Kinetics of uranium leaching process using sulfuric acid for Wadi Nasib ore, South western Sinai, Egypt

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Received: 5/9/2020 \hspace{1cm} Accepted: 28/10/2020

Abstract:

The leaching of uranium from Wadi Nasib ore using sulfuric acid solution has been studied. The effect of different parameters such as leaching time, sulfuric acid concentration, solid/liquid ratio, leaching temperature and mechanical stirring speed were investigated in order to optimize various process parameters for maximizing the recovery of uranium. Uranium dissolution efficiency of 98.5\% was obtained for Wadi Nasib ore after 4 hours contact time leaching using 200 g L\textsuperscript{-1} H\textsubscript{2}SO\textsubscript{4} and solid/liquid ratio 1/4 at leaching temperature 80°C and 400 rpm mechanical stirring speed without any oxidant addition. The kinetic data showed that the leaching process can be described by a shrinking-core model with apparent activation energy equals to 16.2 kJ/mol. The low activation energy supported the findings that the Wadi Nasib leaching rate is controlled by diffusion-controlled process.

Keywords: Kinetic; Leaching; Uranium Ore; Sulfuric Acid; Diffusion Control

1- INTRODUCTION

Uranium is one of the important elements. It represents the corner stone in nuclear energy field through U fuel. It is used as a fuel in nuclear power plants and in manufacture of nuclear weapons. Depleted uranium is a by-product of uranium enrichment. Depleted uranium has a variety of applications because of its high density and its pyrophoric properties. It is used in armour plates in heavy tanks and in armour-piercing ammunition.

Leaching is an important step in the processing of a uranium ore. Uranium ores are treated by either acid or alkaline reagents, where sulfuric acid or sodium carbonate-sodium bicarbonate systems used almost exclusively for commercial uranium recovery. There are two valency states in which uranium occurs naturally, the hexavalent form, the oxide of which is UO\textsubscript{3} and the tetravalent form, the oxide of which is UO\textsubscript{2}. In its hexavalent form uranium goes directly into solution, while in its tetravalent form it does not dissolve at a perceptible rate and requires oxidizing to the hexavalent using an oxidant.

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The most widely used methods for the treatment of uranium ores involves treating finely ground ore slurry with sulfuric acid at elevated temperatures. The choice of sulfuric acid depends on its relatively low cost and wide availability. Other applicable acids, such as hydrochloric and nitric are more costly and cause more serious environmental pollution (Ashbrook, 1978).

In the leaching process, H$_2$SO$_4$ acid dissolves U as UO$_2^{2+}$ and then forms three complexes of uranyl sulfate anion as the following equations:

$$\text{UO}_2^{2+} + \text{SO}_4^{2-} \rightarrow \text{UO}_2(\text{SO}_4) \quad (1)$$

$$\text{UO}_2^{2+} + 2\text{SO}_4^{2-} \rightarrow \text{UO}_2(\text{SO}_4)_2^{2-} \quad (2)$$

$$\text{UO}_2(\text{SO}_4)_2^{2-} + (\text{SO}_4)^{2-} \rightarrow \text{UO}_2(\text{SO}_4)_3^{4-} \quad (3)$$

In Sinai, several leaching studies have been performed upon Abu Zeneima ore from different locations, but with different constituents and grades of the economic valuable elements, the leaching characteristics of U from a siltstone ore from Abu Zeneima area was reported by (Mahdy et al., 1988; Mahdy, 1989; Amer et al., 2000; Abdel Monem et al., 1997). They concluded that valuable elements could be almost completely leached by mineral acid under mild agitation or percolation leaching conditions. In the meantime, carbonate leaching achieved uranium separation from other metals during the leaching step. Also, the agitation acid leaching is preferred to leach most of the metal values with high efficiencies belonging to the sedimentary rocks of gibbsite shale, Abu Zeneima area, (El Hazek et al., 2008). The ore was exposed to 800 g L$^{-1}$ sulfuric acid with a solid/liquid ratio of 1/2 at 100°C for 4 h. leaching efficiency of uranium attained 95.5% and complete leaching of other elements Al, Cu, and Ni.

The leaching process of uranium – rare earth elements ore from El-Missikat in a sulfuric acid solution using hydrogen peroxide as an oxidant was investigated (Moussa et al., 2014). 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.

Uranium leaching from low grade uraniferous Granites, Eastern Desert, Egypt was investigated using 15% sulfuric acid to achieve 94% leaching efficiency, while the other relevant factors of acidic leaching of a technological sample were $-180 + 63 \mu m$ particle size with low in the relatively fine particle of $-63 \mu m$ fraction, 1/4 solid/liquid ratio, 75°C reaction temperature, 240 min. agitation time and 600 rpm agitation speed, and the dissolution rate of uranium using 15% H$_2$SO$_4$ was reported as diffusion controlled, follows the shrinking core model $[1-3(1-X)^{2/3} + 2(1-X)] = kdt$ with an apparent activation energy of 11.914 Kj mol$^{-1}$ (Kraiz et al., 2016).

The leaching process of uranium (VI) from El-Erediya rock sample in sulfuric acid solution using hydrogen peroxide as an oxidant was investigated (Khawassek et al., 2016). The leaching efficiency of uranium (VI) was about 95.2%. The experimental kinetic leaching data were well interpreted with a shrinking core model with diffusion control through a porous product layer.

Kinetic studies of uranium and iron dissolution using sulfuric acid from Abu Zeneima ferruginous siltstone, Southwestern Sinai, Egypt were investigated (Atia et al., 2018). The importance of this study from the fact that, there is a difference in the dissolution rate and activation energy which allows the separation of the two elements from each other through the dissolution units without entering the extraction units. Kinetic data analysis showed that the dissolution mechanism follows the shrinking core model with chemical reaction as a rate determining step with an activation energy (Ea) 31.59 Kj mol$^{-1}$ for uranium and (Ea) 26.02 Kj
mol\(^{-1}\) for iron. The dissolution study showed that dissolution efficiency approximately 81.92% for uranium and 96.94% for iron.

Low grade fluoritized uranium samples from Gabal El-Missikat prospect, Eastern Desert, Egypt was subjected to sulfuric acid leaching (Mohammed et al., 2019). The effects of leaching parameters on uranium dissolution mechanism were investigated. The shrinking core model was used to model leaching reactions. The kinetics equations indicate that the reactions appear to be controlled by layer diffusion process. The activation energy for uranium dissolution was evaluated. Low activation energy value 2.54 Kj mol\(^{-1}\) confirms the diffusion layer mechanism. The extraction efficiency of uranium was about 91%.

In this concern, the main objective of this work is to investigate the kinetics of Wadi Nasib, uranium ore leaching process using sulfuric acid in order to improved understanding the chemical reaction mechanism that is involved in the dissolution of this mineral that influence the leaching process.

2. EXPERIMENTAL

2.1. Materials
The working sample calcareous shale ore material has actually been collected from variably mineralized of Wadi Nasib area, Um Bogma formation Sinai, Egypt. The collected sample has been subjected to chemical analysis involving the major oxides besides the tenor of the economic metal values. Therefore, a representative portion of this sample was properly prepared by crushing, grinding to – 200 mesh size before quartering.

2.2. Control analysis
Chemical analysis of the present ore sample ground to a mesh size of - 200 has been performed. However, the trace elements e.g. Zn, Cu, Pb, Ni, Co, etc. were analyzed using flame atomic absorption spectroscopy FAAS Unicam 969. While the major elements as oxides such as: Fe\(_2\)O\(_3\), Al\(_2\)O\(_3\), MgO and CaO are classically estimated by wet chemistry, (Lyaudet et al., 1987).

On the other hand, 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 (Bodu, 1984).

2.3. Preparation of sulfate leach liquor
Sulfate leach liquor was prepared via agitation leaching of a weighted 250g of the fine ground calcareous shale ore material of Wadi Nasib area using 200 g L\(^{-1}\) H\(_2\)SO\(_4\) acid solution at S/L mixing ratio of 1/4 and stirring for 4h at 80°C. In the filtrate solution, uranium content was determined by titration method.

3. RESULTS AND DISCUSSION

3.1. Mineralogical characterization
The representative studied ore sample was collected from Um Bogma formation at Wadi Nasib area. It was ground to a mesh size ranging from -60 to -150. XRD mineralogical investigations of this sample revealed the presence of minerals such as gypsum, [CaSO\(_4\).2H\(_2\)O]; quartz, (SiO\(_2\)); Malachite, Cu\(_2\)CO\(_3\)(OH)\(_2\); Dolomite, [CaMg(CO\(_3\))\(_2\)]; Brochantite, [Cu\(_4\)(SO\(_4\))\(_6\)]. Azurite, [2CuCO\(_3\).Cu(OH)\(_2\)] and Sklodowskite, [MgO(UO\(_3\)).(SiO\(_2\)).(H\(_2\)O)\(_7\)]. The obtained results revealed the presence of U and Cu minerals as summarized in Table (1).
### Table (1): X-ray diffraction data of the working sample material

| Fraction No. | Identified Minerals | Name      | Chemical formula |
|--------------|---------------------|-----------|------------------|
| 1            | quartz              | SiO₂      |                  |
|              | Azurite             | 2CuCO₃.Cu(OH)₂ |                 |
|              | Brochantite         | Cu₄(SO₄)OH₆ |                 |
|              | Dolomite            | Ca Mg(CO₃)₂ |                 |
| 2            | Quartz              | SiO₂      |                  |
|              | Sklodowskite        | MgO(UO₂)₂(SiO₃)₂(H₂O)₇ |          |
| 3            | Azurite             | 2CuCO₃.Cu(OH)₂ |                 |
|              | Quartz              | SiO₂      |                  |
|              | Gypsum              | CaSO₄.2H₂O |                  |
|              | Dolomite            | Ca Mg(CO₃)₂ |                  |
|              | Malachite           | Cu₂CO₃(OH)₂ |                  |

### 3.2. Chemical composition

Complete chemical composition of Wadi Nasib ferruginous calcareous shale ore material is given in Table (2). This table revealed that, the present ore material is mainly consists of SiO₂, CaO, MgO, Fe₂O₃, CuO and Al₂O₃ as the major oxides beside high concentrations of ZnO, Na₂O and K₂O. As well as considerable concentrations of REEs, Cu and U as the element of interest. The chemical composition reflects the previously mentioned mineralogical composition.

### Table (2): Complete chemical composition of Wadi Nasib calcareous shale sample

| Major oxides | Conc.,% | Trace elements | Conc., mg L⁻¹ |
|--------------|---------|----------------|---------------|
| SiO₂         | 13.0    | U              | 2000          |
| Al₂O₃        | 10.7    | REE            | 1825          |
| TiO₂         | 0.22    | Cu             | 27000         |
| MnO          | 0.40    | Zn             | 5300          |
| Fe₂O₃        | 6.69    | Pb             | 454           |
| CaO          | 25.7    | Ni             | 330           |
| MgO          | 2.9     | Co             | 120           |
| Na₂O         | 0.6     |                |               |
| K₂O          | 0.47    |                |               |
| P₂O₅         | 0.07    |                |               |
| Loss of ignition | 28.5  |                |               |

### 3.3. Leaching investigation

Different effective leaching parameters, stirring speed, H₂SO₄ acid concentration, solid/liquid ratio, leaching time and leaching temperature were investigated. All the leaching experiments were conducted with a constant weight sample of 10 g ground to - 200 mesh.
3.3.1. Effect of stirring speed
This effect was studied at different stirring speed ranged from 150 to 700 rpm while the other applied conditions were kept constant at H$_2$SO$_4$ concentration of 100 g L$^{-1}$, S/L ratio of 1/3 at room temperature (25°C) and stirring for 2 h. The obtained data plotted in Fig. (1) revealed that, the maximum leaching efficiency of U reflected the maximum value, 50% at 400 rpm. Then the steady state was recorded so the applied stirring speed at 400 rpm would be performed.

![Fig. (1): Effect of stirring speed upon uranium leaching efficiency](image)

3.3.2. Effect of H$_2$SO$_4$ acid concentration
Different concentrations of H$_2$SO$_4$ ranged from 100 to 300 g L$^{-1}$ were used to investigate the effect of acid concentrations upon U leaching efficiency from the present ore sample. The other leaching parameters were kept constant at S/L ratio of 1/3 and agitation time of 120 min at room temperature (25°C) at 400 rpm. After washing, filtration and analysis, the obtained data plotted in Fig. (2), show that, the maximum leaching efficiency of U 55% was observed at acid concentration of 200 g L$^{-1}$ then the steady state of leaching efficiency was achieved. The obtained data were explained by the acid consuming due to present of elements as Ca, Mg, Fe and Al. For economic point of view 200 g L$^{-1}$ consider as the optimum leaching concentration.

3.3.3. Effect of S/L ratio
The effect of S/L ratio upon U leaching efficiency was examined at different S/L ratios ranged from 1/1 to 1/6 by using 200 g L$^{-1}$ of H$_2$SO$_4$ and stirring time for 2 h at room temperature (25°C) and at 400 rpm. Fig. (3) indicated that the best leaching efficiency of U, 58% was attained at S/L ratio of 1/4. In the meantime, increasing the acid amount by applying a S/L ratio 1/6, slightly increase the leaching efficiency of U reached to 58.8 Therefore, it can be considered solid/liquid ratio 1/4 is optimum.

3.3.4. Effect of leaching time
Fig. (4) reflected the remarkable influence of varying the agitation leaching time from 20 to 240 min upon U leaching efficiency by using 200 g L$^{-1}$ of H$_2$SO$_4$ at S/L ratio of 1/4, room temperature (25°C) and 400 rpm. The obtained results emphasized that increasing the leaching
time from 40 to 240 min increased the leaching efficiencies of U up to 67.77% thus; 240 min was considered as the optimum leaching time of U.

![Fig. (2): Effect of H₂SO₄ acid concentration upon uranium leaching efficiency](image)

**Fig. (2):** Effect of H₂SO₄ acid concentration upon uranium leaching efficiency

![Fig. (3): Effect of S/L ratio upon uranium leaching efficiency](image)

**Fig. (3):** Effect of S/L ratio upon uranium leaching efficiency

### 3.3.5. Effect of leaching temperature

The leaching temperature changed from 25°C to 90°C. The obtained results in Fig. (5) clearly emphasized the highly improvement in U leaching efficiencies which sharply increased up to 98.5% by increasing the leaching temperature up to 80°C, a slight increase in temperature to reach 90°C the leaching efficiency of the U show a constant state. Therefore, it can be concluded that 80°C leaching temperature would be adequate and considered optimum. Thus, leaching temperature plays a critical role in the leaching process. From the acidic agitation leaching study, it can be concluded that the optimum leaching conditions for dissolving 98.5%
of U are 200 g L⁻¹ H₂SO₄ acid concentration, stirring speed 400 rpm, solid /liquid ratio 1/4 with leaching time 4 h and leaching temperature 80°C.

**Fig. (4):** Effect of time upon uranium leaching efficiency

**Fig. (5):** Effect of temperature upon uranium leaching efficiency

### 3.3.6. Leaching kinetic of uranium

Leaching data was studied in this work to investigate the kinetics of Wadi Nasib, uranium ore leaching process using sulfuric acid in order to improve understanding the chemical reaction mechanism involved in the dissolution of this mineral, where the kinetic of leaching reaction is often described by the shrinking core model.

Traditionally, reactions involving solids and fluids have been modeled using the shrinking core model. The latter envisions a chemical reaction of the form:
F (fluid) + bS (solid) → Products (p)                  (4)

The model has been applied for U which assumes that during the course of the reaction an ash layer is formed around a shrinking core of unreacted solid reactant. As a result of the reaction progresses, the ash layer steadily increases inward towards the shrinking core, until all of the reactant solid reacted (Levenspiel, 1999; Sohn, 2003; Hsu et al., 2009). It is envisioned the major resistance to the reactions are as follows:

i. The diffusion of the reactant fluid F to the surface of the solid through the fluid film surrounding the particle

ii. The diffusion of the reactant fluid through the solid ash layer to the reaction surface at the unreacted core

iii. Reaction of the fluid reactant F at the reaction surface of the unreacted core with solid S

iv. Diffusion of the fluid product from the surface of the unreacted core through the ash layer back to the outer part of the ash layer

v. Diffusion of the fluid product through the fluid film surrounding the particle.

This choice for this model has been applied of U which the main target in our study. Depending on the reaction occurring, one, or a combination of the resistances which control the reaction, and hence can be used to develop a model that will predict how that particular chemical process will proceed with time (Hsu et al., 2009). The reaction will be controlled by one of two possible resistance; the chemical reaction itself, diffusion through the fluid film surrounding the particle, or diffusion through the ash layer of product. But amongst these controlling mechanisms the liquid-film diffusion resistance is eliminated or minimized by effective stirring. So the results in Table (3) and Fig. (6) were analyzed by using the following kinetic rate, equations.

When the chemical reaction controls the process can be modeled as

$$K_c t = 1 - (1 - x)^{1/3}$$  \hspace{1cm} (5)

When diffusion through the solid controls, the reaction can be modeled as

$$K_d t = 1 - 3(1 - x)^{2/3} + 2(1 - x)$$  \hspace{1cm} (6)

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.

The temperature effect was examined in the range of 298 - 363$^o$K under the conditions of 200 g L$^{-1}$ H$_2$SO$_4$, solid/liquid ratio at 1/4, with stirring speed 400 rpm at stirring time 240 min. the results in Table (3) and graphed in Fig. (6) show that the leachability of uranium increases gradually by increasing of time and temperature. The maximum leachability was found to be 98% at 353$^o$K and after leaching time of 240 min.

The relationship between reaction model and leaching time, at different temperatures is given in Fig. (7). 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 (4) vary in the range of 0.0011 – 0.0035 min$^{-1}$ while $K_c$ was between 0.0016 and 0.0031 min$^{-1}$. The $R^2$ values for $K_d$ was 0.925 to 0.988. Such results indicated that linear relationship between $[1 - 3(1 - x)^{2/3} + 2(1 - x)]$ and
leaching time (t) is significant and suggest that the leaching rate of uranium diffusion controlled. While for Kc it was in the range of 0.43 - 0.69. Based on the R² values it can be inferred that the predominant dissolution mechanism of uranium from the Wadi Nasib ore sample is diffusion controlled only. It is inferred that the insoluble oxide minerals (quartz, etc.) associated with uranium play the role of the product layer. This result agrees with the mineralogical analysis for the working Wadi Nasib ore sample.

**Table (3): Effect of temperature on leaching rate of uranium**

| Time, min | 298°K | 323°K | 343°K | 353°K | 363°K |
|-----------|-------|-------|-------|-------|-------|
| 0         | 0     | 0     | 0     | 0     | 0     |
| 20        | 31.4  | 35.7  | 40    | 42.8  | 50    |
| 40        | 41.4  | 47.1  | 54.2  | 57.1  | 60    |
| 60        | 48.5  | 51.25 | 58    | 61.6  | 68    |
| 120       | 58.4  | 60    | 71.4  | 74.2  | 80    |
| 240       | 68.77 | 76.9  | 88.8  | 98.1  | 98.53 |

**Fig. (6):** Plot of Uranium leachability versus leaching time at different temperatures

The apparent activation energy was determined based on the Arrhenius equation:

\[ k = A e^{(E_a/RT)} \]


\[ \ln k = \ln A - E_a/RT \]  \hspace{1cm} (7)

where k is a reaction rate constant, A is the frequency factor in min⁻¹, Eₐ is the apparent activation energy J/mol and R is universal gas constant (8.314 J k⁻¹ mol⁻¹). The ln k versus 1/T plotted in Fig. (8), where the regression analysis showed that the linear relationship is also significant. The apparent activation energy (Eₐ) was, hence determined to be 16.3 KJ mol⁻¹. values it can be
inferred that the predominant dissolution mechanism of uranium from the ore is diffusion controlled. This value is less than the amount reported by (Crundwell, 2013), the activation energy for diffusion-controlled reactions is below 20 Kj mol\(^{-1}\) and it is above 40 Kj mol\(^{-1}\) for chemical controlled reactions.

**Fig (7):** Plot of \([1-3(1-x)^{2/3}+2(1-x)]\) versus time for different temperatures

**Fig. (8):** Plot of \(\ln K\) (min\(^{-1}\)) against reciprocal of absolute temperature (K\(^{-1}\))

\[y = 0.0035x\]
\(R^2 = 0.9879\)

\[y = 0.003x\]
\(R^2 = 0.988\)

\[y = 0.0023x\]
\(R^2 = 0.9829\)

\[y = 0.0014x\]
\(R^2 = 0.9547\)

\[y = 0.0011x\]
\(R^2 = 0.9205\)
The leaching kinetics of uranium of Wadi Nasib ore showed that the rate of dissolution using H$_2$SO$_4$ acid is diffusion controlled and follows the shrinking core model \[1-3(1-X)^{2/3} + 2(1-X) = kdt\] with an apparent activation energy of 16.2 kJ/mol. This data of kinetic leaching of uranium of Wadi Nasieb agrees with others kinetic leaching data of some acidic leaching of uranium ores with acidic leaching for sella uranium ore material, (Khawassek et al., 2016).

Table (4): The apparent rate constant with their coefficient of determination at different Temperatures

| Temperature, (°K) | Apparent rate constant (min$^{-1}$) | Coefficient of determination($R^2$) |
|-------------------|------------------------------------|-----------------------------------|
|                   | Chemical control ($k_c$) | Diffusion control ($k_d$) | Reaction control | Diffusion control |
| 298               | 0.0016                 | 0.0011                          | 0.4377           | 0.9205           |
| 323               | 0.0019                 | 0.0014                          | 0.5307           | 0.9547           |
| 343               | 0.0025                 | 0.0023                          | 0.6926           | 0.9829           |
| 353               | 0.00293               | 0.003                           | 0.7862           | 0.988            |
| 363               | 0.0031                 | 0.0035                          | 0.6988           | 0.9869           |

**Conclusion**

Uranium can be leached from Wadi Nasib ore using sulfuric acid. The optimum leaching conditions for dissolving 98.5% of U are 200 g L$^{-1}$ H$_2$SO$_4$ acid concentration, stirring speed 400 rpm, solid/liquid ratio 1/4 with leaching time 4 h and leaching temperature 80°C.

The leaching kinetics of uranium of Wadi Nasib ore showed that the rate of dissolution using H$_2$SO$_4$ acid is diffusion controlled and follows the shrinking core model \[1-3(1-X)^{2/3} + 2(1-X) = kdt\] with an apparent activation energy of 16.2 kJ/mol.

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