Resource-saving technology of obtaining nanosilica

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Abstract. Possibility of obtaining nanoscale silica from the dust of dry gas cleaning of silicon production by thermal methods is considered in the report. Analysis of the chemical and granulometric compositions of the initial dust, dust after annealing in the muffle furnace is given. A new technology for the thermal-vortex enrichment of dust of dry gas cleaning of silicon to obtain nanosilica is proposed, the results of modeling and laboratory studies are presented.

1. Introduction

Nanosilica is one of the most popular nanomaterials in modern industry. Different types of amorphous nanosilica are used in construction, in the rubber, chemical, tire, food and other industries. There are prospects for its use in metallurgy [1-2]. High-purity nanosilica is used in medicine and is necessary for many scientific studies.

High demand for nanosilica is explained by its unique physico-chemical properties:

- nanosilica is chemically neutral with respect to many known mineral and organic compounds, but it reacts well, for example, with hydrofluoric acid with the creation of hydrofluoric acid;
- has a large specific surface, due to which it is widely used as sorbents, modifiers, etc.

Depending on the purity, granulometric composition, nature of origin and method of production, cost of different varieties of nanosilica can vary thousands of times.

Gas cleaning dust of silicon production. In the production of silicon in ore-thermal furnaces, a large amount of dust is released, which today is one of the accumulating wastes.

2. Gas cleaning dust of silicon production

In the production of silicon in ore-thermal furnaces, a large amount of dust is released, which today is one of the accumulating wastes. Russian enterprises, which produce technical silicon, switch to dry gas cleaning technology, one of the stages of which is dust trapping with the help of bag filters. It is the dust captured by bag filters (hereinafter - dust of bag filters) that is of greatest interest for the producing of amorphous nanosilica.

For researches within the scope of the report, dust samples of bag filters from ZAO “Kremniy” in Shelekhov were selected. Dust of bag filters of ZAO “Kremniy” has the chemical composition given in table 1.
Table 1. Chemical composition of dust of bag filters of ZAO “Kremniy”.

| S No. | Substance  | Content (%) |
|-------|------------|-------------|
| 1     | SiO₂       | > 92        |
| 2     | C          | < 8         |
| 3     | Possible admixtures | < 1        |

Granulometric content of dust of bag filters is given in figure 1 and table 2.

Figure 1. Granulometric content of dust of bag filters of ZAO “Kremniy”.

Table 2. Granulometric content of dust of bag filters of ZAO “Kremniy”.

| x less than, µm | Q (x), % |
|-----------------|---------|
| 0.1             | 10.1    |
| 0.2             | 29.4    |
| 0.3             | 58.6    |
| 0.5             | 77.7    |
| 0.8             | 81.9    |
| 1.3             | 82.6    |
| 2.3             | 85.4    |
| 3.8             | 89.8    |
| 6.5             | 94.3    |
| 10              | 97.3    |
| 18              | 99.4    |
| 30              | 99.6    |

From the chemical and granulometric analyses it can be seen that the dust of bag filters is a mixture of nanostructures based on silicon dioxide and carbon. Isolation of the nanosilica is a promising direction for researches [3-10].

Heat treatment of gas cleaning dust. Based on the chemical composition of dust, it seems appropriate to use heat treatment of dust of bag filters to oxidize (burn out) carbon and obtain silica of a certain purity.

Within the framework of the laboratory experiment, a series of experiments was conducted to burn carbon from dust of bag filters using the laboratory muffle furnace ЭКПС-10. Cuvets with dust of bag
filters were placed in the furnace chamber for a time of 5–45 minutes, at different temperatures 450-800 °C.

A series of experiments showed that the best mode of annealing is holding for 15 minutes at a temperature of 650 °C. A further increase in the annealing time does not lead to a more complete burnout of carbon, and an increase in temperature greater than 650 °C leads to considerable sintering of the particles and the formation of large stable conglomerates.

A visual comparison of the initial and annealed dust of bag filters shows that the annealed dust is much whiter than the original one, which indicates a decrease in the amount of carbon.

Table 3 shows the chemical composition of the dust of bag filters, annealed at 650 °C for 15 minutes.

| S No. | Substance          | Content (%) |
|-------|--------------------|-------------|
| 1     | SiO₂               | > 98        |
| 2     | C                  | < 2         |
| 3     | Possible admixtures| < 1         |

Granulometric composition of dust of bag filters, annealed in a muffle furnace, is shown in figure 3 and in table 2.

Figure 2. Granulometric composition of dust of bag filters, annealed in a muffle furnace.

The results of analyses of dust of bag filters annealed in the muffle furnace show that part of carbon is successfully oxidized, increasing the percentage of silica. However, a significant increase in particle size is observed. These particles can not be decomposed by ultrasound i.e. particle sintering occurred due to prolonged exposure to temperature. Method of annealing the dust of bag filters in the muffle furnace showed its efficiency, however, the following drawbacks were revealed:

- Insufficient burn of carbon;
- Sintering of silica particles into large conglomerates;
- High energy consumption per kilogram of the output.
Table 4. Granulometric composition of dust of bag filters, annealed in a muffle furnace.

| x in µm | Q3(x) in % |
|---------|------------|
| 0.1     | 0          |
| 0.2     | 0.3        |
| 0.3     | 2.1        |
| 0.5     | 3.7        |
| 0.8     | 4.3        |
| 1.3     | 5.2        |
| 2.3     | 7.4        |
| 3.8     | 12.8       |
| 6.5     | 24.8       |
| 10      | 39.8       |
| 18      | 73.4       |
| 30      | 96.7       |

As a result of studying the process of heat treatment of dust of bag filters by the example of annealing in a muffle furnace, the authors proposed a method for burning carbon that does not have the disadvantages mentioned above.

3. Conclusion
Laboratory studies have shown the efficiency of the method of thermo-vortex dust enrichment of bag filters of silicon production. It was possible to achieve a significant reduction in the amount of carbon, as well as to avoid excessive sintering of silica particles. A greater degree of oxidation of carbon without increase of the temperature can be achieved by increasing the time particles spent in the furnace chamber. One way to increase the residence time in the working zone is to increase the size of the working chamber.

A further line of research is the development and construction of a thermal vortex enrichment installation with a large size of working chamber, which will allow make more complete burn of carbon, and also generate more product to study its consumer qualities.

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