Expt. No.: **Date:**

Experiment	**Preparation of nanosilica by sol- gel method and its characterization**
Problem definition	Preparation and characterization of nanoparticles of SiO_2.
Methodology	SiO_2 nanoparticles are prepared by the sol-gel method and characterized by absorption, diffraction and microscopic techniques
Solution	Preparation of SiO_2 nanoparticles
Student learning outcomes	Students will learn to a) prepare SiO_2 nanoparticles by sol-gel method b) analyze the nanoparticles by instrumental techniques

1. Significance of the experiment:

Nanosilica (SiO_2) is a promising material to find applications in various fields such as drug delivery, gene therapy, and detection of biomolecules, photodynamic therapy and bioimaging. The salient features are high thermal, chemical and colloidal stability, high surface area, and good biocompatibility. It was also demonstrated that the immobilization of drugs on silica nanoparticles can reduce adverse side effects.

SiO_2 nanoparticles can be achieved by several methods such as chemical vapour deposition, plasma and combustion synthesis, hydrothermal and sol-gel methods. Each method has its merits and demerits. The sol-gel method is one of the promising methods employed in the preparation of SiO_2 nanoparticles.

2. Principle of sol-gel method:

Silicon dioxide (SiO_2) nanoparticles were prepared by sol-gel method starting from tetraethylorthosilicate ($Si(OEt)_4$) precursor[Reference 1]. After dissolving in ethanol, the solution undergoes hydrolysis with the addition of NaOH pellets and results in very unstable silicon hydroxide [$Si(OH)_4$]. This turns into a turbid colloidal solution called sol. Sols are dispersions of colloidal particles (with diameters 1-100 nm) in a solvent. A gel is an inter-connected, rigid network with pores of sub-micrometer dimensions and polymeric chains whose average length is greater than a micrometer. The sol-gel method for the preparation of nanosilica particles is represented in **Fig. 1**.

[Reference 1] A. M. Buckley & M. Greenblatt, *Journal of Chemical Education*, 71, 599-602 (**1994**).

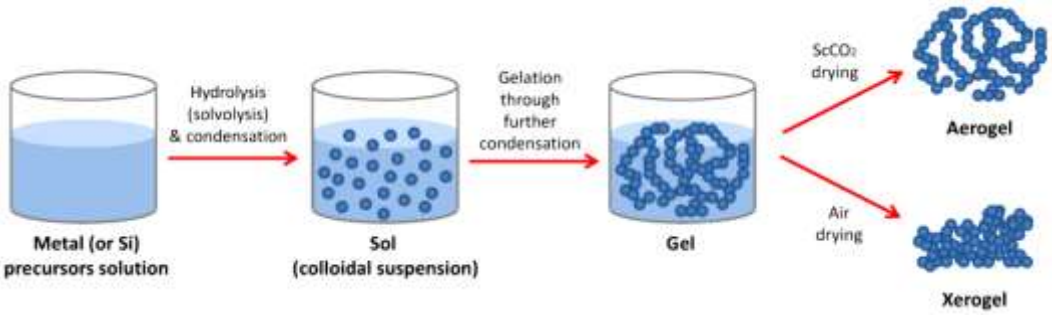


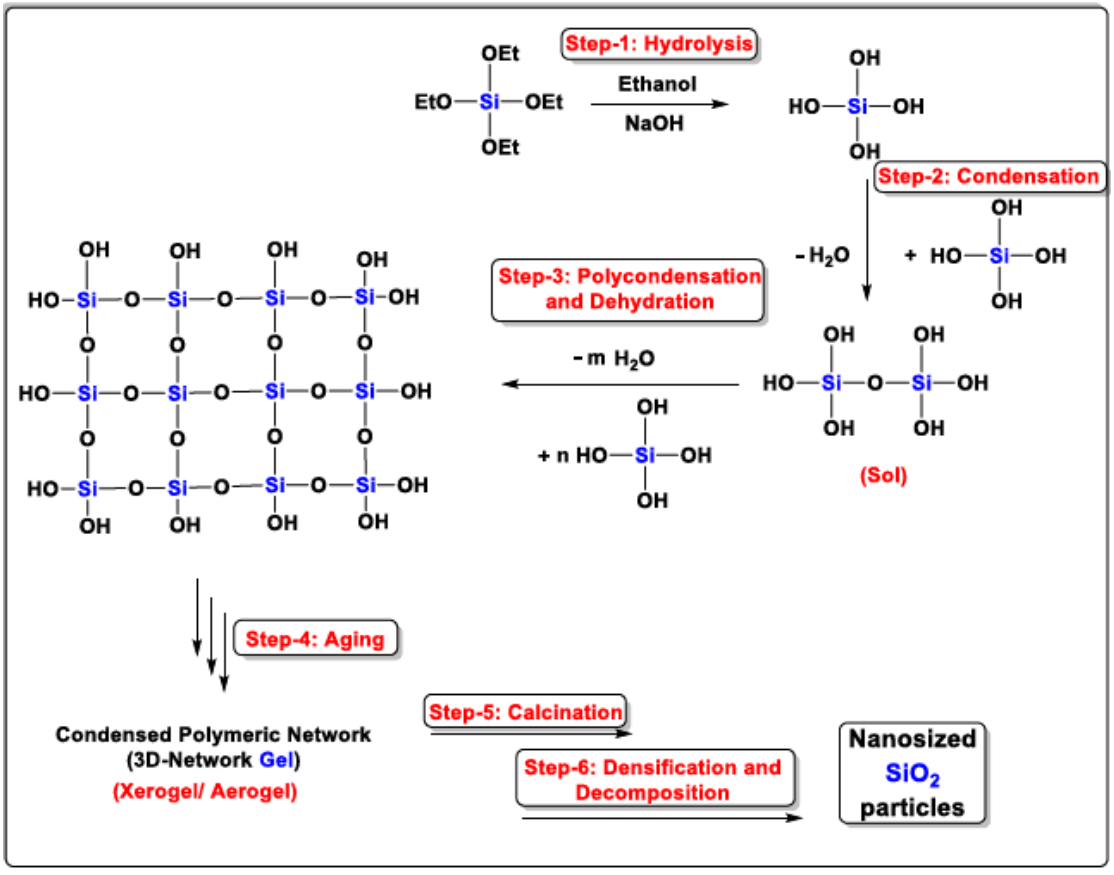
Fig. 1. Simplified chart of the sol-gel method for preparation of nanosilica particles ($ScCO_2$ = supercritical CO_2)

Possible chemical transformations:

$$Si(OC_2H_5)_4 + x\,H_2O \xrightarrow{C_2H_5OH} Si(OC_2H_5)_{4-x}(OH)_x + x\,C_2H_5OH$$

$$Si(OC_2H_5)_{4-x}(OH)_x + Si(OC_2H_5)_{4-x}(OH)_x \xrightarrow{C_2H_5OH} Si(OC_2H_5)_{4-x}(OH)_{x-1}OSi(OH)_{x-1}(OC_2H_5)_{4-x} + H_2O$$

Further, sol is converted into gel by polycondensation of silicon hydroxide [$Si(OH)_4$] into a condensed polymeric network (from sol $\rightarrow$ gel) as shown below.



On drying, the condensed polymeric network gets converted into a three-dimensional network result a gel.

Colloidal solution (sol) → Condensed polymeric network (gel) → Dried form (Three-dimensional network) (gel)

On calcination of the gel, SiO_2 nanoparticles are formed. SiO_2 nanoparticles are characterized by powder XRD and Electron Microscopic techniques.

3. Requirements:

Reagents and solutions: Tetraethylorthosilicate (TEOS), ethanol, NaOH pellets and distilled water.

Apparatus, equipment and Instruments: 50 or 100 mL measuring cylinder, two 100 mL beakers, glass rod for stirring, funnel and filter papers.

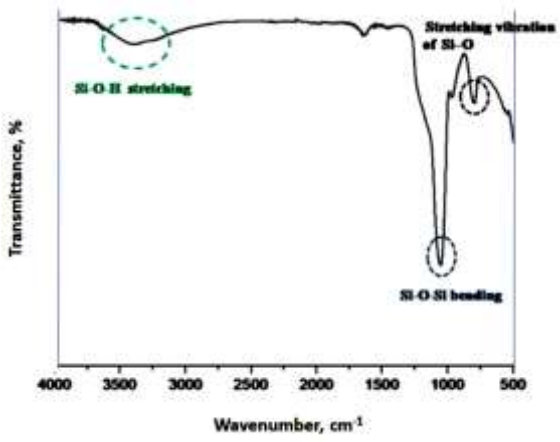
4. Experimental procedure:

In a 100 mL beaker, 5 mL of TEOS is transferred using a measuring cylinder followed by the addition of 5 mL of ethanol. Then 7.5 mL of distilled water is measured through the measuring cylinder and is added to the ethanolic solution of TEOS. Later, **2 pellets** (exactly) of NaOH are added by a spatula to the 100 mL beaker containing the reaction mixture. The two immiscible solutions in the beaker become miscible upon constant mixing them with the glass rod. The pH of the solution should be basic. This sol-gel solution is left as such under stirring for 30 min. The sol-gel obtained silica could be visualized in the bottom of the beaker as a white solid. Later it was filtered using a filter paper.

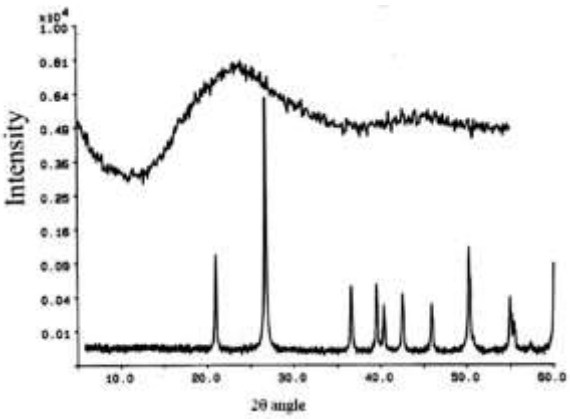
5. Characterization:

The powder XRD pattern of the collected powder sample was recorded on a Bruker D8 Advance (Germany) with Cu Kα radiation source (operating Voltage and current 30 KV, 40MA respectively) in the scan range 10-80º (2Θ). The expected pattern is given in **Fig.2** for reference.

a) Infrared spectrum



b) Powder XRD spectrum



c) Scanning electron microscopic image

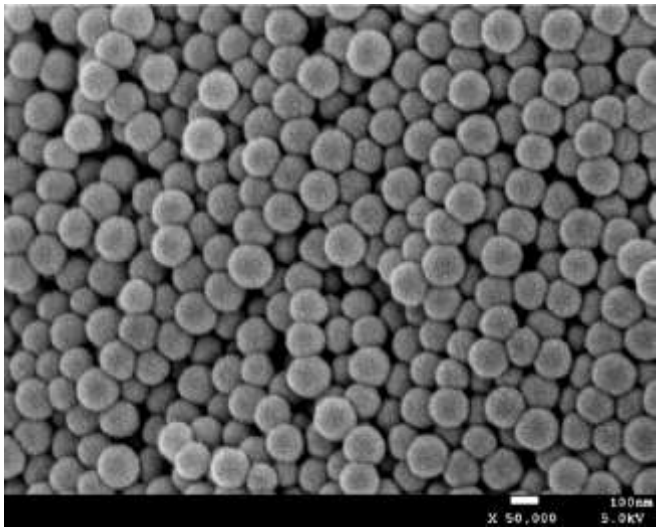


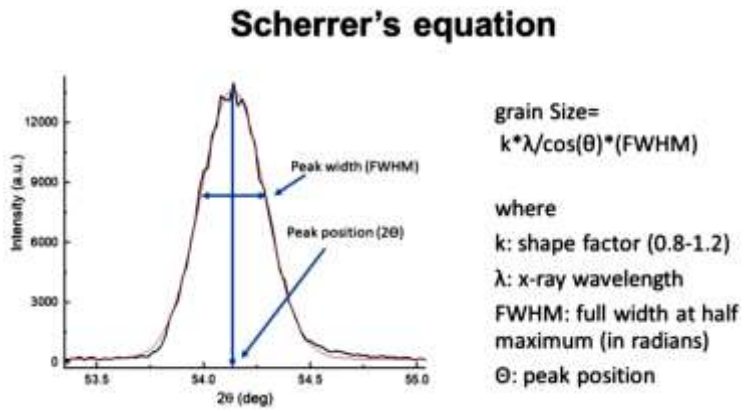
Fig. 2. a) Infrared spectrum of nanosilica particles; b) XRD pattern of SiO_2 nanoparticles mediated in ethanol; c) Scanning electron microscopic image of the surface of nanosilica particles

5. Particle size calculation using Scherrer's equation:

Scherrer equation:

$$\textbf{Grain size} = \frac{k*\lambda}{\cos(\theta)*(FWHM)}$$

where, k = 0.9, λ = 1.0506 Å, and "Θ" and FWHM will be obtained from powder XRD data.



Results:

(i) Quantity of nanosilica material obtained by the sol-gel method after the filtration

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(ii) Particle size calculated as per the "Θ" given_____.