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HIGHLY ACTIVE SILICA FILLER OBTAINING PROCESS BY SULPHURIC ACID PRECIPITATION

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Abstract

The article scientifically substantiates and practically implements an improved three-stage technology for the synthesis of highly active precipitated silicon dioxide (white carbon black), which serves as a domestic analogue to the international standard ULTRASIL® VN 3. The study investigates the influence of «mild» precipitation conditions, including temperature (90–92 °C), sodium silicate density (1.07–1.09 g/cm^3), and controlled technological pauses, on the physicochemical properties of the product. It was established that a clear separation of the nucleation (pH 11.0) and structuring (pH 10.0) stages allows for precise control of the specific surface area within the range of 150–200 m^2/g. Process scaling results on a bench-scale unit with a 400-liter reactor confirmed the stability of quality characteristics: SiO_2 content ≥ 97 %, pH 6.2, and moisture content ≤ 6 %. Product testing at consumer enterprises confirmed its high reinforcing capacity in tire and rubber compounds.

Keywords: silicon dioxide; precipitated silica; liquid glass; sulfuric acid; specific surface area; rubber compounds.

РОЗРОБЛЕННЯ ТЕХНОЛОГІЇ ОДЕРЖАННЯ ВИСОКОАКТИВНОГО КРЕМНЕЗЕМНОГО НАПОВНЮВАЧА СІРЧАНОКИСЛОТНИМ МЕТОДОМ ОСАДЖЕННЯ РІДКОГО СКЛА

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Анотація

У статті науково обґрунтована та практично впроваджена вдосконалена тристадійна технологія синтезу високоактивного осадженого діоксиду кремнію (білої сажі), що є вітчизняним аналогом міжнародного стандарту ULTRASIL® VN 3. У роботі досліджується вплив «м'яких» умов осадження, які включають температуру (90–92 °C), густину силікату натрію (1.07–1.09 г/см^3) та контрольовані технологічні паузи, на фізико-хімічні властивості продукту. Встановлено, що чітке розділення стадій зародження (pH 11.0) та структурування (pH 10.0) дозволяє точно контролювати питому поверхню в діапазоні 150–200 м^2/г. Результати масштабування процесу на лабораторній установці з реактором об'ємом 400 л підтвердили стабільність якісних характеристик: вміст SiO_2 ≥ 97 %, pH 6.2 та вміст вологи ≤ 6 %. Випробування продукту на підприємствах-споживачах підтвердили його високу армуючу здатність у шинних та гумових сумішах.

Ключові слова: діоксид кремнію; осаджений кремнезем; рідке скло; сірчана кислота; питома площа поверхні; гумові суміші.

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Introduction

Due to exceptional hardness and durability silicon dioxide became an irreplaceable component in numerous industries. First it was used as basic construction material. Nowadays it has transformed into a high-technology product with multiple synthetic modifications.

Amorphous non-porous SiO_2 is widely used in food and pharmaceutical industries (additive E551) where it is used as an anticaking agent and structure stabilizer for medicines [1]. In other industries it is a basic component in glass, ceramics, cements, abrasives and fire extinguishers processes and an active filler in industrial rubber products [2–5].

Piezoelectric properties of crystal quartz are especially important as they enable its application in radiotechnics devices and ultrasound equipment. In high technologies silicon dioxide is critical as a dielectric and passivating layer in microelectronics, basis in fiber optical communications and thermal resistant adsorbent in evaporation systems [6].

Synthesis method directly determines morphology and physical and chemical characteristics of final product, namely dispersity, particles geometry and surface porosity. Pyrogenic silicon dioxide (colloidal) known as aerosil or orbisil is an ultralightweight powder with absorbing ability. Its structure makes it an irreplaceable rheologic modifier (thickener) for paints, lubricants and adhesives and an efficient active components carrier in pharmacy [7–9].

Precipitated silicon dioxide (silica white) has hydrated form $mSiO_2 \times nH_2O$. This material is classified as an active mineral filler critically important for improvement of rubber mixtures and car tires mechanical properties [10–11].

Industrial synthesis methods allow to process natural raw materials (sand, minerals) and chemical compounds (halogenides) into functional silica forms by precipitation or high temperatures hydrolysis.

A wide range of silicon dioxide functional characteristics (from piezoelectric effect to polymer matrices armoring ability) allows development of special approaches to controlling structure of this material still at its formation stage. As particles morphology, their size and chemical purity directly depend on selected process scheme selection of the best synthesis method becomes key aspect of modern research.

Highly dispersive SiO_2 synthesis technologies are divided into physical and chemical. Sol-gel method (liquid phase synthesis) and gas phase methods such as pyrolysis and plasma-chemical precipitation are the most widely used methods.

Sol-gel process is the basic one for industrial precipitated silica, glass and ceramics obtaining processes [12–14]. Despite high raw material consumption and multiple stages, this process is demanded as it allows to obtain high purity homogeneous products from mineral raw materials.

Gas phase methods (pyrolysis) require fewer process stages. Plasmatrones and special combustion chambers allow precise control of nano particles size. But basic restriction of these methods are strict requirements to initial reagents purity (mainly halogenides) that makes impossible direct processing of natural raw materials. In mid-20th century pyrogenic methods started to develop. They are based on steam phase hydrolysis of silica halogenides; high temperature oxidation of silica containing compounds; plasma recovery of natural silicas to SiO_2 followed by its oxidation.

Main producers of pyrogenic silica are Degussa AG (now Evonik Industries AG), Germany (product name: aerosil); Cabot Corporation, USA (carbosil); Wacker-Chemie GmbH, Germany (hydrophilic silica dioxide); Fransol-Paris (fransil); TOKUYAMA Corporation, Japan (reolosil).

Despite high processability of pyrogenic methods precipitated silicon dioxide is the most widely used industrial process. As distinct from gas phase methods that require expensive synthetic raw materials precipitation in liquid phase allows to use accessible mineral resources and agricultural wastes.

Flexible control of precipitation methods (temperature, reagents concentration and fluid pH) this method the most preferable for obtaining wide range of materials: from active tires rubber filler to highly pure sorbents. We shall further detail technical peculiarities and chemical properties of precipitated silica called «silica white» in our industry.

The modern industrial precipitated silica process is based on using silicate lump (fluid glass) as basic raw material. The process starts with high temperature melting of quartz sand with sodium alkali at 1700 °C, after that obtained product is dissolved in autoclave under pressure. Further processing includes normalization of SiO_2 content (up to 50–60 g/dm^3) and clarification in sodium silicate solution. Key stage of phase transformation is solution carbonation with gas mixture from lime kilns (CO_2 – 35 % weight). This process flows under isothermal conditions (75–

85 °C) within required time up to attaining required alkalinity values, pH value is corrected in the final with hydrochloric acid. The final stage includes extraction of amorphous phase by filtration, cake washing and thermal processing (drying) in gas flow followed by separation on sleeve filters.

Silica white is usually obtained by interaction of fluid glass and acid reagents such as hydrochloric acid. $CaCl_2$ as a precipitation agent at the first stage allows to obtain hydrated silica slurry that is further demineralized and dried. But the final product obtained by this process often includes undesired calcium compounds inclusions. Such impurities decrease adsorption activity and purity of amorphous SiO_2 which is a critical drawback of this method, as distinct from direct carbonation [15–16].

An alternative approach to silica white synthesis is proposed in [17]. This method is based on phosphor containing raw materials that can be destructed by nitrate acid. Silicon dioxide slurry is formed by intense agitation during 15–20 minutes at moderate heating (50–60 °C). This method combines purification and modification stages: after extraction and washing SiO_2 cake is dried at low temperatures (30–40 °C) and then processed with silicone oil. This allows to obtain hydrophobizated particles directly during the process cycle that are further caught by sleeve filters system.

The proposed method of highly dispersive SiO_2 obtaining is based on controlled interaction of sulfate acid and sodium silicate solution. This process includes pulp formation, its filtration and further repulpation of obtained cake. Key innovation is introduction of active macromolecular agent to liquid phase before spray or thermal drying. This allows to improve particles size and ensure stable high dispersity of the material. Such silicon dioxide as arming agent in rubber mixtures causes synergetic effect: significant decrease of hysteresis heat formation and abrasive wear, at the same time rupture resistance and relative increase of rubber products service life are increased [18].

Ammonia fluoride method of complete silicate raw material processing (such as kyanite concentrates) is based on cyclic ammonia fluoride use for selected oxydes extraction. This process includes high temperature SiO_2 ($T > 150$ °C) fluoridation with formation of ammonia hexaftorsilicate that undergoes ammonia hydrolysis after thermal sublimation ($T > 320$ °C). Closed cycle is the key advantage of this method: recovered fluoride and ammonia liquor are returned to the process ensuring high economic efficiency. Obtained amorphous silicon dioxide has high purity (basic substance 93 % weight) [19].

Innovation method of highly dispersive nanosolica synthesis in membrane dispersion reactor by interaction of H_2SO_4 and Na_2SiO_3 has been proposed. The membrane process allowed to intensify mass exchange and precisely control reaction conditions. This allowed to significantly decrease particles size and their aggregation degree. It was established that addition of surface active substances (sodium carboxymethyl-cellulose and polyethyl glycole) to the system ensures formation of ~20 nm particles with specific surface area 126.2 m^2/g. High dispersity (DI = 1.12) and low coordination figure of the product prove efficiency of this method for functional additions process for tires, lacquers and paints and cosmetics industries [20].

Authors [21] studied silica synthesis by carbonation (CO_2) and acid precipitation (HCl) from sodium silicate solutions. It was established that particles morphology, adsorptive capacity (for dibutylphthalide absorption) and particles aggregation degree directly depend on reaction temperature and precursor initial concentration. The best product characteristics that can compete with the properties of high quality commercial fillers have been attained at a temperature 40 °C and a SiO_2 concentration of 8 %. Comparative analysis of rubber composites proved high armoring potential of obtained silica, as they showed excellent vulcanization and mechanical strength. These results show that precise control of filler properties by synthesis parameters are possible.

Tests proved that two-stage chemical preparation of rice husk (extraction by alcohol-HCl and aqueous H_2SO_4 mixture) allows obtaining precursor fit for prompt synthesis of high purity silica. Optimized burning mode at 600 °C within 5 minutes allows to obtain white amorphous powder with short range of particles sizes (up to 10 μm) and significant adsorption capacity. Due to high specific surface (> 235 m^2/g) obtained silicon dioxide is a prospective matrix for active components immobilization in chemical and medical industries [22].

Leading European companies (Rhodia, France, Evonik Industries AG, Germany) obtain highly active silica filler by the hydrochloric acid process of sodium silicate precipitation followed by filtration, cake washing and final product drying.

Evonik Industries AG (Germany) manufactures precipitated silica filler «ULTRASIL®VN 3» highly demanded by numerous domestic and foreign consumers and is regarded as a standard for similar product development.

This method includes a hydrochloric acid batch feed with constant velocity to sodium silicate solution with molar ratio $SiO_2 : Na_2O = 3.1$. At this stage, silicate monomer transforms to oligomer. All silicon dioxide is precipitated. All stages of silicon dioxide precipitation flow within the temperature range 40–95 °C. The cake is filtered, washed, and dried. According to [23] precipitation mode from sodium silicate solution is important for obtaining active silica filler. In this case, the most disperse, and, consequently, the most active primary silicon dioxide particles are formed. Facilitated precipitation mode should promote gel structure of silicon dioxide particles. In Degussa's patent [24] process equipment and certain peculiarities of silica white synthesis are described. These patents always included quite high precipitation stage temperature, keeping gradual neutralization and crucial impact of concentration and silicate module of sodium silicate solution on surface properties and diameter of silica particles.

Industrial processes of leading European manufacturers such as Evonik Industries AG are based on continuous or batch hydrochloric acid feed to sodium silicate solution under high temperatures [32–33]. Despite stable quality, such methods have serious restrictions: one- or two-stage precipitation often causes quick noncontrolled particles aggregation of due to sharp increase of system viscosity («silicate shock») that complicates obtaining short range of particles sizes.

In order to eliminate these disadvantages and improve the process of silica morphology control, NIOCHIM proposed and tested three-stage precipitation method. As distinct from standard approaches this development is based on accurate separation of solid phase formation stages.

This study is aimed at process parameters adjustment and optimization of three-stage precipitation method developed at NIOCHIM for obtaining domestic analogue of «ULTRASIL®VN 3» silica filler.

Materials and Methods

In order to attain this goal, study of the impact of «soft» synthesis conditions (low hydrochloric acid concentration 15–17 % and fluid glass density 1.07–1.09 g/cm^3) on physical and chemical indices of the final product was required, namely on its specific and arming capacity at the same time keeping high quality and optimal pH level (6–7) of final silica white. Deposition temperature ranged from 80 to 92 °C.

Fluid glass with a density of 1.45 g/cm^3 with silicate module 2.5–4.0 and technical hydrochloric acid according to Ukrainian State Standards (DSTU GOST 2184:2018) were used as raw materials. Fresh diluted unclarified solutions were used as sodium silicate operating solutions.

Silicon dioxide was precipitated from sodium silicate solution in reactor V = 2 dm^3 installed in a liquid thermostat filled with water. The reactor was equipped with an arm agitator. The laboratory unit was also equipped with a nutch filter and a drying box. The set temperature was regulated by contact and checked with mercury thermometer. Silicon dioxide was precipitated with concentrated hydrochloric acid fed to the reactor lid opening through a fission chute. Acid flow was controlled by opening the tap on the chute. Sodium silicate solution was neutralized to pH 3.0±0.5.

Powders specific surface was determined according to ISO 9277 [26]. This method is based on measuring volumes of gas preliminarily adsorbed on the surface of the studied specimen from operating gas mixture flow (nitrogen-helium) at fluid nitrogen temperature followed by desorption in case of temperature increase.

Drying losses, bulk density and pH of aqueous extract is determined according to ISO 787 [27–28], and SiO_2 content is determined according to ISO 3262-19 [29]. Correspondence between selected study indices and results is attained according to standard international methods and mathematical tools for data processing.

Results and Discussion

In order to provide softer precipitation conditions sodium silicate solution was neutralized by concentrated hydrochloric acid in three batches, with two stops during hydrochloric acid feed for 10 and 20 minutes.

First hydrochloric acid feed was stopped at slurry pH = 11±0.1. When slurry pH reached 10±0.1 at stage two hydrochloric acid feed was stopped again.

Third stage of hydrochloric acid feed was till slurry pH = 4.0–7.0. Adjustment tests on silica filler mode were held with the stop in diluted hydrochloric acid feed at slight turbidity of sodium silicate solution, i.e. when first silica dioxide particles precipitated. They were treated as seeds for further SiO_2 particles growth.

Another stop of hydrochloric acid feed was made when silicate monomer transformed into silicon dioxide high-molecular compound and solution viscosity started to increase sharply. These studies show that the first stage should flow smoothly, hydrochloric acid feed should take 10-15 minutes; second precipitation stage should flow till complete silica drop-out from sodium silicate solution, after that stop for 2 minutes is required.

At the third stage hydrochloric acid is fed till slurry pH = 4.0–4.5 in order to ensure its good filtration properties. In this case final product pH value shall be within 6…7. Complete study took 80–100 minutes. Slurry was filtered and silica dioxide cake was dried in Buechner funnel under vacuum, and cake was dried in drying box under 105–120 °C.

It should be noted that prompt acid feed at all three stages allowed to obtain loose gel cake that gave solid product with high absorption specific surface (190–247 m^2/g).

Study conditions and characteristics of obtained specimen are listed in Table 1.

Quality indices of precipitated active silica filler obtained by fluid glass precipitation with hydrochloric acid met requirements listed in Table 2. This Table also contains quality indices of similar foreign product «ULTRASIL®VN 3» by *Evonik Industries AG* [25].

Table 1

Conditions of obtaining and specimen quality by three-stage precipitation

No	Fluid glass module	Fluid glass density, g/cm^2	Study conditions							Nitrogen specific surface m^2/g
			Precipitation temperature, °C	pH			Acid feed time, min			
				Stage I	Stage II	Stage III	Stage I	Stage II	Stage III	
1	3.0	1.09	90	11.1	9	3.3	15	30	10	199
2	3.0	1.09	90	11.3	9	4.0	20	30	10	181
3	3.0	1.23	80	11.1	10	2.5	16	50	20	302
4	3.0	1.23	80	10.9	10	4.6	15	51	20	146
5	3.4	1.11	90	10.9	10	3.5	15	60	20	190
6	3.4	1.11	90	10.9	9	2.9	20	30	10	170
7	3.4	1.22	90	11.0	10	4.0	21	44	20	130
8	3.4	1.22	80	11.1	10	4.4	10	60	20	189
9	3.7	1.10	90	11.0	10	4.6	11	30	16	210
10	3.7	1.10	80	10.9	10	4.2	15	40	13	153
11	3.7	1.25	90	11.1	10	4.3	14	40	14	139

It should be noted that silica filler structure is characterized not only by specific surface value of nitrogen adsorption, but also by external specific surface value determined according to cetyltrimethylammonium bromide (m^2/g), product porosity – dibutilphtalate absorption TBN (cm^3/100 t) and pH value of silica filler aqueous extract [30–31].

Table 2

Comparative characteristics of obtained product and its foreign analogue

No	Description	Our product	«ULTRASIL®VN 3» by *Evonik Industries AG*
1.	Mass weight of silica dioxide (as burned substance)	98	≥97
2.	Specific nitrogen absorption area, m^2/g	150-190	180
3.	Aqueous extract pH (5% slurry in water)	6.0-7.5	6.2
4.	Drying losses, %,	6.5	5.5
5.	Bulk density, g/cm^3	220	270

These results show that in all specimens obtained under laboratory conditions only specific surface at nitrogen absorption was determined (Sorbi unit was used as an example).

First stage of laboratory studies included test of fluid glass module and density, precipitation temperature and pH value of third stage precipitation for specific surface of hydrochloric silica filler specimen.

Analysis of obtained results allows to determine obvious dependency of specific surface on:

- Precipitation temperature (80–90 °C);
- Fluid glass density (γ = 1.07–1.24 g/cm^3);
- Fluid glass module (2.5–4.5);

- pH value of third precipitation stage (2.5–4.5).

On the basis of above assumptions series of silica filler syntheses were held with fluid glass module γ = 1.07–1.24 g/cm^3. Results of these tests are given in table 1.

Analysis of obtained data allowed to select the best conditions for precipitation process for obtaining active silica filler – analog of «ULTRASIL VN 3»:

- Fluid glass module: 3.0–3.7;
- Precipitation temperature: 90–91 °C;
- Fluid glass density: γ = 1.07–1.09 g/cm^3;
- pH value of precipitation stages: Stage I – 10.9–11.0; Stage II – 9.9–10.0; Stage III – 4–4.5.

Laboratory studies allowed to select process standards for active silica filler hydrochloric process:

1. Active silica filler with nitrogen adsorption specific surface 150–200 m^2/g can be obtained from fluid glass with module 3.0–3.7. (Standard ULTRASIL VN 3 has value 180 m^2/g).
2. Fluid glass concentration range can make SiO_2 = 60–100 g/dm^3 at density 1.09–1.11 g/cm^3.
3. Temperature of precipitation stage should be maintained 90–92 °C.
4. Precipitation should flow in three stages with two breaks:
   - pH Stage I – 11.0–11.1;
   - pH Stage II – 9.9–10.0;
   - pH Stage III – 4.0–4.5.
5. Drying in laboratory box at t = 105–110 °C should continue till moisture content of final product makes max. 6.0 % (according to ISO 787-2 standard moisture content makes 5.5 %).

So, silica acid filler process consists of (Fig.):

- preparation and treatment of operation sodium silicate and hydrochloric acid solutions;
- SiO_2 precipitation from fluid glass with hydrochloric acid;
- filtration and washing of silicon dioxide cake;
- slurry repulpation with artesian water and partial slurry neutralization by hydrochloric acid;
- drying of precipitated silicon dioxide slurry;
- final product packing.

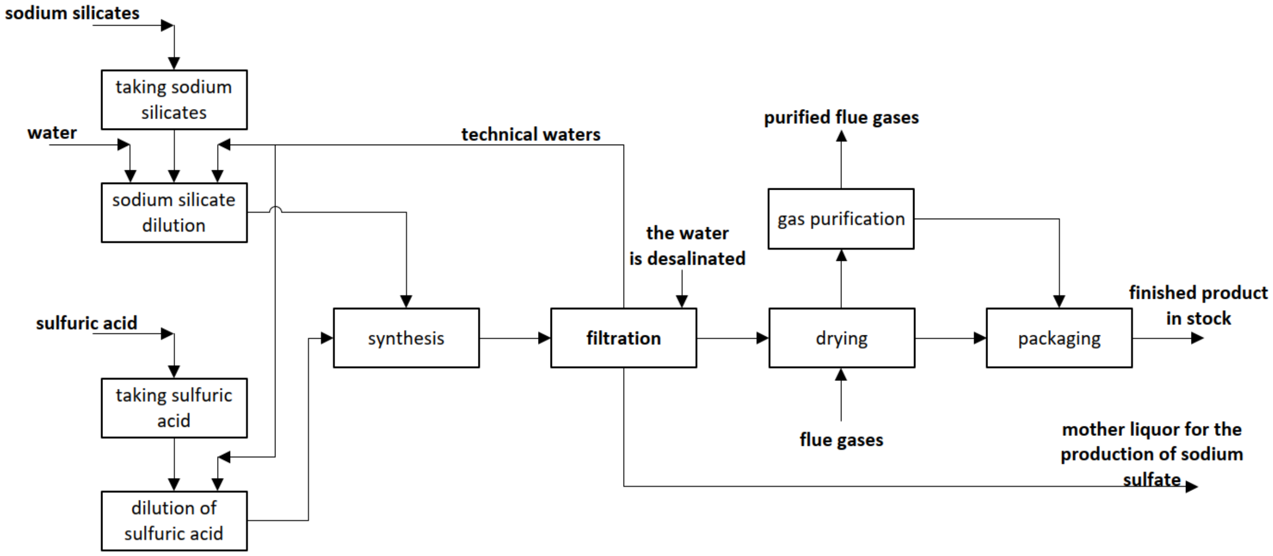

Figure. Principal process diagram of silica acid filler process

At the second stage of this study test set was created aimed at synthesis process scaling and manufacture of product test batches.

Jacketed reactor was used as the basic part of test unit. It is a 400 dm^3 stainless steel tank with conic bottom with a two-tier arm agitator. Agitator rotation frequency made 300 rpm that corresponded to rotary velocity 2.8 m/s. Water was used as a heat carrier. Hydrochloric acid was fed through the opening in reactor lid.

Obtained slurry was filtered on chamber press with filtration surface 6.2 m^2. Wet cake was washed with process water at the rate of 2 volumes of mother solution. Then wet cake was repulped and dried in dryer.

Manufactured batch was tested by the customers who confirmed that it is completely fit as an acid silica filler in tier industry and technical rubber products as an efficient replacement of import analogue «ULTRASIL VN 3».

Conclusions

The work contains scientifically grounded and actual improvement of three-stage process for highly active precipitated silicon dioxide synthesis similar to ULTRASIL® VN 3.

Basic results of the study:

1. It was established that «soft» precipitation modes (temperature 90–92 °C, fluid glass density 1.07–1.09 g/cm^3) with regulated technical pauses at nucleation (pH 11.0) and structuring (pH 10.0) stages allows precise control of product specific surface within 150–200 m^2/g range.
2. Test unit with 400 l reactor and chamber press filter was developed based on laboratory data, thus confirming process scaling without compromising filler quality.
3. Obtained test batch completely meets international standards with SiO_2 (≥ 97 %), pH (6.2) and moisture content (≤ 6.0 %).
4. The product was tested at the customers' plants that confirmed its high arming capacity for tires and technical rubber products. Developed process allows to obtain competitive domestic product that can completely replace imported analogues at industrial scale.

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