

Kinetics of SiO₂ leaching from Al₂O₃ extracted slag of fly ash with sodium hydroxide solution

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Abstract: Kinetics of SiO₂ leaching from Al₂O₃ extracted slag of fly ash with sodium hydroxide solution was studied. The effect of leaching temperature, mass ratio of NaOH to SiO₂ and stirring speed on SiO₂ leaching rate was investigated. The results show that increasing leaching temperature, mass ratio of NaOH to SiO₂ and stirring speed increases SiO₂ leaching rate. The SiO₂ leaching rate is 95.66% under the optimized conditions. There are two stages for the SiO₂ leaching process, and the leaching reaction is very rapid in the first stage but quite slow in the second stage. The whole leaching process follows the shrinking core model, and the outer diffusion of no product layer is the rate-controlling step. The activation energies of the first and second stages are calculated to be 8.492 kJ/mol and 8.668 kJ/mol, respectively. The kinetic equations of the first and the second stages were obtained, respectively.

Key words: fly ash; leaching; kinetics; SiO₂

1 Introduction

Fly ash is a fine ash separated from the flue gas of coal-fired power plant burning pulverized coal [1]. The contents of Al₂O₃ and SiO₂ in fly ash are very high. However, fly ash is mainly used in the building industry, highway road bases and grout mixes [2,3], which cause huge waste of resources. So, it will be very necessary to improve value-added utilization of fly ash. Through recovering Al₂O₃ and SiO₂ from fly ash, the status of lacking metal resources in China would be greatly relieved.

Many typical metallurgical methods on the comprehensive utilization of fly ash have been reported. Limestone sintering method is similar to the major method currently used in the Al₂O₃ plant. But due to the addition of a large amount of limestone in the sintering process, a considerable amount of calcium silicate residue can be used for cement production only [4,5]. Thus the SiO₂ resource is wasted. Acid leaching method has been a hot research topic in recent years [6–8]; however, by which the recovery rate of Al₂O₃ is lower [9–11]. The Al₂O₃, which exists in Al₂O₃ extracted slag,

could decrease the quality and yield of SiO₂ product. There are also some other methods. WANG et al [12,13] studied extracting SiO₂ from fly ash by alkali leaching, and then Al₂O₃ was extracted from desiliconized fly ash by alkali lime sintering process. The recovery rates of SiO₂ and Al₂O₃ were 72% and 90%, respectively. From the above, it can be seen that improving the recovery rate of SiO₂ is the key for the utilization of fly ash.

In the present work, a novel method for the comprehensive utilization of fly ash was established, which not only takes full advantage of Al₂O₃, but also makes SiO₂ enriched in Al₂O₃ extracted slag. In this method, NH₄HSO₄ roasting technology was used to treat fly ash, by which Al and Si can be effectively separated. The content of SiO₂ in Al₂O₃ extracted slag could reach 90%, which is obviously higher than that in other reports (about 68.9% or 86.72%) [8,14]. In this study, the Al₂O₃ extracted slag was used as raw material to recover SiO₂ with NaOH solution.

In the actual production process, the reaction rate and the reaction effect are mainly depended on the kinetics factors. Nevertheless, there are no reports on kinetics of SiO₂ leaching from Al₂O₃ extracted slag of fly ash. Therefore, it is very necessary to study the kinetics

of SiO_2 leaching process. The objectives are to investigate the main factors involved in the leaching process, such as leaching temperature, mass ratio of NaOH to SiO_2 and stirring speed, and also to determine the kinetics of SiO_2 leaching from Al_2O_3 extracted slag.

2 Experimental

2.1 Materials

NH_4HSO_4 and NaOH were in industrial grade; the reagents used for analysis and detection were in analytical grade. Deionized water was used throughout the experiments whenever needed. Representative sample of fly ash was taken from a coal-fired power plant in Inner Mongolia, China, the size fraction of which mainly ranged from 61 to 74 μm . Fly ash was mixed with NH_4HSO_4 at a mole ratio of Al_2O_3 to NH_4HSO_4 of 1:8. The mixture was roasted at 400 $^\circ\text{C}$ for 60 min in air atmosphere. The roasted clinker was leached with deionized water at 90 $^\circ\text{C}$ for 60 min, and then filtered. The Al_2O_3 extracted slag was obtained. The detailed chemical compositions of fly ash and Al_2O_3 extracted slag were examined by ICP, and the analytical results are shown in Table 1. Compared with fly ash, the Al_2O_3 content in Al_2O_3 extracted slag is lowered while the SiO_2 content is improved. The mineral phases of fly ash and Al_2O_3 extracted slag were analyzed by XRD. The XRD analysis was performed using Cu K_α radiation ($\lambda=1.5406$ nm) at 40 kV and 30 mA. The XRD results are shown in Fig. 1. It can be seen that the major mineral phases of fly ash are mullite and $\alpha\text{-SiO}_2$. At the same time, a broad characteristic diffraction peak appears from 20 $^\circ$ to 25 $^\circ$, indicating the existence of glass phase.

Table 1 Compositions of fly ash and Al_2O_3 extracted slag (mass fraction, %)

Item	Al_2O_3	SiO_2	Fe_2O_3	CaO	TiO_2
Fly ash	38.53	45.41	2.38	2.73	1.85
Al_2O_3 extracted slag	4.82	90.00	0.03	0.10	0.44

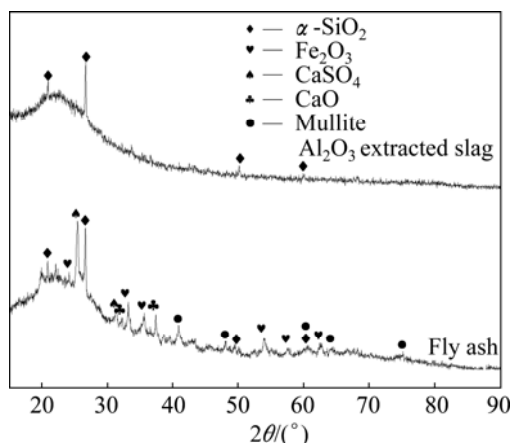


Fig. 1 XRD patterns of fly ash and Al_2O_3 extracted slag

Whereas, there is no mullite phase in Al_2O_3 extracted slag, which shows that the Al_2O_3 in mullite has been extracted, and the mineral phase of Al_2O_3 extracted slag is $\alpha\text{-SiO}_2$. The morphologies of fly ash and Al_2O_3 extracted slag were examined by SEM. It can be seen from Fig. 2(a) that fly ash presents a rough surface. Figure 2(b) shows that there are many small holes in the particle of Al_2O_3 extracted slag, which were eroded by NH_4HSO_4 .

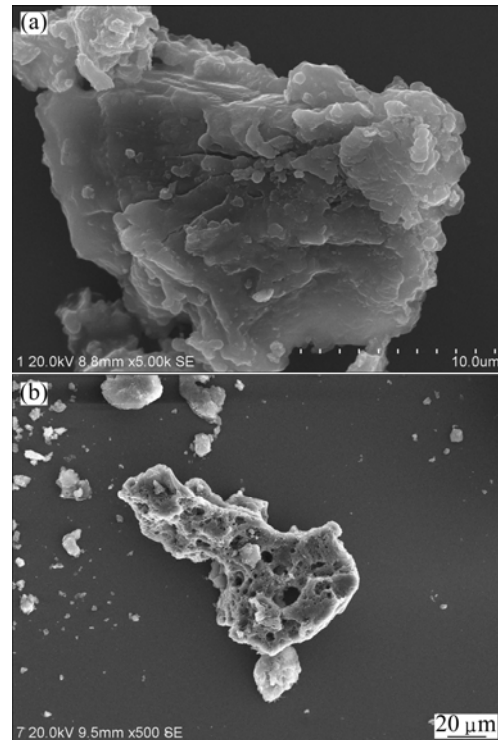


Fig. 2 SEM images of fly ash (a) and Al_2O_3 extracted slag (b)

2.2 Method

The leaching experiments were performed in a 1 L polytetrafluoroethylene (PTFE) beaker with a mechanical stirrer and heated in a water bath with a temperature controller. The accuracy of leaching temperature was ± 0.5 $^\circ\text{C}$. 50 g of Al_2O_3 extracted slag was used for each experiment. According to the mass ratio of NaOH to SiO_2 , the calculated mass of NaOH was added to the PTFE beaker containing 500 mL water, and then the NaOH solution was heated to the desired temperature. When the temperature reached and remained stable, the Al_2O_3 extracted slag was added under the condition of continuously stirring. After selected time intervals, approximately 10 mL of sample of the reaction mixture was taken out, and then quickly filtered. The content of SiO_2 in residue was analyzed by titration, and SiO_2 leaching rate was calculated as

$$\alpha(\text{SiO}_2) = \frac{m(\text{SiO}_2) - m'(\text{SiO}_2)}{m(\text{SiO}_2)} \times 100\% \quad (1)$$

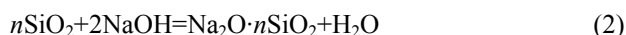
where $\alpha(\text{SiO}_2)$ is the SiO_2 leaching rate; $m(\text{SiO}_2)$ is the

mass of SiO_2 in Al_2O_3 extracted slag; $m'(\text{SiO}_2)$ is the mass of SiO_2 in residue.

3 Results and discussion

3.1 Leaching process and controlling step for reaction rate

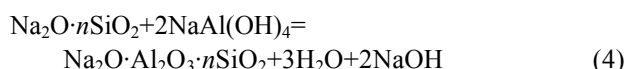
The main chemical reaction that occurs between Al_2O_3 extracted slag and NaOH is as follows:



Due to the existence of a small amount of Al_2O_3 , the following reaction will take place in the leaching process:



With the increase of Al content, the reaction between $\text{NaAl}(\text{OH})_4$ and $\text{Na}_2\text{O} \cdot n\text{SiO}_2$ will occur, which can be written as



The particle of Al_2O_3 extracted slag is regarded as dense spherical type. During the process of SiO_2 leaching from Al_2O_3 extracted slag, the particle surface area decreases as the reaction progresses. So, the liquid–solid leaching process can be analyzed with the shrinking core model, as shown in Fig. 3.

The reaction includes four steps as follows: 1) outer diffusion of leaching agent (NaOH solution) through the liquid boundary layer; 2) inner diffusion of leaching agent (NaOH solution) and liquid product ($\text{Na}_2\text{O} \cdot n\text{SiO}_2$) through the residue layer; 3) chemical reaction between

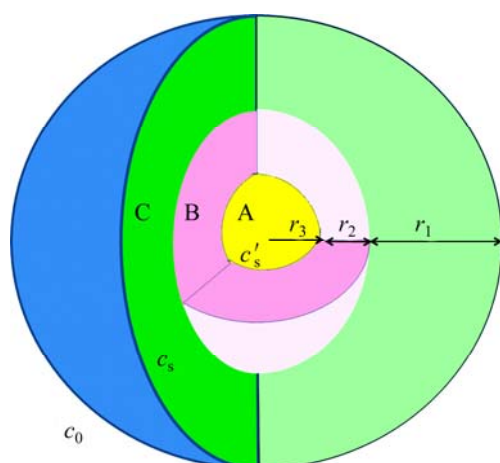


Fig. 3 Illustrative diagram of leaching process: A—Non-leached particle of Al_2O_3 extracted slag; B—Layer of residue; C—Diffusion region of solvent; c_0 —Concentration of leaching reagent in fluid phase; c_s —Concentration of leaching reagent on surface of particle; c'_s —Concentration of leaching reagent in region of reaction; r_1 —Thickness of diffusion layer of leaching agent; r_2 —Thickness of residue; r_3 —Radius of non-leached particle of Al_2O_3 extracted slag

particle of Al_2O_3 extracted slag and leaching agent (NaOH solution); 4) diffusion of liquid product ($\text{Na}_2\text{O} \cdot n\text{SiO}_2$) to liquid of leaching system.

During the process of SiO_2 leaching, the main product is liquid product ($\text{Na}_2\text{O} \cdot n\text{SiO}_2$), so the inner diffusion resistance is small. Thus the rate-controlling step is outer diffusion of no product layer or surface chemical reaction.

When the leaching process is controlled by outer diffusion of no product layer, the following expression of shrinking core model can be used to describe the kinetics [15]:

$$1 - (1 - \alpha)^{2/3} = k't \quad (5)$$

When the leaching process is controlled by surface chemical reaction, the following expression of shrinking core model can be used to describe the kinetics [15,16]:

$$1 - (1 - \alpha)^{1/3} = k''t \quad (6)$$

where α is the SiO_2 leaching rate; k' and k'' are the apparent rate constants; t is the leaching time.

3.2 Effect of leaching temperature

The influence of temperature on SiO_2 leaching rate was studied in a mass ratio of NaOH to SiO_2 2:1 at stirring speed of 300 r/min, and the results are shown in Fig. 4. The temperature has a noticeable influence on SiO_2 leaching rate. The SiO_2 leaching rate increases with the increase of leaching temperature. When the temperature increases from 25 °C to 99 °C, the leaching rate of SiO_2 after 30 min improves from 34.75% to 96.08%. It seems that high temperature makes the reaction molecules move faster and strengthens the liquid–solid mass transfer. In order to achieve a better effect on SiO_2 leaching reaction and avoid excessive energy consumption, leaching temperature of 90 °C is chosen. Figure 4 also shows that the leaching reaction is very rapid in 1–5 min but quite slow in 5–30 min. Such as at 90 °C, the leaching rate of SiO_2 reaches 91.85% in

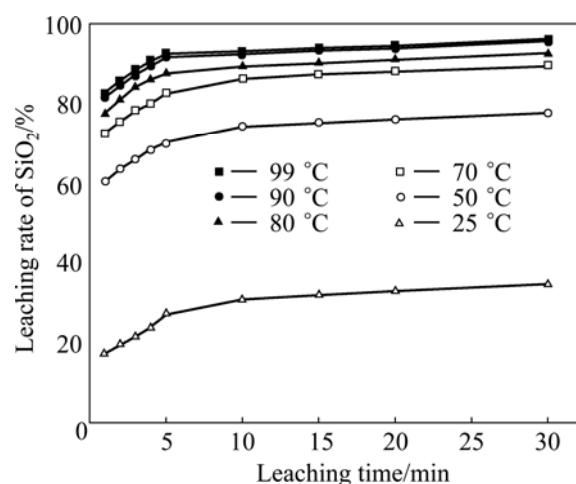


Fig. 4 Effect of leaching temperature on SiO_2 leaching rate

1–5 min, while increases by just 3.81% in 5–30 min. Thereby, the effect of leaching temperature on SiO_2 leaching rate is divided into two parts: 1–5 min is the first stage; 5–30 min is the second stage [17].

In order to determine the kinetic parameters and rate-controlling step for SiO_2 leaching from Al_2O_3 extracted slag, the experimental data presented in Fig. 4 were analyzed on the shrinking core model. By fitting the experimental data in Fig. 4 and Eq. (5) or Eq. (6), the results are shown in Fig. 5 and Fig. 6, respectively. In Table 2, the apparent rate constants and correlation

coefficient values are given for outer diffusion of no product layer and surface chemical reaction.

Table 2 shows that the apparent rate constants of the first stage are bigger than that of the second stage in the two rate-controlling steps, and there is an order of magnitude between the two stages. It also illustrates that the leaching reaction is very rapid in the first stage but quite slow in the second stage in the experimental temperature range.

According to Arrhenius equation, it is represented as [18]

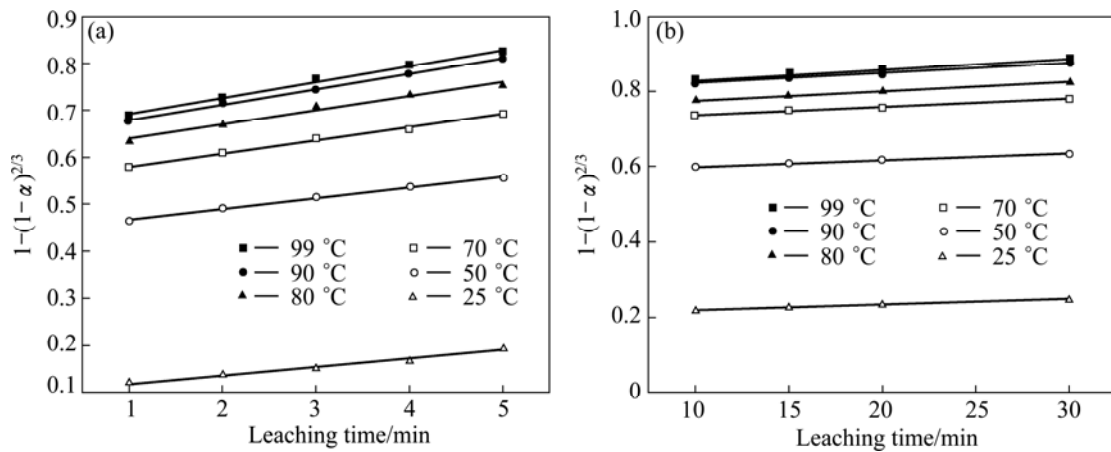


Fig. 5 Plots of $1-(1-\alpha)^{2/3}$ vs time at different temperatures: (a) The first stage; (b) The second stage

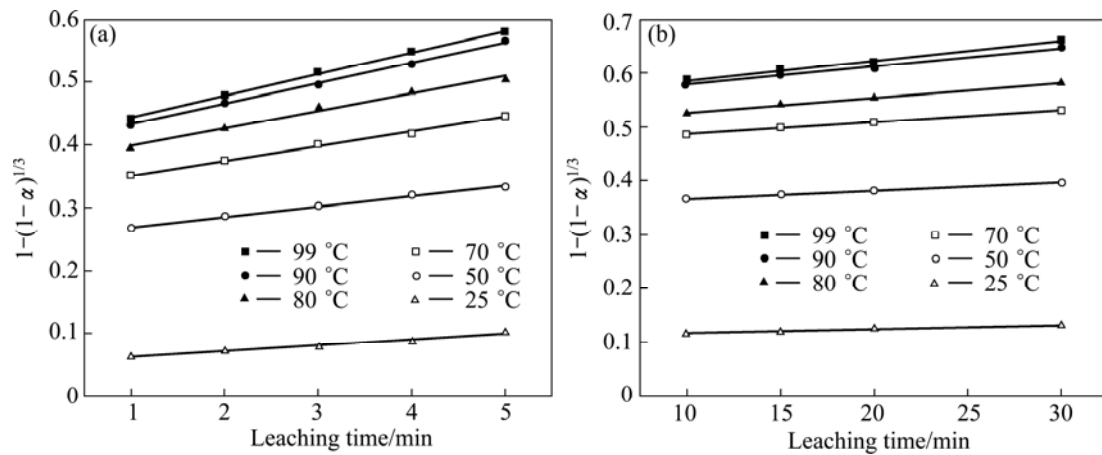


Fig. 6 Plots of $1-(1-\alpha)^{1/3}$ vs time at different temperatures: (a) The first stage; (b) The second stage

Table 2 Apparent rate constants and correlation coefficients at different temperatures

Reaction temperature/°C	Outer diffusion controlling				Surface chemical reaction controlling			
	The first stage		The second stage		The first stage		The second stage	
	k'/min^{-1}	R^2	k'/min^{-1}	R^2	k''/min^{-1}	R^2	k''/min^{-1}	R^2
99	0.0340	0.9920	0.0028	0.9924	0.0348	0.9981	0.0037	0.9890
90	0.0327	0.9980	0.0026	0.9915	0.0326	0.9966	0.0034	0.9905
80	0.0301	0.9697	0.0024	0.9951	0.0273	0.9790	0.0027	0.9971
70	0.0279	0.9933	0.0022	0.9944	0.0230	0.9938	0.0023	0.9961
50	0.0230	0.9907	0.0018	0.9971	0.0164	0.9931	0.0015	0.9978
25	0.0174	0.9762	0.0014	0.9903	0.0094	0.9740	0.0008	0.9906

$$\ln k = \ln A - E/(RT) \quad (7)$$

where k is the reaction rate constant; A is the pre-exponential factor; E is the reaction activation energy; R is the mole gas constant; T is the thermodynamic temperature.

To calculate the activation energy, the experimental data presented in Table 2 were used to make a plot of $\ln k$ vs T^{-1} . The Arrhenius plots of outer diffusion of no product layer and surface chemical reaction are shown in Fig. 7 and Fig. 8, respectively. And the activation energies are listed in Table 3.

Table 3 shows that when the SiO_2 leaching process is controlled by outer diffusion of no product layer, the

reaction activation energies of the first and second stages are 8.492 kJ/mol and 8.668 kJ/mol, respectively. Whereas, when the SiO_2 leaching process is controlled by surface chemical reaction, the reaction activation energies of the first and second stages are 16.566 kJ/mol and 19.088 kJ/mol, respectively. As we all know, the activation energy of outer diffusion controlled process is typically between 4 and 12 kJ/mol [15,16], while, for a chemically controlled process, the value is usually greater than 41.8 kJ/mol [15]. Therefore, the value of activation energy indicates that the SiO_2 leaching process from Al_2O_3 extracted slag with NaOH solution is controlled by outer diffusion of no product layer.

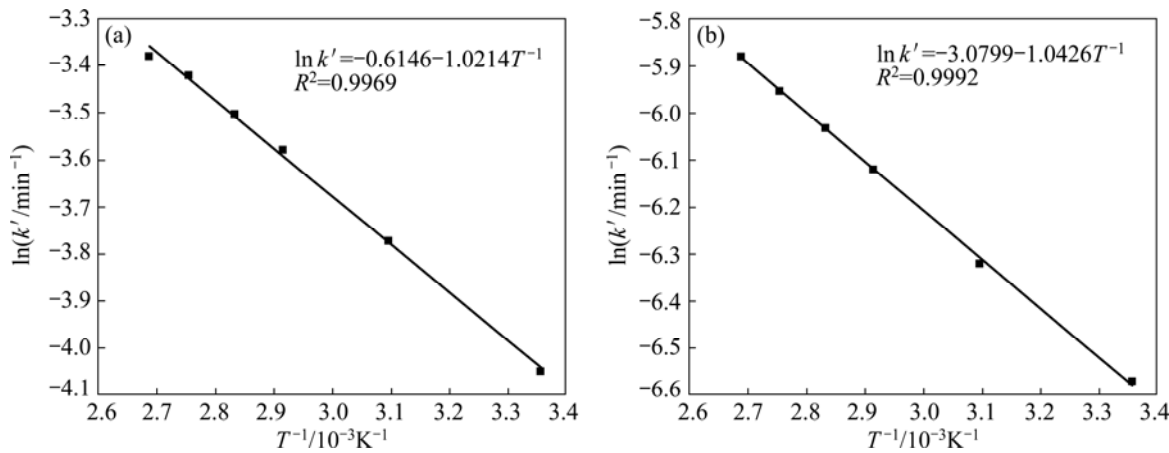


Fig. 7 Arrhenius plot of outer diffusion controlling: (a) The first stage; (b) The second stage

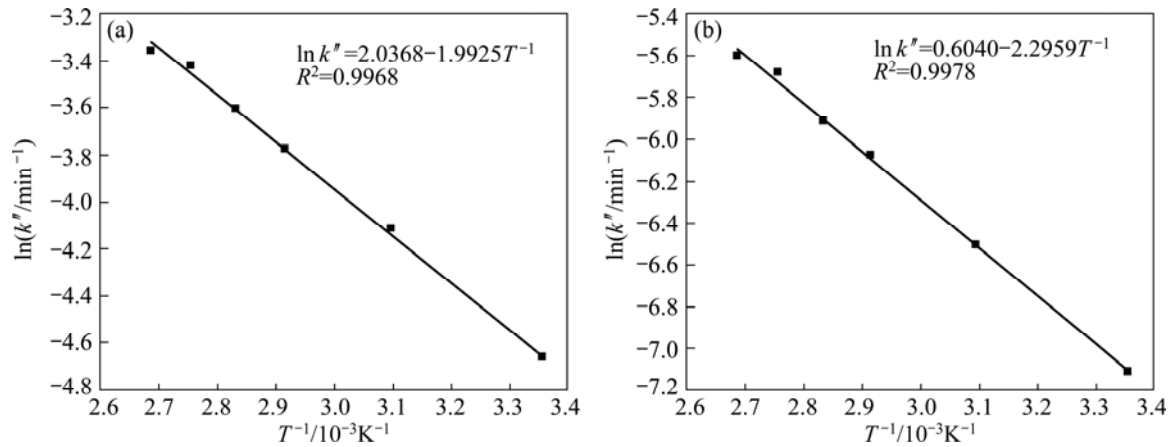


Fig. 8 Arrhenius plot of surface chemical reaction controlling: (a) The first stage; (b) The second stage

Table 3 Activation energies and kinetic equations of SiO_2 leaching from Al_2O_3 extracted slag

Item	Outer diffusion controlling		Surface chemical reaction controlling	
	The first stage	The second stage	The first stage	The second stage
A	0.5409	0.0460	7.6660	1.8294
$E/(\text{kJ}\cdot\text{mol}^{-1})$	8.492	8.668	16.566	19.088
Kinetic equation	$1-(1-\alpha)^{2/3}=$	$1-(1-\alpha)^{2/3}=$	—	—
	$0.5409\exp[-8492/(RT)]t$	$0.046\exp[-8668/(RT)]t$		

3.3 Effect of mass ratio of NaOH to SiO₂

The effect of the mass ratio of NaOH to SiO₂ on SiO₂ leaching rate was examined under the conditions of leaching temperature 90 °C and stirring speed 300 r/min, and the results are shown in Fig. 9. When the mass ratio of NaOH to SiO₂ increases from 1:1 to 3:1, the SiO₂ leaching rate improves with increasing the mass ratio of NaOH to SiO₂. Excess NaOH can make the reaction molecules contact better and ensure sufficient reactions. However, when the mass ratio of NaOH to SiO₂ is 4:1, the reaction between NaOH and Al₂O₃ is accelerated. When the concentration of Al in the solution reaches a certain value, NaAl(OH)₄ could react with Na₂O·nSiO₂ to generate sodium–silicon slag, which will lead to the decrease of SiO₂ leaching rate. When the mass ratio of NaOH to SiO₂ is 2:1, SiO₂ leaching reaction can achieve a better effect and the cost is low, so the mass ratio of NaOH to SiO₂ of 2:1 is chosen. Figure 9 shows that leaching reaction is rapid in 1–5 min while slow in 5–30 min. Therefore, the effect of mass ratio of NaOH to SiO₂ on SiO₂ leaching rate is also divided into two parts.

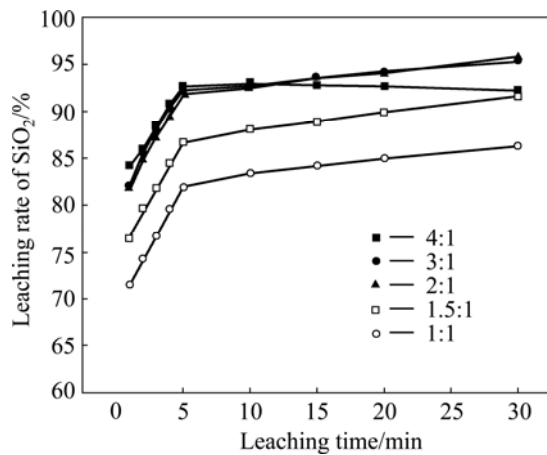


Fig. 9 Effect of mass ratio of NaOH to SiO₂ on SiO₂ leaching rate

The experimental data presented in Fig. 9 were brought into Eq. (5) for linear fitting, and the results are shown in Fig. 10. It can be seen that there is a good linear relationship between $1-(1-\alpha)^{2/3}$ and time. It is found that the SiO₂ leaching process is controlled by outer diffusion of no product layer at different mass ratios of NaOH to SiO₂. The apparent rate constants and correlation coefficient values are shown in Table 4.

3.4 Effect of stirring speed

Experiments with various stirring speeds were carried out under typical test conditions of leaching temperature 90 °C and mass ratio of NaOH to SiO₂ 2:1. The results are shown in Fig. 11. It is found that the stirring speed has a remarkable influence on SiO₂ leaching rate. The SiO₂ leaching rate increases with the

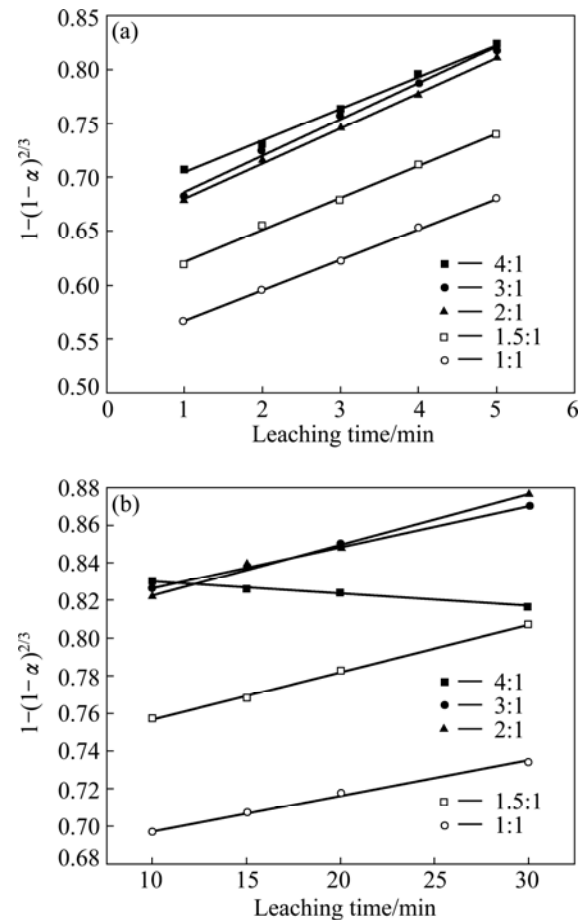


Fig. 10 Plots of $1-(1-\alpha)^{2/3}$ vs time at different mass ratios of NaOH to SiO₂: (a) The first stage; (b) The second stage

Table 4 Apparent rate constants and correlation coefficients at different mass ratios of NaOH to SiO₂

Mass ratio of NaOH to SiO ₂	Outer diffusion controlling			
	The first stage		The second stage	
	k'/min^{-1}	R^2	k'/min^{-1}	R^2
4:1	0.0296	0.9950	−0.0006	0.9929
3:1	0.0336	0.9928	0.0022	0.9984
2:1	0.0327	0.9980	0.0026	0.9915
1.5:1	0.0298	0.9971	0.0025	0.9973
1:1	0.0286	0.9992	0.0019	0.9941

increase of stirring speed. A typical characteristic of outer diffusion is that increasing stirring speed increases the leaching rate [16]. Thus, Fig. 11 also illustrates that the rate-controlling step of SiO₂ leaching process is outer diffusion. When the stirring speed is 300 r/min, SiO₂ leaching reaction can achieve a good effect and the energy consumption is low, so the stirring speed of 300 r/min is chosen. Figure 11 shows that SiO₂ leaching rate is rapid in 1–5 min while slow in 5–30 min. Therefore, the effect of stirring speed on SiO₂ leaching rate is also divided into two parts.

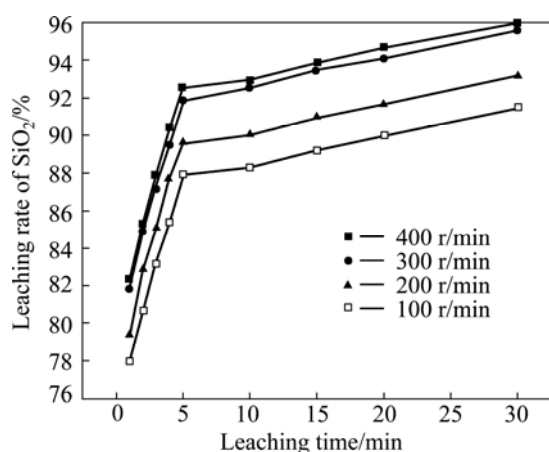


Fig. 11 Effect of stirring speed on SiO₂ leaching rate

The experimental data presented in Fig. 11 were brought into Eq. (5) for linear fitting, and the results are shown in Fig. 12. It is evident that there is a good linear relationship between $1-(1-\alpha)^{2/3}$ and time. This result manifests that the SiO₂ leaching process is controlled by outer diffusion of no product layer at different stirring speeds. The apparent rate constants and correlation coefficients are shown in Table 5.

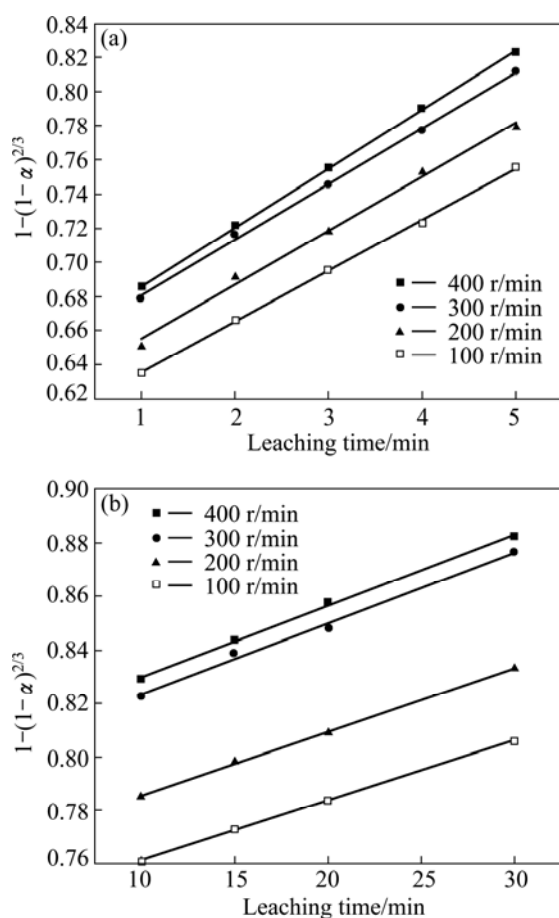


Fig. 12 Plots of $1-(1-\alpha)^{2/3}$ vs time at different stirring speeds: (a) The first stage; (b) The second stage

Table 5 Apparent rate constants and correlation coefficients at different stirring speeds

Stirring speed/ (r·min ⁻¹)	Outer diffusion controlling			
	The first stage		The second stage	
	k'/min^{-1}	R^2	k'/min^{-1}	R^2
400	0.0344	0.9997	0.0027	0.9968
300	0.0327	0.9980	0.0026	0.9915
200	0.0317	0.9926	0.0024	0.9987
100	0.0298	0.9991	0.0022	0.9994

3.5 Characterization of residue

The residue, which reacted for 30 min under the optimized conditions of leaching temperature 90 °C, mass ratio of NaOH to SiO₂ 2:1 and stirring speed 300 r/min, was characterized by chemical analysis, XRD pattern and SEM image.

The chemical analysis of residue is shown in Table 6. Compared with Al₂O₃ extracted slag, the SiO₂ content in the residue is lowered while the Al₂O₃ content is improved. From Table 6, it can be seen that only 22.33% of SiO₂ remains in the residue. WU et al [19] studied extracting Al₂O₃ from fly ash by acid method, and then investigated the dissolving behavior of SiO₂ in Al₂O₃ extracted slag with high concentrated NaOH solution. In their work, the mass fraction of SiO₂ in residue was 37.79%. Thereby, SiO₂ leaching effect is better by the present method.

Table 6 Composition of residue (mass fraction, %)

Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	TiO ₂
11.87	22.33	2.69	0.50	6.21

The XRD pattern of residue is shown in Fig. 13. As illustrated in Fig. 13, there is no mineral phase of sodium-silicon slag, indicating that the SiO₂ leaching reaction is efficient and less SiO₂ is lost under the optimized conditions. Additionally, Fig. 13 also shows

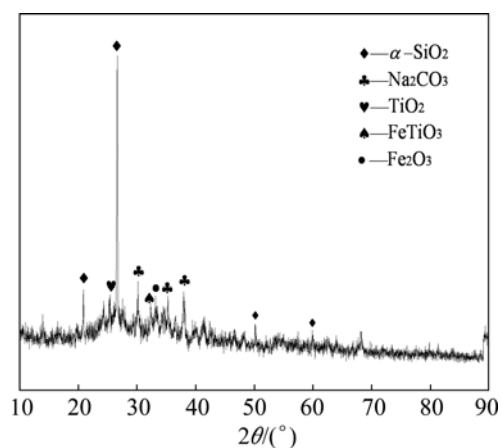


Fig. 13 XRD pattern of residue

that the glass phase disappears, whereas, α - SiO_2 phase is still there. This result manifests that the reaction activity of glass phase with NaOH is higher than that of α - SiO_2 .

The morphology of residue was examined by SEM as shown in Fig. 14. It can be seen that the particles of Al_2O_3 extracted slag were almost completely broken after NaOH leaching.

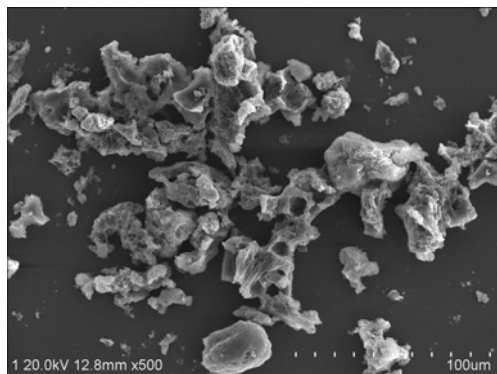


Fig. 14 SEM image of residue

4 Conclusions

1) The effect of leaching temperature, mass ratio of NaOH to SiO_2 and stirring speed on SiO_2 leaching rate from Al_2O_3 extracted slag is noticeable. The SiO_2 leaching rate increases with increasing the leaching temperature, mass ratio of NaOH to SiO_2 and stirring speed. The SiO_2 leaching rate is up to 95.66% under the optimized conditions of leaching temperature 90 °C, mass ratio of NaOH to SiO_2 2:1, stirring speed 300 r/min and leaching time 30 min.

2) The leaching process can be divided into two stages: 1–5 min is the first stage; 5–30 min is the second stage. The leaching reaction is very rapid in the first stage but quite slow in the second stage. The whole leaching process follows the shrinking core model, and the outer diffusion of no product layer is the rate-controlling step. The activation energies of the first and second stages are calculated to be 8.492 kJ/mol and 8.668 kJ/mol, respectively. The kinetic equations of the first and second stages can be expressed as $1-(1-\alpha)^{2/3}=0.5409\exp[-8492/(RT)]t$ and $1-(1-\alpha)^{2/3}=0.046\exp[-8668/(RT)]t$, respectively.

3) After NH_4HSO_4 roasting of fly ash, the Al_2O_3 extracted slag was used as raw material to recover SiO_2 with NaOH solution. Compared with other methods, the SiO_2 leaching process is more efficient in this work, which has many merits of convenient for recycling SiO_2 , less energy consumption and low cost.

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粉煤灰提铝渣碱浸提硅动力学

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摘 要: 对粉煤灰提铝渣碱浸提硅过程的动力学进行研究。实验考察碱浸温度、碱硅比(NaOH 与 SiO_2 质量比)及搅拌速度对二氧化硅浸出率的影响。实验结果表明, 升高碱浸温度、加大碱硅比及加强搅拌速度对浸出过程有利, 在最优条件下, 二氧化硅浸出率为 95.66%。该浸出过程分为两个阶段, 在第一阶段二氧化硅的浸出速率快, 在第二阶段二氧化硅的浸出速率慢, 两阶段均符合收缩核模型且受不生成固体产物层的外扩散控制。碱浸提硅过程第一阶段和第二阶段的反应活化能分别为 8.492 kJ/mol 和 8.668 kJ/mol, 分别求得这两个阶段的动力学方程。

关键词: 粉煤灰; 浸出; 动力学; 二氧化硅

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