

Properties of Amorphous Silica Synthesized from Copper-Smelting Slags

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Highly dispersed powders of amorphous silica (silicon dioxide) with a purity of 99.98 % using ammonium fluoride were synthesized from the man-made waste of copper-smelting production. The amorphous state of the obtained silica samples was confirmed by XRD analysis. It was found that the particle size distribution of SiO₂ in an aqueous medium is mainly represented by aggregates (100-220 nm) and agglomerates of aggregates (1300 and 5500 nm). It is shown that an increase in the time of ultrasonic treatment does not significantly affect the bi- or trimodal character of the particle size distribution. The effective diameter D_{ef} of agglomerates of silica particles decreases by 3 times during the first 10 min of ultrasonic treatment. With a subsequent increase in the processing time, D_{ef} does not change significantly, but fluctuates around some specific value. IR spectra of silica samples have characteristic bands related to bending vibrations of Si–O–Si at 468 cm⁻¹ (intense) and O–Si–O at 800 cm⁻¹ (average intensity), as well as bands related to stretching vibrations of Si–O–Si in the range 1000-1200 cm⁻¹, which are responsible for the reactivity. It was found that the process of thermal desorption from the surface of the SiO₂ sample proceeds in several stages, with the most intense release of the following main volatile products: O (m/z 16), OH (m/z 17), H₂O (m/z 18), and CO (m/z 28). The textural characteristics of the synthesized powders were determined by the method of low-temperature nitrogen adsorption. It is shown that the shape of the adsorption isotherm and the hysteresis loop corresponds to the texture porosity of aggregates of non-porous nanomaterials. The adsorption isotherm is well linearized, which made it possible to calculate the specific surface area according to BET (64 m²/g) and Langmuir (93 m²/g), the total pore volume, micropores, and the total area of micropores.

Keywords: Synthesis, Ammonium fluoride, Industrial waste, Highly dispersed powders, Amorphous silicon dioxide, Particle size distribution.

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1. INTRODUCTION

Amorphous silica, silicon dioxide (SiO₂), can be obtained naturally or synthetically, and is known to have a wide range of applications. Despite numerous studies over the past few decades, the investigation of the structural properties of amorphous silica and composite materials based on it continues to arouse great interest among both researchers and consumers of this product. This is due, firstly, to its widespread use in various industries (construction, rubber industry, production of adhesives, gels and sealants, as well as the creation of heat-resistant materials, polymers, hydrophobic and antifriction coatings, etc.) [1-5]. Composite materials based on highly dispersed amorphous silica are also widely used in optoelectronics and optotelecommunication technologies [6, 7]. Secondly, the study of the amorphous silica structure makes it possible to reveal its new properties, which depend heavily on the method and conditions of synthesis, as well as on the characteristics of the raw materials [7, 8].

At present, to obtain highly dispersed powders of silicon dioxide, both chemical (sol-gel, pyrolysis, plasma-chemical) and physical (dispersion, electron-beam, laser ablation and plasma-arc) methods are used [9-14]. The traditional methods used for the synthesis of highly dispersed SiO₂ powders have their own advantages, but also disadvantages, including the multistage process, high cost of equipment, special and expensive raw materials used, etc.

In this work, the study of the properties of highly dispersed SiO₂ powders synthesized from industrial waste (copper smelting slags) with a specific chemical composition and structure using ammonium fluoride was carried out [15-17].

Thus, the question of the connection between the structural and other characteristics of silicon dioxide is of purely practical importance when using materials based on it as a filler, since the definition of the "structure – property" dependence will allow direct control over the synthesis of new materials.

2. EXPERIMENT

2.1 Materials

Slags from the copper-smelting production of the Almalyk Mining and Metallurgical Combine (AMMC) are the product of the smelting process of copper concentrates containing copper from 10 to 30 %, which are obtained by beneficiation of sulfide copper-molybdenum ores. In this work, dump slags of copper production were used. According to the chemical composition, their main macrocomponents are silicon dioxide SiO₂ and Fe with a content (wt. %) of 29.8-40.0 and 30.7-41.4, respectively. The content of other components, Al₂O₃ (up to 6.0 %), CaO (up to 2.5 %), Cu (up to 0.9 %), MgO (1.3 %), Zn (1.2 %) etc., is insignificant.

Thus, the use of dump slags from AMMC is aimed at developing resource-saving technologies and solving

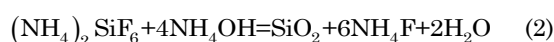
environmental problems associated with the utilization of man-made waste from mining and metallurgical industries.

2.2 Synthesis

For the synthesis of highly dispersed particles of amorphous silica dioxide, ammonium fluoride (NH_4F , Guangzhou Yijia Supply Chain Co Ltd, China) $\geq 98\%$ purity) was used. Fluorination was carried out in a specially designed reactor made of nickel alloy according to the following reaction:



After filtration, by separation from the $(\text{NH}_4)_2\text{SiF}_6$ solution under the action of ammonia water (NH_4OH), according to the reaction



a highly dispersed powder of amorphous SiO_2 was obtained.

2.3 Characterization

X-ray diffraction (XRD) patterns were obtained on a DRON-4-07 X-ray diffractometer (NPO Burevestnik, St. Petersburg, Russia) in filtered $\text{CoK}\alpha$ radiation with the Bragg-Brentano survey geometry. Diffraction patterns were recorded in the 2θ angle range from 10 to 70° at room temperature with a step of 0.05° and exposure at a point of 1 s. A glass cell was used as a substrate.

Particle size distribution (PSD) was studied using a Zetasizer Nano ZS (Malvern Instruments) apparatus using a universal dip cell (ZEN1002) with a wavelength of $\lambda = 633$ nm and a scattering angle of 13 and 176° . Accuracy and reproducibility of particle size are $\pm 1-2\%$. Suspensions of synthesized silicon dioxide samples with a concentration of $C_{\text{SiO}_2} = 1$ wt. % were prepared in de-ionized water using ultrasonic dispersion (Sonopuls HD 2040 homogenizer, Bandelin, 20 kHz, 80 W). To carry out measurements and to process the results obtained, a computer program from Malvern Instruments was used, which assumes that the particles are spherical.

The transmittance infrared (IR) spectra were recorded in the range of $4000-300$ cm^{-1} using a Specord M80 (Carl Zeiss, Germany). The samples were mixed with KBr (Sigma-Aldrich, for spectroscopy) in a ratio of $1:100$, stirred and pressed into thin pellets 8 ± 28 mm in size and 20 ± 0.5 mg in mass.

Thermal desorption mass spectra were studied using one-pass (OP) temperature programmed desorption (TPD) time-of-flight (ToF) method with mass-spectrometry (MS) control using a MSC-3 ("Electron", Sumy, Ukraine) ToF mass-spectrometer [18]. The pressure in the chamber was 4×10^{-6} Pa, the sample mass was 7 mg (with a short distance (~ 0.5 cm) between the sample and the MS detector), and the heating rate (β) was 1.2 K/s. The sample was heated to a maximum temperature of 600°C in 75 min linearly in time at a rate of 0.15°C/s . At the same time, with the heating of the sample, the mass spectra were recorded on a computer, from which the data on the time variation of the amplitude of the required components were then ex-

tracted and the dependences $I_m(t)$ were plotted.

To analyze the textural characteristics of the obtained amorphous silicon dioxide powders, low-temperature (77.4 K) nitrogen adsorption-desorption isotherms were recorded on a Kelvin-1042 adsorption analyzer (Costech Microanalytical). The samples were degassed at 110°C for 2 h in a vacuum chamber. The values of the specific surface area (S_{BET}) were calculated according to the standard BET method [19]. The total pore volume V_p was evaluated from the nitrogen adsorption at $p/p_0 \approx 0.98-0.99$, where p and p_0 denote the equilibrium and saturation pressures of nitrogen at 77.4 K, respectively [20].

3. RESULTS AND DISCUSSION

3.1 Synthesis of Amorphous SiO_2 Particles

The synthesis of amorphous SiO_2 particles was carried out according to reaction (2). For this, ammonium hexafluorosilicate $(\text{NH}_4)_2\text{SiF}_6$ was treated with ammonia water with a concentration of $10-25\%$ at a pH of $8-9$ and a temperature of 25°C . After filtration from the $(\text{NH}_4)_2\text{SiF}_6$ solution, a highly dispersed powder of amorphous SiO_2 was obtained, having a purity of 99.98% , according to mass spectrometry data.

The efficiency of the process of ammonia hydrolysis $(\text{NH}_4)_2\text{SiF}_6$ depends on the values of its concentration in the solution [16]. $(\text{NH}_4)_2\text{SiF}_6$ solutions were prepared with a concentration of $3, 10, 20,$ and 30 wt. %, from which SiO_2 particles were synthesized at the above temperature. The indicators of silicon dioxide extraction were as follows: at a concentration of 3 wt. % of $(\text{NH}_4)_2\text{SiF}_6$ – 98% ; at 10 wt. % – 75% ; at 20 wt. % – 87.5% , and at 30 wt. % – 75% . Thus, a 10 -fold increase in the concentration of ammonium hexafluorosilicate leads to a decrease in product extraction by 25% .

3.2 X-ray Powder Diffraction Analysis

The XRD pattern of synthesized amorphous silicon dioxide powder is characterized by the presence of a wide halo in the range of $2-20^\circ$, which corresponds to the presence of X-ray amorphous material in the sample (Fig. 1).

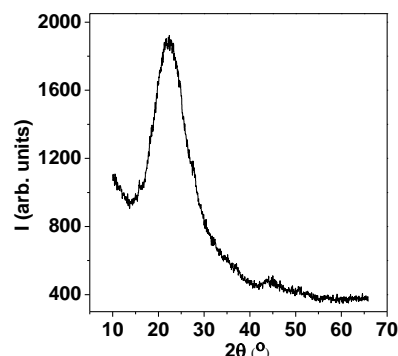


Fig. 1 – XRD pattern of amorphous silicon dioxide powder obtained from dump slags of copper smelting production

3.3 Particle Size Distribution in Aqueous Medium

It was revealed by laser correlation spectroscopy that the PSD of amorphous SiO_2 in an aqueous medium

is polymodal and is represented mainly by aggregates of 100-220 nm and agglomerates with a maximum at 1300 and 5500 nm (Fig. 2). An increase in the time of ultrasonic treatment of aqueous suspensions of silicon dioxide samples from 1 to 25 min does not significantly affect the PSD nature. This indicates the formation of sufficiently strong micron-sized agglomerates, which is apparently due to the type of synthesis and subsequent material processing. At the same time, it can be seen from Fig. 2 that with an increase in the processing time, the maximum values of PSD decrease almost by more than 2 times, for example, in the Intensity curve from 1300 (at 1 min) to 600 nm (at 25 min) and in the Number curve – from 220 to 100 nm, which can be explained by the partial destruction of agglomerates.

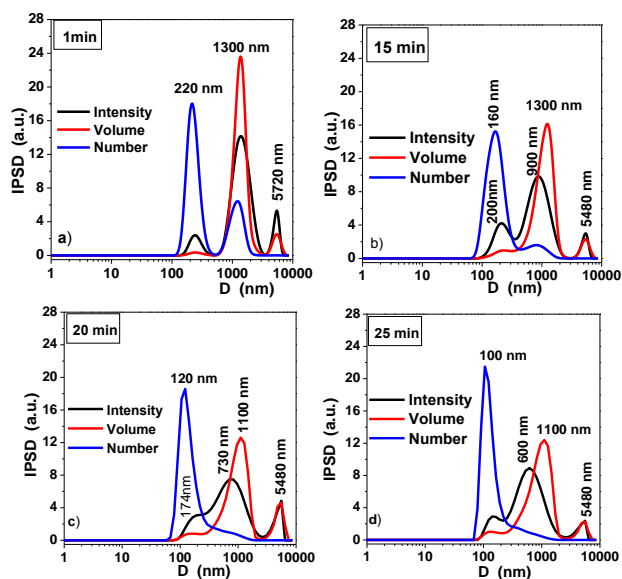


Fig. 2 – PSD related to the intensity of light scattering (I), the volume (V) and the number of synthesized silica particles (N), depending on the time of ultrasonic treatment in an aqueous medium ($C_{\text{SiO}_2} = 1$ wt. %)

The effective diameter (D_{ef}) is the average value of the diameter of the particles in the dispersion, which makes it easier to estimate the change in particle size during processing. D_{ef} values were determined by the intensity of the scattered light

$$D_{ef} = \frac{\sum_i N_i d_i^6}{\sum_i N_i d_i^5}$$

where d_i and N_i are the diameter and number of the i -th particle, respectively [21, 22].

The effective particle diameter of the studied silica samples depends on the time of ultrasonic treatment (Fig. 3). During the first 10 min, there is a significant decrease in D_{ef} from 1600 to 659 nm (2.4 times). With a further increase in the ultrasonic treatment time, the D_{ef} value is in the range of 633-467 nm. This effect can be associated with balancing between the processes of destruction of aggregates and their formation: disaggregation \leftrightarrow aggregation. Therefore, in this case, according to the D_{ef} value, it is correct to estimate not the particles size in the suspension, but the equilibration time between the processes of destruction and formation of aggregates (when D_{ef} stops decreasing).

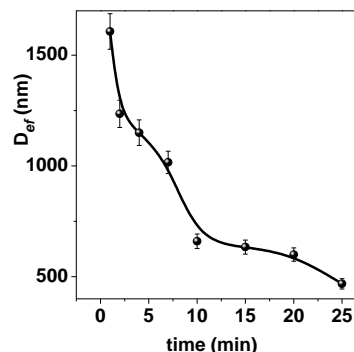


Fig. 3 – The effective diameter of SiO_2 particles (determined from the scattered light intensity) as a function of the time of ultrasonic treatment in an aqueous medium ($C_{\text{SiO}_2} = 1$ wt. %)

3.4 IR Spectroscopy

The IR spectra of silica showed the presence of characteristic bands of bending vibrations Si–O–Si at 468 cm^{-1} (intense) and O–Si–O at 800 cm^{-1} (medium intensity) and bands of stretching vibrations Si–O–Si in the range of $1000\text{-}1200 \text{ cm}^{-1}$ (Fig. 4a). This distinct band characterizes the reactivity of silica. The bands at $1636, 3200\text{-}3500 \text{ cm}^{-1}$, related to adsorbed water, which is associated with silanol groups by hydrogen bonds, indicate its significant content in the sample (Fig. 4b).

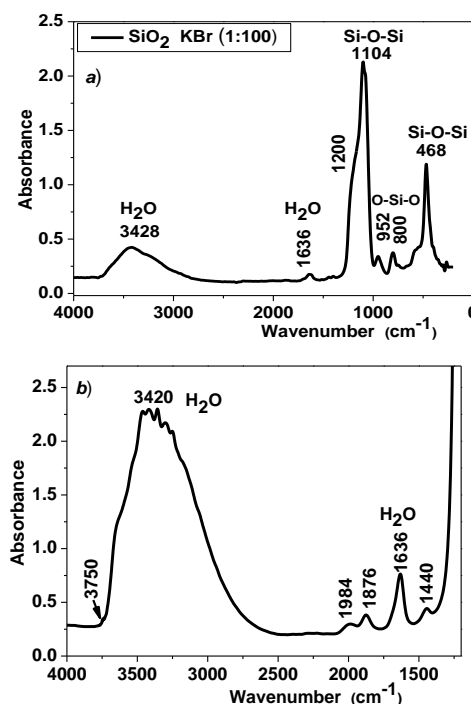


Fig. 4 – The IR spectra of amorphous silicon dioxide powder: a) mixed with KBr in a ratio of 1:100, b) initial, taken for transmission

3.5 TPD Mass Spectra

The mass spectra of the products of thermoprogrammed desorption from the surface of the synthesized SiO_2 sample at two temperatures ($T = 27 \text{ }^\circ\text{C}$ and $T = 600 \text{ }^\circ\text{C}$) are shown in Fig. 5a. Interpretation of the most intense signals in the mass spectra indicates that the most probable volatile products are H_2 (m/z 2), O

(m/z 16), OH (m/z 17), H₂O (m/z 18), CO (m/z 28), CO₂ (m/z 44), NO₂ (m/z 44). Analysis of the TPD mass spectra shows that at low temperatures of the sample, the lines of H₂ (m/z 2) and CO₂ (m/z 44) are the main ones. At high temperatures, the lines of $m/z = 17, 18$ indicate that hydroxyl radicals OH and water molecules H₂O are the main products of TPD.

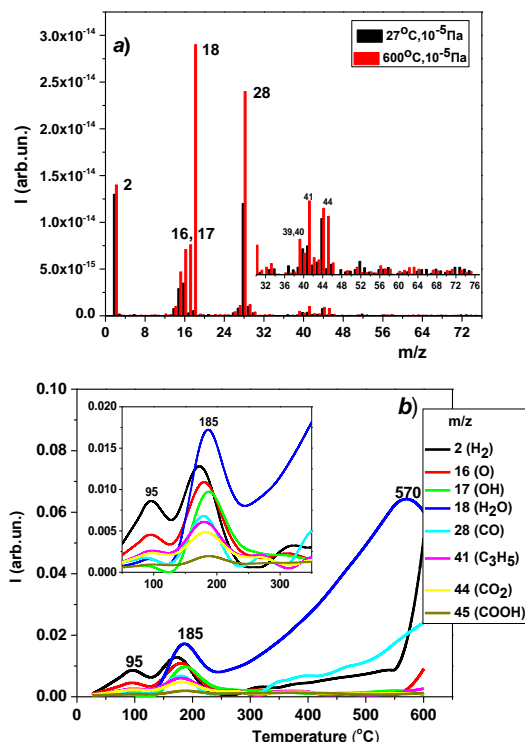


Fig. 5 – TPD mass spectra obtained during thermolysis of the SiO₂ sample at $T = 27$ and 600 °C (a) and thermograms of the main desorption products: H₂ (m/z 2), O (m/z 16), OH (m/z 17), H₂O (m/z 18), CO (m/z 28), CO₂ (m/z 44), NO₂ (m/z 44) (b)

According to the analysis of the thermograms of the main products (Fig. 5b) desorbed from the surface of the SiO₂ sample, the desorption process occurs in two or three stages. Molecular hydrogen is desorbed in three stages at temperatures of 95, 170 and 600 °C, apparently due to the type of bonds with the surface (low-temperature hydrogen may be responsible for van der Waals forces and chemisorption, high-temperature hydrogen – for diffusion from the volume). For curves of oxygen, hydroxyl radical and water, only two desorption stages are observed. The desorption curve of H₂O as the main component confirms the presence of a large amount of adsorbed water in the sample (Fig. 4). The process of H₂O desorption occurs in three stages at different temperature maxima: at $T_{\max} = 95$ and 185 °C, adsorbed water is released, the release area 250 - 570 °C is probably associated with the process of dehydroxylation of the surface of the SiO₂ sample, since there are several types of OH groups on the silica surface (single, vicinal and geminal OH groups, hydrogen-bonded and free) [22]. The desorption stage at $T_{\max} = 185$ °C (Fig. 5b) presumably is related to the products of CO

and CO₂. The wide maximum for m/z 44 can be responsible for both COOH and NO₂. This may be due to the fragmentation of hydrocarbons under the action of an electron beam used as an ionization source.

3.6 Textural Characterization

The nitrogen adsorption isotherm for the obtained silicon dioxide sample is S-shaped. This isotherm shape implies the presence of polymolecular adsorption. The curved initial section indicates a strong interaction of the adsorbate with the silica surface. At high pressures, the isotherm has a bend with a hysteresis loop (type IV) corresponding to the textural porosity of aggregates of nonporous nanoparticles (Fig. 6a). The shape of the hysteresis loop indicates the dominant contribution of mainly cylindrical mesopores (filled with adsorbed nitrogen during measurements) [19, 21].

A study of the specific surface area and sorption capacity of the samples by the method of low-temperature nitrogen adsorption showed that SiO₂ samples have a maximum specific surface area of about 64 m²/g according to BET and 93 m²/g according to Langmuir. The total pore volume is 218 mm³/g, including 3.7 mm³/g of micropores. The total area of micropores is 10.5 m²/g, the non-microporous surface is 53.4 m²/g. The pore size distribution curves calculated from nitrogen adsorption-desorption isotherms show three characteristic maxima related to mesopores ($R = 2$ - 50 nm) (Fig. 6b). Narrow voids between particles closely located in the same aggregates can also contribute to the porosity of the tested sample.

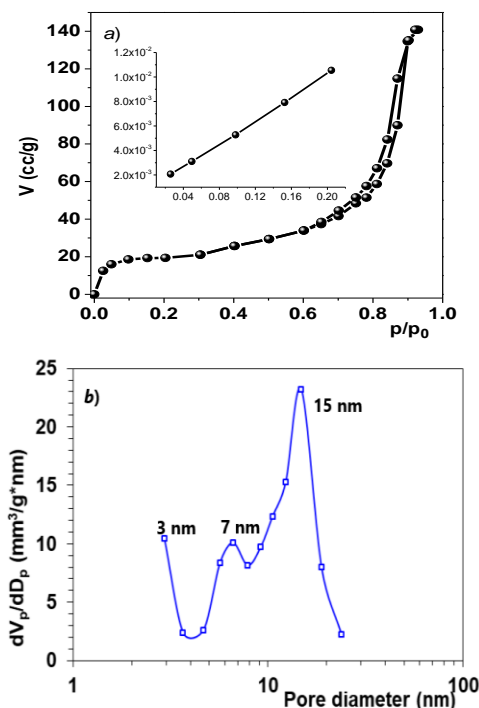


Fig. 6 – Nitrogen adsorption-desorption isotherm (a), incremental pore size distribution for synthesized silica dioxide (b)

4. CONCLUSIONS

The efficiency of hydrolysis of $(\text{NH}_4)_2\text{SiF}_6$ depends on time and concentration of ammonia. In this case, the maximum extraction of SiO_2 , equal to 98 %, occurs at a concentration of ammonium hexafluorosilicate of 3 wt. %. Further increase in the concentration of $(\text{NH}_4)_2\text{SiF}_6$ up to 30 wt. % leads to a decrease in the amount of recovered silicon dioxide to 75 wt. %. Thus, an increase in the concentration of $(\text{NH}_4)_2\text{SiF}_6$ in the solution during hydrolysis by a factor of 10 leads to a decrease in the yield of silicon dioxide by 25 %.

Using the method of X-ray phase analysis, the amorphous state of the obtained SiO_2 samples was confirmed. It was found that the PSD of amorphous SiO_2 in an aqueous medium at a concentration of 1 wt. % is polymodal and is mainly represented by aggregates (100-220 nm) and agglomerates of aggregates (1300-5500 nm). An increase in the time of ultrasonic treatment does not significantly change the nature of the PSD. However, with an increase in the time of ultrasonic treatment to 25 min, a shift of the PSD curves related to intensity towards lower values is observed, which may be associated with partial destruction of agglomerates. The effective diameter D_{ef} of agglomerates of silicon dioxide particles decreases significantly during the first 10 min of ultrasonic treatment (by

2.4 times). With a further increase in processing time, the value of D_{ef} fluctuates around a certain value.

According to thermal desorption mass spectrometry data, the main volatile products of desorption of SiO_2 powders up to 600 °C are H_2O , H_2 , O, OH, CO_2 , CO_5 .

It was shown that the synthesized SiO_2 powders have a maximum specific surface area of 64 m^2/g according to BET and 93 m^2/g according to Langmuir. The total pore volume is 218 mm^3/g , including 3.7 mm^3/g of micropores. The total area of micropores is 10.5 m^2/g , the non-microporous surface is 53.4 m^2/g .

So, the proposed synthesis method allows to process the man-made waste of the copper-smelting production of AMMC and obtain from it a dispersed high-purity silicon dioxide powder (99.98 %) with micron-sized particles and a moderate specific surface area that fully meets the requirements for fillers used in the rubber industry.

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REFERENCES

1. M. Davraz, L. Gunduz, *Cement Concrete Res.* **35** No 7, 1251 (2005).
2. N.C. Camacho, J.R. Vega Baudrit, Y.C. Urena, *Organic & Medicinal Chem. I J.* **5** No 1, 555654 (2018).
3. P.V. Grishin, *Vestnik Kazanskogo Tehnologicheskogo Universiteta* **17** No 18, 239 (2014).
4. *The Surface Properties of Silica* (Ed. by A.P. Legrand) (John Wiley & Sons Ltd: 1998).
5. *Himija Poverhnosti Kremnezema: v 2 ch.*, (Ed. by A.A. Chuiko) (Kiev: Nauk. Dumka: 2001).
6. S. Das, J. Manam, S.K. Sharma, *J Mater Sci: Mater Electron.* **27** No 12, 13217 (2016).
7. T. Uchino, *J. Ceram. Soc. Jpn.* **113** No 1, 17 (2005)
8. L. Khouchaf, K. Boulahya, P. Pratim Das, S. Nicolopoulos, V.K. Kis, J.L. Labar, *Materials* **13**, 4393 (2020).
9. E. Tranquillo, F. Barrino, G.D. Poggetto, I. Blanco, *Materials* **12** No 1, 155 (2019).
10. H.N. Azlinaa, J.N. Hasnidawania, H. Noritaa, S.N. Surip, *Acta Physica Polonica A* **129** No 4, 842 (2016).
11. N.X. Huan, T.N. Anh, N.T. Hang, D.T. Nhung, N.V. Thanh, *J. Viet. Env.* **9**, 255 (2018).
12. S. Stopic, F. Wenz, T.-V. Husovic, B. Friedrich, *Metals* **11**, 463 (2021).
13. V.G. Il'ves, M.G. Zuev, S.Yu. Sokovnin *J. Nanotechnology*, **2015**, 417817 (2015).
14. S.P. Bardakhanov, *Questions of Atomic Science and Technology. Series: Nuclear Physics Research* **5**, 165 (2008).
15. A.N. Dyachenko, R.I. Kraydenko, *Processing for Profit, Social and Environmental Stewardship Proceedings of the 51 Annual Conference of Metallurgists of CIM (COM 2012)*, 238 (Canada: Niagara, ON: 2012).
16. B.M. Abdurakhmanov, M.Sh. Kurbanov, S.A. Tulaganov, M. Ernazarov, L.S. Andriyko, A.I. Marinin, A.Yu. Shevchenko, *Uzbek. Phys. J.* **23** No 1, 65 (2021).
17. V.S. Rimkevich, A.A. Pushkin, I.V. Girenko, *Basic Mental Research* **2**, 6055 (2015).
18. V.M. Gun'ko, V.I. Zarko, B.A. Chuikov, V.V. Dudnik, Yu.G. Ptushinskii, E.F. Voronin, E.M. Pakhlov, A.A. Chuiko, *Int. J. Mass Spectrom. Ion Proces.* **172**, 161 (1998).
19. S.J. Gregg, K.S.W. Sing, *Adsorption, Surface Area and Porosity* (London: Academic Press: 1982).
20. A.W. Adamson, A.P. Gast, *Physical Chemistry of Surface*, 6th ed. (Wiley, New York: 1997).
21. V.M. Gun'ko, *Appl. Surf. Sci.* **307**, 444 (2014).
22. V.M. Gun'ko, V.V. Turov, P.P. Gorbik, *Voda na Mezhdaznoy Granice* (Naukova dumka: Kiev: 2009).

Властивості аморфного кремнезему, синтезованого із мідеплавильних шлаків

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З техногенних відходів мідеплавильного виробництва синтезовано високодисперсні порошки аморфного кремнезему (діоксиду кремнію) чистотою 99,98 % з використанням фториду амонію. Аморфний

стан отриманих зразків кремнезему було підтверджено рентгеноструктурним аналізом. Встановлено, що гранулометричний склад SiO_2 у водному середовищі в основному представлений агрегатами (100-220 нм) і агломератами агрегатів (1300 та 5500 нм). Показано, що збільшення часу ультразвукової обробки не впливає істотно на бі- або тримодальний характер гранулометричного складу. Ефективний діаметр D_{ef} агломератів частинок кремнезему зменшується в 3 рази протягом перших 10 хв ультразвукової обробки. При подальшому збільшенні часу обробки D_{ef} істотно не змінюється, а коливається навколо деякого конкретного значення. ІЧ спектри зразків кремнезему мають характерні смуги, пов'язані з коливаннями вигину Si–O–Si при 468 cm^{-1} (інтенсивні) та O–Si–O при 800 cm^{-1} (середньої інтенсивності), а також смуги, пов'язані з коливаннями розтягу Si–O–Si в діапазоні $1000\text{-}1200 \text{ cm}^{-1}$, які відповідають за реакційну здатність. Встановлено, що процес термічної десорбції з поверхні зразка SiO_2 протікає в кілька етапів, з найбільш інтенсивним виділенням таких основних летких продуктів: O (m/z 16), OH (m/z 17), H_2O (m/z 18) та CO (m/z 28). Текsturні характеристики синтезованих порошків визначали методом низькотемпературної адсорбції азоту. Показано, що форма ізотерми адсорбції та петлі гістерезису відповідає текстурній пористості агрегатів непористих наноматеріалів. Ізотерма адсорбції добре лінеаризована, що дозволило розрахувати питому площу поверхні за методами BET ($64 \text{ m}^2/\text{g}$) і Ленгмюра ($93 \text{ m}^2/\text{g}$), загальний об'єм пор, мікропор і загальну площу мікропор.

Ключові слова: Синтез, Фторид амонію, Промислові відходи, Високодисперсні порошки, Аморфний діоксид кремнію, Гранулометричний склад.