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New Approach to Obtaining of Nanosilica and Its Properties

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Experimental procedure, research method and materials

The development of new cost-effective methods for the synthesis of amorphous silica is of great practical importance, since highly dispersed silica is used in various industries in its initial or modified form both to improve the properties of materials and to create new composite materials. Therefore, special attention is paid to resource- and energy-efficient technologies, not requiring some special raw materials or the application of many expensive reagents and equipment

In this work, we study the characteristics of disperse silica synthesized from two types of metallurgical wastes using energy-efficient, practical (i.e., only one reagent is used) and environmentally safe – ammonium fluorine technology. The silica characteristics were analyzed and compared to those of fumed silica A-60 prepared using the pyrogenic synthesis

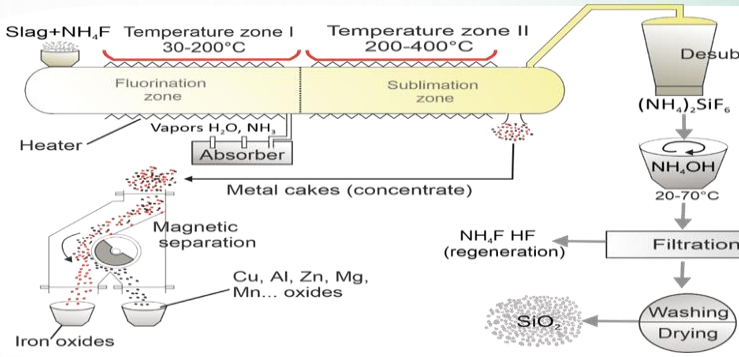
Amorphous SiO_2 synthesis:

- fluorinating reagent - NH_4F (or NH_4HF_2).
- Sintering of the original silica-containing raw material at T = 90 -180 ° C;
- Sublimation of $(NH_4)_2SiF_6$ at T = 320 - 400 ° C

$$SiO_2 + 6NH_4F \xrightarrow{200^0C} (NH_4)_2SiF_6 + 4NH_3 + 2H_2O$$

$$(NH_4)_2SiF_6 + 4NH_4OH \xrightarrow{320\text{-}400^\circ C} SiO_2 + 6NH_4F + 2H_2O$$

Technological scheme of the amorphous SiO_2 synthesis

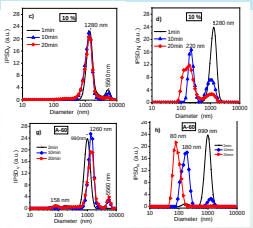


Results and discussion

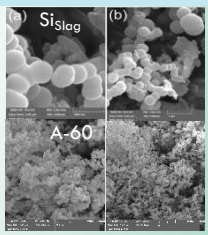
Table. Textural characteristics of synthesized silica samples

Sample	S_{BET} m^2/g	S_{SAXS} m^2/g	S_{micro}, m^2/g	S_{meso}, m^2/g	S_{macro}, m^2/g	V_{micro}, cm^3/g	V_{meso}, cm^3/g	V_{macro}, cm^3/g	V_p, cm^3/g	R_p, nm
Si_{Slag}	58	109	8.5	55	0	0.002	0.22	0	0.22	**7.6**
Si_{MS}	10	91	0.2	0.1	10.2	0	0.06	0.02	0.08	**25.6**
A-60	69	73	32	45	3.2	0.014	0.13	0.07	0.22	**23.8**

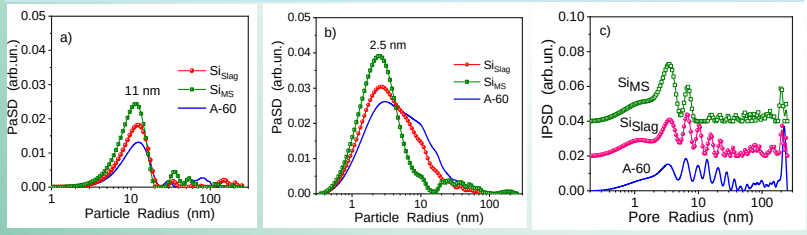
PSDs of the silica samples



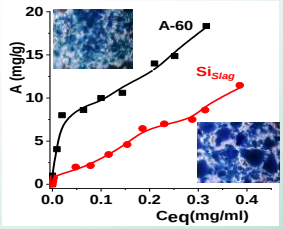
SEM images of A-60, Si_{Slag}



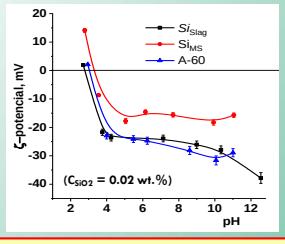
Particle size distributions with (a) a model of spherical particle, (b) a complex model of particles, and (c) incremental pore size distributions (based on SAXS data)



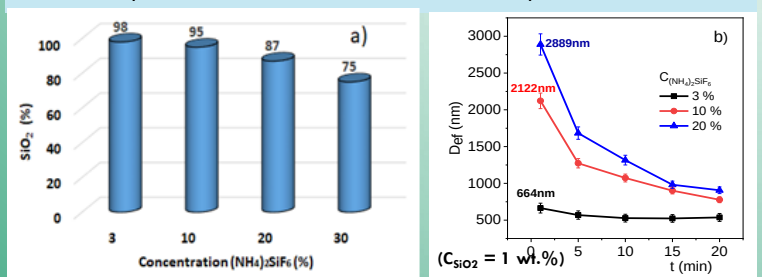
MB adsorption isotherms on the silica samples surface



ξ- potential as a function of pH



Extraction of silicon dioxide at various HFSA concentration in solution (a) and effective diameter of SiO_2 particle as a function of the time of ultrasonic treatment in aqueous medium



Conclusion: High disperse silica powders can be synthesized from metallurgical waste. The silica samples have the amorphous structure and purity of ~99.97 %. The specific surface area is 64 m^2/g and 10 m^2/g for Si_{Slag} and Si_{MS}, respectively. The total pore volume is 0.22 cm^3/g (for Si_{Slag}) and 0.08 $cm3/g$ (for Si_{MS}). There is a fraction of pores inaccessible (closed) for nitrogen molecules that can be detected with SAXS since there is a relation $S_{SAXS} > S_{BET}$. During the silica synthesis, the increase in the $(NH_4)_2SiF_6$ concentration by 10 times led to increasing D_m of the particles more then by 4 times (from 664 nm to 2889 nm) and a decrease in the silica yield by ~25 %. The PSD relative to the volume and numbers of silicas particles synthesized by fluoride technology depend weakly on the HFSA concentration and the sonication time. For all studied silica samples, it is bimodal. But in contrast to Si_{Slag}, for A-60, an increase in the sonication time results in the formation of agglomerates. For the studied silica samples the morphological and textural characteristics (particle and pore size distributions) are similar. Thus, the use of the developed equipment with application of the fluoride synthesis makes it possible to process man-made silicon-containing metallurgical waste and to simultaneously obtain three important products: iron concentrate; concentrate of precious and non-ferrous metals; and amorphous silica of high purity that significantly increase the economic feasibility of this technology usage.

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