Preparation of Ultrafine $\text{SiO}_2$ from Acid Leaching Residue of Zinc Oxide Ore

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ABSTRACT: Ultrafine $\text{SiO}_2$ powder was successfully obtained by carbonating the NaOH leaching solution of the acid leaching residue of zinc oxide ore. The influences of flow rate of CO$_2$ and carbonation temperature on the properties of $\text{SiO}_2$ were discussed. Ultrafine $\text{SiO}_2$ powder was characterized using XRD, SEM and FT-IR. The thermal analysis of ultrafine $\text{SiO}_2$ powder was also investigated from room temperature to 1000 $^\circ$C. The results showed that the ultrafine $\text{SiO}_2$ powder was amorphous, uniformly spherical particles and distributed in a narrow range.

1 INTRODUCTION

Ultrafine $\text{SiO}_2$ powder, as a functional material, has attracted much attention and shows good application value due to excellent and distinguished performance [1-2]. Now, Ultrafine $\text{SiO}_2$ powder is widely applied in many fields including of filler, ceramics, optics and so on [2-4]. There are a variety of methods to prepare ultrafine $\text{SiO}_2$ powder, such as sol-gel method, alkoxide hydrolysis method, precipitation method etc [5-8]. In this paper, the NaOH leaching solution of the acid leaching residue of zinc oxide ore was used as raw material. The carbonation method, as a simple and effective wet chemical method, was applied in preparing ultrafine $\text{SiO}_2$ powder. The influences of solution temperature, flow rate of CO$_2$ and solution pH value were discussed. XRD, SEM, FT-IR and TG-DTA were adopted to characterize the ultrafine $\text{SiO}_2$ powder.

2 EXPERIMENTS

2.1 Materials and apparatus

The NaOH leaching solution of the acid leaching residue of zinc oxide ore was used as raw material. The leaching solution was mixed solution of $\text{Na}_2\text{SiO}_3$ and NaOH. CO$_2$ was used as carbonation reactant. Beaker, stirrer and thermostat water bath were used.

2.2 Experimental procedure

A certain amount of acid leaching residue of zinc oxide ore and NaOH solution were put into a 2000 mL beaker which was fixed in a thermostat water bath. The thermostat water bath was kept at the desired temperature. The solution was agitated using a double-leaf agitating at 400 rpm controlled by a digital variable speed motor. When the leaching reaction ended, the slurry was filtered to obtain the filter cake and filtrate. The filter cake was washed and dried in oven.

The solution after purification was added into a 1000 mL beaker fixed in a thermostat water bath. When the desired temperature reached, the CO$_2$ was flowed into the solution at a certain speed. The pH value of the solution was monitored by using the pH meter and the precise pH indicator paper. When the reaction ended, the mixture was filtrated to separate the precipitation and solution. The ultrafine $\text{SiO}_2$ powder was obtained after drying the precipitation.

3 CHARACTERIZATION

The phase of the ultrafine $\text{SiO}_2$ powder was examined on a Japan Rigaku X-ray powder diffractometer. The morphology of the ultrafine $\text{SiO}_2$ powder was observed on a Japan shmadzu SSX-550 scanning electron microscope. The thermal analysis of the ultrafine $\text{SiO}_2$ powder was carried out on a SDT 2960 synchronous DSC-TGA.
4 DISCUSSIONS

4.1 Influences of flow rate of CO\textsubscript{2} and temperature on the precipitation ratio of SiO\textsubscript{2}

The influences of flow rate of CO\textsubscript{2} and carbonation temperature on the precipitation ratio of SiO\textsubscript{2} were shown in Fig.1. It can be seen that the precipitation ratio increased along with the flow rate of CO\textsubscript{2} and carbonation temperature rising. When the flow rate of CO\textsubscript{2} was higher than 4 L·min\textsuperscript{-1}, the precipitation ratio was stable. The precipitation ratio increased below 65 °C. So the appropriate carbonation conditions were reaction temperature 65 °C and flow rate of CO\textsubscript{2} 4 L·min\textsuperscript{-1}.

![Figure 1. Influences of flow rate of CO\textsubscript{2} and temperature on the precipitation ratio of SiO\textsubscript{2}](image)

4.2 Characterization of ultrafine SiO\textsubscript{2} powder

Fig.2 shows the XRD pattern and SEM image of ultrafine SiO\textsubscript{2} powder. The XRD pattern was obtained using X-Ray diffractometer, employing Cu Ka radiation with a acceleration voltage 40kV, scanning rate 6°·min\textsuperscript{-1}, 2θ range from 10° to 70°. The obtained sample was amorphous silica from XRD pattern. No sharp diffraction peak was observed, but only a gentle diffraction peak. The particles were regular sphere with uniform granularity.

![Figure 2. XRD pattern, SEM image of ultrafine SiO\textsubscript{2} powder](image)

4.3 Thermal analysis of ultrafine SiO\textsubscript{2} powder

Fig.3 shows the Thermal analysis and XRD pattern of ultrafine SiO\textsubscript{2} powder. It was obvious that there were two obvious endothermic peaks around at 80 °C and 510 °C, which corresponded to those two obvious weight loss. The first one endothermic peak was attributed to the elimination of absorbed water. The second one was attributed to the elimination of constitution water. There was no endothermic peak from 600 °C to 1000 °C. Only a fluctuation disappeared at around 725 °C. The XRD pattern of the ultrafine SiO\textsubscript{2} powder obtained at 1000 °C showed that the roasted ultrafine SiO\textsubscript{2} powder was still amorphous. The ultrafine SiO\textsubscript{2} powder had good thermal stability.

Fig. 4 shows the FT-IR spectra of ultrafine SiO\textsubscript{2} powder dried and roasted in the wavenumber range from 4000 cm\textsuperscript{-1} to 400 cm\textsuperscript{-1}. There are broad absorption bands nearby at 1100, 800 and 490 cm\textsuperscript{-1}, which are attributed to the asymmetric stretching vibration, symmetric stretching vibration and bending vibration of Si-O-Si bond, respectively.
The absorption bands due to silanol stretching and bending vibration at 1650 and 950 cm\(^{-1}\) are observed in fig. 4(a), but disappeared in Fig. 4(b). A broad absorption band around 3462 cm\(^{-1}\) is designated to O-H vibration of the absorbed H\(_2\)O. The FT-IR spectrum reveals much hydroxyl existing in ultrafine SiO\(_2\) powder.

5 CONCLUSIONS

The appropriate carbonation conditions were reaction temperature 65 °C and flow rate of CO\(_2\) 4 L·min\(^{-1}\). The obtained ultrafine SiO\(_2\) powder was amorphous with regular sphere shape. The ultrafine SiO\(_2\) powder had good thermal stability. The ultrafine SiO\(_2\) powder roasted at 1000 °C was still amorphous. The absorption bands nearby at 1100, 800 and 490 cm\(^{-1}\) are attributed to the asymmetric stretching vibration, symmetric stretching vibration and bending vibration of Si-O-Si bond.

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