

PRODUCTION OF HIGHLY DISPERSED AMORPHOUS SiO_2 FROM INDUSTRIAL ALKALINE SOLUTIONS USED IN THE REGENERATION OF ION-EXCHANGE RESINS

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ABSTRACT

The possibility of obtaining precipitated silicon dioxide from industrial alkaline silicate solutions, formed during the regeneration of ion-exchange resins contaminated with silicon compounds in uranium hydrometallurgy, has been investigated. SiO_2 precipitation was carried out using sulphuric acid, with the effects of precipitant concentration, temperature, process duration, pH of the medium and drying temperature evaluated. It was established that the optimal conditions for obtaining the precipitate are: a 40% H_2SO_4 solution, a precipitation temperature of 25 °C, a process duration of 2 hours, pH 6–8 and subsequent drying at 300 °C. Under these conditions, an amorphous, highly dispersed silicon dioxide was obtained with a SiO_2 content of up to 86.7% and a specific surface area of up to 357 m^2/g . Based on low-temperature nitrogen adsorption data and BET, BJH and NLDFT calculations, the formation of a highly developed micro-mesoporous structure was confirmed. The results obtained demonstrate the potential for processing alkaline silicate solutions from the regeneration of ion-exchange resins to produce a functional silica material and reduce the volume of industrial waste.

INTRODUCTION

Silicon dioxide is a highly sought-after functional material used in the production of rubbers, paints and varnishes, construction composites, sorbents and catalysts. One of the most common methods of producing it is precipitation from silicate solutions using mineral acids (Pan et al., 2022; Mujkanovic & Jovanovic, 2021; Mamfoumbi et al., 2024; Nazharova & Galimbekova, 2010; Salimzhanova et al., 2019). Both traditional silicate materials and various silicon-containing industrial wastes can be used as raw materials for the production of SiO_2 (Cheng et al., 2019; Bochevskaya et al., 2017; Kutishcheva et al., 2021; Karimova et al., 2024; Hamidu et al., 2025; Deng et al., 2025; Shahbaz et al., 2023; Karlina et al., 2023).

Alkaline solutions formed during the regeneration of ion-exchange resins used in uranium hydrometallurgy represent a promising source of silicon. During the operation of anion exchangers, silicon compounds accumulate within the structure and on the surface of the sorbent, impairing its process properties. During alkaline regeneration, some of the silicon-containing compounds are released into solution in the form of silicate species, which allows such solutions to be considered as a secondary raw material for the production of amorphous silicon dioxide.



The formation of precipitated silica from silicate solutions is governed by the hydrolysis of silicate species, the polycondensation of silicic acid, and the formation of a Si–O–Si network. The properties of the final product depend on the pH, temperature, acid concentration, duration of the process and drying conditions (Matinfar & Nychka, 2023; Quang et al., 2012; Sazali et al., 2022; Montini et al., 2023; Belton et al., 2012). Despite the existence of studies on the production of SiO₂ from silicate solutions, the specific features of processing real industrial solutions used for the regeneration of ion-exchange resins have not been sufficiently investigated. In view of this, the aim of this study was to investigate the influence of process parameters on the precipitation of amorphous silicon dioxide from industrial alkaline solutions used for the regeneration of ion-exchange resins and to evaluate the physicochemical characteristics of the resulting material.

The scientific novelty of this work lies in a comprehensive investigation of the deposition parameters of amorphous silicon dioxide from alkaline silicate solutions used in the regeneration of ion-exchange resins in uranium hydrometallurgy, the identification of optimal process conditions, and a comparison of the structural characteristics of the resulting material with those described previously in the literature. For the first time, the production of a functional silica material with a well-developed micro-mesoporous structure and a high specific surface area has been demonstrated, indicating the potential for using industrial solutions as a source of secondary raw materials.

MATERIALS AND METHODS

The starting material used was an alkaline silicate solution obtained following the regeneration of macroporous anion exchangers contaminated with silicon compounds, using a sodium hydroxide solution with a mass concentration of 50 g/dm³. The mass fraction of silicon in the initial sorbent was 10.6%. Composition of the resulting silicate solution, g/dm³: Na – 3.39; Si – 43.19; Mg – 0.005; K – 0.004; Ca – 0.008; Fe – 0.003; Al – 0.0001; Sb – 0.0005; Cu – 0.0003; Zn – 0.0001.

SiO₂ precipitation was carried out using sulphuric acid under constant stirring at 500 rpm. The concentration of H₂SO₄ was varied within the range 20–50 %, the process temperature from 25 to 50 °C, the precipitation duration from 1 to 3 hours, and the precipitation pH within the ranges 6–8 and 9–10. The resulting precipitate was filtered, washed with distilled water and dried at 105 °C and 300 °C. Repeated rinsing with distilled water was carried out using a volume at least 10 times greater than the volume of the precipitate, whilst stirring. Rinsing was continued until the electrical conductivity of the rinse water fell below 5 µS/cm and the pH was close to neutral (7 ± 0.2), which ensured that the residual sodium content in the resulting material was reduced to <0.4% Na₂O.

The chemical composition of the solutions and precipitates was determined using titrimetric, spectrophotometric and spectrometric methods. The phase composition was determined by X-ray diffraction on a DRON-4-07 diffractometer using CuKα radiation over a scan range of 2θ = 10–80° with a step size of 0.02°. The porous structure was evaluated by low-temperature nitrogen adsorption at 77 K on a Thermo Scientific SURFER (Italy) gas adsorption porosimeter. The specific surface area was calculated using the BET (Brunauer–Emmett–Teller) method in the range P/P₀ = 0.05–0.30; mesopore volume using the BJH (Barrett–Joyner–Halenda) method, and micropore volume using the NLDFT (Non-Local Density Functional Theory) method. The experiments were carried out in triplicate; the error in the results did not exceed ± 3 %.

Analysis of the raw materials and the final product for uranium and heavy metal content, carried out for safety monitoring purposes using the ICP-MS method, showed that the metal concentrations were below the limit of detection.

RESULTS AND DISCUSSION

To investigate the effect of sulphuric acid concentration on the precipitation of amorphous silicon dioxide, the alkaline solution—after the ion-exchange material had been purified of silicon—was heated to 30 °C and, whilst being stirred continuously (500 rpm), acidified with a solution of H_2SO_4 of a specified concentration until the required pH was reached. The results are presented in Table 1.

Table 1. Relationship between the characteristics of the SiO_2 precipitate and changes in the process parameters of the precipitation process

Parameter	Value	SiO_2 concentration in the filtrate, g/dm ³	SiO_2 recovery, %	SiO_2 content in the residue, %
Concentration of H_2SO_4 precipitant, %	20	5,3	83,7	84,4
	30	9,1	71,9	82,6
	40	5,9	81,8	85,3
	50	12,1	62,3	85,0
Precipitation pH	6-8	0,3	85,1	85,8
	9-10	1,0	80,7	83,2
Temperature, °C	25	0,04	84,9	85,0
	30	1,3	81,5	84,8
	40	1,5	81,1	85,0
	50	0,8	78,4	80,4
Time, h	1	1,4	72,1	75,3
	2	0,3	84,6	85,3
	3	1,9	79,8	84,8
<i>Note – compiled by the authors</i>				

The most favourable results were achieved using a 40 % H_2SO_4 solution: the SiO_2 content in the precipitate was 85.3 %, and the recovery rate was 81.8 %. Increasing the acid concentration to 50 % led to a decrease in recovery to 62.3 %, which may be due to a change in gelation conditions and the partial transfer of silicon into the filtrate. A 40 % H_2SO_4 concentration was selected for further studies.

The concentration of the precipitant influences the rate of hydrolysis of silicate forms and the polycondensation process, which determine the formation of the Si-O-Si gel network. At low acid concentrations, hydrolysis is slowed down, leading to a less dense structure and a reduction in specific surface area. High concentrations accelerate aggregation, causing the formation of large particles and reducing porosity.

The temperature of the precipitation process affects the kinetics of precipitate formation.

It has been established (Sarawade et al., 2010) that an increase in process temperature usually leads to an increase in the degree of aggregation, pore size and volume, and a decrease in the specific surface area of the precipitate. The effect of temperature was investigated at a H_2SO_4 concentration of 40 %, a pH of 6–8, a process duration of 3 hours and a stirring speed of 500 rpm (Table 1).

The most favourable results were obtained at 25 °C, where maximum extraction and the lowest residual SiO_2 concentration in the filtrate were observed. At this temperature, aggregation proceeds more slowly, resulting in the formation of particles with a high specific surface area and a well-developed porous structure. An increase in temperature from 25 to 50 °C reduced the SiO_2 recovery from 84.9 % to 78.4 %, accelerated aggregation and coagulation, leading to an increase in pore size and a decrease in specific surface area, which has a negative impact on the sorption properties.

The effect of settling time was investigated at a temperature of 25 °C, a H_2SO_4 concentration of 40 %, a pH of 6–8 and a stirring speed of 500 rpm. The results are presented in Table 1.

The duration of precipitation determines the extent of gel formation, particle growth and sediment aggregation. When the process time was increased to 2 h, SiO_2 recovery reached 84.6%, and the SiO_2 content in the sediment was 85.3%. A duration of 2 hours has been selected as the optimum for the process, during which a well-developed porous structure is formed. A further increase in duration to 3 hours led to a decrease in recovery to 79.8 %, which may be due to the coalescence of aggregates, particle overgrowth, changes in the structure of the precipitate and a deterioration in its properties.

The pH of the medium controls the distribution of silicate forms and the charge stability of the particles. The optimum pH range was 6–8. Under these conditions, the residual concentration of SiO_2 in the filtrate was lower than at pH 9–10, whilst the recovery reached 85.1 % (Table 1). This indicates more complete precipitation of silicon dioxide in the slightly acidic and near-neutral regions. At high pH, the solubility of the particles increases and the yield of precipitate decreases.

Thus, the optimal combination of parameters – an acid concentration of 40 %, a temperature of 25 °C, a reaction time of 2 hours, and a pH of 6–8 – ensures the formation of amorphous, highly dispersed silicon dioxide, comparable in quality to the best commercial samples.

The content of impurity components in the precipitates varied within the following ranges, mass %: Fe_2O_3 – 0.01–0.02; Na_2O – 2.9–3.7; SO_4^{2-} – 0.76–3.15; CaO – 0.25–0.39; losses on ignition – 7.8–17.6.

Table 2 and Figure 1 present the results of the chemical analysis of the precipitated silica sample, whilst the results of the phase analysis are shown in Figure 2.

Table 2. Results of the chemical analysis of a precipitated silica sample, % by mass

Spectrum	O	Na	Si	S	Conclusion
1	58,38	0,97	40,38	0,27	100,00
2	59,00	0,80	39,82	0,38	100,00
3	58,34	1,05	40,14	0,47	100,00
Average value	58,37	0,94	40,11	0,37	100,00

Note – compiled by the authors

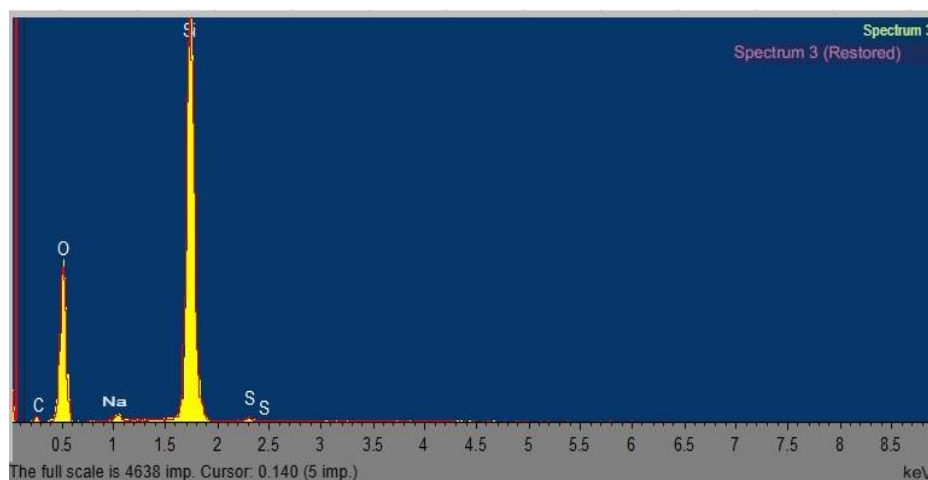


Figure 1. Spectral image of the chemical analysis results.

Note – compiled by the authors

Chemical analysis data show that oxygen and silicon predominate in the composition of the precipitate, which is consistent with the siliceous nature of the material. The low sodium and sulphur content indicates that the sediment has been effectively washed to remove soluble impurities. According to X-ray phase analysis, a broad diffuse halo is observed in the diffractogram in the region of $2\theta \approx 20\text{--}25^\circ$, which indicates a predominantly amorphous structure of silicon dioxide.

The porous structure of the sample obtained was investigated using low-temperature nitrogen adsorption at 77 K, with data processed using the BET, BJH and NLDFT methods.

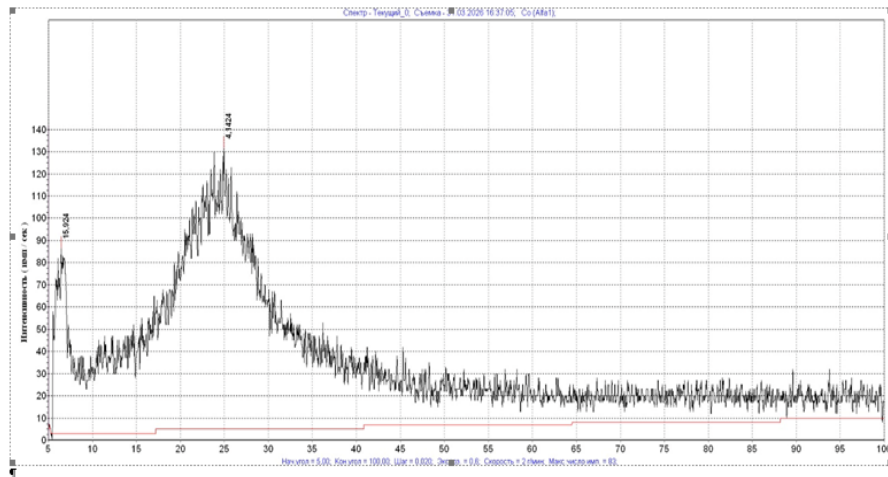


Figure 2. Diffractogram of a sample of precipitated silicon dioxide

Note – compiled by the authors

Analysis of the pore distribution using the NLDFT method (Figures 3 and 4a) revealed the presence of a pronounced microporous component with a main peak at a radius of approximately 0.6–0.7 nm.

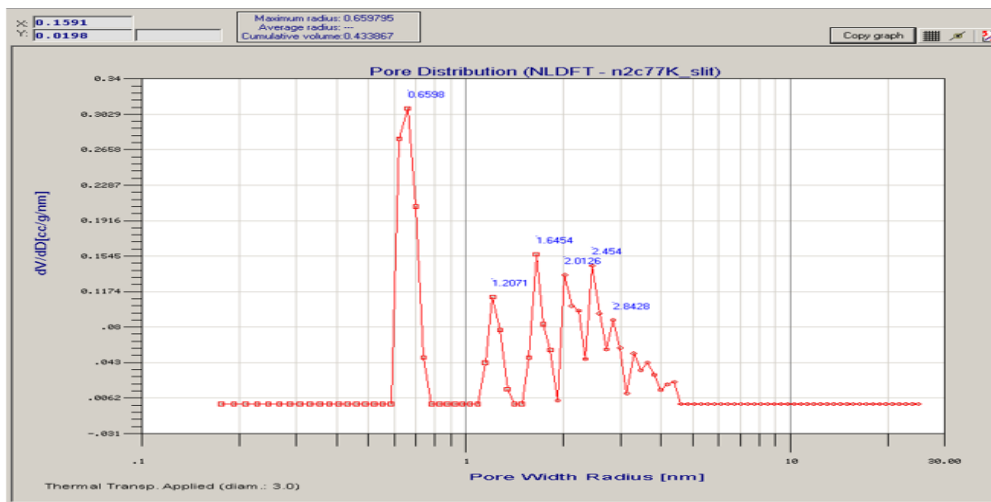
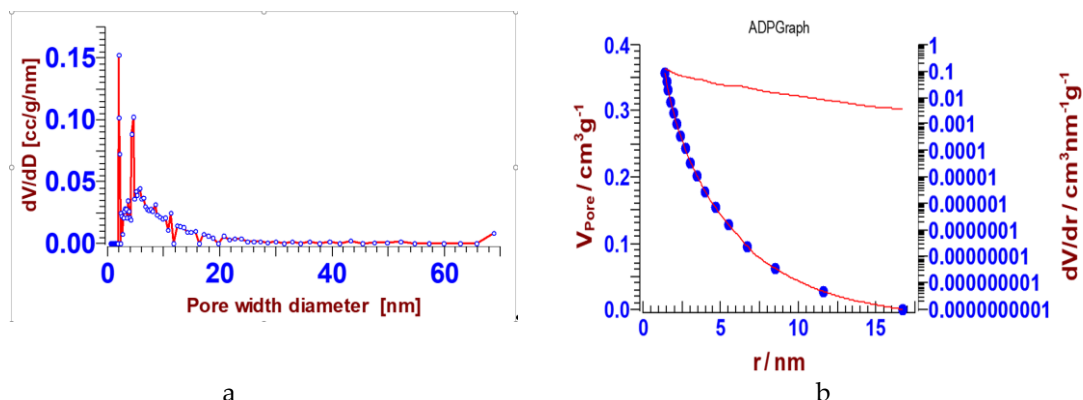


Figure 3. Pore volume distribution calculated using the NLDFT method (slit pore model)

Note – compiled by the authors



a – using the NLDFT method; b – using the BJH method

Figure 4. Calculation of the pore distribution of the sample obtained

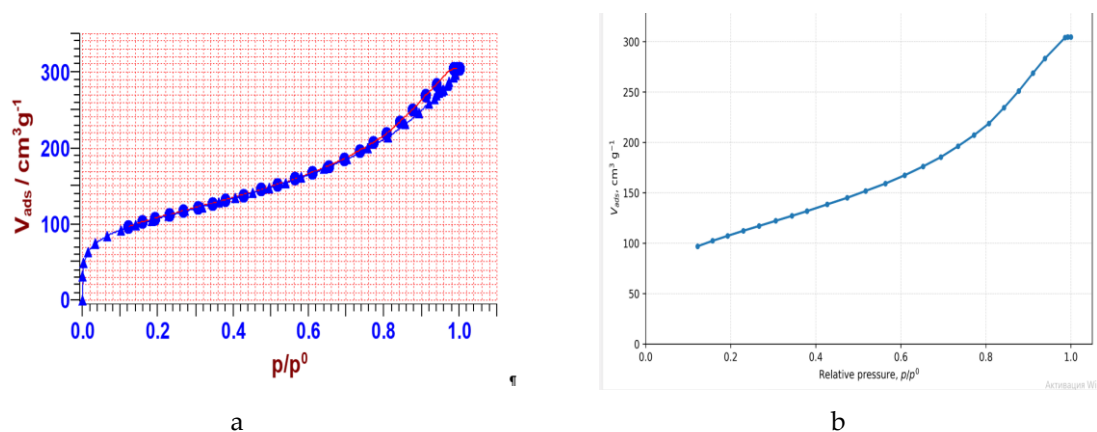
Note – compiled by the authors

In addition, a distribution in the 1–3 nm range is observed, associated with transitional pores and inter-aggregate voids. This distribution pattern indicates a disordered porous structure formed by the aggregation of primary amorphous SiO_2 nanoparticles.

The BJH method (Figure 4b) confirmed the presence of a mesoporous component, attributable primarily to interparticle and interaggregate voids.

The average pore radius is approximately 3.0 nm, the hydraulic radius is approximately 2.88 nm, and the cumulative pore volume is $0.5147 \text{ cm}^3/\text{g}$. The specific surface area of the sample, calculated using the BET method, is $357 \text{ m}^2/\text{g}$. The high specific surface area is attributable to the well-developed pore structure and the high degree of dispersion of the material.

The shape of the nitrogen adsorption and desorption isotherms (Figure 5) also confirms the presence of a mixed micro-mesoporous structure.



a – adsorption isotherm; b – desorption isotherm

Figure 5. Adsorption–desorption isotherms for nitrogen in the sample under investigation at 77 K

Note – compiled by the authors

A comparison of the properties of the synthesised silicon dioxide with those of commercial functional silicas [Pan et al., 2022; Karimova et al., 2024; Hamidu et al., 2025; Deng et al., 2025] shows that, in terms of specific surface area ($200\text{--}360 \text{ m}^2/\text{g}$ [Karimova et al., 2024; Hamidu et al., 2025]) and micro-mesopore structure, the material obtained is most similar to highly dispersed amorphous silicas.

The sharp increase in adsorption at low relative pressures indicates a contribution from microporosity, whilst the further increase in adsorbate volume at medium and high p/p_0 values is associated with the filling of mesopores and interparticle voids.

Thus, the resulting material is characterised by an amorphous structure, a high specific surface area and a well-developed micro-mesoporous system. In terms of specific surface area, it is comparable to highly dispersed silica materials; however, in terms of its method of preparation, it belongs to the category of precipitated silicon dioxide.

The drying temperature influences the compositional make-up and degree of dehydration of the resulting precipitate. Precipitates obtained at pH 6–8 were dried at 105 and 300 °C in a muffle furnace for one hour. The results are presented in Table 3.

Table 3. Composition of silicon dioxide precipitates

Drying temperature, °C	Content in the sediment, %					
	SiO ₂	Fe ₂ O ₃	Na ₂ O	SO ₄ ²⁻	CaO	losses on ignition
105	82,1	0,01	0,3	0,41	0,33	9,1
300	86,7	0,02	0,4	0,24	0,31	8,2
<i>Note – compiled by the authors</i>						

Raising the drying temperature to 300 °C increases the SiO₂ content from 82.1 to 86.7 % and reduces the loss on ignition. This is due to the removal of physically bound water and partial surface dehydration. Consequently, drying at 300 °C yields a precipitate with a higher SiO₂ concentration and stable physicochemical properties.

CONCLUSIONS

This study demonstrates the possibility of producing amorphous, highly dispersed silicon dioxide from industrial alkaline silicate solutions generated during the regeneration of ion-exchange resins in uranium hydrometallurgy.

The optimal precipitation conditions are: H₂SO₄ concentration – 40 %, temperature – 25 °C, process duration – 2 hours, pH – 6–8. Under these conditions, SiO₂ recovery reaches up to 85.1 %, and the SiO₂ content in the precipitate after drying at 300 °C is 86.7 %.

The resulting material has an amorphous structure, a specific surface area of 357 m²/g and a mixed micro-mesoporous system. This confirms the potential for using industrial alkaline solutions as a secondary raw material for the production of silica materials.

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DATA AVAILABILITY STATEMENT: The original materials presented in the study are included in the article; further requests may be directed to the corresponding author

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