

Article

Effect of Pyrite on the Leaching Kinetics of Pitchblende in the Process of Acid In Situ Leaching of Uranium

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Abstract: In the process of acid in situ leaching of sandstone uranium ore, pyrite, which is a common associated mineral of pitchblende, would inevitably participate in the reaction. Therefore, it is important to study the influence of pyrite on the leaching kinetics of pitchblende. In this study, we compared the difference leaching rates of pitchblende in the systems of sulfuric acid–hydrogen peroxide, sulfuric acid–hydrogen peroxide–pyrite and sulfuric acid–pyrite and studied the influence of temperature and pyrite quantity on the leaching rate of pitchblende. The results show that the leaching process of pitchblende follows the shrinking particle model controlled by a chemical reaction, and the apparent activation energy E_a of the leaching reaction is $(3.74 \pm 0.40) \times 10 \text{ kJ/mol}$. Pyrite itself cannot promote the dissolution of pitchblende; however, it can promote the leaching of pitchblende in the presence of an oxidizer. Increasing the quantity of pyrite in a certain range can increase the leaching rate of pitchblende, and the reaction order of pyrite is 0.36.

Keywords: pitchblende; pyrite; acid leaching mining; shrinking particle model



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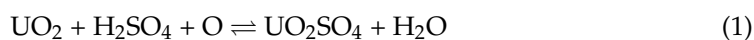
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1. Introduction

Sandstone uranium deposits are of great economic value and strategic significance, and they have been the major object of uranium mining in China nowadays [1], due to their characteristics of shallow burial, large reserves, suitable for in situ leaching and low cost [2,3]. The composition of a sandstone-type uranium ore is complex, including pitchblende (UO_2), coffinite ($\text{U}(\text{SiO}_4)_{1-x}(\text{OH})_{4x}$, ($x < 0.5$)), ningyoite ($\text{Ca}_{2-x}\text{U}_x(\text{PO}_4)_2 \cdot n\text{H}_2\text{O}$, ($x \leq 1$, $n = 1, 2$)) and autunite ($\text{Ca}(\text{UO}_2)(\text{PO}_4)_2 \cdot (10\text{--}12)\text{H}_2\text{O}$). UO_2 often occurs in symbiosis with pyrite (FeS_2), chalcopyrite (CuFeS_2) and marcasite (FeS_2), among which pyrite (FeS_2) is the most common symbiosis mineral in UO_2 deposits [4].

Acid in situ leaching uranium mining is an important uranium mining method, in which the leaching solution is directly injected into the underground ore-bearing strata through drilling, and uranium is obtained by the chemical reaction of the mineral and aqueous solution. Sulfuric acid (H_2SO_4) is widely used in acid in situ leaching, because it is cheaply priced and has a quick reaction [5–7]. In spite of the advantages, such as low production cost and reduced damage to the surface of the ecological environment, a large amount of sulfuric acid injected will reach the underground ore aquifer and cause serious pollution to the groundwater environment. Furthermore, sulfuric acid leaching is a nonselective process resulting in other minerals being dissolved into the groundwater and affects the in situ leaching process. Uranium minerals containing uranyl (e.g., autunite) in the ore-bearing strata can react directly with H_2SO_4 and dissolve, while only a small part of U(IV) can dissolve in H_2SO_4 under natural conditions. However, most of the uranium in sandstone uranium deposits is UO_2 [8]. The leaching of UO_2 first requires oxidizing it

into the U(VI) redox state, where it can more readily dissolve, as shown in Equation (1) [9]. Hydrogen peroxide (H_2O_2) is commonly used as the oxidant [10].



Some research on the effect of iron on the in situ leaching of uranium has been studied. Amme [11] investigated the impact of the reactions between hydrogen peroxide (H_2O_2) and iron ($\text{Fe}^{2+}/\text{Fe}^{3+}$) on UO_2 dissolution in an oxygen-free batch reactor. The interaction in the absence of UO_2 gave a stoichiometric redox reaction of Fe^{2+} and H_2O_2 when the H_2O_2 and Fe^{2+} were present in equal concentrations; however, the predomination of H_2O_2 resulted in the delayed catalytic decomposition of H_2O_2 . With UO_2 present, it either dissolved slowly or precipitated as uranium peroxide ($\text{UO}_4 \cdot n\text{H}_2\text{O}$), depending strongly on the ratio of H_2O_2 and Fe^{2+} . Zhou et al. [12] studied the kinetics of uranium dissolution and migration under the action of an acidic solution containing Fe^{3+} and its relationship with Fe^{3+} . They found that the uranium oxidized by Fe^{3+} migrates from the ore to the solution within 10 h; in addition, the reaction rate of uranium was positively correlated with the transformation rate of Fe^{2+} and Fe^{3+} . When the transformation rate of Fe^{3+} to Fe^{2+} reached zero, the oxidation and dissolution of uranium nearly ceased, and the uranium concentration in the solution achieved an equilibrium. The reaction rate of uranium, $v(\text{U})$, with respect to Fe^{3+} , $v(\text{Fe}^{3+})$, in the solution was shown to follow $v(\text{U}) = 0.0206 + 0.0429 \exp[-v(\text{Fe}^{3+})/5.07]$. Filippov [13] studied the manganese dioxide oxidation of UO_2 in the absence of iron ions, showing that the redox potential cannot be used as the only standard to judge the oxidation rate, and the real reaction rate depends on the reaction mechanism. After the addition of Fe^{3+} , the dissolution percentage of UO_2 and the redox potential rise sharply, which proves that iron ion plays a catalytic role in the process of oxidizing UO_2 . Kinetics can be interpreted as a tool for investigating the rates of chemical reactions and understanding the ways different processes are affected. The most commonly employed method for analyzing the kinetics of uranium leaching is shrinking particles with the shrinking core model [14,15]. There has been no report on the influence of the FeS_2 coexistence in sandstone uranium ore on the leaching of UO_2 under the conditions of a strong acid and oxidant in acid in situ leaching mining. Therefore, this study explores the influence of FeS_2 on the leaching of U from FeS_2 to obtain the basic kinetics of H_2SO_4 .

2. Experiment

2.1. Materials

Both UO_2 and FeS_2 were powders below 74 μm (passed through a 74- μm sieve) from 272 Uranium Industry Co. Ltd., China National Nuclear Corporation. H_2SO_4 and H_2O_2 (30%) were purchased from Hengyang Kaixin Chemical Reagent Co., Ltd (Hengyang, China). The reagents used in the experiment were all analytically pure, and the water used was deionized water.

2.2. Methods

A 250-mL mixed solution of 5 g/L H_2SO_4 and 0.06 mol/L H_2O_2 was added into a 500-mL three-neck flask equipped with a condenser and was heated in an electric thermostatic water bath (Shanghai Kuntian, Shanghai, China) to the desired reaction temperature (15–45 $^\circ\text{C}$). Different amounts of FeS_2 (0.1, 0.4, 0.8 and 1.2 g) and 0.2 g UO_2 were added to the flask. The supernatant was extracted over a range of leaching times and filtered to obtain a 1-mL solution. The uranium concentration was analyzed by an atomic absorption spectrophotometer (Thermo Fisher, Waltham, America). The total iron and Fe^{2+} concentrations were analyzed by a UV spectrophotometer (Beifen-Ruili, Beijing, China); the difference between them was the concentration of Fe^{3+} . The Eh value was measured by a redox potentiometer from Mettler (Zurich, Switzerland). Each experiment was repeated twice, and the average value was used.

Equation (2) was used to calculate the leaching rate of UO_2 ,

$$\eta = \frac{cV}{m_0} \times 100\% \quad (2)$$

where c is the concentration of U in the solution (mg/L), V is the volume of the solution (mL) and m_0 is the initial mass of U in UO_2 (mg).

3. Results and Discussion

In the cases of 5 g/L H_2SO_4 , 0.06 mol/L H_2O_2 and 0.2 g UO_2 at 25 °C, the influence of a pyrite addition amount on different systems of H_2SO_4 - H_2O_2 , H_2SO_4 - H_2O_2 - FeS_2 was investigated. The concentrations of U and Fe under different FeS_2 additions are shown in Figure 1. Without the addition of FeS_2 , the leaching rate of UO_2 was slow. The maximum leaching rate was only 36.88%, with a U concentration of 260.06 mg/L at 360 min. After the addition of 0.1 g FeS_2 , there was no obvious rate change in the initial stage of the reaction; however, the relative rate of the reaction slowly increased after 120 min compared to that without FeS_2 , and the final leaching rate was 48.40% with a U concentration of 341.31 mg/L. With further increases of the FeS_2 mass, the Fe^{3+} ion concentration quickly increased, resulting in an increase of the U concentration. When the Fe concentration was less than 5 mg/L, it had no obvious effect on the UO_2 leaching. When the Fe concentration reached 5 mg/L, the reaction rate of U was obviously higher than that without Fe. When the Fe concentration was about 20 mg/L, the reaction rate of UO_2 reached the maximum. The slope of the U concentration versus time gradually became smaller, indicating that the reaction rate of UO_2 gradually slowed down at the end of the experiment, and the final leaching rates of U were 64.79%, 76.34% and 79.58%, corresponding to the FeS_2 amounts of 0.4 g, 0.8 g and 1.2 g, respectively.

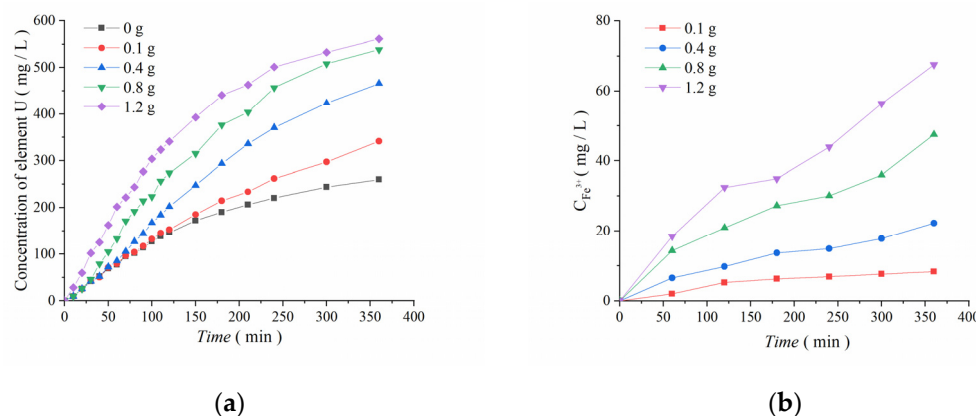


Figure 1. Element U (a) and Fe^{3+} ion (b) concentration changes with time under different pyrite masses.

Figure 2 shows the fitted curves of the uranium leaching rate and the Eh value of the solution at 360 min versus the quantity of FeS_2 . Generally, the uranium leaching rate increased with the pyrite quantity added; however, the acceleration of the pyrite quantity on the leaching rate became small at 360 min. The Eh value decreased with the increase of pyrite, which may have been caused by the consumption of hydrogen peroxide in the dissolution of pyrite. The decrease of Eh, which was attributed to the decrease in the concentration of H_2O_2 , may be the reason why the slope of the uranium leaching rate curve slowed down at the end of the experiment.

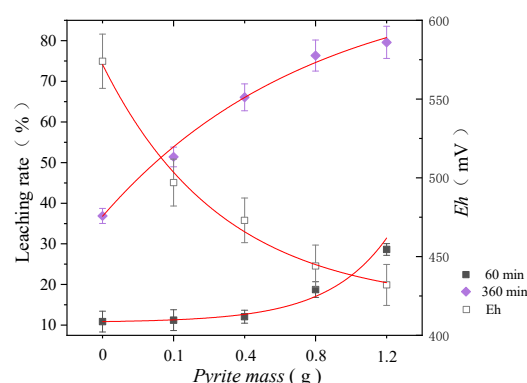
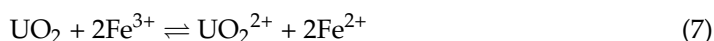
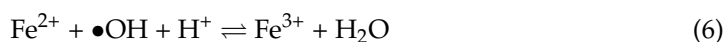
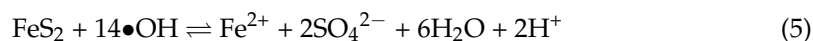
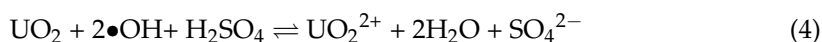


Figure 2. Uranium leaching rate versus the quantity of FeS₂ added.

4. Reaction Mechanism

The following reactions may occur in the reaction system [16–19]:

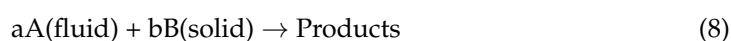


Hydrogen peroxide first dissociates into $\bullet\text{OH}$ (Equation (3)) and participates in the reactions. In the absence of FeS₂, part of UO₂ can be oxidized directly by $\bullet\text{OH}$ (Equation (4)). With the addition of FeS₂, Fe plays an intermediary role in the leaching of UO₂, as shown in Equations (5)–(7). Here, $\bullet\text{OH}$ first oxidizes FeS₂ into 2SO₄^{2−} and Fe²⁺ (Equation (5)) and then Fe²⁺ to Fe³⁺ (Equation (6)), which, in turn, oxidizes UO₂ into the more soluble uranyl (UO₂²⁺) ion (Equation (7)), resulting in U leaching into the surrounding solution. The oxidation of Fe²⁺ by $\bullet\text{OH}$ (Equation (6)) regenerates the Fe³⁺ ion concentration, enabling the further leaching of U through Equation (7).

To prove the above speculation, we analyzed the FeS₂ particles after leaching under the conditions of 5 g/L H₂SO₄, 0.2 g UO₂ and 0.8 g of FeS₂ without H₂O₂ at 25 °C. As we suspected, neither U (VI) nor Fe³⁺ were observed in the solution without the presence of an oxidizer. It shows that FeS₂ itself cannot oxidize UO₂. Since FeS₂ does not contain any oxygen, it cannot be oxidized directly into another species. The only role of FeS₂ in this study was to provide a source of Fe²⁺ ions (Equation (5)), which oxidize to Fe³⁺ (Equation (6)) through the reaction with $\bullet\text{OH}$ from the decomposition of H₂O₂ (Equation (3)). FeS₂ promotes the leaching of UO₂ only in the presence of an oxidizing agent.

5. Apparent Activation Energy and Kinetics Model

In the leaching reaction, the particles shrink, and the surface is not covered with other solids, which conforms to the shrinking particle model (SPM) [14]. According to the leaching kinetics model, uranium leaching is controlled by reactant diffusion and/or a surface chemical reaction. SPM was used to fit the leaching data at different temperatures with the kinetic reaction model [20–22]. For the following reactions:



If the leaching process is mainly determined by diffusion of the reactant inside the solid, the rate expression is:

$$1 - 2\alpha/3 - (1 - \alpha)^{2/3} = kt \quad (9)$$

However, if the leaching process is mainly determined by the fluid–solid chemical reaction, then the rate expression becomes:

$$1 - (1 - \alpha)^{1/3} = kt \quad (10)$$

where k is the apparent reaction rate constant, min^{-1} , and α is the reaction fraction.

The reaction fractions of UO_2 under the conditions of 5 g/L H_2SO_4 , 0.2 g UO_2 , 0.8 g of FeS_2 and 0.06 mol/L H_2O_2 at different temperatures versus time are fitted in Figure 3. It can be seen that Equation (10) can better fit the experiment data, as the maximum R^2 was 0.97 in the fitting results controlled by diffusion of the reactant inside the solid (i.e., Equation (9) and Figure 3a), but all the values of R^2 were greater than 0.99 in the fitting results controlled by the chemical reaction (i.e., Equation (10) and Figure 3b). Therefore, the leaching process was controlled by the chemical reactions.

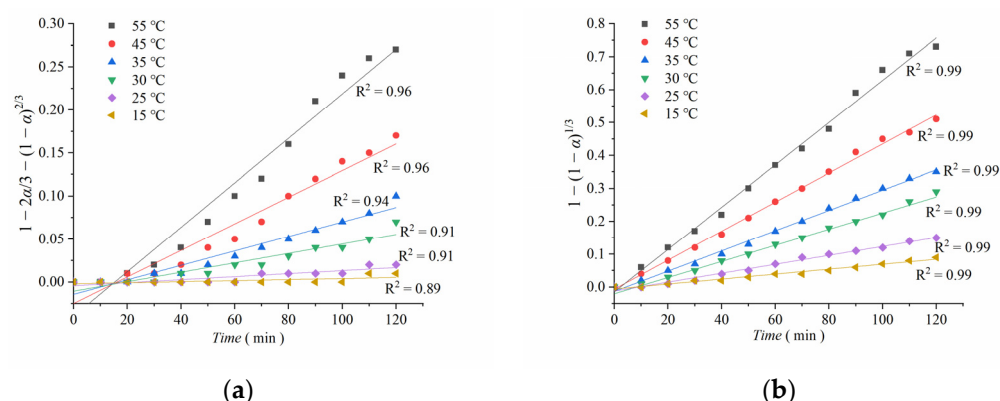


Figure 3. The fit of the diffusion rate expression versus time for (a) diffusion inside the solid and (b) chemical reaction.

The reaction rate constant k obtained from the time-dependent gradients in Figure 3b was substituted into the following Arrhenius equation [10,14]:

$$\ln k = \ln A - \frac{E_a}{RT} \quad (11)$$

where A is the pre-index factor; E_a is the apparent activation energy, kJ/mol; T is the thermodynamic temperature of the reaction, K and R is the molar gas constant ($\text{J mol}^{-1} \cdot \text{K}^{-1}$).

According to the fitting results in Figure 4, we calculated the pre-exponential factor A to be $e^{(9.15 \pm 1.56)} \text{min}^{-1}$ and the apparent activation energy E_a to be $(3.74 \pm 0.40) \times 10 \text{ kJ/mol}$. The evaluated activation energies were lower than $4.86 \times 10 \text{ kJ/mol}$ calculated by Park et al. [23].

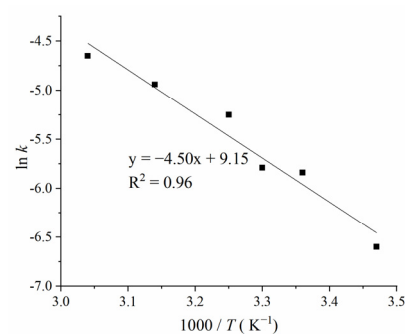


Figure 4. Arrhenius diagram of dissolved UO_2 .

The relationship between the leaching rate constant of UO_2 and the quantity of FeS_2 in the control stage of the chemical reactions can be expressed as:

$$k_m = A \exp\left(-\frac{Ea}{RT}\right) C_0 m^p \quad (12)$$

where k_m is the rate constant based on the amount of pyrite added. C_0 is the constant of the other experimental parameters, m is the mass of FeS_2 and p is the reaction order of FeS_2 .

Set $A \exp\left(-\frac{Ea}{RT}\right) C_0$ as k' , and Equation (12) can then be simplified as

$$k_m = k' m^p \quad (13)$$

Figure 5a displays the $1 - (1 - \alpha)^{1/3}$ versus time with different FeS_2 additions. The slopes of these data are the reaction rate constant of UO_2 , which increases with the increasing amount of the coexistent Fe. This further demonstrates that the addition can accelerate the dissolution of UO_2 . We then plotted the reaction rate constant at different FeS_2 additions in Figure 5b, showing that the influence of a FeS_2 addition on the reaction rate becomes less obvious as the Fe amount increases; this is probably due to the leaching reaction tending towards completion, with an order of the reaction of 0.36.

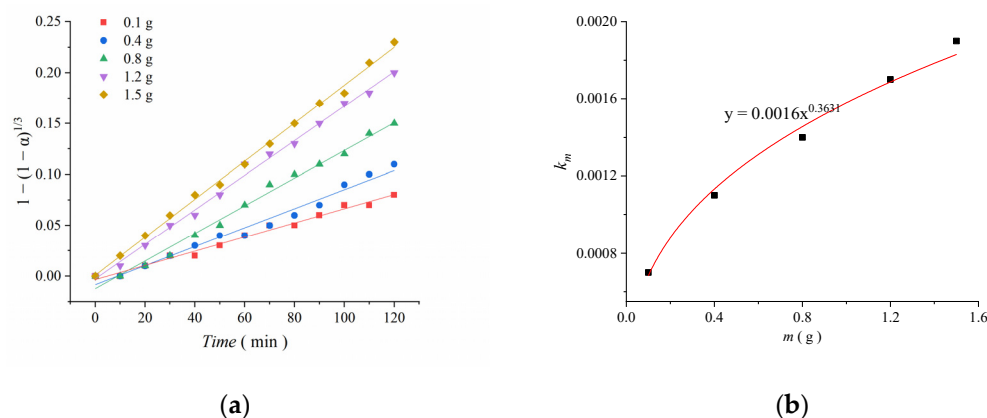


Figure 5. (a) Curve of $1 - (1 - \alpha)^{1/3}$ with time under different FeS_2 masses. (b) Fitting curve of k_m and m .

6. Conclusions

The experiment of UO_2 dissolution in $\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2$, $\text{H}_2\text{SO}_4\text{-H}_2\text{O}_2\text{-FeS}_2$ and $\text{H}_2\text{SO}_4\text{-FeS}_2$ was conducted, and the effect of FeS_2 on the dissolution of UO_2 was investigated.

FeS_2 can promote the dissolution of UO_2 well in the presence of H_2O_2 . When Fe^{3+} is 5 mg/L, the promoting effect of Fe^{3+} can be observed. With the increase of the concentration of Fe^{3+} in the solution, the promoting effect on the dissolution of UO_2 will be more obvious. When Fe^{3+} was 20 mg/L, the reaction rate of UO_2 reached the maximum, and any further increase of the Fe^{3+} concentration could not increase the reaction rate of UO_2 . When the mass of FeS_2 increased from 0 g to 1.5 g, the uranium leaching rate increased by 45.7%. However, FeS_2 cannot promote the dissolution of UO_2 in the absence of an oxidant.

The dissolution of UO_2 was controlled by a chemical reaction, and the apparent activation energy Ea was $(3.74 \pm 0.40) \times 10$ kJ/mol. The leaching process followed the shrinking particle model controlled by the chemical reaction. The reaction order of FeS_2 was 0.36 at 25 °C.

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Data Availability Statement: The data presented in this study are available on request from the corresponding author.

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