



Reaction mechanism of roasting Zn_2SiO_4 using NaOH

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Abstract: The reaction kinetics of roasting zinc silicate using NaOH was investigated. The orthogonal test was employed to optimize the reaction conditions and the optimized reaction conditions were as follows: molar ratio of NaOH to Zn_2SiO_4 of 16:1, reaction temperature of 550 °C, and reaction time of 2.5 h. In order to ascertain the phases transformation and reaction processes of zinc oxide and silica, the XRD phase analysis was used to analyze the phases of these specimens roasted at different temperatures. The final phases of the specimen roasted at 600 °C were Na_2ZnO_2 , Na_4SiO_4 , Na_2ZnSiO_4 and NaOH. The reaction kinetic equation of roasting was determined by the shrinking unreacted core model. Aiming to investigate the reaction mechanism, two control models of reaction rate were applied: chemical reaction at the particle surface and diffusion through the product layer. The results indicated that the diffusion through the product layer model described the reaction process well. The apparent activation energy of the roasting was 19.77 kJ/mol.

Key words: reaction mechanism; kinetics; Zn_2SiO_4 ; NaOH roasting; reaction process; phase transformation

1 Introduction

Zinc is one of the important transition metals and widely used in galvanizing and battery industry. With the gradual exhaustion of zinc sulfide ore and the rising demand for zinc, low-grade zinc oxide ore has attracted much attention in zinc metallurgy [1,2]. As the secondary ore of zinc, it is difficult to float due to high gangue, complex phases and serious pelitization [3,4]. Zinc oxide ore with utilization value mainly includes smithsonite ($ZnCO_3$), hemimorphite [$Zn_4Si_2O_7(OH)_2 \cdot H_2O$], zincite (ZnO), willemite (Zn_2SiO_4), and hydrozincite [$2ZnCO_3 \cdot 3Zn(OH)_2$], etc. Zinc in these minerals usually exists as multiphase rather than a single phase, such as $ZnCO_3$ and Zn_2SiO_4 . Lead, iron, silica, aluminum and cadmium also exist in these minerals [5,6].

Great amount of zinc oxide ore, especially low-grade zinc oxide ore, is available in China. For example, the zinc oxide ore in Lanping, Yunnan Province is abundant. However, the recovery ratios of valuable

metals are low and the cost is high when the mineral is treated directly by some traditional methods due to low grade, complex compositions and mineral intergrowth. The typical pyrometallurgy methods are gradually losing their attraction for high energy consumption and heavy pollution [7,8]. On the contrary, hydrometallurgical routes become more and more attractive. Normally, hydrometallurgical routes include acid leaching and alkaline leaching (involving NaOH leaching and ammonium leaching) [9]. All of those methods have their advantages and disadvantages. Acid leaching is a method widely studied and applied. However, silica gel is easily formed and enters into the leaching solution, causing filtration difficulty [10,11]. When NaOH leaching is applied, silica and lead dissolve in solution together with zinc. However, the decomposition of Zn_2SiO_4 is slow [12,13]. Ammonia is an attractive reagent. When ammonium leaching is adopted, the leaching vessels are rigorously required in order to avoid ammonium volatilization [14,15].

Many researchers have focused on comprehensive

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utilization of low-grade zinc oxide ore by improving the current routes or developing new method or new reaction medium in recent years, such as $(\text{NH}_4)_2\text{SO}_4$ roasting [16], NaOH roasting [17] and solvent extraction [18]. NaOH roasting is a process combining the advantages of pyrometallurgy and hydrometallurgy routes. The stable Zn_2SiO_4 can be decomposed in this simple process with low energy consumption. As the key step of utilizing low-grade zinc oxide ore, it is necessary to investigate the roasting process and reaction mechanism. In this work, Zn_2SiO_4 was roasted by using NaOH and the reaction conditions were optimized. The phase transformation and reaction process of zinc and silica were examined. The reaction kinetic equation of roasting zinc silicate using NaOH was deduced finally.

2 Experimental

2.1 Materials and characterization

Analytic grade Zn_2SiO_4 with purity $\geq 99.0\%$ was used as raw material and NaOH was used as reactant. Distilled water was homemade in laboratory.

2.2 Procedure

Appropriate masses of Zn_2SiO_4 with 74–104 μm in particle size and NaOH were weighted according to molar ratio (8:1, 12:1 and 16:1) and mixed uniformly in a medicine grinder. The mixture was put into a crucible, and then the crucible was placed in a roasting furnace. The roasting procedure was controlled and monitored by an intelligent temperature control instrument. When the roasting process ended, the specimens were water-leached at 80 °C for 1.5 h, filtrated and washed. Zinc oxide and silica were distributed both in leaching solution and water leaching residue. The contents of zinc oxide and silica both in solution and residue were examined by titration method to calculate the recovery ratios.

The mixed material was put into a series of crucibles. When the desired temperature was reached, the crucibles were placed into the roasting furnace. After the temperature was stabilized, the crucibles were orderly taken out at a predetermined time interval and cooled rapidly in order to reduce deviation. After leaching and filtration, the recovery ratios of zinc oxide and silica were calculated.

The recovery ratio of zinc oxide or silica was calculated using

$$\eta = \frac{V \cdot c}{C} \quad (1)$$

where η is the recovery ratio of zinc oxide or silica, V is the leaching solution volume, c is the concentration of

zinc oxide or silica, and C is the initial content in Zn_2SiO_4 .

3 Results and discussion

3.1 Characterization of zinc silicate

Figure 1 shows the XRD pattern of zinc silicate. The position and intensity of the diffraction peaks are consistent with those of the zinc silicate (JCPDS No. 37–1485).

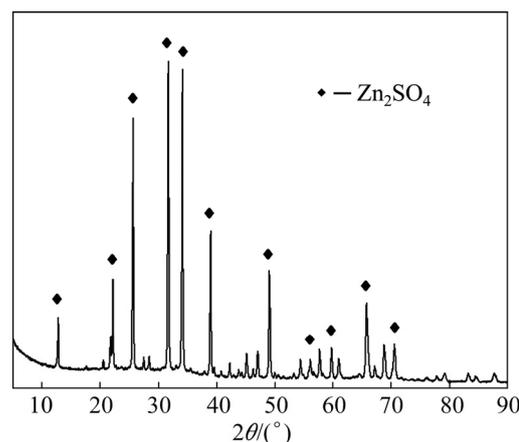


Fig. 1 XRD pattern of zinc silicate

3.2 Orthogonal test

The orthogonal test was designed to optimize the reaction conditions of roasting Zn_2SiO_4 by using NaOH. The factors and levels are listed in Table 1, and the results are listed in Table 2. The recovery ratios of zinc oxide and silica were used as evaluation indexes.

Table 1 Factors and levels of orthogonal test

Level	Molar ratio of NaOH to Zn_2SiO_4	Reaction temperature/°C	Reaction time/h
1	8	450	2
2	12	500	2.5
3	16	550	3

The optimized reaction conditions were as follows: molar ratio of NaOH to Zn_2SiO_4 of 16:1, reaction temperature of 550 °C and reaction time of 2.5 h. The sequence influencing the recovery ratios of ZnO and SiO_2 in molten NaOH system is molar ratio, reaction temperature and reaction time. The verification experiments were carried out and the recovery ratios of ZnO and SiO_2 were stable at approximate 96% and 92%, respectively. This is because $\text{Na}_2\text{ZnSiO}_4$ is generated in roasting, which is the main component in leaching residue. From the residual $\text{Na}_2\text{ZnSiO}_4$ formula, Zn is involved in the reaction; however, Si has no change. Thereby, the recovery ratio of ZnO is higher than that of SiO_2 .

Table 2 Results of orthogonal test

No.	Molar ratio of NaOH to Zn ₂ SiO ₄	Reaction temperature/°C	Reaction time/h	Recovery ratio/%	
				Zinc oxide	Silica
1	8:1	450	2	88.20	76.46
2	8:1	500	2.5	90.70	81.43
3	8:1	550	3	88.31	76.66
4	12:1	450	2.5	88.76	77.50
5	12:1	500	3	94.07	88.12
6	12:1	550	2	93.04	86.07
7	16:1	450	3	92.71	85.45
8	16:1	500	2	94.84	89.68
9	16:1	550	2.5	96.03	92.10
<i>d_{ij}</i>	Zn/Si	Zn/Si	Zn/Si		
<i>d_{1j}</i>	89.07/78.18	89.89/79.80	92.03/84.07		
<i>d_{2j}</i>	91.96/83.90	93.20/86.41	91.83/83.68		
<i>d_{3j}</i>	94.53/89.08	92.46/84.94	91.70/83.41		
<i>R</i>	5.46/10.9	3.31/6.61	0.33/0.66		

d_{ij} (*i, j*=1,2,3) is the average value of various factors at levels

The recovery ratios of ZnO and SiO₂ increased with molar ratio rising due to the improvement of interface area between Zn₂SiO₄ and NaOH. Raising reaction temperature was contributed to the mass transfer between the liquid and solid phases.

3.3 Reaction process analysis

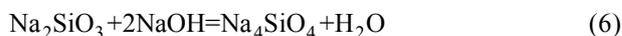
Zn₂SiO₄ was roasted for 2 h by using NaOH at molar ratio of 16:1 at different temperatures. The mineral phases of the specimens are displayed in Fig. 2.

The existence of Na₂ZnO₂, Na₂ZnSiO₄ and Na₂Zn(OH)₄ in Fig. 2(a) indicated that the reaction between Zn₂SiO₄ and NaOH had started at 250 °C. When the reaction temperature was 300 °C, the diffraction peaks of Zn₂SiO₄ disappeared in the XRD pattern, indicating that Zn₂SiO₄ had been completely reacted. The phases were Na₂ZnO₂, Na₂ZnSiO₄, Na₂Zn(OH)₄ and ZnO. Na₂Zn(OH)₄ was an intermediate product, which could be seen as Na₂ZnO₂.

The main compositions in Fig. 2(c) were Na₂ZnSiO₄, Na₂Zn(OH)₄ and NaOH, which were similar with those in Fig. 2(d). Na₂Zn(OH)₄ disappeared when the temperature was 400 °C. At 450 °C, Na₂SiO₃ and Na₄SiO₄ were synthesized. Na₂SiO₃ disappeared when the temperature reached 500 °C because of the transformation from Na₂SiO₃ to Na₄SiO₄. The XRD patterns in Figs. 2(g) and (h) were almost the same. The final phases in the specimen roasted at 600 °C were Na₂ZnO₂, Na₄SiO₄ and Na₂ZnSiO₄. Residual NaOH still existed. However, the reaction between Zn₂SiO₄ and NaOH was not complete, which could be confirmed from Na₂ZnSiO₄ phase in the XRD pattern. The existence of

Na₂ZnSiO₄ explained why the recovery ratios of ZnO and SiO₂ were only about 96% and 92%. NaOH·H₂O was attributed to the moisture adsorption capacity of NaOH in air. Na₂ZnO₂ and Na₄SiO₄ are easily soluble in water and enter into the liquid phase; however, Na₂ZnSiO₄ dissolves slowly in alkaline solution, so Na₂ZnSiO₄ enters into the leaching residue after filtration.

From above, the reaction process could be summarized as follows: Na₂ZnO₂ [Na₂Zn(OH)₄] and Na₂ZnSiO₄ were obtained when zinc silicate reacted with NaOH. The reaction between Zn₂SiO₄ and NaOH proceeded with reaction temperature rising. When the reaction temperature was above 450 °C, Na₂SiO₃ and Na₄SiO₄ were synthesized. In addition, Na₂SiO₃ was transformed into Na₄SiO₄ finally. The final phases in the specimen were Na₂ZnO₂, Na₄SiO₄, Na₂ZnSiO₄ and NaOH. Na₂ZnSiO₄ was more stable than Zn₂SiO₄ in the chemical property. The chemical equations in roasting could be summarized as follows:



3.4 Dynamics computation

The reaction between NaOH and Zn₂SiO₄ can be regarded as liquid–solid reaction in experiments because

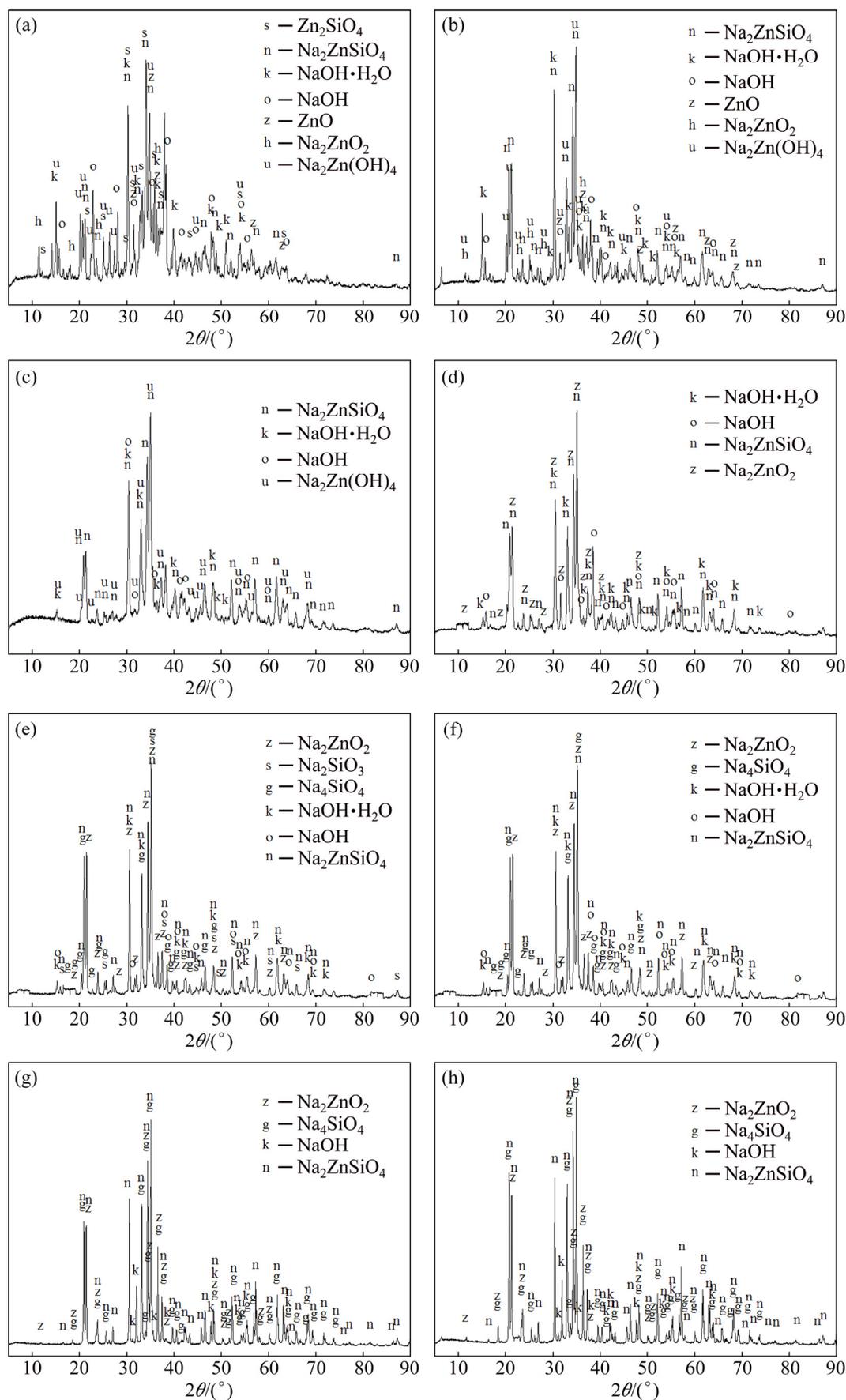


Fig. 2 XRD patterns of specimens obtained at different temperatures: (a) 250 °C; (b) 300 °C; (c) 350 °C; (d) 400 °C; (e) 450 °C; (f) 500 °C; (g) 550 °C; (h) 600 °C

the melting point of NaOH is 318 °C. Therefore, the roasting process can be examined by the shrinking unreacted core model if Zn₂SiO₄ particles are sphere-shaped [19]. The reaction rate of molten NaOH roasting Zn₂SiO₄ may be controlled by two different models: the chemical reaction at the particle surface or diffusion through the product layer [19,20]. Equation (7) can describe the reaction rate controlled by chemical reaction at the particle surface. The reaction rate controlled by the diffusion through the product layer can be expressed as Eq. (8):

$$k_c t = 1 - (1 - \alpha)^{1/3} \tag{7}$$

$$k_p t = 1 - (1 - \alpha)^{2/3} - 2\alpha/3 \tag{8}$$

where t is the reaction time (min), α is the recovery fraction of zinc oxide or silica, and k_c and k_p are the reaction rate constants.

3.4.1 Effect of molar ratio of NaOH to zinc silicate on recovery ratios of ZnO and SiO₂

The effect of molar ratio of NaOH to zinc silicate on the recovery ratios of zinc oxide and silica was investigated. The roasting temperature was kept at 500 °C, and the results are plotted in Fig. 3.

The recovery ratios of zinc oxide and silica increased with rising molar ratio of NaOH to Zn₂SiO₄.

Increasing the NaOH dosage was contributed to the improvement of the real contact area. The recovery ratios of zinc oxide and silica kept increasing within 100 min, and the maximum values were 81% and 73%, respectively.

In order to identify the rate-controlling step, the kinetics of molten NaOH roasting Zn₂SiO₄ was analyzed with the two different models. Based on experimental data in Fig. 3, the relationships of the right-hand sides of Eq. (7) and Eq. (8) against reaction time are shown in Fig. 4 and Fig. 5, respectively. The apparent reaction rate constants k_c and k_p could be obtained from the slopes of those straight fitting lines. These constants with corresponding correlation coefficients R are recorded in Table 3. The straight lines in Fig. 5 are closer to zero point than those in Fig. 4, indicating that the reaction rate might be controlled by the diffusion through the product layer.

3.4.2 Effect of roasting temperature on recovery ratios of ZnO and SiO₂

The effect of roasting temperature on the recovery ratios of zinc oxide and silica was investigated in a temperature range of 350–500 °C. The molar ratio of NaOH to zinc silicate was 16:1. The experimental results are plotted in Fig. 6.

It was obvious that the roasting temperature played

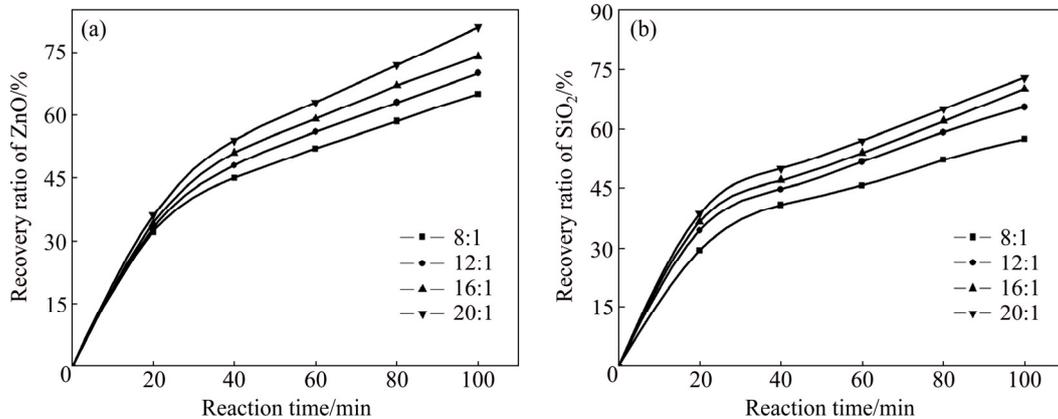


Fig. 3 Influence of NaOH to Zn₂SiO₄ molar ratio on recovery ratios of zinc oxide (a) and silica (b)

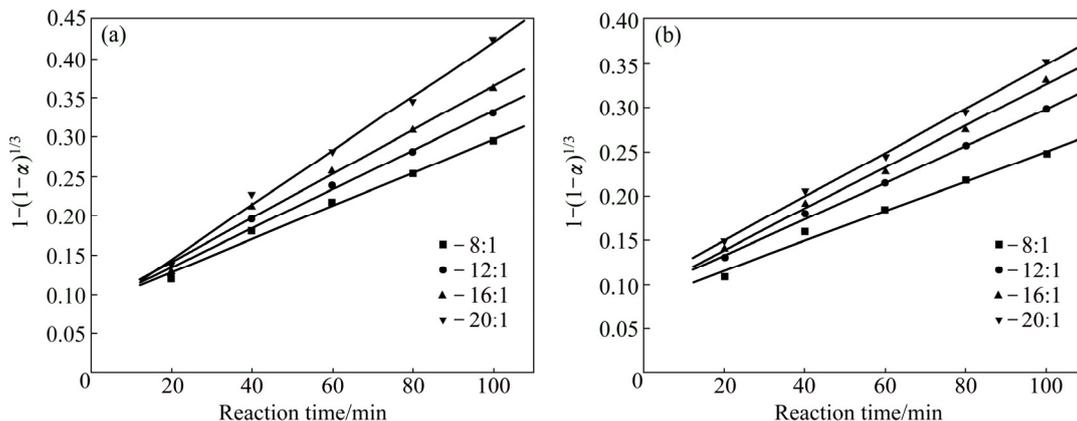


Fig. 4 Plots of $1 - (1 - \alpha)^{1/3}$ against reaction time at different NaOH to Zn₂SiO₄ molar ratios: (a) ZnO; (b) SiO₂

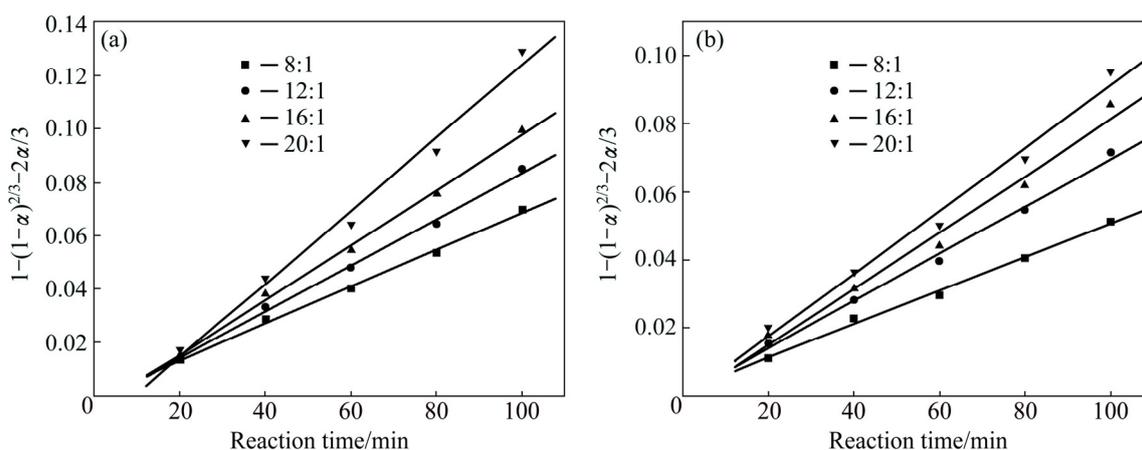


Fig. 5 Plots of $1-(1-\alpha)^{2/3}-2\alpha/3$ against reaction time at different NaOH to Zn_2SiO_4 molar ratios: (a) ZnO; (b) SiO_2

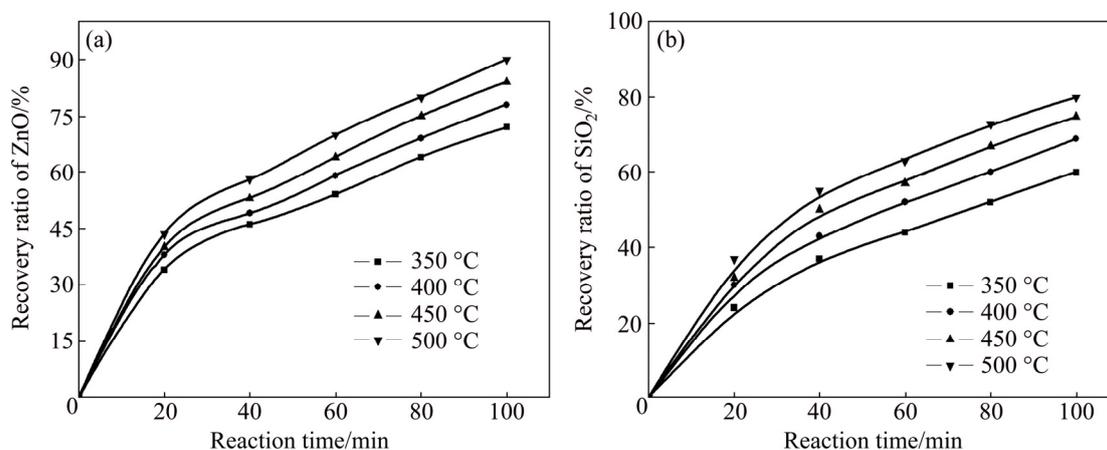


Fig. 6 Effect of reaction temperature on recovery ratios of zinc oxide (a) and silica (b)

Table 3 Apparent rate constants and correlation coefficients at different molar ratios

Molar ratio	ZnO				SiO ₂			
	$1-(1-\alpha)^{1/3}$		$1-(1-\alpha)^{2/3}-2\alpha/3$		$1-(1-\alpha)^{1/3}$		$1-(1-\alpha)^{2/3}-2\alpha/3$	
	k_c	R	k_p	R	k_c	R	k_p	R
8:1	0.00211	0.9950	0.000691	0.9984	0.00168	0.9931	0.000492	0.9975
12:1	0.00249	0.9947	0.000865	0.9986	0.00207	0.9992	0.000692	0.9973
16:1	0.00281	0.9941	0.00103	0.9984	0.00233	0.9981	0.000828	0.9911
20:1	0.00346	0.9969	0.00136	0.9943	0.00249	0.9980	0.000921	0.9927

a significant role in roasting. The recovery ratios of zinc oxide and silica increased with the roasting temperature rising. About 90% zinc oxide and 80% silica were recovered at 500 °C for 100 min. The two models were used to investigate the kinetics of roasting process. The plots of the right-hand sides of Eq. (7) and Eq. (8) against reaction time are shown in Fig. 7 and Fig. 8, respectively. The apparent rate constants k_c and k_p were calculated from the slopes of those fitting straight lines in Figs. 7 and 8, as listed in Table 4 together with the corresponding correlation coefficients R . All the fitting straight lines in Fig. 8 were closer to zero point than

those in Fig. 7. In other words, the results were better fitted by Eq. (8) than by Eq. (7).

From above, the reaction rate of roasting Zn_2SiO_4 in molten NaOH might be controlled by the diffusion through the product layer. However, as we know, there must be deviation when the experiments were carried out. And the reaction started before the roasting temperature was stable.

3.4.3 Calculation of apparent activation energy

The reaction rate constant k is a function of temperature. The relationship between them is expressed by the Arrhenius equation [21]:

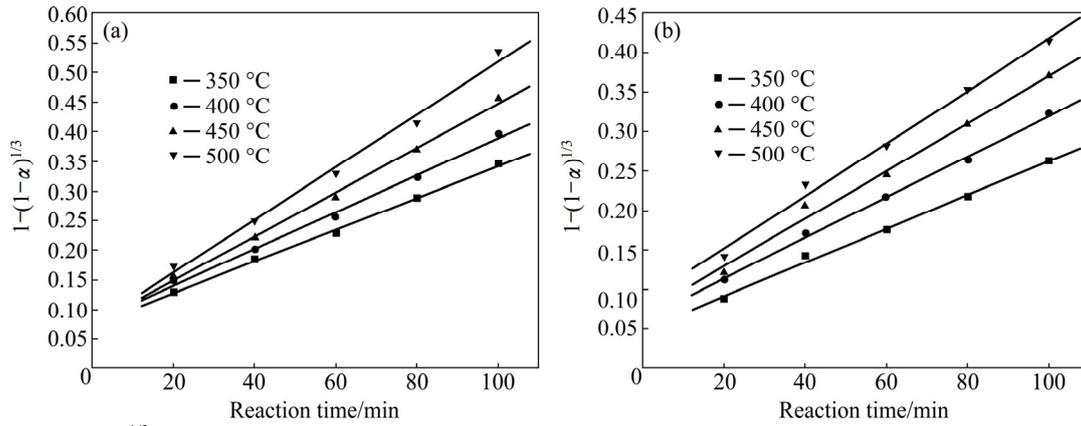


Fig. 7 Plots of $1-(1-\alpha)^{1/3}$ against reaction time at different roasting temperatures: (a) ZnO; (b) SiO₂

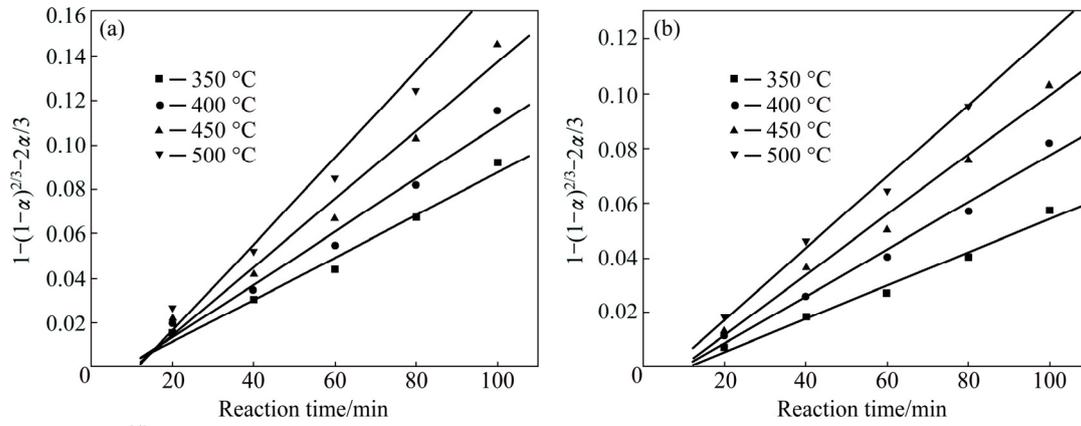


Fig. 8 Plots of $1-(1-\alpha)^{2/3}-2\alpha/3$ against reaction time at different roasting temperatures: (a) ZnO; (b) SiO₂

Table 4 Apparent rate constants and correlation coefficients at different roasting temperatures

Roasting temperature/°C	ZnO				SiO ₂			
	$1-(1-\alpha)^{1/3}$		$1-(1-\alpha)^{2/3}-2\alpha/3$		$1-(1-\alpha)^{1/3}$		$1-(1-\alpha)^{2/3}-2\alpha/3$	
	k_c	R	k_p	R	k_c	R	k_p	R
350	0.00268	0.9986	0.000953	0.9912	0.00213	0.9973	0.000608	0.9928
400	0.00310	0.9976	0.00119	0.9882	0.00257	0.9987	0.000860	0.9923
450	0.00374	0.9978	0.00154	0.9889	0.00301	0.9948	0.00109	0.9942
500	0.00445	0.9955	0.00194	0.9864	0.00332	0.9962	0.00131	0.9971

$$k=A\exp[-E/(RT)] \tag{9}$$

where A is the frequency factor, E is the apparent activation energy, and R is the gas constant.

The activation energy E determined by the plots of $\ln k$ against $1/T$ in Figs. 9(a) and (b) (calculated by using Eq. (7)) was 13.61 and 11.99 kJ/mol, respectively. And the frequency factor A was 0.0363 and 0.0218, respectively. The average values of E and A were 12.80 kJ/mol and 0.0291, respectively.

The activation energy E determined from the plots of $\ln k$ against $1/T$ in Figs. 10(a) and (b) (calculated by using Eq. (8)) was 19.07 and 20.46 kJ/mol, respectively. And the frequency factor A was 0.0371 and 0.0323, respectively. The average values of E and A were 19.77 kJ/mol and 0.0347, respectively.

The values of the activation energy E calculated using Eq. (7) and Eq. (8) were both less than 20 kJ/mol. According to the literatures [21,22], the roasting process was controlled by diffusion through the product layer when the activation energy was less than 20 kJ/mol. So, the kinetic equation could be expressed as

$$1-(1-\alpha)^{2/3}-2\alpha/3=0.0347\exp[-19.77/(RT)]t \tag{10}$$

3.5 Characterization of leaching residue

Figure 11 shows the XRD pattern and SEM image of leaching residue. The position and intensity of the diffraction peaks were consistent with those of the sodium zinc silicate (JCPDS No. 76-0714). No other phases were detected. Na₂ZnSiO₄ particles were regular and in a narrow size distribution.

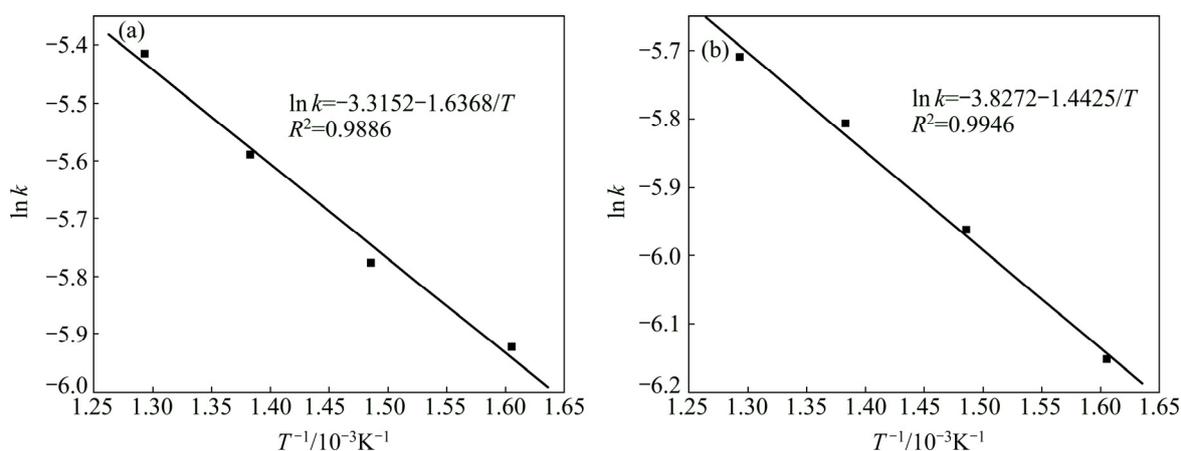


Fig. 9 Plots of $\ln k$ against $1/T$ at different roasting temperatures using Eq. (7): (a) ZnO; (b) SiO₂

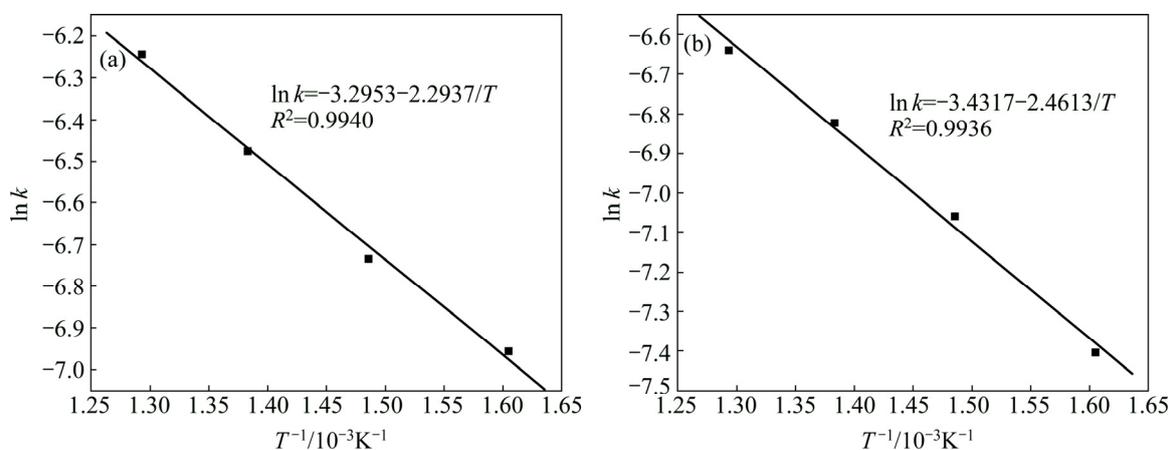


Fig. 10 Plots of $\ln k$ against $1/T$ at different roasting temperatures using Eq. (8): (a) ZnO; (b) SiO₂

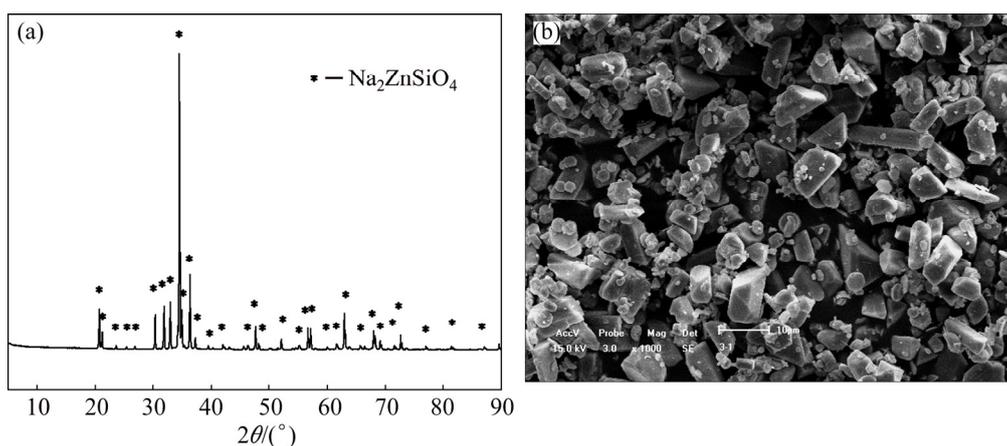


Fig. 11 XRD pattern (a) and SEM image (b) of leaching residue

4 Conclusions

1) The optimized reaction conditions of roasting zinc silicate by using NaOH were as follows: molar ratio of NaOH to zinc silicate of 16:1, reaction temperature of 550 °C and reaction time of 2.5 h. The influencing sequence was molar ratio, reaction temperature and

reaction time. Under optimized reaction conditions, the recovery ratios of ZnO and SiO₂ were approximately 96% and 92%, respectively. The apparent activation energy of the roasting was 19.77 kJ/mol. The reaction rate of roasting zinc silicate was controlled by the diffusion through the product layer.

2) Na₂ZnO₂ [Na₂Zn(OH)₄] and Na₂ZnSiO₄ were

obtained when Zn_2SiO_4 reacted with NaOH. The reaction between Zn_2SiO_4 and NaOH proceeded with rising reaction temperature. When the reaction temperature was above 450 °C, Na_2SiO_3 and Na_4SiO_4 were obtained, and Na_2SiO_3 was transformed into Na_4SiO_4 finally. The final phases in the specimen obtained at 600 °C were Na_2ZnO_2 , Na_4SiO_4 , Na_2ZnSiO_4 and NaOH. Na_2ZnSiO_4 was more stable than Zn_2SiO_4 in the chemical property.

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NaOH 焙烧 Zn_2SiO_4 反应机理

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摘要: 研究氢氧化钠焙烧硅酸锌的反应动力学, 采用正交试验优化反应条件, 优化反应条件为: NaOH 和 Zn_2SiO_4 摩尔配比 16:1、反应温度 550 °C 以及反应时间 2.5 h。为了确定氧化锌和二氧化硅的物相转化和反应过程, 采用 XRD 技术分析不同温度焙烧样品的物相。600 °C 焙烧样品的最终物相为 Na_2ZnO_2 、 Na_4SiO_4 、 Na_2ZnSiO_4 和 NaOH。通过未反应收缩核模型研究焙烧过程的动力学方程, 选取 2 种反应速率控制模型考察反应机理, 分别为颗粒表面化学反应控制和通过固体产物层的扩散控制模型。结果表明: NaOH 焙烧 Zn_2SiO_4 的反应过程受通过固体产物层的扩散控制, 反应的表现活化能为 19.77 kJ/mol。

关键词: 反应机理; 动力学; Zn_2SiO_4 ; NaOH 焙烧; 反应过程; 物相转化