



Kinetics of leaching process of Uranium from EL-Missikat shear zone Eastern Desert, EGYPT

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Article Information

Abstract

Received; 25 October. 2013

In Revised form; 20 November, 2013

Accepted; 20 November, 2013

Keywords:

Leaching

Uranium

REE

Shrinking core model

Kinetics

Diffusion

The leaching process of uranium- REE ore from El-Missikat in a sulfuric acid solution using hydrogen peroxide as an oxidant was investigated. The leaching condition temperature, hydrogen peroxide concentration, sulfuric acid concentration, contact time, particle size, solid-liquid ratio and agitation rate were studied. The optimum process operating parameters were: ore particle size 74 μm ; sulfuric acid concentration 2.5 M; contact time 240 min; solid-liquid ratio 1:2; H₂O₂ concentration 0.5 M and agitation rate 600 rpm at temperature 25°C. The leaching efficiency of REE was about 70%, while the uranium leaching efficiency was about 95%. The experimental data were well interpreted with a shrinking core model with diffusion control through a porous product layer. The leaching process follows the kinetic model:

$1-3(1-X)^{2/3} + 2(1-X) = k_1t$ with an apparent activation energy of 13.16 kJ/mole.

1. Introduction

The study area of Gabal El-Missikat is one of the most important localities in the central Eastern Desert of Egypt for occurrence of uranium deposits; El-Missikat covers an area of about 80 km² and includes Gabal El-Garra and Gabal El-Gidami in addition to Gabal.El-Missikat [1]. El- Missikat uranium prospect area lies at about 3 km, midway between Safaga, on the red Sea coast and Qena in the Nile Valley. It is roughly bound by longitudes 33° 15' - 33° 28' E and latitudes 26° 24' - 26° 30' N where the mineralogical studies revealed the presence of uranium minerals such as uranophane, uraninite, soddyite and renadite [2].

Gabal El-Missikat consists of rocks are essentially compound of quartz, potash feldspars, sodic plagioclase and biotitic. The main accessory minerals are sulfides, magnetite, zircon, apatite, fluorite, titanite, monazite, xenotime, uranothorite, rutile and uraninite. Hematite, epidote, muscovite and chlorite are present as secondary minerals [3].

Recovery of metal values from the ores includes three main processes namely the physical upgrading, leaching and finally the metals recovery then purification with a great deal of chemical treatments through the extraction of metals from the obtained solution. This would be applied upon some minerals such as monazite heavy minerals from ores or residues either by gravitational differences or slight difference in magnetism ability. Leaching process is the dissolution of uranium from the uranium containing minerals in the ore. The selection of leaching procedure for dissolving uranium minerals is dependant in part on the physical characteristics of the ore such as: type of mineralization, ease of liberation and the nature of other constituent minerals presents [4].

During the past few decades, different types of leaching processes based on acid leaching and alkaline leaching methods were developed for processing uranium ore of different characteristics [5].

Leaching (or solid extraction) is defined as the hydrometallurgical process which is used to dissolve valuable matter from its mixture with an insoluble solid by means of suitable reagent. In case of uranium ore material, uranium exists mainly in the hexavalent or tetravalent states. The hexavalent uranium oxide (UO_3) is the main constituent in secondary uranium minerals and may be considered as the amphoteric uranyl oxide $[(\text{UO}_2)\text{O}]$ which is capable to form salts with both acid and alkaline reagents[6].

Many precious metals have been leached from their ores such as batch leaching of uranium ore in Canada [7], Oxidative leaching of molybdenum-uranium ore in Wadi Sikait, Egypt [8], Dissolution of nickel from lateritic nickel ore in Eskisehir region of Turkey [9], Column leaching of lanthanides from Abu Tartur phosphate ore in Egypt [10], leaching process of sella uranium ore in Egypt [11], dissolution of total gold from Ijero-ekiti (Nigeria) gold ore deposit [12], leaching of TiO_2 from Egyptian ilmenite [13], leaching of nickel and cobalt from Chinese laterite ore [14]. leaching of Cobalt and copper have been leached successfully by HCl from Co-Cu ores in the Democratic Republic of Congo [15] and phosphorus leaching from high phosphorus iron ores in China [16].

Several leaching methods were studied and industrially applied for the breakdown of the principal REE minerals. There are two main breakdown methods was worked out namely; the sulfuric acid and the caustic soda methods [17]. The former was actually used most extensively in the USA. Depending on the acid/ore ratio, temperature and acid concentration, either Th or the REEs can be selectively solubilized or else both Th and REEs can be totally solubilized for later separation. The alkali method of monazite breakdown was mainly applied in Brazil and India [18].

The aim of this work is to investigate a simple leaching process of relatively low grade uranium-REE ore material from EL-Missikat, Eastern Desert, Egypt by using H_2SO_4 acid in the presence of H_2O_2 as an oxidant. This paper considers the kinetic aspects of uranium leaching. The effects of the main system variables on the leaching rate were examined, the kinetic model and the apparent activation energy were determined

2. Experimental

2.1. Characterization of the uranium-REE ore

Samples were collected from El-Missikat which is located in the Eastern Desert, Egypt. The ore sample was firstly crushed to 297 μm and 149 μm then ground to 74 μm particle size. Sample characterization by XRF showed the presence of Ba, Pb, Fe, Mo, Nb, Zr, Y, Sr, U, Zn, Mn, Ca, K, S, Si and Al. The experimental work was performed upon the ore with sulfuric acid. The mineralization is considers mainly as uranium ore materials which is also associated with other economic minerals such as REE and other elements. Several experiments were carried out upon the ore to specify some of these economic minerals (REE). Series of experiments was carried out upon 100 g sample portion of heavy concentrate ground to 74 μm size. After each experiment, the leach slurry was filtered, washed thoroughly with hot distilled water and then both filtrate and washing were made up to 1000 ml. Uranium and REE leaching efficiency was calculated according to the following equation:

$$\text{Leaching efficiency of Uranium or REE, \%} = \frac{\text{Leached Metal ion conc.}}{\text{Original Metal ion conc.}} \times 100$$

2.2 Analytical procedures and Instrumentation

The ore was analyzed for its major and minor elements using the reported methods and the results are shown in the table (1)

- 1- Generally, the samples used in this work were weighed using an analytical balance produced by Shimadzu (AY 220).
- 2- The hydrogen ion concentration of the different solutions was measured accurately using the pH- meter model (HAANA pH-mV-temp).
- 3- Total REE was determined by arsenazo III where the absorbance of its complex was measured at the wavelength 650nm [19] by using UV-spectrophotometer "single beam multi-cells-positions model SP-8001", Metretech Inc., version 1.02.
- 4- Icp model (Prodigy Axial high dispersion ICP-OES-USA) used for determination individual REE.
- 5- An atomic absorption model G.B.C.A.A, was used for measuring trace elements.
- 6-The X-ray fluorescence technique (XRF) was used to identify the unknown minerals using a PHILIPS X'UNIQUE II spectrometer Rh-target tube, 70KV, 15mA, LiF-220 analyzing crystal and Rh- target tube, 30 KV,60 mA , PE-001 analyzing crystal.
- 7- Uranium was determined by titration against ammonium meta vanadate [20]

Table (1): Chemical analysis of El-Missikat ore

Major oxides	%	Trace Element	ppm
SiO ₂	78	U	700
Al ₂ O ₃	10.2	∑REEs	500
Fe ₂ O ₃	4.4	Zn	400
P ₂ O ₅	0.75	Pb	417
CaO	1.2	Cd	92
MgO	1.16	Cu	100
Na ₂ O	0.078	Ni	80
K ₂ O	0.068	Nb	87.5
MnO	0.1	Sr	201
TiO ₂	0.15	Zr	200
L.O.I**	1.7	Th	20.5
Total	98.506	Ba	200

L.O.I**Total loss in ignition

3. Results and Discussion

3.1 Leaching results

3.1.1 Effect of H₂SO₄ concentration

A series of leaching experiments was carried out using different H₂SO₄ concentrations (0.5 to 3 M). The other leaching conditions were kept constant, where the ore particle size was 74 μm, a solid/liquid ratio of 1:3, contact time 300 min and a stirring rate of 700 rpm at 25°C. The leaching efficiencies are shown in Fig (1). From this Figure, it is clear that as the H₂SO₄ concentration increased from 0.5 M to 3 M, the uranium dissolution efficiency increased from 33% to 70% while the dissolution efficiency of REE increased from 23% to 54%. However, 2.5 M H₂SO₄ was the H₂SO₄ concentration choice applied in the subsequent leaching experiments achieving the highest dissolution efficiencies.

The leaching residue contained mainly SiO₂ and Fe₂O₃. These substances are not leached because SiO₂ does not undergo dissolution under normal acidic conditions. In addition, for a low concentration of Fe₂O₃ when H₂SO₄ is used, the iron oxide will not dissolve.

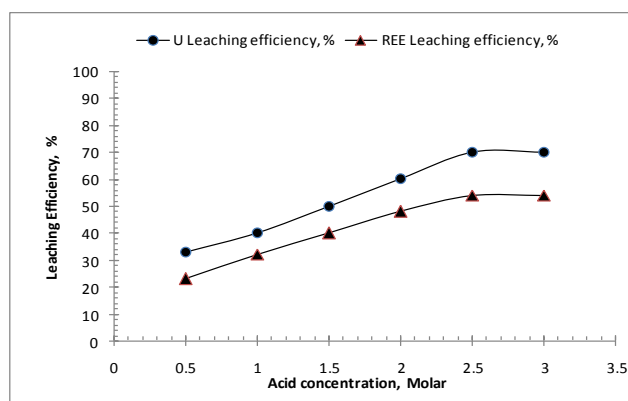


Fig 1: Effect of H₂SO₄ concentration on the leaching efficiencies of uranium and REE (ore particle size 74μm, solid/liquid ratio 1:3, stirring speed 700 rpm, contact time 300 min at 25°C)

3.1.2. Effect of Contact time

In these experiments, different leaching times (30 to 300 min) was tested. The other leaching conditions were kept fixed, namely; 74 μm ore grain size, 2.5 M H_2SO_4 , 700 rpm stirring speed, solid/ liquid ratio of 1:3 at 25 $^{\circ}\text{C}$. For a leaching time of 300 min, the leaching efficiencies of uranium and REE reached 70% and 54%, respectively. As the leaching time was extended, the leaching efficiency of uranium and REE did not increase. Therefore, it can be concluded that 240 min contact time represents the preferred condition for the subsequent uranium and REE ore dissolution experiments. The results are shown in Fig (2).

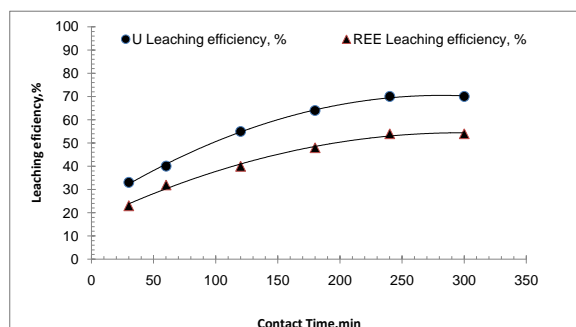


Fig 2: Effect of Contact Time on the leaching efficiencies of uranium and REE (ore particle size 74 μm , 2.5 M H_2SO_4 , solid/liquid ratio 1:3, stirring rate 700 rpm at 25 $^{\circ}\text{C}$)

3.1.3. Effect of H_2O_2 concentration

The uranium and REE minerals present in the working ore need to be oxidized before dissolving because they are in oxidative states that cannot be dissolved under normal conditions. For this reason, H_2O_2 was chosen as a strong oxidizing agent [21]. It has been shown that hydrogen peroxide under acidic conditions can oxidize the low valence states of uranium and REE to high valence states which are readily soluble in the leaching solution.

In order to evaluate the effect of H_2O_2 , series of leaching experiments were carried out using 2.5 M H_2SO_4 . These experiments were performed in the absence of and in the presence of different concentrations of H_2O_2 varying from 0 M to 0.6 M. The other leaching conditions were fixed at: a solid/liquid ratio of 1:3, contact time 240 min, stirring rate 700 rpm, 74 μm ore grain size at 25 $^{\circ}\text{C}$. The results are shown in Figure (3). About 70% and 54% of uranium and REE respectively dissolved in the absence of H_2O_2 . As the milliliters H_2O_2 added increased the leaching efficiency of uranium and REE increased to 95% and 70% respectively. Therefore, 0.5 M H_2O_2 /100g ore represents the preferred condition for dissolution experiments.

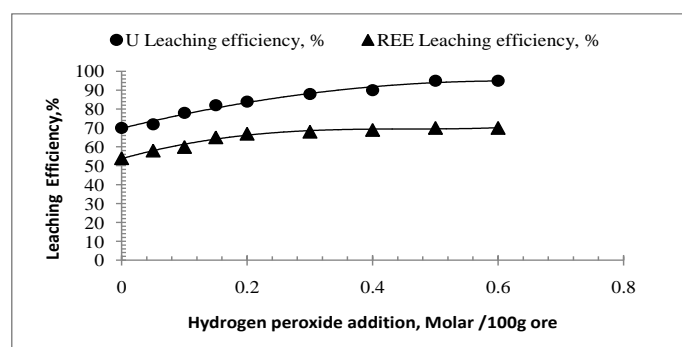


Fig 3: Effect of the H_2O_2 concentration on the leaching efficiencies of uranium and REE (ore particle size 74 μm , 2.5 M H_2SO_4 , solid/liquid ratio 1:3, stirring rate 700 rpm, contact time 240 min at 25 $^{\circ}\text{C}$)

3.1.4 Effect of stirring rate

The effect of the stirring rate was studied using conditions of: 74 μm particle size, 2.5M H_2SO_4 , 0.5 M H_2O_2 , 1: 3 solid/ liquid ratio at 25 $^{\circ}\text{C}$ for 240 min. Stirring rate of 200 rpm, 300 rpm, 400 rpm, 500 rpm, 600 rpm and 700 rpm were examined. The leaching rates for uranium and REE increased as the stirring rate increased to 600 rpm reaching 95% and

70% leaching efficiencies respectively and then remained almost constant above 600 rpm. Therefore, the preferred speed was 600 rpm which was used for all the subsequent tests.

3.1.5 Effect of solid/liquid ratio

Working with fixed concentrations of 2.5 M H₂SO₄ and 0.5 M H₂O₂, the effect of eight other solid/liquid ratios (1:1, 1:1.25, 1:1.5, 1:75, 1:2, 1:3, 1:4 and 1:5) were tested under the same leaching conditions used for the solid/liquid ratio of 1:2. The results are shown in Figure (4). The leaching efficiencies of uranium and REE decreased at solid/liquid ratios of 1:1 and 1:75. Increasing the amount of acid by using a solid/liquid ratio of, 1:2 to 1:5 the extraction efficiency not increased. Therefore, a solid/liquid ratio of 1:2 was the preferred ratio.

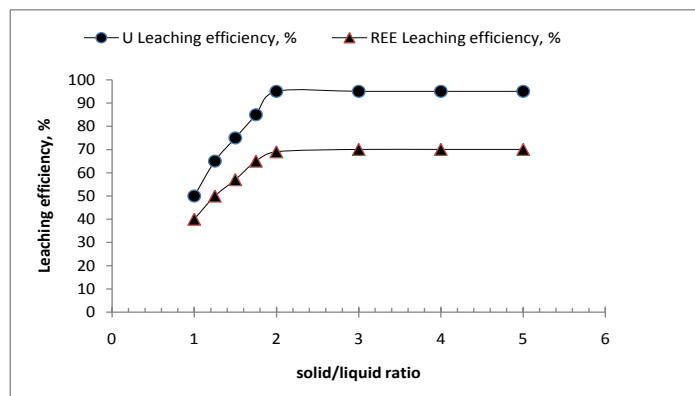


Fig 4: Effect of solid/liquid ratio on the leaching efficiencies of uranium and REE (ore particle size 74 μm, 2.5 M H₂SO₄, 0.5 M H₂O₂, contact time 240 min, stirring rate 600 rpm at 25°C)

3.1.6 Effect of particle size

The effect of particle size on the leaching of uranium and REE was studied using three different size fractions namely; 297 μm, 149 μm and 74 μm. It was found that, the leaching efficiencies of uranium and REE increased as the particle size of the working ore decreased. When a particle size of 297 μm was used, the leaching efficiency of uranium and REE sharply decreased to 57.14% and 36% respectively. As a result, the fraction with the smallest particle size 74 μm gave the highest dissolution result. This is due to the highest surface area of the smallest particle size fraction; the conversion rates are inversely correlated with average initial diameter of the particles. The results are shown in table (2).

Table (2): Results of Grain Particle Size Effect on Leaching Efficiency

Time, min.	U. Leaching Efficiency, %			REE Leaching Efficiency, %		
	Grain Size, μm			Grain Size, μm		
	297	149	74	297	149	74
30	26.42	30	50	14	26	29
60	31.42	40	61.42	20	34	40
120	42.85	57.14	78.57	27	42.2	54
180	51	65.71	85.71	32	50	64
240	56.42	71.42	95	36	56	70
300	57.14	71.42	95	36	60	70

3.1.7 Effect of temperature

Leaching experiments were carried out at room temperature, 40°C, 60°C, 80°C and 100°C using the same conditions. The resulting leaching efficiencies are shown in table (3) and indicate that the temperature plays a critical role in the leaching of uranium and REE. For example, when working at room temperature, the obtained leaching efficiencies for uranium and REE were only 70% and 54% respectively. By increasing the temperature from 40°C to 80°C, the uranium and REE leaching efficiencies gradually increased from 74.28% to 81.42% and from 60% to 76%, respectively. A further increase in temperature to 100°C, gave uranium and REE leaching efficiencies of 95.7 % and 88 %, respectively. Therefore, it can be concluded that higher leaching temperatures resulting higher leaching efficiencies.

Table (3): Effect of temperature on the leaching efficiencies for uranium and REE

Temperature, °C	25	40	60	80	100
U Leaching Efficiency, %	70	74.28	78.57	81.42	95.71
REE Leaching Efficiency, %	54	60	70	76	88

3.2 Leaching kinetics of Uranium

3.2.1 Effect of temperature

Figure (5) presents the effect of the reaction temperature on the uranium leaching rate in the range of 25°C–100°C under conditions of 74 μm particles, 2.5M H₂SO₄, 0.5 M H₂O₂ with a 1:2 solid/liquid ratio. The results show that the leaching rate of uranium increases as the temperature increases. In order to obtain the kinetic equation and the apparent activation energy for the dissolution of uranium in the presence of H₂O₂, the experimental data in **Figure (5)** were correlated to various kinetic models for solid-liquid reactions. Several equations were studied including [22, 23]:

$$1-3(1-X)^{2/3} + 2(1-X) = k_1t, \tag{1}$$

$$1 - (1 - X)^{1/3} = k_2t, \tag{2}$$

$$X = k_3t, \tag{3}$$

where k_1 , k_2 and k_3 are the apparent reaction rate constants (min^{-1}) for each case respectively and t is the leaching time (min) and X is the fraction reacted expressed as:

$$X = \% \text{ extraction}/100. \tag{4}$$

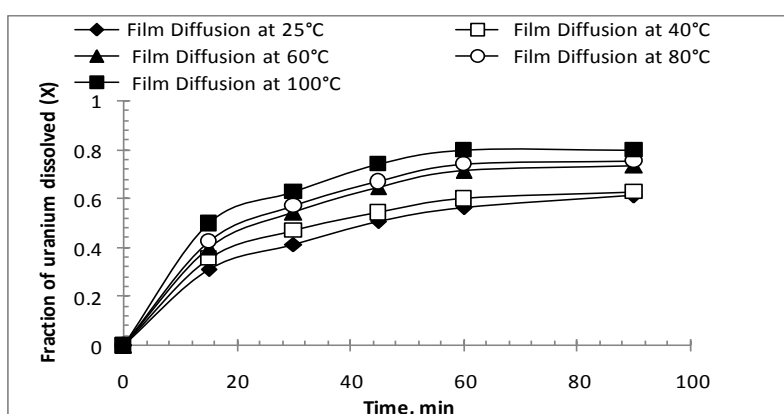


Fig 5: Effect of different temperatures on the uranium leaching efficiency (ore particle size 74μm, 2.5M H₂SO₄, 0.5 M H₂O₂, 1:2 solid/liquid at stirring rate 600 rpm)

The data did not fit Eqs. (2,3). The best fit for the data from 0 min to 90 min was for Eq. (1), a diffusion controlled kinetic equation. The relationships between equation 1-3 $(1-X)^{2/3} + 2(1-X)$ and the leaching time for uranium at various temperature are plotted in Figure (6). The R squared values for all the lines are greater than 0.9. This indicates that the linear relationship between $1-3(1-X)^{2/3} + 2(1-X)$ and the leaching time (t) is significant and suggests that the leaching rate of uranium is diffusion controlled. The apparent activation energy was determined from the Arrhenius equation [24]:

$$\ln k = \ln A - E_a/RT \tag{5}$$

Where k is the reaction rate constant, A is the frequency factor, E_a is the apparent activation energy and R is the ideal gas constant. The data for the four temperatures are plotted and given in Figure (7), and the regression analysis for these plots also shows that the linear relationship is significant. The apparent activation energy (E_a) was determined to be 13.16 KJ/mol.

It is worthy to mention that the calculated activation energy suggests a diffusion controlled process for EL-Missikat low grade uranium-REE ore material at temperatures 25°C, 40°C, 60°C, 80°C and 100°C. The different values of the apparent rate constants k_1 and k_2 at different temperatures and their corresponding correlation coefficient rate are summarized in table (3).

Table 4: The value of the apparent rate constants, K_1 and K_2 , min^{-1} with the correlation coefficient at different temperature range.

Temperature, °C	Apparent K_2 , min^{-1}	R^2	Apparent K_1 , min^{-1}	R^2
25	0.004	0.882	0.002	0.994
40	0.005	0.837	0.003	0.999
60	0.006	0.903	0.004	0.999
80	0.006	0.896	0.005	0.992
100	0.007	0.875	0.006	0.998

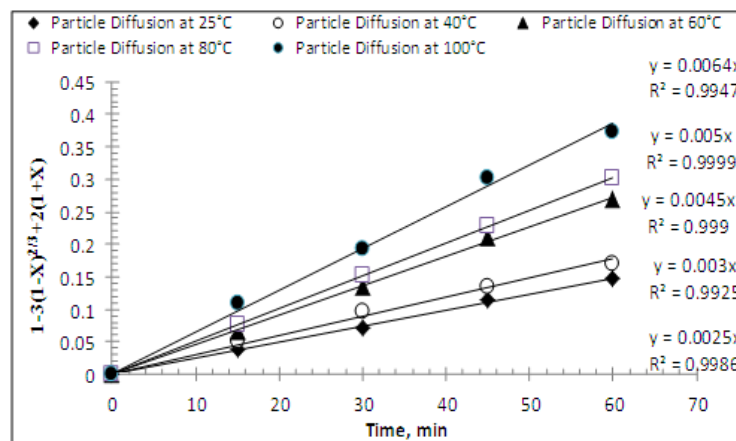


Fig 6: Relationship between $1-3(1-X)^{2/3} + 2(1-X) = k_1t$ and leaching time for uranium at various temperatures (ore particle size 74 μm , 2.5M H_2SO_4 , 0.5 M H_2O_2 , 1:2 solid/liquid, at stirring rate 600 rpm)

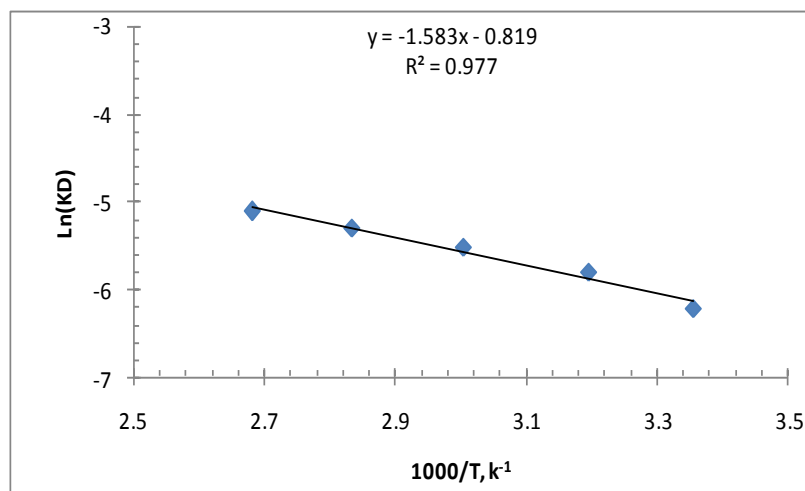


Fig 7: Arrhenius plot for uranium leaching (ore particle size 74 μm , 2.5 M H_2SO_4 , 0.5 M H_2O_2 , 1:2 solid/liquid, at stirring rate 600 rpm)

3.2.2 Effect of H₂SO₄ concentration

The effect of the H₂SO₄ concentration was studied from 0.5 to 2.5 M on the uranium leaching rate in the absence of H₂O₂ with a 1: 3 solid/liquid ratio at 25°C There is a general increase in the leaching rate as the H₂SO₄ concentration increases. The corresponding plots of $1-3(1-X)^{2/3} + 2(1-X)$ versus time at various concentrations are graphed in Figure (8) It can be seen that an initial H₂SO₄ concentration of 2.5 M is necessary to obtain a high dissolution rate of uranium. In order to obtain the reaction order for the total H₂SO₄ concentration, log-log plots of the rate constants versus the total H₂SO₄ concentration are plotted and given in Figure (9) .The slope of the line, or the reaction order of the total H₂SO₄ concentration, is 0.96. Hence the leaching rate of uranium strongly depends on the acid concentration.

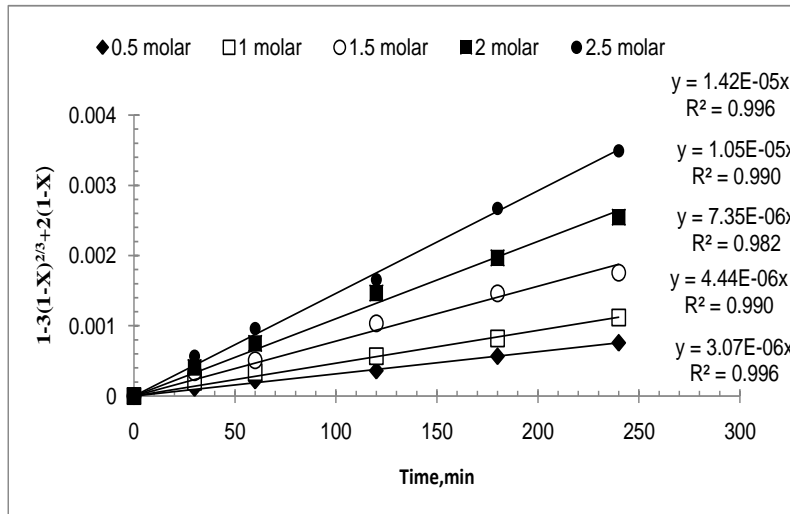


Fig 8: Relationship between $1-3(1-X)^{2/3} + 2(1-X) = k_1t$ and leaching time for uranium leaching at various H₂SO₄ concentration (ore particle size 74µm, contact time 240 min, 1:3 solid/liquid, stirring rate 700rpm at 25°C)

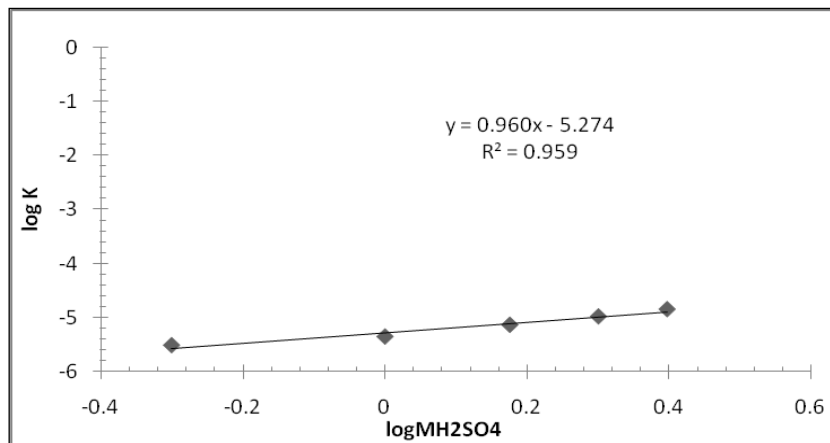


Fig 9: log-log plot of the rate constant versus H₂SO₄ concentration (ore particle size 74µm, contact time 240 min, 1:2 solid/liquid, stirring rate 600 rpm at 25°C)

3.2.3 Effect of particle size

The effect of particle size (74 µm , 149 µm and 297 µm) on the rate of the uranium leaching reaction in the presence of 2.5 M H₂SO₄, 0.5 M H₂O₂ and a 1:2 solid/liquid ratio is illustrated in table (5). There is a general increase in the leaching rate as the particle size decreases. One reason for this is that for smaller particle sizes, there is an increase in the reaction surface area which enhances the mass transfer process of leaching. Another reason is that the solid particles were activated during grinding. The plots of $1-3(1-X)^{2/3} + 2(1-X)$ against time for the various particle sizes are graphed in Figure (10). The apparent rate constant was determined and plotted versus the inverse of the initial average particle diameter d and the results are shown in Figure (11). The linear relationship between the rate constant k, and the inverse

of (d) indicates that the ash layer diffusion reaction on the particle surface is the rate-limiting step of the dissolution process.

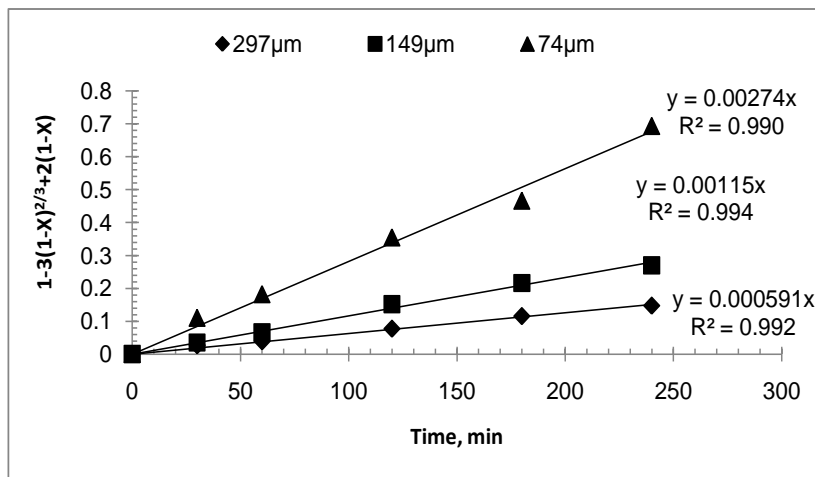


Fig 10: Relationship between $1-3(1-X)^{2/3} + 2(1-X)$ and leaching time for uranium leaching with different particle sizes (2.5M H_2SO_4 , 0.5M H_2O_2 , contact time 240 min, 1:2 solid/liquid ratio, stirring rate: 600rpm at 25°C)

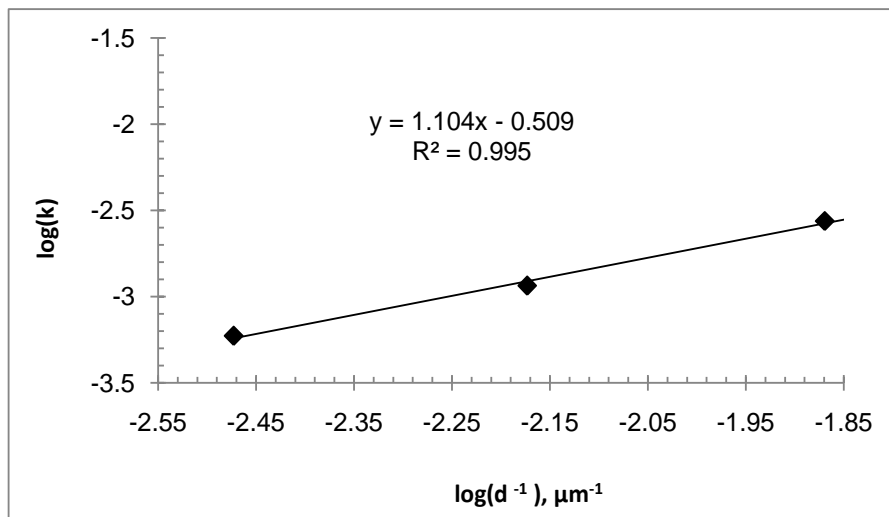


Fig 11: plot of the rate constant versus the inverse of the particle diameter (2.5 M H_2SO_4 , 0.5M H_2O_2 , contact time 240 min, 1:2 solid/liquid ratio, stirring rate 600 rpm at 25°C)

The application of diffusion model for different particle size fractions is shown in Figure (11). From the corresponding k_1 and particle size values, plots of $\log k$ versus $\log d^{-1}$ were obtained. The results also showed that is the order of the reaction was found inversely proportional to power 1.104 of particle size ($[r_0]^{-1.104}$) from Figure (10).

4 Conclusions

Uranium and REE can be easily leached from Uranium/ REE ore by using H_2SO_4 acid in the presence of H_2O_2 as the oxidant. Using conditions of: 2.5 M H_2SO_4 ; 0.5 M H_2O_2 ; a stirring rate of 600 rpm, a solid/liquid ratio of 1 : 2, 240 min and a particle size of 74 μm , gave leaching efficiencies of about 95% for uranium and 70% for REE. The reaction order of the total H_2SO_4 concentration, is 0.96. Hence the leaching rate of uranium strongly depends on the acid concentration. The order of the reaction was found inversely proportional to power 1.104 of particle size ($[r_0]^{-1.104}$).

The leaching kinetics of uranium showed that the rate of uranium leaching using H_2SO_4 acid in the presence of H_2O_2 as an oxidant is diffusion controlled and follows the shrinking core model

$$1-3(1-X)^{2/3} + 2(1-X) = k_1 t \text{ with an apparent activation energy of 13.16 kJ/mol.}$$

The kinetics study also showed that the leaching reaction has a strong dependence on the concentrations of acid and hydrogen peroxide. The linear relationship between the rate constant, k , and the inverse of the initial particle diameter indicates that the rate of uranium leaching is diffusion controlled.

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