



## Leaching kinetics of zinc silicate in ammonium chloride solution

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**Abstract:** The leaching kinetics of zinc silicate in ammonium chloride solution was investigated. The effects of stirring speed (150–400 r/min), leaching temperature (95–108 °C), particle size of zinc silicate (61–150 μm) and the concentration of ammonium chloride (3.5–5.5 mol/L) on leaching rate of zinc were studied. The results show that decreasing the particle size of zinc silicate and increasing the leaching temperature and concentration of ammonium chloride can obviously enhance the leaching rate of zinc. Among the kinetic models of the porous solids tested, the grain model with porous diffusion control can well describe the zinc leaching kinetics. The apparent activation energy of the leaching reaction is 161.26 kJ/mol and the reaction order with respect to ammonium chloride is 3.5.

**Key words:** zinc silicate; ammonium chloride; leaching; kinetics; porous diffusion control

### 1 Introduction

With the depletion of world supplies of high grade zinc sulfides ores, more and more attention will be focused on the extraction of zinc from the zinc oxide ores. Zinc oxide ores are generally found in the forms of carbonate and silicate such as smithsonite ( $\text{ZnCO}_3$ ), hydrozincite ( $2\text{ZnCO}_3 \cdot 3\text{Zn}(\text{OH})_2$ ), zincite ( $\text{ZnO}$ ), willemite ( $\text{Zn}_2\text{SiO}_4$ ) and hemimorphite ( $\text{Zn}_4\text{Si}_2\text{O}_7(\text{OH})_2 \cdot \text{H}_2\text{O}$ ) in the world [1,2]. Within the foreseeable future, the annual production of zinc from non-sulfide sources will exceed 10% of global zinc production. Currently, the leaching agents, such as sulfuric acid [3–5] and ammoniacal solutions [6,7], have been used to extract zinc from zinc oxide ores. Calcium/magnesium carbonate gangues containing in zinc oxide ores cause tremendous increase in acid consumption and gypsum harden in acid-leaching process, while ammonia is an attractive reagent for its no reaction with carbonate gangues, low inventory cost and amenability to regeneration [7]. Ammonia is commonly used in the extraction industry of cobalt, nickel and copper due to the formation of stable metal ammine complexes, leading to higher solubility in most cases. Other harmful impurities such as Fe, Pb, Ca and Mg precipitate as oxides because of their poor complexation

ability with ammonia [8].

The leaching kinetics of zinc silicate is far less studied than that of zinc sulfides and zinc carbonates ores. In acid leaching, TERRY and MONHEMIUS [4] studied the leaching kinetics of both natural and synthetic willemite ( $\text{Zn}_2\text{SiO}_4$ ) and hemimorphite. They found that the acidic dissolution was controlled by diffusion for hemimorphite and primarily controlled by chemical and diffusion step for willemite. ABDEL-AAL [9] studied the leaching kinetics of low grade zinc silicate, and proposed that the process was controlled by diffusion on an “ash” layer and the apparent activation energy was calculated as 13.4 kJ/mol. In alkaline leaching, SANTOS et al [10] studied the leaching kinetics parameters of zinc silicate ores in sodium hydroxide solutions. They found that the leaching process was a progressive reduction in particle size controlled by chemical step with an apparent activation energy of 67.8 kJ/mol, which was in good agreement with the results of CHEN et al [11]. In ammoniacal solution, DING et al [8] found that the Elovich equation typified the dissolution behavior of hemimorphite in ammoniacal solution with an activation energy of 57.6 kJ/mol which was characteristic for a chemically controlled process.

The most significant characteristics of leaching in an ammonium chloride solution is the practically

constant and almost neutral pH, generally in the 6–7 range. This makes the calcium, magnesium, silicon, iron and other minor elements (such as As, Sb, Bi) containing in the minerals remain in the residues during the leaching process [12]. Zinc could be extracted by purification or solvent extraction and electrowinning [12–14]. As the leaching kinetics of zinc silicate in ammonium chloride solutions has not been well studied, the purpose of the present work is to assess the leaching kinetic parameters of the zinc silicate in the ammonium chloride solutions, and it will be a guidance for leaching zinc silicate using ammonium chloride solutions.

## 2 Experimental

### 2.1 Materials

Zinc silicate was synthesized according to Ref. [15]. The X-ray diffraction (XRD) revealed that the sample produced was zinc silicate (Fig. 1). The mineralogical composition is presented in Table 1. Analytical grade of ammonium chloride and deionized water were used in the experiments.

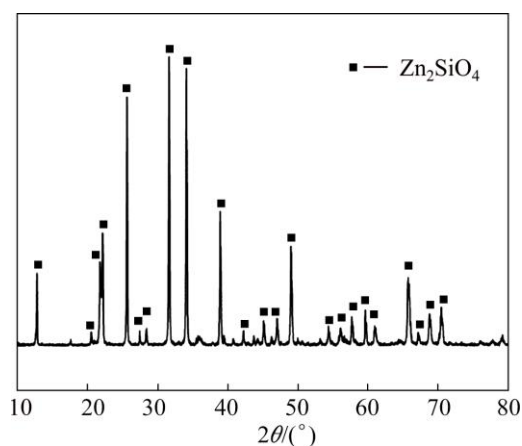


Fig. 1 XRD pattern of zinc silicate

Table 1 Mineralogical composition of zinc silicate

Phase	Mass fraction/%	Occupancy/%
ZnSO <sub>4</sub>	0.08	0.18
ZnO	0.09	0.20
Zn <sub>2</sub> SiO <sub>4</sub>	44.83	99.62
Zn <sub>T</sub>	45.00	100

### 2.2 Leaching methods

The leaching solutions were prepared from reagent-grade ammonium chloride, dissolved in deionized water, according to the desired ammonium chloride concentration (3.5–5.5 mol/L). All experiments were carried out by using zinc silicate particles with 45–61 μm in size, except in experiments where effect of particle size on zinc extraction was investigated. The leaching temperature was in the range of 95–108 °C and the stirring speed was in the range of 150–400 r/min.

The leaching process was carried out in a flat-bottomed spilt flask (2000 mL total volume), with a three-necked top, equipped with a mercury thermometer, a delivery tube for the leaching solutions and a condenser tube which decreased the evaporation loss of the solution. Temperature control of the flask contents within ±0.5 °C was achieved by a thermostat oil bath cauldron. Agitation was provided by a magnetic stirrer that enabled adequate dispersion of the mineral particles. The solution volume was 1000 mL and the solid concentration was 5 g/L. At selected time intervals, a known amount (5 mL) of slurry was withdrawn by sampling port and filtered quickly. 0.5 mL of filtrate was then diluted as 250 mL for zinc analysis using an atomic absorption spectrometer (TAS-990).

The leaching rate of zinc,  $R$ , is defined by

$$R = cV/(m\omega) \quad (1)$$

where  $c$ ,  $V$ ,  $m$  and  $\omega$  are the zinc concentration in filtration, the volume (we assumed it constant), mass and zinc content of the added material, respectively.

The zinc silicate and leaching residues were analyzed by XRD (Japan Rigaku Model TTRIII + 40 kV/250 mA with Cu K<sub>α</sub> radiation), SEM and backscattered electron micrographs (BEM) (Japan Jeol JSM-6360LV).

## 3 Results

### 3.1 Effect of stirring speed on leaching rate of zinc

To investigate the effect of stirring speed on the leaching rate, experiments were carried out at five different stirring speeds in the range of 150–400 r/min. The results, as presented in Fig. 2, show little effect of stirring speed on zinc extraction. Therefore, the leaching process does not seem to be controlled by mass transfer through the liquid boundary layer. As a result, the stirring speed was kept at 350 r/min, unless otherwise stated in this work.

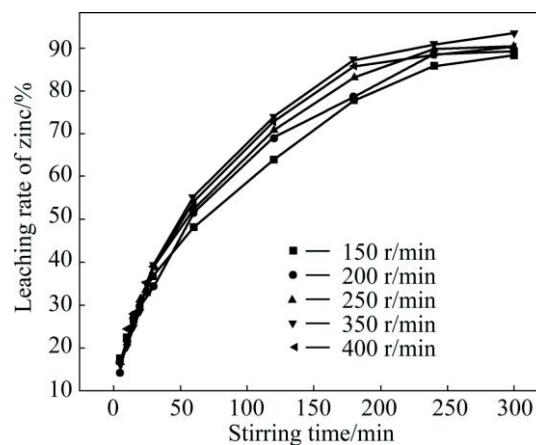
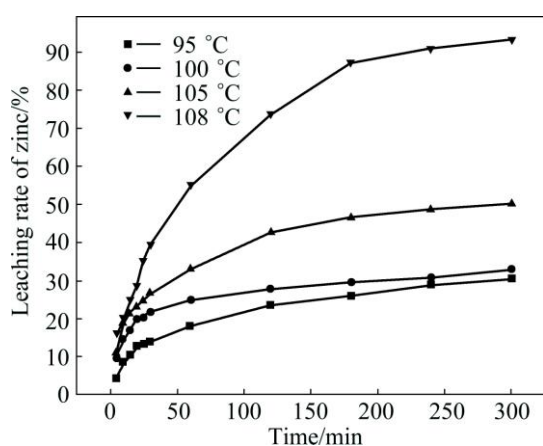


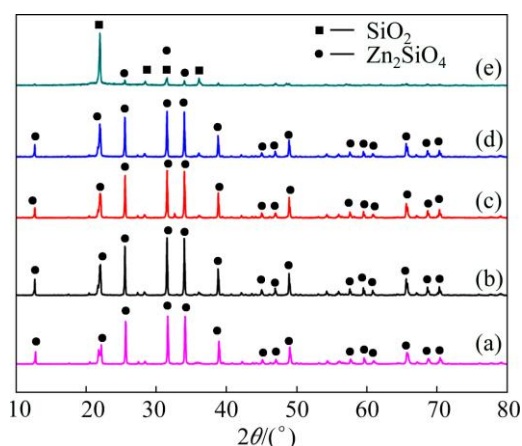
Fig. 2 Effect of stirring speed on leaching rate of zinc (5.0 mol/L NH<sub>4</sub>Cl, solid/liquid ratio 5 g/L, temperature 108 °C and particle size 45–61 μm)

### 3.2 Effect of leaching temperature on leaching rate of zinc

The effect of leaching temperature from 95 to 108 °C on the leaching rate of zinc was studied. The results, as presented in Fig. 3, show that the leaching rate of zinc increases significantly with the increase of temperature. After 300 min, the leaching rate of zinc increases from 50% to 93% as the temperature increases from 105 to 108 °C. Figure 4 shows the XRD patterns of the leaching residues obtained at different leaching temperatures. The major phase of leaching residue is silica dioxide when the leaching temperature is higher than 108 °C.



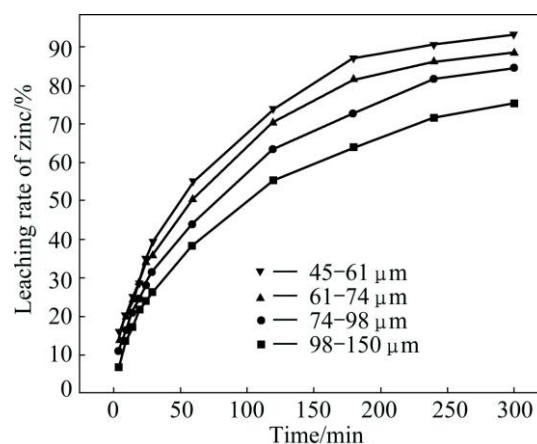
**Fig. 3** Effect of leaching temperature on leaching rate of zinc (5.0 mol/L  $\text{NH}_4\text{Cl}$ , solid/liquid ratio 5 g/L, stirring speed 350 r/min and particle size 45–61  $\mu\text{m}$ )



**Fig. 4** XRD patterns of leaching residues at different leaching temperatures: (a) Material; (b) 95 °C; (c) 100 °C; (d) 105 °C; (e) 108 °C

### 3.3 Effect of particle size on leaching rate of zinc

The influence of particle size from 45–61 to 98–150  $\mu\text{m}$  on the leaching rate of zinc was investigated. The results, as presented in Fig. 5, show that decreasing particle size enhances zinc extraction. As expected, the smaller the size of the particles, the faster the leaching

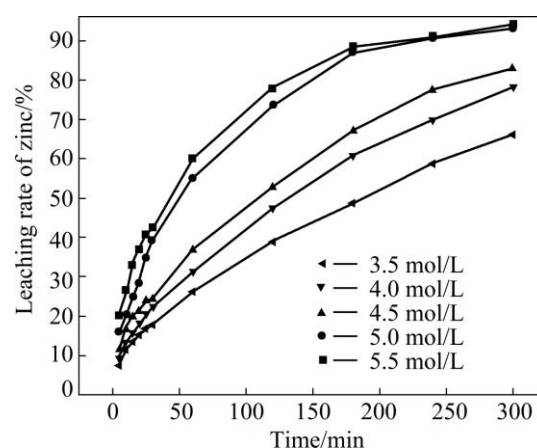


**Fig. 5** Effect of particle size on leaching rate of zinc (5.0 mol/L  $\text{NH}_4\text{Cl}$ , solid/liquid ratio 5 g/L, stirring speed 350 r/min and temperature 108 °C)

rate. It seems that decreasing the particle size can improve the reaction area.

### 3.4 Effect of ammonium chloride concentration on leaching rate of zinc

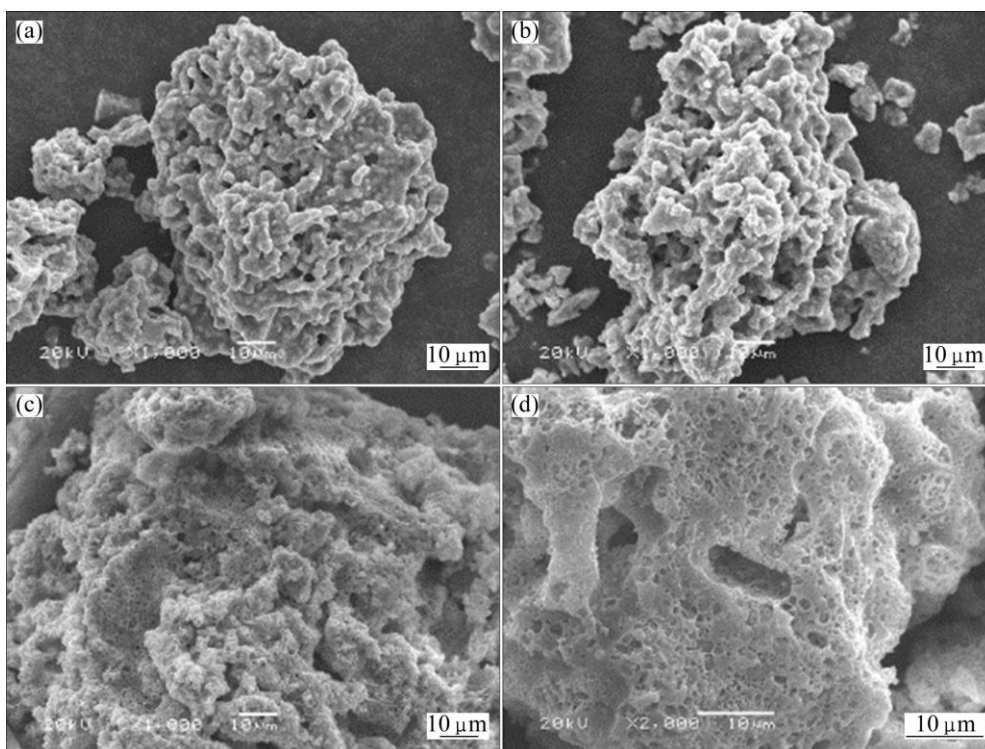
The effect of ammonium chloride concentration on the leaching rate of zinc was studied in the range of 3.5 to 5.5 mol/L. The results, as presented in Fig. 6, show that the leaching rate of zinc increases significantly with increasing the ammonium chloride concentration. About 94% of zinc is extracted in 5.5 mol/L ammonium chloride solution after 300 min.



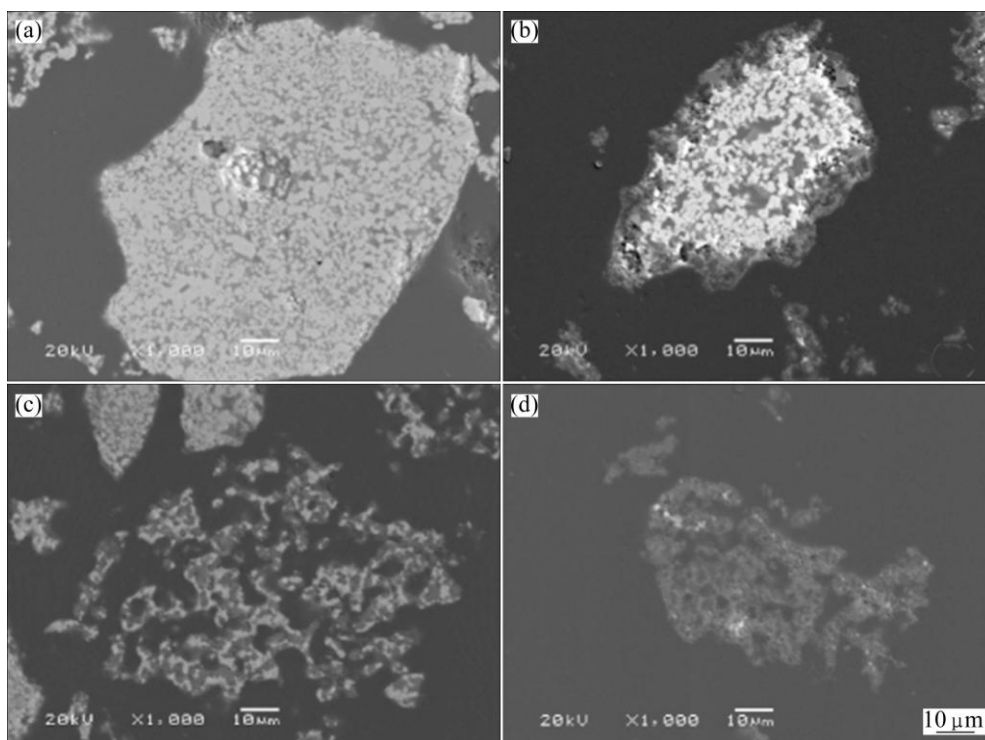
**Fig. 6** Effect of ammonium chloride concentration on leaching rate of zinc (Temperature 108 °C, solid/liquid ratio 5 g/L, stirring speed 350 r/min and particle size 45–61  $\mu\text{m}$ )

### 3.5 Characterization of leaching residues

The zinc silicate samples before and after leaching were characterized by SEM (see Fig. 7) and BEM (see Fig. 8). The solid particles present a rough and porous surface, as shown in Fig. 7(a). The micrographs of the leaching residues exhibit a further increase in the



**Fig. 7** SEM images of zinc silicate before leaching (a), after 30% leaching rate of zinc (b), after 75% leaching rate of zinc (c), and after 93% leaching rate of zinc (d)

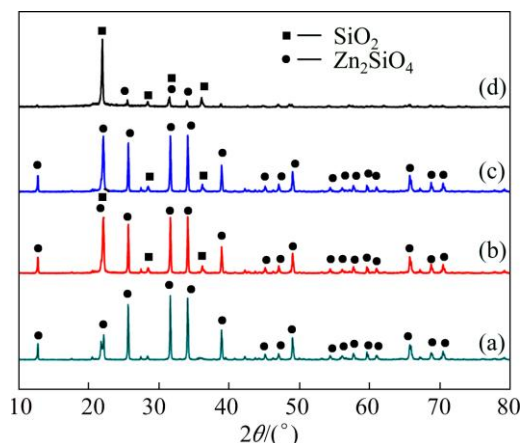


**Fig. 8** BEM images of zinc silicate (polished section) before leaching (a), after 30% leaching rate of zinc (b), after 75% leaching rate of zinc (c) and after 93% leaching rate of zinc (d)

roughness and porosity of the solid. For instance, comparing Figs. 7(c) and (d) with (a) reveals the occurrence of more holes on the surface of the solid particles. The BEM images (Fig. 8) of the polished

section of the zinc silicate before and after leaching exhibit that the reticulate structure appears but no reaction product layer occurs on the particle surface. Figure 9 shows the XRD patterns of the leaching

residues at different reaction time. Therefore, it is deduced that the zinc leaching process might be controlled by diffusion of the reagent in the porous structure of the particles, as observed by GEORGIOU and PAPANGELAKIS [16] in pressure leaching experiments carried out with limonitic laterite ores.



**Fig. 9** XRD patterns of leaching residues at different leaching time: (a) Material; (b) 0.5 h leaching; (c) 2 h leaching; (d) 5 h leaching

#### 4 Kinetic discussion

The comparison of the SEM (Fig. 7) and BEM images (Fig. 8) of the zinc silicate and the leaching residues shows that the residues become very porous with an increase in leaching rate of zinc. This suggests that the leaching process can be described according to the shrinking core model. The reaction rate might be controlled by chemical reaction on the particle surface, diffusion through the inert particle pore, diffusion through liquid film on the surface of the particle, or a combination of two or three of these processes [17].

When the surface chemical process alone is the rate limitation step, the following expression of the shrinking core model can be used to describe the leaching kinetics of the process:

$$1-(1-R)^{1/3}=k_R t, \quad k_R = \frac{bk[c(\text{NH}_4\text{Cl})]^n}{\rho_{\text{silicate}} r_0} \quad (2)$$

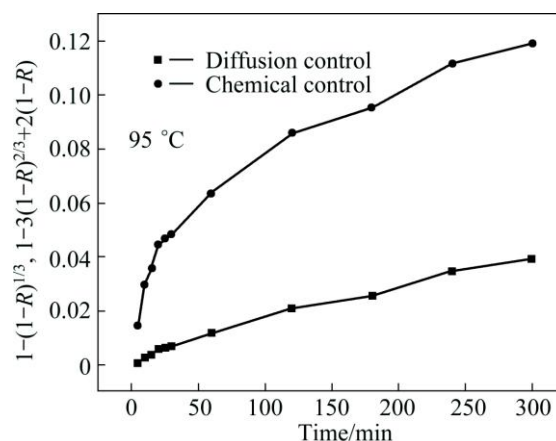
where  $k_R$  is the reaction rate constant;  $t$  is time;  $R$  is the reacted fraction of zinc;  $b$  is the stoichiometric coefficient;  $k$  is the chemical rate constant;  $c(\text{NH}_4\text{Cl})$  is the concentration of ammonium chloride;  $n$  is the reaction order;  $\rho_{\text{silicate}}$  is the silicate molar density; and  $r_0$  is the initial particle radius.

Similarly, when the diffusion of the reagent through a product layer is the rate-controlling step, the following expression of the shrinking core model is achieved to describe the dissolution kinetics:

$$1-3(1-R)^{2/3}+2(1-R)=k_d t, \quad k_d = \frac{6bD_{\text{eff}}[c(\text{NH}_4\text{Cl})]^n}{\rho_{\text{silicate}}} \quad (3)$$

where  $k_d$  is the apparent diffusion rate constant for nonporous particles; and  $D_{\text{eff}}$  is the effective diffusion coefficient.

Figure 10 presents the fitting curves of the shrinking core model with chemical reaction control and product diffusion control to the experimental data. It shows that the model for product diffusion control is better than that for chemical reaction control. However, the dissolution of the zinc silicate occurs without the formation of a product layer, as shown in Figs. 7 and 8. Therefore, this model is not consistent with the SEM and BEM analysis of the leaching residues. Thus, a different model, which takes into account the effect of porosity, should be built so that the zinc silicate leaching can be adequately described.



**Fig. 10** Fitting shrinking core models with chemical reaction control and product diffusion control (conditions as shown in Fig. 3)

Several models have been proposed to describe the leaching kinetics of porous solids, such as the random pore model, the uniform pore model and the grain model. The random pore model could not be applied to representing the leaching process as observed elsewhere [18]. The uniform pore model with chemical control was also tested to describe the leaching kinetics, but did not produce a good fit to the experimental data, as observed in other hydrometallurgical systems [18,19]. The grain pore model will be discussed below [5].

The grain model can also be applied to describing the leaching kinetics of porous solids. The model gives the following expression for spherical particles:

$$1-3(1-R)^{2/3}+2(1-R)=k_D t, \quad k_D = \frac{3bD_{\text{eff}}[c(\text{NH}_4\text{Cl})]^n}{r_0^2(1-\varepsilon_0)\rho_{\text{silicate}}} \quad (4)$$

where  $k_D$  is the reaction rate constant, and  $\varepsilon_0$  is the initial porosity of the solid. The plot of  $1-3(1-R)^{2/3}+2(1-R)$



versus leaching time,  $t$ , at different temperatures, is given in Fig. 11. As shown in Fig. 11, a good fit is observed when  $1-3(1-R)^{2/3}+2(1-R)$  is applied to the experimental data. Figure 12 shows the Arrhenius plot constructed with the reaction rate constant value,  $k_D$ , calculated from the data presented in Fig. 11. The calculated apparent activation energy determined for the pore diffusion control is 161.26 kJ/mol.

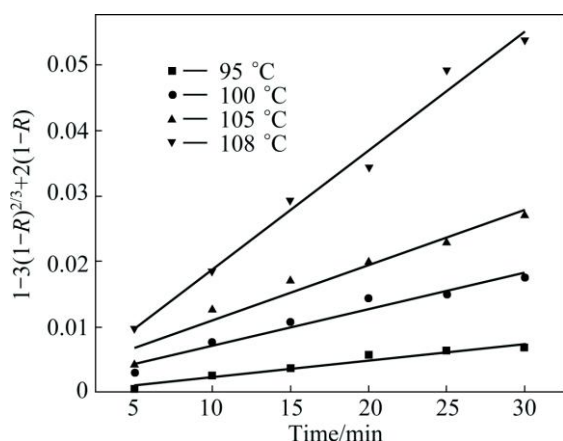


Fig. 11 Plot of  $1-3(1-R)^{2/3}+2(1-R)$  vs  $t$  at different temperatures

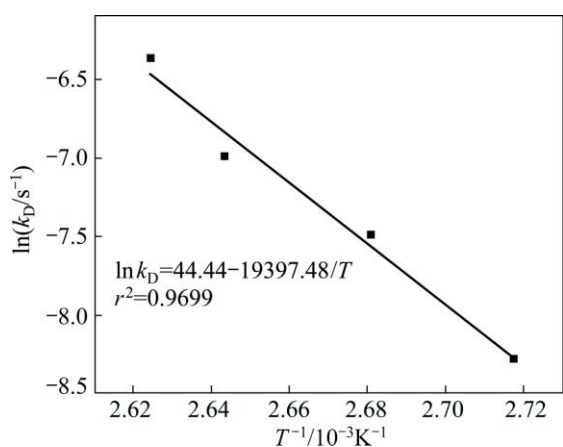


Fig. 12 Arrhenius plot for leaching of zinc silicate

The dissolution of zinc occurs as a consequence of the formation of ternary complexes in neutral ammonium chloride solutions with the general formula of  $ZnCl_n(NH_3)_{4-n}^{2-n}$  [20]. The ion radius of ternary complexes is larger than that of single ion  $Zn^{2+}$ , which results in the lower diffusion speed, thus the higher apparent activation energy of the pore diffusion control is obtained than that in other leaching media (Table 2).

According to the data of Fig. 5, the plot of Eq. (4) vs leaching time,  $t$ , for different particle sizes is given in Fig. 13. Clearly, the model also fits well. Furthermore, from the analysis of Eq. (4), there is a clear dependence of the reaction rate constant,  $k_D$ , with the inverse square of initial particle radius ( $1/r_0^2$ ).

Figure 14 shows the plot of  $k_D$  vs  $1/r_0^2$  that is obtained from the linear fitting of the data in Fig. 5 into Eq. (4). As shown in Fig. 14, a linear relationship between the rate constant and the square of the inverse of

Table 2 Selected values of activation energies reported for leaching of zinc silicates

Experimental condition	Model-controlling step	Activation energy/(kJ mol <sup>-1</sup> )	Ref.
Willemite, franklinite (43.5% Zn, 23.3% SiO <sub>2</sub> ), 0.2–1.0 mol/L H <sub>2</sub> SO <sub>4</sub> ; 30–60 °C, 1% solid, 2.5 m <sup>2</sup> /g	Grain pore model GPM-pore diffusion control	51.9	[5]
Willemite, franklinite (35.6% Zn, 23.3% SiO <sub>2</sub> ), 0.2–1.0 mol/L H <sub>2</sub> SO <sub>4</sub> ; 10–50 °C, 1% solid, 0.6 m <sup>2</sup> /g	GPM-pore diffusion control	78.2	[21]
Willemite, franklinite (46.9% Zn, 3.2% Fe, 29.6% SiO <sub>2</sub> ), 0.2–1.0 mol/L H <sub>2</sub> SO <sub>4</sub> ; 30–70 °C, 1% solid, 0.5 m <sup>2</sup> /g	GPM-pore diffusion control	67.0	[21]

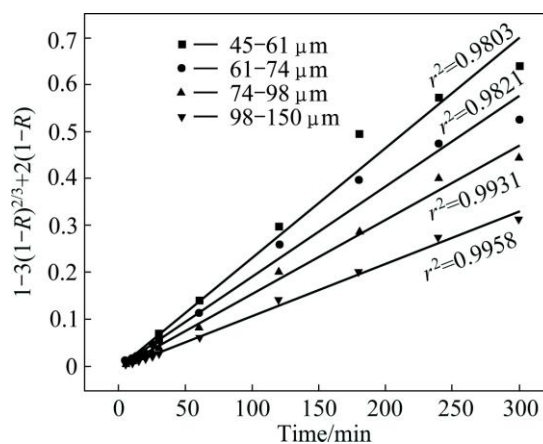


Fig. 13 Plot of  $1-3(1-R)^{2/3}+2(1-R)$  vs  $t$  for different particle sizes

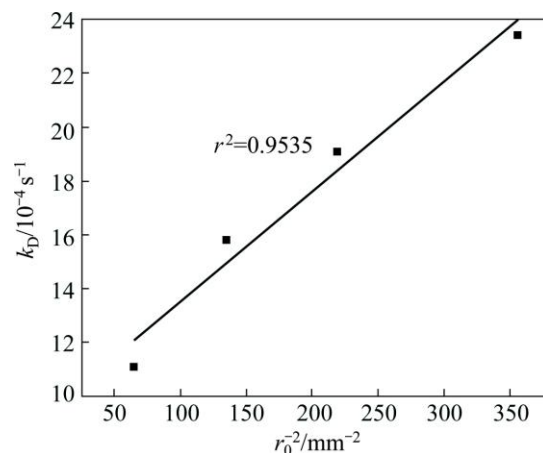


Fig. 14 Plot of  $k_D$  vs  $1/r_0^2$

the initial particle radius,  $1/r_0^2$ , is obtained. This figure might support the application of the grain model with pore diffusion control to describe the dissolution kinetics of the zinc silicate.

According to the data in Fig. 6, the plot of Eq. (4) vs leaching time,  $t$ , at different ammonium chloride concentrations is given in Fig. 15, which is in good agreement with the grain model. Using the fitted  $k_D$  values, plot of  $\ln k_D$  vs  $\ln[c(\text{NH}_4\text{Cl})]$  at different ammonium chloride concentrations is given in Fig. 16. The slope of the regressive line is 3.5, which is equal to the reaction order of ammonium chloride.

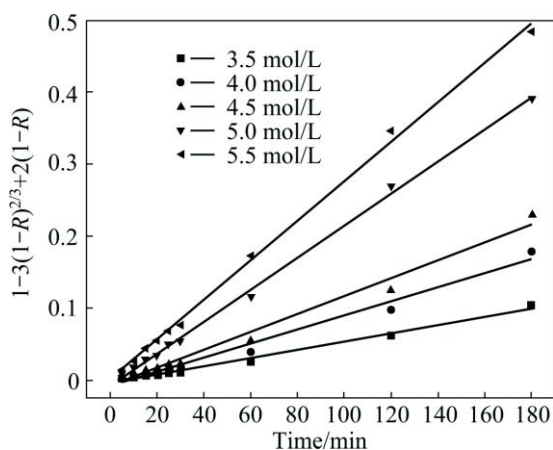


Fig. 15 Plot of  $1-3(1-R)^{2/3}+2(1-R)$  vs  $t$  at different ammonium chloride concentrations

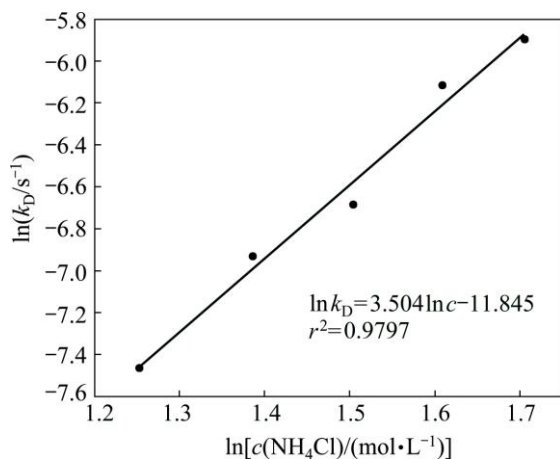


Fig. 16 Plot of  $\ln k_D$  vs  $\ln[c(\text{NH}_4\text{Cl})]$

## 5 Conclusions

1) The leaching kinetics of synthesized zinc silicate in ammonium chloride solutions was studied. The results reveal that the leaching rate of zinc is enhanced slightly with increasing stirring speed but increases significantly with the increase of specific surface area, temperature and ammonium chloride concentration.

2) The shrinking core model with diffusion control fits the experimental results, but it could not physically

represent the leaching kinetics.

3) The grain model, which can successfully describe the dissolution of zinc in ammonium chloride solutions, is proposed since no reaction products occurred on the particle surfaces according to the analysis of SEM and BEM. The reaction rate constant has a linear relationship with the square of the inverse of the initial particle radius.

4) The apparent activation energy of the leaching reaction and reaction order with respect to ammonium chloride are determined as 161.26 kJ/mol and 3.5, respectively.

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## 硅酸锌在氯化铵溶液中的浸出动力学

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**摘 要:** 研究硅酸锌在氯化铵溶液中的浸出动力学, 讨论搅拌速度(150~400 r/min)、浸出温度(95~108 ℃)、硅酸锌粒度(61~150 μm)以及氯化铵浓度(3.5~5.5 mol/L)对锌浸出率的影响。结果表明, 减小硅酸锌粒度、提高浸出温度和氯化铵浓度可以显著地提高锌的浸出率。在多孔颗粒的动力学模型中, 颗粒模型的孔隙扩散控制能很好地描述锌的浸出动力学。浸出反应的表现活化能为 161.26 kJ/mol, 氯化铵的反应级数为 3.5。

**关键词:** 硅酸锌; 氯化铵; 浸出; 动力学; 孔隙扩散控制

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