

The effect of ultrasonic treatment on alumina leaching from calcium aluminate slag

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2.1. Materials and apparatus

Abstract

The effect of ultrasonic treatment on the alumina leaching properties of calcium aluminate slags was investigated. The effects of the main leaching parameters such as ultrasonic power, leaching time, leaching temperature, and the concentration of sodium carbonate on alumina leaching rate were determined. The mechanism of ultrasonic effects on leaching is also discussed. It was found that the increase in leaching rate was slight, but leaching conditions were improved significantly when ultrasonic treatment was used in the leaching process. Leaching temperature and sodium carbonate concentration decreased by 30°C and $60geL^{-1}$ respectively with the addition of ultrasonic treatment. Ultrasonics would not promote the decomposition of γ -2CaO-SiO₂. The agglomeration of leaching residue was prevented by the cavitation and mechanical effect of ultrasonic treatment.

Introduction

With the rapid development of the steel and aluminum industries, the shortage of suitable resources of iron ore and bauxite has attracted considerable attention [1]. Therefore, the utilization of low grade ores becomes imperative. Leaching alumina from resources which are low grade and refractory, such as iron-bearing bauxite, red mud and fly ash, has become a research focus in alumina production [2-4]. Iron-bearing bauxite is treated by the process of "Blast furnace Sintering-". After smelting, a calcium aluminate slag is obtained, and then it is used to leach alumina [5-7].

However, there are many problems in alumina leaching of calcium aluminate slag. The required concentration of sodium carbonate in the leaching solution is high, leaching time is long, and the alumina leaching rate is low [8-9]. Solving these problems effectively could improve the comprehensive utilization value of iron-bearing bauxite.

Ultrasonic wave treatment has advantages in leaching processes of ores which are difficult to treat [10-12]. Both the leaching rate of refractory ores and the leaching conditions are improved with the cavitation effect of the ultrasonic wave [13, 14]. The application of ultrasonics in hydrometallurgy is concentrated in Heavy metal and precious metal metallurgy, but little research has been carried out in alumina production, especially in treating iron-bearing bauxite using ultrasonics.

Therefore, in order to find an effective method to improve the leaching rate and to decrease the concentration of sodium carbonate and leaching temperature enhancing leaching of calcium aluminate slag with ultrasonic treatment was studied.

Experimental

The material used in the leaching process is a calcium aluminate slag which is obtained by treating iron-bearing bauxite with a blast furnace. Table 1 shows the chemical composition of the slag, while Fig.1 shows the XRD results from the slag. The main phases in the slag are $12CaO^{-7}Al_2O_3$ and γ -2CaO-SiO₂. After grinding, the particle size of the slag is below 74µm.



The ultrasonic cleaner (KQ-100) used in the experiments is produced by Kunshan ultrasonic instrument Ltd. The frequency of the ultrasonic wave is 40 kHz, and the power, which is adjustable, is between 40 and 100W. Stirring of the solution is achieved using a high speed mixer. The tri-mouth glass flask is fitted with a thermometer, stirrer and a reflux condenser. Scanning electron microscopy (SHIMADZU SSX-550) is used to observe the surface morphology of the slag.

2.2. Procedure

The sodium aluminate solution obtained from the slag is treated with the carbonization precipitation process, and circulating mother liquid is used to leach new calcium aluminate slag. The conditions of the leaching solution for alumina digestion are listed below: caustic alkali concentration $N_k=7g\cdot L^{-1}$, $\alpha_k=1.6$ (molecular ratio between Na₂O and Al₂O₃), liquid-solid ratio L/S=5. Other conditions are variable in different experiments such as sodium carbonate concentration, leaching time, leaching temperature, and ultrasonic power. The leaching solution is preheated to the required temperature and then poured into the tri-neck glass flask in which the weight of calcium aluminate slag is 10 g. The leaching process begins when the speed of mixer is up to 300r•min⁻¹.

After leaching and dry filtration, the filtrate is used to analyze the composition concentration of solution, and the filter residue is washed and dried for analysis.

Results and discussion

3.1 Effect of ultrasonic power on leaching rate of slag

Variations in ultrasonic power will cause large changes in the sound intensity and the maximum radius of cavitation bubbles. These changes will affect the leaching process of the slag.

The effect of ultrasonic power on leaching rate of slag was investigated under the following conditions: 1), sodium carbonate concentration (Nc)=120g•L⁻¹, leaching temperature (T)=75°C; 2), Nc= $60g•L^{-1}$, T=45°C. Leaching time (t) was 80min, and other conditions were as shown in section 2.2. The results are shown in Fig.2.



Fig. 2 Effect of ultrasonic power on alumina leaching rate

The alumina leaching rate of the slag was improved slightly with ultrasonic treatment, compared with that of slag without ultrasonics, when ultrasonic power was below 180w under condition 1. But if ultrasonic power was increased to 200W, the leaching rate could be improved by 1.5%. That was because the viscosity of the solution decreased and the movement of grains became violent when the power was high. Therefore, the leaching reaction was easy to carry out. For ultrasonic treatment at high temperature, cavitation occurred readily, but the cavitation intensity decreased with increasing temperature. In other words, because the leaching rate was high temperature, the effect of ultrasonic on leaching rate was slight.

The leaching rate increased significantly with increasing ultrasonic power under condition 2. The leaching rate changed little when ultrasonic power was up to 80W. The alumina leaching rate was improved by 5% with ultrasonic treatment. The optimal leaching rate obtained under condition 1 was close to that obtained under condition 2. This suggested that the ultrasonic

treatment could improve the leaching conditions, but could not increase the maximum leaching rate.

In addition, the increase of the useless bubble and the formation of a sound barrier, were the reason why the leaching rate changed slightly when the ultrasonic power was higher under both conditions.

3.2 Effect of leaching time on leaching rate of slag

Leaching rates with different leaching times were investigated under the following conditions: Nc=120g•L⁻¹, T=75°C and 45°C. Ultrasonic power, fixed according to section 3.1 was 120W in the following experiments. The results are shown in Fig.3.



Fig. 3 Effect of leaching time on leaching rate

The leaching rate of slag with ultrasonic and without ultrasonic treatment increased slightly with increasing leaching time when the leaching temperature was 75°C. That was because the reaction speed was fast at this temperature.

With ultrasonic assistance, alumina leaching rate was improved visibly with increasing leaching time when leaching temperature was 45° C. Extended leaching time had little effect on leaching rate. That was because the temperature and the initial leaching rate were low, so the cavitation effect was significant when time was less than 80min. When leaching time was over 80min most of the alumina was leached from the slag, so leaching rate increased slightly. The optimal leaching time was 80min, under this optimal condition the leaching rate with ultrasonic was close to the leaching rate which was obtained with or without the existence of ultrasonic when the temperature was 75°C.

Therefore, although ultrasonic could not increase the maximum leaching rate when leaching temperature was 45° C and leaching time was 80min, it has decreased the required leaching temperature by 30° C.

3.3 Effect of Nc on leaching rate of slag

Nc(defined in section 3.1, passage 2) was high in the leaching process without ultrasonics, and this would increase the consumption of sodium carbonate. Experiments with different Nc were carried out under the following conditions: t=80min, L/S=5. The ultrasonic power was 120w. The results are shown in Fig. 4.



Fig.4 shows that with the increase of Nc, alumina leaching rate When leaching temperature was 75°C and Nc increased. increased from 60 $g \cdot L^{-1}$ to 120 $g \cdot L^{-1}$ the alumina leaching rate without ultrasonic increased by 3.3%, but leaching rate with ultrasonic increased by 1.3%. The results indicated that Nc had little effect on leaching rate and the existence of ultrasonic weakened the slight effect when leaching temperature was high.

Nc had a great effect on alumina leaching rate when leaching temperature was 45°C and ultrasonic was not used. Leaching rate increased obviously with the increase of Nc. But with the addition of ultrasonics, Nc had a little effect on leaching rate. The use of ultrasonic not only increases the leaching rate but also weakens the effect of Nc on leaching rate. The leaching rate of slag with ultrasonic under 45° C and $60 \text{ g} \cdot \text{L}^{-1}$ was close to that of slag without ultrasonic under 75°C and 120 g•L⁻¹. In another word, under the circumstances that the extraction rate is guaranteed not to fall, the existence of ultrasonic could not only decrease leaching temperature by 30°C but also decrease Nc by 60 g•L⁻¹.

3.4 Effect of leaching temperature on leaching rate of slag

The effect of leaching temperature on leaching rate of slag was studied when Nc was 60g•L⁻¹, leaching time was 80min, and L/S was 5. The results were shown in Fig.5.



Fig 5 Effect of leaching temperature on leaching rate

Leaching rate of slag without ultrasonic improved with increasing leaching temperature. Leaching rate with ultrasonic assistance was better than that without ultrasonics. When leaching temperature was 45° C and 75° C leaching rate of slag with ultrasonic increased by 10% and 4%. Effect of leaching temperature on leaching rate of slag with ultrasonic was not obvious. This meant that the addition of ultrasonic treatment weakened the effect of leaching temperature on the leaching rate of the slag.

Generally, the increase of temperature will decrease the viscosity and promote molecular motion and Mass Transfer of liquid systems. So leaching rate without ultrasonic increased obviously with the increase of leaching temperature. When there was ultrasonic treatment, the vibration and cavitation effect also accelerated the speed of molecular motion and mass transfer in the liquid system. And under this condition the increase of temperature could not promote the leaching process unceasingly, so the effect of temperature was weakened.

3.5 Effect of ultrasonic on stability of y-2CaO+SiO2 in slag

Although γ -2CaO-SiO₂ was stable, some of it in slag was still decomposed in sodium carbonate solution. The cavitation effect of ultrasonic treatment would cause instantaneous high local temperatures and pressures. Shock waves and micro jets would be generated which increase the reaction activity of solids [14]. Meanwhile, the shock wave could intensify the relative motion and collision probability of reactants. If the decomposition of γ - $2CaO \cdot SiO_2$ was intensified with the existence of ultrasonic, the loss of alumina would increase, and these results were not expected [15]. Therefore, the content of silica in leaching solution was investigated under different ultrasonic power. Leaching time was 80min and L/S of slurry was 5. Other conditions and results are shown in Fig.6.



Silica content decreased with the increase of ultrasonic power when leaching temperature was 45° C and Nc was $60g \cdot L^{-1}$. The silica content changed little when temperature was 75°C and Nc was 120g•L⁻¹. This suggests that ultrasonic treatment does not promote the decomposition of y-2CaO•SiO₂. Silica content under the former condition was much lower than that under the latter condition. So the influencing factors of decomposition of y-2CaO•SiO₂ were leaching temperature and sodium carbonate concentration Ultrasonics did not appear to affect the

decomposition of γ -2CaO•SiO₂. On the contrary, the improved leaching conditions decreased the decomposition of γ -2CaO•SiO₂.

3.5 Effect mechanism of ultrasonic on alumina leaching property of slag

In addition to the cavitation effect, a mechanical effect would be generated by ultrasonics. The shock wave whose speed could reach 1000m•ns⁻¹ damaged and sheared the surface of solid particles, this helps to sustain a high reaction activity. In order to study the mechanism of ultrasonic treatment, micro morphology of the leaching residue was observed by SEM. The results were shown in Fig.7 and Fig.8.



Fig.7 SEM image of slag with ultrasonic wave ($\times 300$)



Fig.8 SEM image of slag without ultrasonic wave ($\times 300$)

The dispersion of leaching residue was better when ultrasonic was used in leaching process. The particle size of most of the residue was small. But the dispersion of leaching residue was worse when ultrasonic was not used in the leaching process. The agglomeration phenomenon was serious under these conditions. In addition, other changes such as cavities caused by shock waves and micro jets were not found in the images. Therefore, the effect mechanism of ultrasonic which improved the leaching process was that ultrasonic enhanced the dispersion of solid particle and prevented the agglomeration of leaching residue.

Conclusions

(1) Ultrasonic could not increase the alumina leaching rate greatly, but it could improve the leaching condition obviously. The leaching temperature decreased from 75° C to 45° C, and

sodium carbonate concentration decreased from 120 geL 1 to 60 geL $^1.$

(2) Ultrasonic didn't promote the decomposition of γ -2CaO•SiO₂. On the contrary, the improved leaching condition decreased the decomposition of γ -2CaO•SiO₂.

(3) The effect mechanism of ultrasonic was that ultrasonic enhanced the dispersion of solid particle and prevented the agglomeration of leaching residue.

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