INNOVATIVE AND CLEANER INORGANIC TECHNOLOGIES
Module supervisor: dr hab. inz. Marcin Banach
Laboratory Instruction No 4
APPLICATION OF RENEWABLE RAW MATERIALS IN THE PRODUCTION OF NANOSILICA

REPORT:
ONE WEEK AFTER THE END OF THE CLASS

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1. Introduction

Silicon dioxide is classified as an inorganic oxide compound in which silicon is present in the IV oxidation stage. In nature it is widespread - it is one of the main components of the Earth's crust. It occurs in the form of crystalline polymorphs, mainly as quartz, tridymite, cristobalite, and in the amorphous form of mineral substances containing water, such as opals. Silicon dioxide is formed by crystallization from supersaturated silicate solution. It has the character of an acid anhydride. It is resistant to chemicals, except for hydrogen fluoride and alkali.

Silicon dioxide nanoparticles are also known as nanosilica. Due to their durability, low toxicity and ability to combine with various polymers, they have many applications. Nanosilica is mainly used as a rubber and plastics additive, as a filler for concrete and other construction composites. It is also the subject of many biomedical studies on using nanosilica as a non-toxic platform for drugs delivery. Nanosilica is also used in the production of silicate-lignin biocomposites, as well as epoxy resins modified with silicate fillers. Silicon dioxide is used for the manufacture of refractories, glass and building materials such as silicate bricks and Portland cement. Its properties allow it to be used as an adsorbant, an anti-aging agent, a drying agent, as a supplement, stabilizer, gelling agent, viscosity regulator and as a water-repellent agent.

2. **Aim of experiment**

The purpose of the experiment is obtain silica from waste material and to compare its quality with silica obtained using conventional methods.

3. **Chemical reagents** redistilled water

- sodium metasilicate

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- hydrochloric acid (d=1.19 g/ml)
- sodium hydroxide
- ash from rice bran

3. The course of the exercise

Stages of experiment (for both types of process):

- Analysis of raw materials
- Preparation of main products
- Analysis of product properties

3.1. Analysis of raw materials

- Appearance → consistency, colour
- Microstructure → based on optical microscopy, define the basic features of the surface of the raw material (homogeneity, grain size, presence of foreign inclusions)
- X-ray diffraction → compare and interpret the obtained spectra

3.2. Obtaining the silica

3.2.1. Conventional process

The basic point of the method is to conduct the sol-gel process. The idea is to precipitate the silica dioxide in an acidic environment, according to the following chemical equation:

$$Na_2SiO_3 + 2HCl \rightarrow SiO_2 + 2NaCl + H_2O$$

Calculate the required mass of raw material ($M_{Na2SiO3)}$= 122,06 g/mol), so as to obtain 3 g of product, assuming that the reaction yield is equal to 90%.

Run under the fume hood

Introduce the calculated mass of sodium metasilicate and 10 ml of water to a beaker placed on a magnetic stirrer, at room temperature. After dissolution of the total amount of

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the sodium silicate, under conditions of continuous stirring, introduce the calculated amount of hydrochloric acid. Note the pH of the mixture. In order to obtain the microcrystalline product, the resulting mixture is transferred to a Teflon vessel and then placed in a microwave reactor.

Process parameters (temperature, pressure, microwave power) will be set by your tutor. After completing the process, based on the graph obtained, read the actual process parameters. After cooling, filter the mixture under reduced pressure and, in order to remove the sodium chloride, wash the precipitate twice with distilled water. Dry at 70°C and weigh the product.

3.2.2. Alternative process – according to US 7897648 B2

To be performed in a fume hood

The ash obtained after the combustion of rice bran will be provided by your tutor.

Weigh 5 g of ash and transfer it to a beaker. Add 60 ml of an aqueous solution of NaOH at a concentration of 30%. While stirring the mixture with a magnetic stirrer, heat it up to 90°C. Filter out the pulp under reduced pressure, take a known volume of filtrate and transfer it to a beaker. In order to obtain the hydrogel under continuous stirring conditions, add hydrochloric acid so that the pH of the reaction mixture was around 4-5. Note the final value of the pH of the mixture. Transfer the whole suspension to a teflon vessel which is then placed in a microwave reactor. Process parameters (temperature, pressure, microwave power) will be set by your tutor. After completing the process, based on the graph obtained, read the actual process parameters. After cooling, filter the mixture under reduced pressure and, in order to remove sodium chloride, wash the precipitate twice with distilled water. Dry at 70°C and weigh the product.

3.3. Analysis of product properties

- Appearance → consistency, colour

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4. Report

The report should include: an introduction (properties and use of microcrystalline silica dioxide), all reaction equations describing the processes carried out, calculations, analysis and interpretation of the results in relation to the literature, conclusions.

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