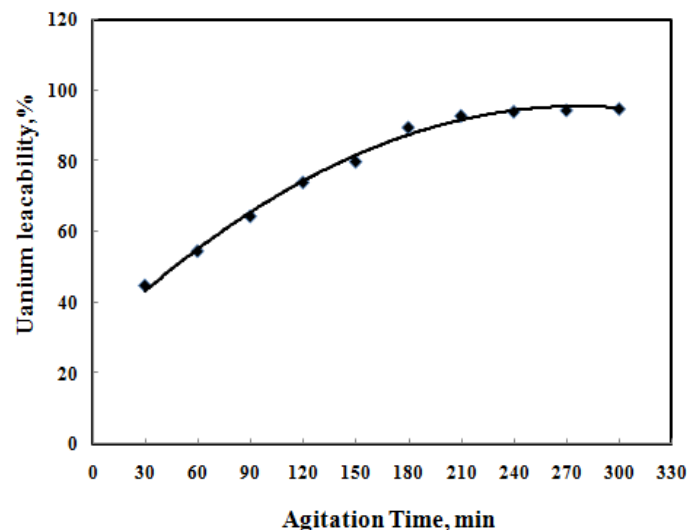


Fig -10: Effect of agitation time on the Uranium leachability

[Test conditions comprised: acid concentration 15%; particle size (-180+36μm); S /L ratio 1:4; agitation speed 600; temperature 75 °C]

From these data it is clear that a substantial amount of uranium was leached within the 60min. 54.52% and increased to 94 % in 240min., above this period, there was only a slight increasing in uranium leachability until 270 min. The manner of uranium leaching with time in the absence of an oxidant has actually been expected after the

mineralogical study which indicated that more than 90% of the uranium present as secondary uranium mineral. The remained insoluble uranium may be present in minerals e.g. zircon, fluorite, etc.

3.3 Leaching kinetics

The leaching of uranium ore was carried out at optimum conditions in the presence of 15% sulfuric acid concentration, -180+63μm particle size, solid/ liquid ratio 1:4 and 600 r.p.m. agitation speed at temperature range between 35°C and 95°C. Figure 11 shows that the leachability of uranium increases gradually by increasing of time and temperature. The maximum leachability was found to be 94% at 75°C and after leaching time of 240 min.

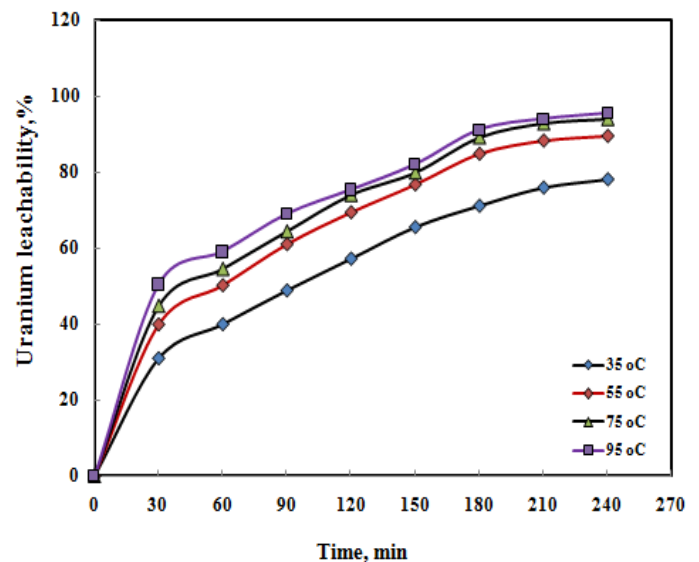


Fig -11: Plot of the Uranium leachability versus time of leaching at different temperatures.

The un-reacted shrinking-core model is the most commonly used mathematical model to describe the heterogeneous reactions like mineral leaching from ores. The solid-liquid-gas phase reactions like leaching; the rate of reaction is controlled by the following steps: solid or product layer diffusion and chemical reaction. One or more of these factors might control the rate of the reaction [10]. In order to understand the leaching mechanism prevalent for uranium ore, Eq. (1) and (2) models were used to describe this mechanism.

$$[1-3(1-X)^{2/3} + 2(1-X)] = k_d t \dots\dots\dots (1)$$

$$[1-(1-X)^{1/3}] = k_c t \dots\dots\dots (2)$$

Where [X] is the conversion fraction of solid particle, [k_d] is the rate constant (min⁻¹) for diffusion through the product layer, [k_c] is the apparent rate constant (min⁻¹) for the surface chemical reaction and [t] is the reaction time.

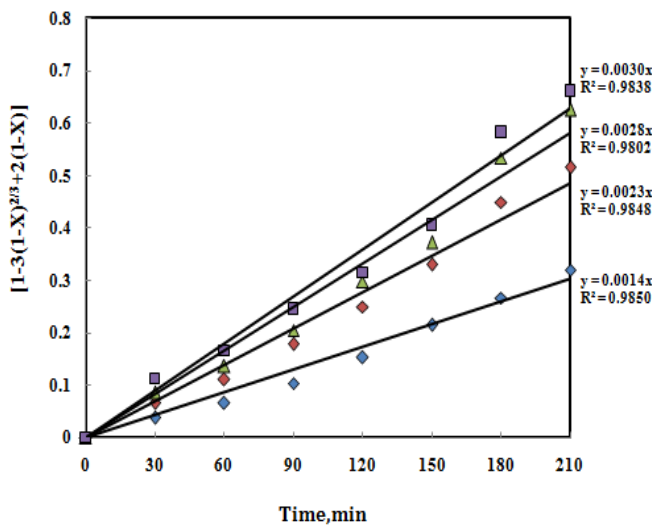


Fig -12: Plot of $1-3(1-x)^{2/3} + 2(1-x)$ versus time for different temperature

The relationship between reaction model and leaching time, at different temperatures is given in Fig.12. The mean values of the reaction rate constants [K] were determined from the slopes of the straight line of the relation between kinetic model and time.

The best fit has R^2 of 1.0. The k_d values given in table 5 vary in the range of 0.0014–0.003 min⁻¹ while the k_c was between 0.0019 and 0.003 min⁻¹. The R^2 values for k_d was 0.983 to 0.985 while for k_c it was in the range of 0.91–0.92. Based on the R^2 values it can be inferred that the predominant dissolution mechanism of uranium from the Gattar ore sample is diffusion controlled only.

Table -5: The apparent rate constant with their coefficient of determination at different temperature

Temperature (°C)	Apparent rate constant (min ⁻¹)		coefficient of determination(R^2)	
	Chemical control (k_c)	Diffusion control (k_d)	Reaction control	Diffusion control
35	0.0019	0.0014	0.9257	0.985
55	0.0025	0.0023	0.9269	0.9848
75	0.0028	0.0028	0.9392	0.9802
95	0.003	0.003	0.9157	0.9838

From the obtained data, the logarithmic values of these reaction rate constants [k_d] were plotted against the reciprocal of the absolute reduction temperature according to the Arrhenius equation as shown in Fig.14. The apparent activation energy (E_a) was calculated from the slope of straight line obtained to be 11.914 for diffusion controlled reaction model. Based on the (E_a)

values it can be inferred that the predominant dissolution mechanism of uranium from the ore is diffusion controlled only. This value is less than the amount mentioned by [14] who pointed out that the activation energy for diffusion-controlled reactions is below 20 kJ/mol and it is above 40 kJ/mol for chemical controlled reactions.

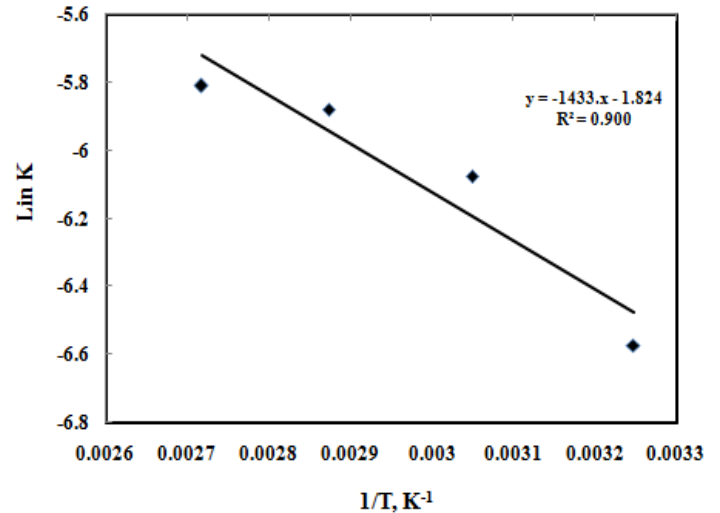


Fig -13: Plot of $\ln K$ (min⁻¹) against reciprocal of absolute temperature (K^{-1}).

4. CONCLUSION

- Proper agitation leaching process has been achieved for Gattar II uraniumiferous altered granite samples. The uranium value of this ore material is 150 ppm U. The optimum working conditions have been determined using sulfuric acid agitation leaching within 15% acid concentration, -180+63 μ m particle size with low in the relatively fine particle of -63 μ m fraction, 1/4 Solid/Liquid ratio, 75°C reaction temperature, 240 min. agitation time and 600 r.p.m. agitation speed.
- The leaching kinetics of uranium showed that the rate of dissolution using H₂SO₄ acid is diffusion controlled and follows the shrinking core model $[1-3(1-X)^{2/3} + 2(1-X)] = k_d t$ with an apparent activation energy of 11.914 kJ/mol.

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BIOGRAPHIES



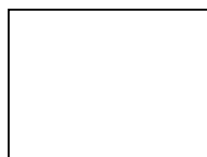
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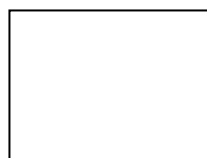
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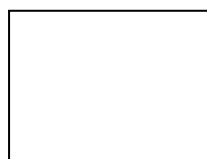
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