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Purification of Silica Micro Powder and Application in Building Structural Adhesive†

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Silica powder is by-products from the ferrosilicon smelting industry. The material contains some of trace amounts of iron oxide and carbon and other impurities, so its application scope has been blocked. Hydrochloric acid pickling can remove the original iron oxide powder effectively. It was found that iron removal ratio increased with increasing of the amount of hydrochloric acid, when hydrochloric acid was more than original powder 10 g/150 mL (20 % hydrochloric acid), removal iron reaction was basic balance. The carbon can be effectively removed by calcinations and carbon removal ratio increased with increasing of calcinations temperature, the sample colour turned from dark gray to light gray and until white. When the temperature was higher than 400 ºC, the calcinations time was greater than 3 h, carbon combustion reaction was basic balance, which can achieve satisfactory bleaching effect. It was found that particle diameter increased with increasing of calcinations temperature, because in the calcinations process particles occurred fusion and copolymerization phenomenon. It was found that the adding amount of silicon powder appropriately can improve the tensile and bending strength of structural adhesive. The optimum calcinations temperature range was between 400 and 500 ºC, the optimum addition of silicon powder was 6 %.

Keywords: Silica powder, Calcinations, Decarburization, Pickling, Structural adhesive.

INTRODUCTION

Silica powder is a kind of non-crystalline fine particles. It is formed during the smelting of ferrosilicon and silicon, because there is a large number of volatile SiO_2 and Si product in the reduction furnace, when the gas oxidize highly with air and then condensate, finally, it is carried outside furnace by flue gas. The main ingredient is SiO_2, which with an excellent performance of volcanic ash[1]. Currently, commercially available silica powder is the silica fume collected by the dust collector bag. The commercially available silica powder is usually presented as grayish and white colour, this is because it contains carbon, iron oxide and other impurities. The impurities also lead to its narrower usage and low added value of products; while purified silica powder not only can improve the content of active ingredient, but also can broaden their application areas and improve its added value. The purified silica powder is widely used in plastics, paints, rubber and paper industries[2-4].

Structural adhesive is a kind of new materials which is widely used in high-rise and underground construction in recent years. It is gradually received by building construction units for its convenient construction and good performance. Silicon dioxide is a good kind of filler which was produced by structural adhesive, it is not only can significantly change the mobility of the glue, but also enhance the bonding strength and mechanical properties.

The authors studied the application of purified silica powder in building structural adhesive. It not only improves the physical properties of the building structure, but also develops a new application field for industrial silica powder.

EXPERIMENTAL

Industrial silica powder, silicon ferroalloy smelting company in Zhangzhou in Fujian; hydrochloric acid (AR); deionized water (home made); unsaturated polyester resin; curing agent; promoter; fine powder and so on. Muffle furnace SX225212, scanning electron microscopy S23000N, atomic AA2630, absorption spectrophotometer, vacuum filtration machine PC3025N, electronic balance BS224S, universal tensile machine.

Experimental methods: Commonly, methods of silica purification are used and it uses hydrochloric acid pickling to remove the iron[5] and calcination to remove carbon[6]. First of

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all 100 g silica powder was soaked in hydrochloric acid solution, pickling and left for 24 h and then leaching. After that washing the filter cake several times with deionized water until the filtrate was neutral, at last detecting the concentration of ions in the filtrate with an atomic absorption spectrophotometer; the cake was dried at 100 ºC for 2 h and reserved for using.

Putting dried filter cake, respectively in muffle furnace for calcination at different temperatures, measuring the sample weight before and after calcination, calculating the weight loss rate under different calcination temperatures and different calcination time. Weight loss = (weight of samples before calcination - weight of samples after calcination)/weight of samples before calcination. Testing microstructure of calcined samples by SEM; testing strength of structural adhesive by universal tensile machine[7,8].

RESULTS AND DISCUSSION

Pickling to remove iron: The industrial silica powder is the by-product formed in the ferrosilicon smelting, which contains a certain amount of iron oxides, the experiment used hydrochloric acid to remove it.

Putting 10 g samples into 5 beakers, respectively, adding 50, 100, 150, 200 and 250 mL diluted hydrochloric acid solution (concentration 20 %), stirring to fully reaction, then aging for 24 h; after that filtering and washing to neutral, the filtrate was adjusted to the same volume. At last, testing the iron concentration of the filtrate by atomic absorption spectrophotometer and converting to the iron content. The relationship between dosage of hydrochloric acid and iron concentration of the filtrate was showed as Fig. 1.

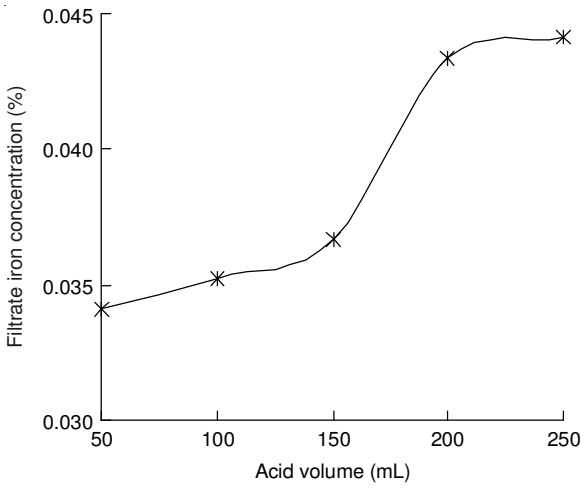

Fig. 1. Curve of acid usage and iron content

Fig. 1 shows that, with the increasing of dosage of hydrochloric acid, the iron content in filtrate increases, when the amount of the acid is greater than 150 mL, the iron concentration tends to be stabilized. From which we know that hydrochloric acid solution can effectively remove the iron of original powder and with the amount of hydrochloric acid increasing, the removal rate of iron increases; when the amount of hydrochloric acid is greater than 150 mL, the removal rate of iron basically achieved a balance.

Calcined to remove carbon: The original silica powder contains little of carbon, which colour is gray. The carbon is oxidized to CO_2 at a high-temperature, thus can achieve the purpose of bleaching. In this experiment, first of all, we put 20 g sample which was washed with acid in the crucible, then calcined in a muffle furnace for 2 h, the calcination temperatures, respectively were 300, 400, 500, 600, 700 and 800 ºC. The relationship between calcination temperature and weight loss shows in Fig. 2.

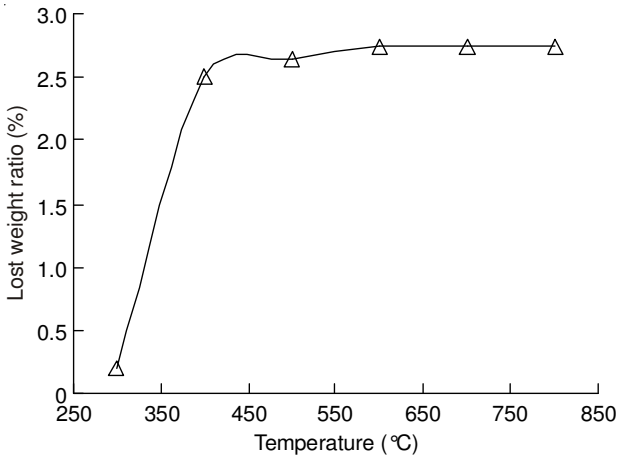

Fig. 2. Curve of calcination temperature and mass loss rate

As Fig. 2 shows, with increasing of calcination temperature, weight loss rate of the sample increased, the sample exterior colour gradually changed from dark gray to light gray and eventually white. When the temperature is lower than 400 ºC, the weight loss rate increased rapidly with extending of time; while the temperature is greater than 400 ºC, the weight loss rate changes gently, which indicating the carbon combustion reaction of sample basically achieved balance. Under the conditions of unchanged calcination time, with the increasing of calcination temperature , the colour of the sample gradually turned from deep to light. At the same time ,the size of the particle diameter became lager, because in the burning process particles get together and converge. When the temperature is lower than 400 ºC, the low temperature has some effect on the removal of carbon. When the temperature is too high, although the sample colour is lighter, particles easily get together again and particles diameter become larger, thus affecting the next application, so this study suggests that the best calcination temperature range from 400 to 500 ºC.

Effect of silica powder dosage on the strength of building adhesive: Adding 4 % of curing agent and right amount of fine powder into 100 g unsaturated polyester resin and fully mixing; respectively adding a certain amount (1, 2, 3, 4, 5, 6, 7, 8 and 9 % in mass ratio) of calcined silica powder (at 400 ºC and calcined 3 h), ultrasound heating (80 ºC) Stir, then adding 0.4 % of the accelerator and stirring. Made into standard test block by the standard test criteria of national building structural adhesive mechanical properties test. The test of tensile strength, bending strength and tensile properties conduct according to GB /T256821995; test of bending performance is according to GB/T257021995. The results show in Figs. 3 and 4, respectively.

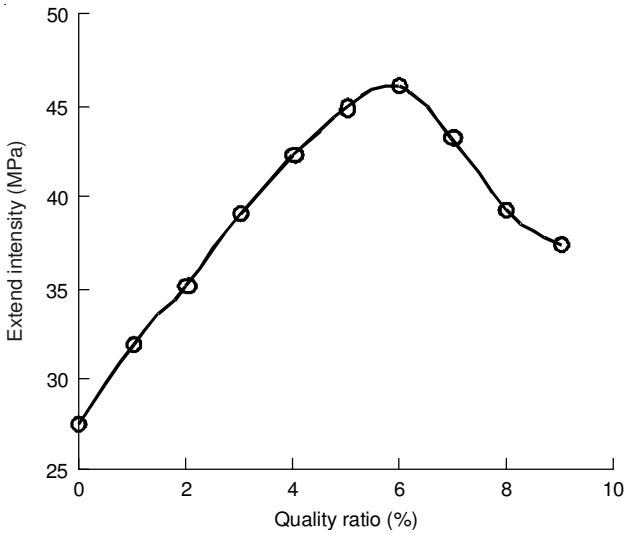

Fig. 3. Effect of adding amount of silica powder on tensile strength of structural adhesive

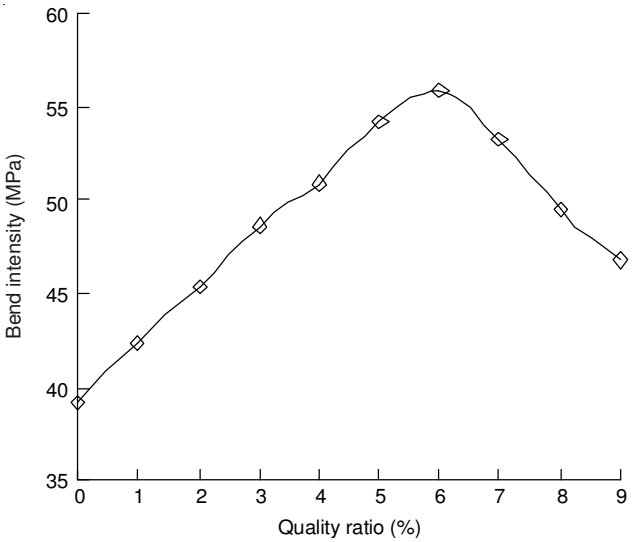

Fig. 4. Effect of adding amount of silica powder on bending strength of structural adhesive

From Figs. 3 and 4, it is clear that with increasing of silica powder dosage, the tensile strength and flexural strength have the same trend, they both reached the maximum at the dosage of 6 % (mass percentage) and the maximum, respectively were 4612 and 5616 MPa, compared with pure poly-unsaturated ester resin, the value, respectively increased by 18146 and 1717 MPa. The mechanical performance decreased with adding silica powder dosage. This phenomenon indicates that silica powder can effectively improve the mechanical performance of structural adhesive. The effect of ultrasonic dispersion is better with adding small amount of silica powder; if dosage is too more, it will affect the uniformity of ultrasonic dispersion, resulting in some silica reunion, which not only reduce its compatibility with structural adhesive and the adhesion of interface, but also reduce percentage of the continuous phase of the structural adhesive. In this experiment the best amount of silica powder is around 6 %.

Conclusion

The study found that hydrochloric acid pickling can remove the iron oxide of original powder effectively, the iron removal ratio increased with increasing of the amount of hydrochloric acid, when hydrochloric acid was more than original powder 10g/150 mL (20 % hydrochloric acid), removal iron reaction was basic balance, the carbon removal ratio increased with increasing of calcinations temperature. The sample colour turned from dark gray to light gray, until white. When the temperature is lower than 400 ºC, the removal rate of carbon increased rapidly with extending of time; with increasing of the calcination temperature, the colour of the sample gradually changed from deep to light. The size of the particle diameter became lager, this because in the burning process particles get together and converge, thus the best calcination temperature of this experiment is ranging from 400 to 500 ºC. In this experiment we also found silica powder can effectively improve the tensile strength and bending strength of structural adhesive.

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