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Dissolution kinetics of hemimorphite in methane sulfonic acid

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Abstract: Hemimorphite has a large content of zinc, but its recovery using flotation alone is low. Nowadays, hydrometallurgical and pyrometallurgical methods are used to treat zinc ores. In this work, the leaching and dissolution kinetics of hemimorphite by using methane sulfonic acid (MSA) as an alternative leaching reagent was investigated. The effects of several experimental parameters including reaction temperature, MSA concentration, particle size, and stirring speed were also analyzed. Results showed that zinc leaching increased with increased reaction temperature, MSA concentration, and stirring speed, as well as decreased particle size. The mechanism of hemimorphite dissolution in MSA solutions may be a new variant of the shrinking-core model. Based on experimental data and kinetics, the apparent activation energy was determined to be 49.50 kJ/mol. The rate of reaction equation was also obtained to describe the process and found that the MSA concentration largely influenced the leaching of hemimorphite.

Keywords: hemimorphite, methane sulfonic acid, dissolution, kinetics

1. Introduction

Zinc, as the most important nonferrous metal following copper and aluminum, has good physical and chemical properties, including high ductility, corrosion resistance, and wear resistance. Zinc is also widely applied in the production of alloys, rubber, die casting, galvanizing, medical, and chemical engineering (Liu, et al., 2012; Feng et al., 2015; Chen et al., 2016; Feng et al., 2017; Li et al., 2013). Similar to copper and lead, zinc metal is produced mostly from zinc sulfide ores because sulfides are easy to separate from gangue minerals by flotation techniques (Feng et al., 2017, 2018). Zinc sulfide ores are being rapidly exhausted, so zinc metal production from oxidized zinc ores are becoming increasingly important. Oxidized zinc minerals mainly contain zincite ZnO), smithsonite ($ZnCO_3$), hydrozincite ($Zn_5(CO_3)_2(OH)_6$), willemite (Zn_2SiO_4), hemimorphite ($Zn_4Si_2O_7(OH)_2 \cdot H_2O$), and zincsilite ($Zn_3Si_4O_{10}(OH)_2 \cdot 4H_2O$) (Yin et al., 2010; Wu et al., 2015; Vida et al,. 2009; Yang et al., 2016).

Flotation is an effective method for the recovery of oxide minerals using different surfactant as collector and lead ions as activator (Liu et al., 2016; Feng et al., 2017). Extensive investigations have been conducted on the treatment of zinc silicate ores using hydrometallurgical and pyrometallurgical methods. Pyrometallurgical method can treat low-grade ores; however, this method is hardly accepted because of some shortcomings, such as heavy pollution, high capital investment, and high energy consumption. Hydrometallurgical method, on the other hand, has been used to extract Zn through various leaching agents, such as acids, caustic soda, sodium hydroxide, and ammoniacal solution (Chen et al., 2009; Ding et al., 2010; Yuan et al., 2010). Sulfuric acid is the most common leaching reagent used to leach Zn. However, for the low-grade zinc ores containing high Fe-bearing and carbonate minerals contents, the use of sulfuric acid will result in high acid consumption, poor leaching, and complex purification process (Mahdi et al., 2013; Zhao et al., 2000). Xu et al. (2012) used sulfuric acid to leach complex sulfide and silicate-containing zinc ore. Their results indicated that complex zinc ore can be

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easily dissolved using sulfuric acid as leaching reagent when the temperature is 413 K and the leaching time is 150 min. In addition, the zinc extraction was above 97% and leachate contained minimal amount of impurities, such as iron and silica. A series of acid leaching of hemimorphite ores was also conducted by several researchers (Terry et al., 1983; Xu et al., 2014; Rao et al., 2017). The hemimorphite dissolution kinetics and the effects of temperature, pH, acid type, and surface area were further investigated in acidic solutions. The results showed that hemimorphite is considerably more reactive toward mineral acids compared to willemite, and the surface chemical reaction rate for hemimorphite dissolution is significantly higher than willemite. Thus, the leaching process is predominantly diffusion-controlled for hemimorphite (Terry et al., 1983). Xu et al. (2014) investigated the hemimorphite dissolution kinetics and mathematical model in dilute sulfuric acid solution at high temperature. Their results showed that zinc leaching is influenced by temperature, sulfuric acid concentration, particle size, and stirring speed. In addition, the leaching process is controlled by the shrinking core model and shrinking core model layer diffusion. The activation energy was E=44.90 kJ/mol in the temperature range of 373– 413 K. The thermodynamic simulations revealed that the partially dissociated protons and amino carboxylate anions synergistically affect the hemimorphite dissolution. Rao et al. (2017) used iminodiacetic acid to leach Zn from refractory hemimorphite. Their experimental results showed that Zn can be dissolved in the leaching solution selectively and efficiently using iminodiacetic acid as a complexing agent. The leaching extraction of zinc and iron are 88.15% and 2.82%, respectively; the isoelectric point of iminodiacetic acid also was recovered by adding dilute sulfuric acid. Thus, the process used in this study is an environmental friendly.

Yuan et al. (2010) investigated hemimorphite alkaline leaching, and their results showed that the zinc recoveries of unmilled, dry milled, and mechanochemically milled hemimorphite are improved with increase in the reaction time to 420 min. Zhao et al. (2017) studied the hemimorphite leaching kinetics in ammonium chloride solution; the leaching time used is 160 min under optimal condition, and the leaching process can be successfully described by the Elovich equation. The leaching kinetics of hemimorphite in ammonium chloride solution with apparent activation energies of 405.14 kJ/mol in leaching temperature ranging from 348 K to 363 K and 239.61 kJ/mol in leaching temperature ranging from 363 K to 381 K is a chemically controlled process.

The objective of the present work is to provide an alternative method to dissolve insoluble hemimorphite. Methane sulfonic acid (MSA) is an organic leaching reagent, which has been used to leach copper-, lead-, and zinc-bearing minerals through the formation of soluble metal methane sulfonate (Feng et al., 2015; Wu et al., 2014). We used MSA to dissolve the refractory hemimorphite to investigate the leaching reagents and kinetics of this leaching process. The effects of complexing agent concentration, reaction temperature, particle size, and stirring speed on the hemimorphite dissolution ratio are also evaluated.

2. Experimental

2.1. Material and dissolution procedure

Hemimorphite samples were obtained from Yunnan, China after the manual removal of gangue minerals. Chemical analysis of the sample showed that hemimorphite contains 54.71% Zn, indicating the high purity of the hemimorphite samples. The chemical compositions of materials are shown in Table 1. The X-ray diffraction analyses of materials are shown in Fig. 1, showing that only hemimorphite was detected. The MSA agents used in this study were of analytical grade. Pure deionized water was used for all experiments; in addition, the zinc concentration was determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

All dissolution experiments were conducted in a laboratory apparatus, which is composed of a 500 cm^3 wide-mouth beaker batch reactor, digitally controlled mechanical stirrer, condenser to prevent evaporation loss, thermometer to control the temperature, and thermostatically controlled water bath for heating. For each experiment, the solid/liquid ratio was maintained at a constant 1:100 g/cm^3, i.e., 5 g of hemimorphite with the required particle was stirred in 500 cm^3 of freshly prepared MSA solutions of various concentrations and heated to the required temperature. A 5 cm^3 portion of the resulting solution was accurately withdrawn at appropriate time intervals and was subjected to quantitative analysis to the Zn aconcentration by using ICP-MS.

Table 1. Chemical composition of materials/%

Component	Zn	Fe	Pb	SiO_2	CaO	MgO
Content	54.71	0.02	0.01	22.85	0.275	0.116

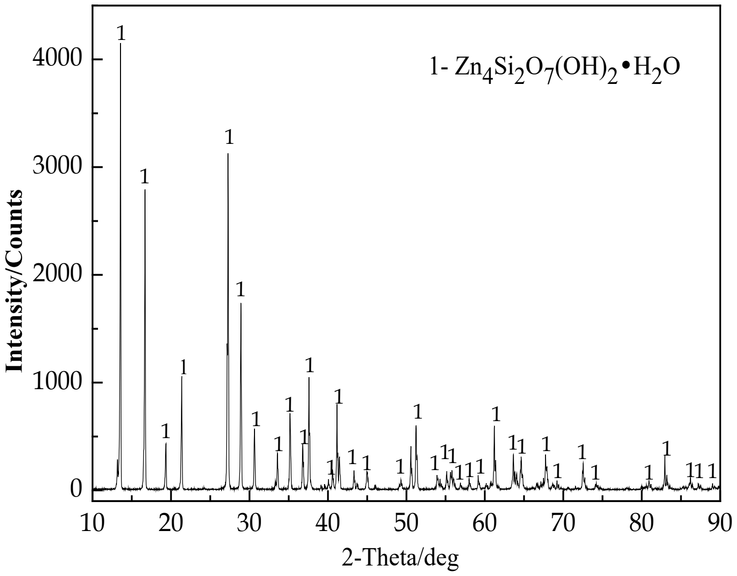

Fig. 1. XRD pattern of pure hemimorphite samples

2.2. Dissolution principle

Hemimorphite [$Zn_4Si_2O_7(OH)_2 \cdot H_2O$] can be dissolved in MSA (CH_3SO_3H) solutions, resulting to the formation of a water-soluble salt. The reaction between hemimorphite and MSA may be expressed as follows:

$$Zn_4Si_2O_7(OH)_2 \cdot H_2O(S) + 8CH_3SO_3H(aq) \rightarrow 4Zn(CH_3SO_3)_2(aq) + Si_2O(OH)_6(aq) + 3H_2O(l). \quad (1)$$

3. Results and discussion

3.1. Effect of temperature

The effect of reaction temperature on zinc extraction as a function of time is show in Fig. 2(a). Samples with particle size in the range of 125–177 μm (it is a middle particle size used in this work) were dissolved in the initial concentration of 0.150 mol/dm^3 and stirring speed of 500 rpm. The solid/liquid ratio was kept constant at 1:100 g/cm^3. As shown in Fig. 2(a), zinc extraction was increased with increased reaction temperature, which significantly affected zinc extraction. At the reaction temperature of 308 K, the leaching rate of zinc reached up to 92.22% in dissolution time of 5 min, whereas only 79.41% Zn was leached within the same dissolution time at 293 K.

3.2. Effect of MSA concentration

Leaching reagent concentration is a very important factor affecting the dissolution kinetics. Each test lasting for 5 min in several experiments were conducted to determine the effect of MSA concentration on hemimorphite dissolution with particle size, reaction temperature, and stirring speed kept at 125–177 μm (it is a middle particle size used in this work), 298 K, and 500 rpm, respectively. The MSA concentration is in the range of 0.125– 0.199 mol/dm^3. As shown in Fig. 2(b), the zinc leaching fraction was increased with an increase in the initial MSA concentration because more leaching reagents can react with the mineral particle. At a MSA concentration of 0.125 mol/dm^3, 72.33% Zn was leached. On the other hand, 93.88% Zn was extracted within the same dissolution time at a MAS concentration of 0.199 mol/dm^3; in addition, hemimorphite was almost completely dissolved. Thus, the initial MSA concentration is a major factor to dissolve hemimorphite. Moreover, when hemimorphite samples interact with MSA, the leaching agents will be consumed with reaction time resulting in the reduction of MSA concentration, in the interaction, which slows down the hemimorphite leaching.

3.3. Effect of particle size

Samples with experimental particle sizes in the range of 250–177, 177–125, 125–88, and 88–74 µm were used in the dissolution experiments at the initial MSA concentration of 0.150 mol/dm^3, reaction temperature of 298 K, and stirring speed of 500 rpm; the results are shown in Fig. 2(c). On the basis of the results shown in Fig.2c, the zinc dissolution rate was evidently increased as the particle size decreased. Within the particle sizes ranging from 74 µm to 250 µm with a decrease in the particle size, the contact area between the particles and fluid were increased. The zinc leaching rates obtained after 5 min of reaction were 75.94% at 250 µm to 174 µm and 92.37% at 88−74 µm particle size. Therefore, the particle size significantly affected the zinc dissolution.

3.4. Effect of stirring speed

Hemimorphite samples with particle size in the range of 125–177 µm were used in the dissolution process within the stirring speed of 250, 500, and 750 rpm. The initial MSA concentration and reaction temperature constant were 0.15 mol/dm^3 and 298 K, respectively; the results are shown in Fig. 2(d). After 5 min of dissolution, the experimental proved that the effect of stirring speed in the zinc leaching rate. The zinc leaching rate was increased from 71.77% to 89.78% when the stirring speed increased from 250 rpm to 750 rpm, which indicated that the stirring speed would affect the dissolution rate. When the stirring speed is low, the mineral particles cannot be dispersed in the solutions and remains at the bottom of the reactor; thus, the zinc dissolution efficiency was influenced. However, no significant difference was observed with further increase in the stirring speed, which can be attributed to the fact that the mineral particles have already been fully suspended.

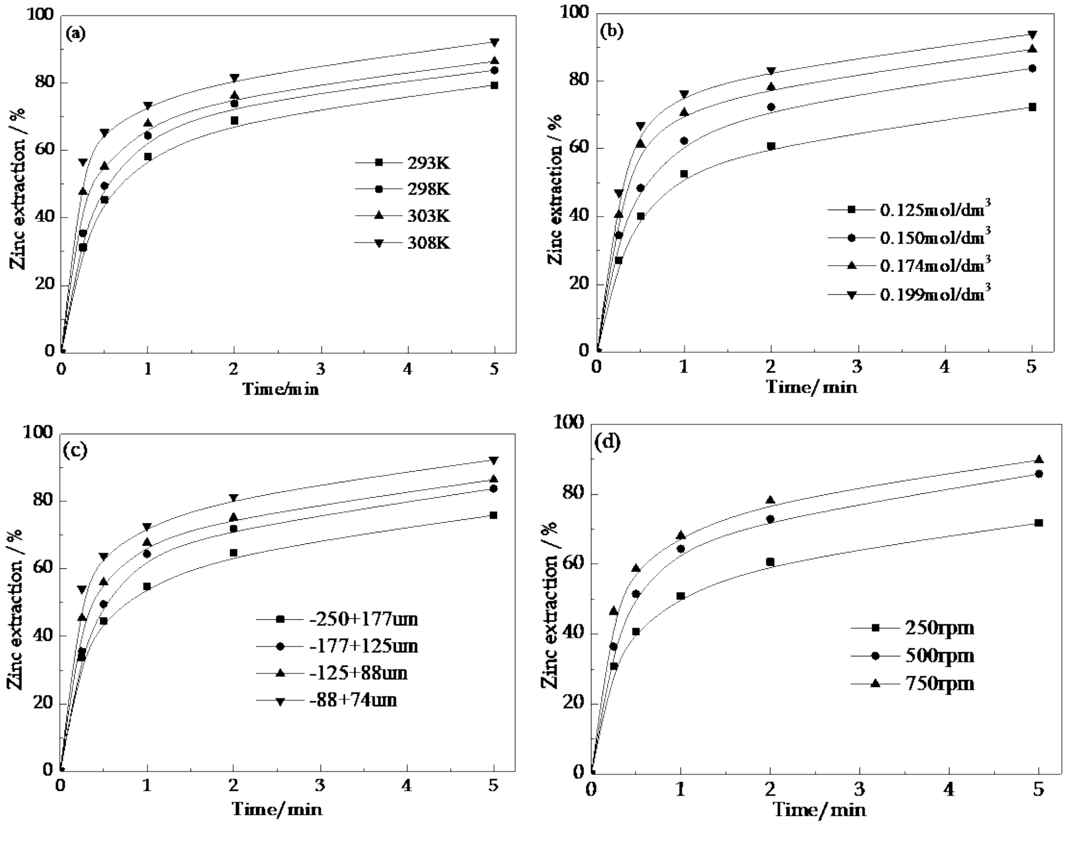

Fig. 2. Effect of operation parameters on the leaching efficiency of Zinc: (a) temperature, (b) MSA concentration, (c) particle size, (d) stirring speed

3.5. Kinetics study

Leaching rate facilitates practical operation conditions and enhances the productivity of zinc extraction. Therefore, extracting Zn economically is significant. However, little information on the leaching kinetics of hemimorphite in MSA has been reported yet. The dissolution of hemimorphite leaching kinetics in MSA is important to study the reaction course and dissolution rates.

Hemimorphite dissolution in MSA is a complicated reaction. The leaching reaction of mineral particles by a solid-fluid reaction is represented by the following reaction:

$$\text{a A fluid} + \text{b B solid} \rightarrow \text{product} \tag{2}$$

where a, b, A, and B represent the stoichiometric coefficient, fluid reactant, and solid undergoing dissolution. The relevant kinetics follows the described shrinking core model. According to this model, the leaching rate process is controlled by either diffusion through the liquid film, diffusion through the product layer, or surface chemical reaction (Ahmet et al., 2012; Feng et al., 2015). If the process is controlled by diffusion through the liquid film, the integrated rate equation of the shrinking core model can be described as follows:

$$k_1 t = x \ . \tag{3}$$

If the reaction is controlled by a surface chemical reaction, the integrated rate equation of the shrinking core model can be described as follows:

$$k_r t = 1- (1-x)^{1/3} \tag{4}$$

If the reaction is controlled by diffusion through the product layer, the integrated rate equation of the shrinking core model can be described as follows:

$$k_d t = 1-3(1-x)^{2/3}+2(1-x) \tag{5}$$

where x is the solid particle conversion fraction, k_1 is the apparent rate constant for diffusion through the fluid film, k_r is the apparent rate constant for the surface chemical reaction, k_d is the apparent rate constant for diffusion through the product layer, and t is the reaction time.

In general, the kinetics of any leaching reaction fits one of the above model. When hemimorphite is placed in MSA solutions, the mineral particles and leaching agent will react, forming the soluble zinc methane sulfonic; insoluble products were not formed during hemimorphite leaching. In the models above, when the rate controlling step is represented by diffusion through the fluid film or surface chemical reaction, the plot of x and $1-(1-x)^{1/3}$ versus the reaction time should be a straight line with a slope of k_l or k_r. For the kinetic analysis, shrinking core model with diffusion through the fluid film and surface chemical reaction as the rate controlling steps, $kt = x$, $kt = 1-(1-x)^{1/3}$ and $kt = 1-3(1-x)^{2/3}+2(1-x)$, was evaluated. The kinetics data obtained were assessed using the correlation coefficient R^2 values in Table 2. The data in Table 2 show that the R^2 values for $1-3(1-x)^{2/3}+2(1-x)$ are below 0.91, which implies poor fit. Therefore, diffusion through the fluid film and surface chemical reaction models also failed to represent the rate-controlling step. Thus, other kinetic models were used to describe the dissolution reaction.

On the basis of the various kinetic models published in the literature, we can find the following model we proposed suitable to describe hemimorphite dissolution in MSA solutions.

$$k_m t = [1-3(1-x)^{2/3}+2(1-x)]^2 \tag{6}$$

where k_m is the apparent rate constant for the surface chemical reaction model. Plots of $[1-3(1-x)^{2/3}+2(1-x)]^2$ versus time as a function of temperature, MSA concentration, particle size, and stirring speed are shown in Fig. 3. The apparent rate constants km for $[1-3(1-x)^{2/3}+2(1-x)]^2$ and its corresponding correlation coefficient values R^2 at various experimental variables are shown in Table 3. The results shown in Fig. 3 and Table 3 illustrate that hemimorphite dissolution followed the kinetic mode presented in Eq. (6). Combined with the experimental data analysis, we concluded that this model is an intermediate mode; its rate is determined by the reagent diffusion to the particle surface in the boundary solution layer and the rate of the surface dissolution reaction. The rate of the process was proportional to the reagent concentration in the solution and the concentration in the degree corresponding to the reaction order with regard to the reagent.

Table 2. The correlation coefficient values (R^2) of three kinetics models at various experimental variables

	R^2		
Experimental variable	Diffusion through the liquid film x	Surface chemical reaction $1-(1-x)^{1/3}$	Diffusion through the product layer $1-3(1-x)^{2/3}+2(1-x)$
Temperature (K)			
293	0.68046	0.77234	0.91121
298	0.66015	0.76885	0.90583
303	0.61621	0.73092	0.87436
308	0.5817	0.7232	0.86008
MSA concentration (mol/dm^3)			
0.125	0.69288	0.76938	0.91644
0.150	0.67334	0.77928	0.91628
0.174	0.62485	0.74851	0.88044
0.199	0.60372	0.75279	0.87537
Particle size (um)			
-250+177	0.67093	0.7572	0.91048
-177+125	0.66219	0.7619	0.90553
-125+88	0.6214	0.73693	0.88129
-88+74	0.59584	0.74107	0.87832
Stirring speed (rpm)			
250	0.68149	0.75945	0.9161
500	0.66443	0.7796	0.91934
750	0.62705	0.76042	0.90143

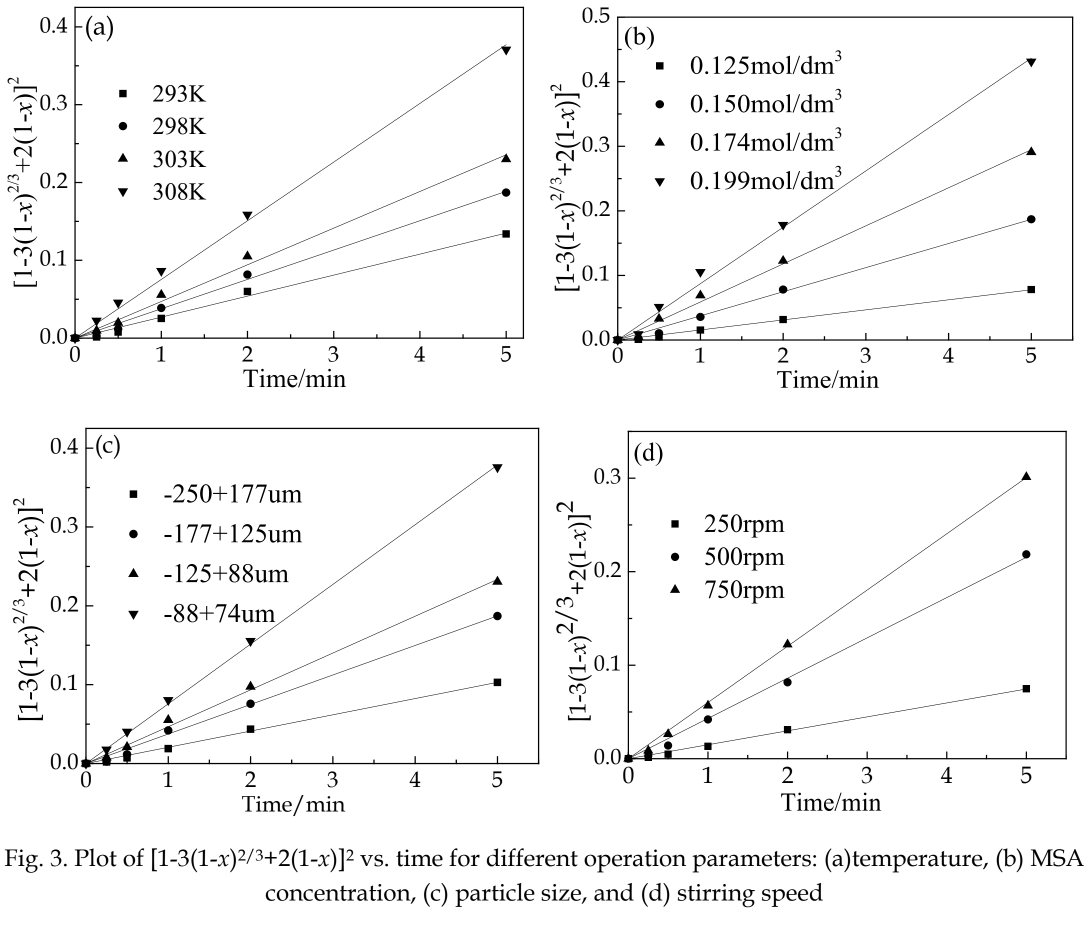

Fig. 3. Plot of $[1-3(1-x)^{2/3}+2(1-x)]^2$ vs. time for different operation parameters: (a)temperature, (b) MSA concentration, (c) particle size, and (d) stirring speed

Table 3. The apparent rate constants km for $[1-3(1-x)^{2/3}+2(1-x)]^2$ and its corresponding correlation coefficient values R2 at various experimental variables

Experimental variable	$[1-3(1-x)^{2/3}+2(1-x)]^2$	
	km (min^{-1})	R^2
Temperature (K)		
293	0.027	0.99464
298	0.03772	0.99602
303	0.04712	0.9957
308	0.0754	0.99793
MSA concentration (mol/dm^3)		
0.125	0.01556	0.99651
0.150	0.03737	0.99622
0.174	0.05893	0.99713
0.199	0.08729	0.99696
Particle size (um)		
-250+177	0.0206	0.99736
-177+125	0.03744	0.99664
-125+88	0.04671	0.99783
-88+74	0.07571	0.99963
Stirring speed (rpm)		
250	0.01491	0.99701
500	0.04307	0.99682
750	0.06012	0.99923

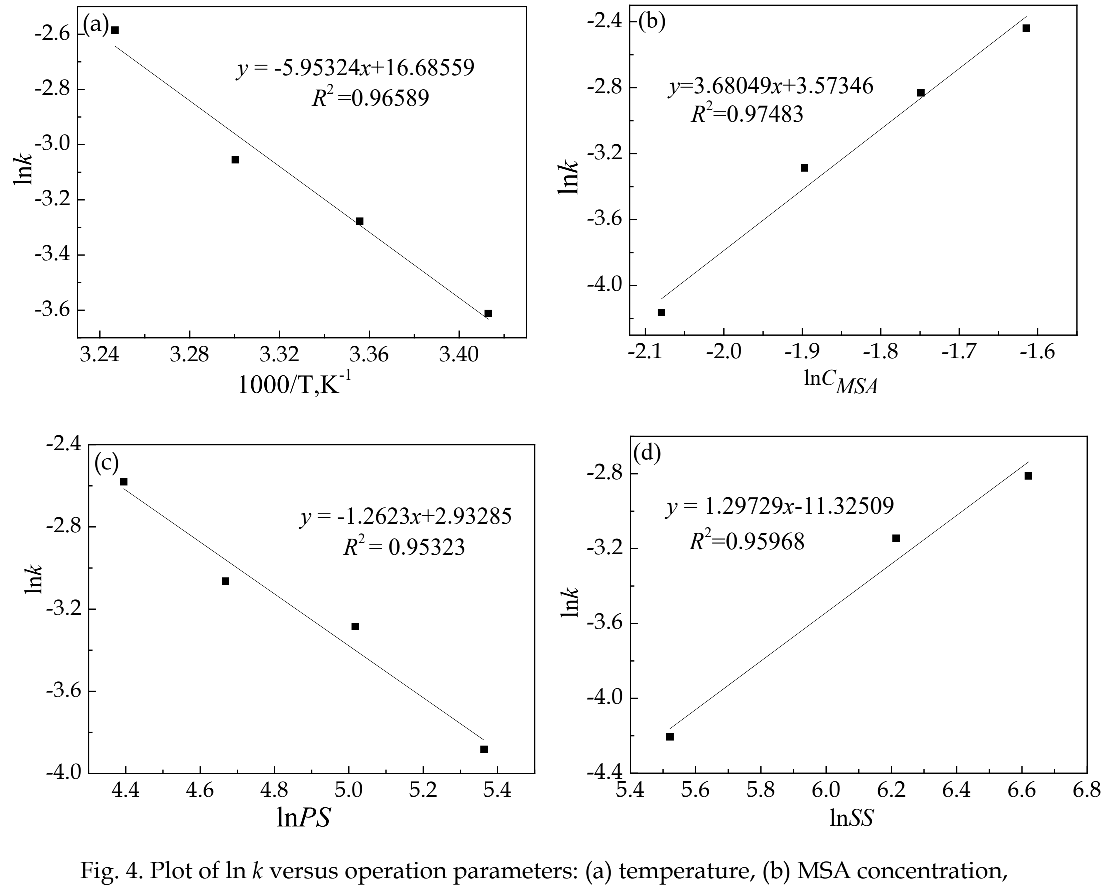

Fig. 4. Plot of ln *k* versus operation parameters: (a) temperature, (b) MSA concentration, (c) particle size, and (d) stirring speed

The kinetic equation of zinc extraction from hemimorphite can be expressed as follows:

$$[1-3(1-x)^{2/3}+2(1-x)]^2 = k_0(C)^{\alpha}(PS)^{\beta}(SS)^{\gamma}\exp(-Ea/RT)t \tag{7}$$

where C, PS, SS, Ea, R, and T represent MSA concentration, particle size, stirring speed, activation energy, universal gas constant, and temperature, respectively. The constants α, β, and γ are the reaction orders for the related parameters, while k_0 is the frequency or pre-exponential factor.

The values of α, β, and γ were 3.68049, −1.2623, and 1.29729, respectively. According to the results shown Fig. 4, the activation energy can be calculated by the Arrhenius equation. In addition, the Arrhenius plot of the dissolution process is shown in Fig. 4(a). According to the data shown in Fig. 4(a), the activation energy is 49.50 kJ/mol. By substituting the value of α, β, γ, and Ea into Eq. (7), the value of ko was calculated to be approximately 9.13. Thus, the equation representing the hemimorphite dissolution kinetics in the MSA solution can be expressed as follows:

$$[1-3(1-x)^{2/3}+2(1-x)]^2 = 9.13(C)^{3.68049}(PS)^{-1.2623}(SS)^{1.29729}\exp(-49.50/RT)t \tag{8}$$

4. Conclusions

MSA is a feasible leaching agent to treat hemimorphite, and its dissolution kinetics was investigated. Hemimorphite was dissolved easily in the MSA solutions, which can be obtained by high zinc extraction with a short reaction time. On the basis of the analysis results, the leaching rate of hemimorphite in MSA solutions was increased with increased reaction temperature, leaching reagent concentration, and stirring speed, as well as decreased particle size. The dissolution process followed the kinetic law of the shrinking core model, and a new kinetic model was found suitable to represent the rate controlling step, with an apparent activation energy value of 49.50 kJ/mol.

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