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# Kinetics of pressure leaching of niobium ore by sulfuric acid

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#### ARTICLE INFO

### ABSTRACT

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*Keywords:* Niobium ore Pressure leaching Sulfuric acid Kinetics The current study was carried out to investigate the kinetics of pressure leaching of niobium ore in sulfuric acid media. The effects of stirring speed, oxygen pressure, sulfuric acid concentration, particle size and temperature on the leaching efficiency of niobium were studied. It was found that over 98% niobium was leached by mixing the ground ore to  $-74 \,\mu\text{m}$  with sulfuric acid concentration 10 M, and heating for 2 h at 200 °C. The pressure leaching of niobium ore in sulfuric acid solution depends largely on the temperature and follows a surface reaction control kinetic model. An activation energy value of 43 kJ/mol was determined for the temperature range of 100 °C to 200 °C.

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#### Introduction

The niobium resources on the planet are scarce and their grades are low, so that niobium is classified as a rare metal. Niobium has good corrosion resistance, high melting point (2741 K), good thermal conductivity [0.53 J/(cm·s·K), under room temperature], etc., so they are widely used in metallurgy, nuclear, aerospace, and electronic industries [1–3]. Moreover, some alkali metal niobates have been used in laser technology [4,5].

The decomposition of the ore is the key step in extracting niobium from niobium ores, and it has been investigated for many years. Several processing methods have been reported, including hydrofluoric acid leaching, alkali fusion method, etc. Currently, most niobium ores are processed by the hydrofluoric acid method [6]. However, due to its high volatility, about 10% HF is lost during the decomposition process, which is hazardous [7]. Furthermore, a large amount of wastewater containing fluoride is generated that needs to be treated. The alkali fusion method was the earliest one used in industry. In alkali fusion, the niobium ore is mixed with sodium hydroxide or potassium hydroxide, and then the mixtures are roasted. In addition, a certain amount of corresponding carbonate is also added for lowering the melting point and viscosity of smelt. The main disadvantage of this method is the high alkali consumption, which is 6 to 8 times the reaction requirement [8]. In 2005 and 2009, Zhou et al. [9] and Wang et al. [10] proposed sub-molten salt method and mechanical activation-sub-molten method respectively. With these methods, the leaching rate of niobium was 10% higher than that of the hydrofluoric acid process, but the alkali-to-ore ratio

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was as high as 7:1. Moreover, a large amount of energy was consumed in the recycling process of excessive alkali.

However, up to now, the sulfuric acid leaching of niobium ores have not been reported detailedly except by El-Hussaini and Mahdy [11]. El-Hussaini and Mahdy reported that almost complete recovery of niobium was achieved under the conditions of finely ground ore  $-74 \mu m$ , sulfuric acid 10.8 M, nitric acid 5.3 M, ore-to-acid weight ratio 1:3, temperature 200 °C and 2 h. The niobium ores dissolution occurred with the sulfuric acid according to the following reaction:

$$Nb_2O_5 + 2H_2SO_4 = Nb_2O_3(SO_4)_2 + 2H_2O.$$
(1)

However, so far the leaching kinetics of niobium ores by sulfuric acid have not been studied yet, so the present work focuses on investigating the leaching kinetics of niobium ores by sulfuric acid.

Because detailed information on the effect of leaching parameters on the kinetics of the leaching of the niobium ores by sulfuric acid is still not available, the present research was conducted to study such effects on leaching kinetics of the niobium ores in sulfuric acid.

#### Experimental

#### Materials

Niobium ore for the experiments was obtained from Jiangxi Yichun Mine, China. The ore was classified into narrow size fractions using a cyclosizer to obtain various clean size fractions. The chemical analysis of this sample  $(-74 \,\mu\text{m})$  is shown in Table 1. The sulfuric acid was purchased from Ganzhou Jinfu Chemicals.

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Table 1

Elemental analysis of the niobium ore.									
Constitue	nt Nb <sub>2</sub>	0 <sub>5</sub> Ta <sub>2</sub> O	5 FeO	MnO <sub>2</sub>	$Al_2O_3$	SnO <sub>2</sub>	REE	ThO <sub>2</sub>	LOI
Content,	% 14.2	3 1.24	25.09	21.08	21.58	14.36	0.09	0.03	0.42

#### Procedure

All experiments were carried out in a stainless pressure vessel equipped with a mechanical stirrer. A schematic of the reactor is shown in Fig. 1. The experiments were carried out intermittently, where one experiment was performed for each single time interval and the other was designed for the next time interval. For each the exact amount of niobium ore was introduced into the reactor, followed by adding sulfuric acid. After sealing, the reactor was purged and the temperature was raised under argon pressure. After each experiment, the precipitates and the filtrate were separated by a sintered glass. The precipitates were washed by diluted sulfuric acid, dried, and weighed. The niobium content of the precipitate was analyzed by ICP-AES.

The niobium leaching efficiency was calculated by mass balance, as follows:

Leaching efficiency = 
$$\left(1 - \frac{m}{m_o}\right) \times 100$$
 (2)

where m was the weight of niobium, iron, manganese or aluminum in precipitate, and  $m_o$  was the weight of niobium in niobium ore.

#### **Results and discussion**

The experiments were carried out with the niobium ore, to study the main variables that could affect the leaching of niobium: i.e. stirring speed, concentration of sulfuric acid, particle size, oxygen pressure, temperature and time.

#### Effect of stirring speed

The effect of stirring rate on niobium leaching was studied at 3 levels (400 rpm, 500 rpm and 600 rpm). All leaching experiments were carried out at T = 200 °C, P = 0.3 MPa, initial sulfuric acid concentration = 10 M and particle size =  $-74 \,\mu$ m. The results are shown in Fig. 2. Clearly, stirring speed improves leaching efficiency. An increase in stirring speed



Fig. 1. Schematic representation of pressure reactor.



**Fig. 2.** Effect of stirring speed on niobium. Leaching condition: T = 200 °C, P = 0.3 MPa, initial sulfuric acid concentration = 10 M, particle size =  $-74 \mu m$ .

usually increases the rate of leaching due to the suspension of the mineral particles and decreases the thickness of the mass transfer boundary layer on the surface of the particle [12]. Thus, a stirring speed of 600 rpm was used thereafter to assure that the leaching reaction was not under external diffusion control.

#### Effect of oxygen pressure

The effect of oxygen pressure was investigated under three conditions (0.3 MPa, 0.5 MPa and 0.7 Mpa). All tests were performed at T = 200 °C, initial sulfuric acid concentration = 10 M, stirring speed = 600 rpm and particle size =  $-74 \,\mu$ m. Fig. 3 demonstrates the effect of oxygen pressure on the leaching efficiency of niobium. As shown in Fig. 3, the variations in the amount of oxygen pressure had no significant effect on the leaching efficiency under the conditions employed. Oxygen here is required for increasing temperature of the pulp.

#### Effect of sulfuric acid concentration

By applying the sulfuric acid concentration at three levels (5 M, 10 M and 15 M), the effect of sulfuric acid concentration on the leaching of niobium was investigated. All experiments were carried out at T = 200 °C, P = 0.3 MPa, stirring speed = 600 rpm and particle size =  $-74 \mu$ m. The results are presented in Fig. 4. It is clear that the leaching rate increased with the increase in sulfuric acid concentration from 5 M



**Fig. 3.** Effect of oxygen pressure on niobium. Leaching condition: T = 200 °C, particle size  $= -74 \mu m$ , stirring speed = 600 r pm, sulfuric acid concentration = 10 M.

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