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Fabrication of Silica Nanoparticles by Sol-Gel Method and Effect of TEOS Precursor

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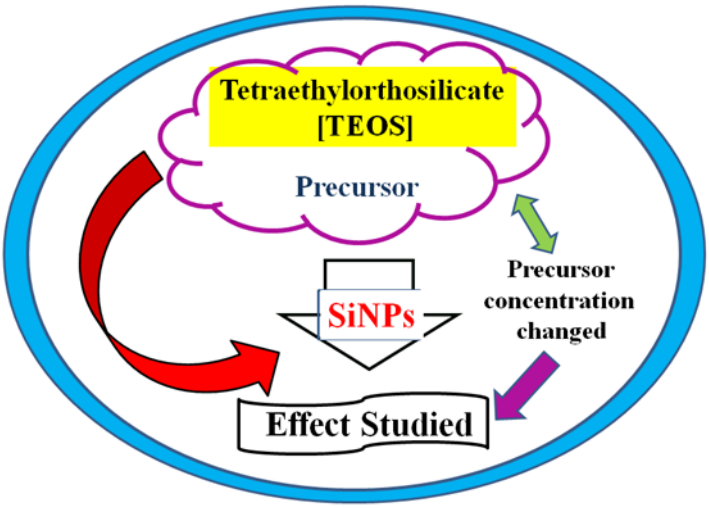
Abstract:
Background*: Silica nanoparticles (SiNPs) have gained significant attention due to their wide applications in catalysis, drug delivery, sensing, and nanotechnology. The sol-gel method is a widely used approach for synthesizing SiNPs due to its simplicity, cost-effectiveness, and ability to control particle size and morphology. Tetraethyl orthosilicate (TEOS) is a key precursor in the sol-gel process, and its concentration plays a crucial role in determining the physicochemical properties of the synthesized nanoparticles. This study investigates the effect of varying TEOS concentrations on the size, morphology, and dispersibility of SiNPs.*
Materials and Methods*: Silica nanoparticles were synthesized via the sol-gel method using TEOS as the silica source, ethanol as the solvent, ammonium hydroxide as the catalyst, and millipore water. The TEOS concentration was varied systematically while keeping other reaction parameters constant. The synthesized nanoparticles were characterized using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), dynamic light scattering (DLS), and X-ray diffraction (XRD) to determine their structural and morphological properties.*
Results*: The results indicate that an increase in TEOS concentration leads to a significant variation in nanoparticle size and agglomeration. At lower TEOS concentrations, uniformly dispersed nanoparticles with smaller diameters (~50 nm) were obtained, whereas higher TEOS concentrations resulted in larger, more aggregated particles. FTIR confirmed the formation of Si–O–Si bonds, while SEM analyses revealed the morphology of SiNPs. DLS measurements indicated a correlation between TEOS concentration and particle size distribution.*
Keyword: *Silica nanoparticles; sol-gel method; TEOS Precursor; Advanced Materials, Nanoparticles Synthesis*



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I. Introduction

Nanotechnology has gained significant attention in recent years due to its wide-ranging applications in various fields, including medicine, catalysis, environmental science, and materials engineering.[1] Among

different nanomaterials, silica nanoparticles (SiNPs) have emerged as one of the most versatile and widely studied due to their unique physicochemical properties such as high surface area, tunable pore structure, biocompatibility, and chemical stability.[2] These properties make silica nanoparticles ideal candidates for applications in drug delivery, biosensors, catalysis, and adsorption.[3-5] The controlled synthesis of silica nanoparticles with desired properties requires a precise understanding of fabrication techniques and precursor effects. The sol-gel method is one of the most widely used techniques for synthesizing silica nanoparticles due to its simplicity, cost-effectiveness, and ability to control particle size and morphology.[6-8] This method involves the hydrolysis and condensation of a silicon alkoxide precursor in the presence of a catalyst and solvent. Tetraethyl orthosilicate (TEOS) is the most commonly used precursor in sol-gel synthesis, as it provides a controlled reaction pathway for forming silica nanoparticles.[9] By adjusting reaction parameters such as precursor concentration, pH, catalyst type, reaction time, and temperature, the size, shape, and surface properties of the silica nanoparticles can be finely tuned. The study of the effect of TEOS concentration is crucial to understanding its role in nanoparticle formation and optimizing synthesis conditions for specific applications.[10-12] Recently, the use of silica nanoparticles has gained significant popularity in various fields due to their numerous advantages over alternative materials, including consistent size, composition, and shape, a surface conducive to modifications, and no adverse effects on human health. The most commonly employed method for producing silica particles of a specific size is the synthesis introduced by V. Stöber and his team in 1968. This method involves the hydrolysis of tetraethoxysilane (TEOS) in a mixture of alcohol and water, followed by the condensation of the resulting silanols. Typically, ammonia is utilized to adjust the pH of the environment (which should range from 11 to 13) in the reaction mixture, where it also serves as a catalyst.[13-16] However, this synthesis can also be conducted in an acidic environment with a pH of 1 to 4 by incorporating HCl and NaF. In this study, we synthesized silica nanoparticles by hydrolyzing TEOS in the presence of different catalysts within an ethanol solvent medium, followed by condensation and polymerization. The physicochemical characteristics of the sols created using this method are influenced by various factors that affect the rates of hydrolysis and condensation reactions, including pH, temperature, initial reactant ratios, order of reactant addition, rate of reactant supply, type and concentration of catalyst, the molar ratio of H_2 O to alkoxysilane, and the conditions and duration of the aging step. By intentionally adjusting these parameters, it is possible to modify the properties of the final product across a broad spectrum.[17] Hydrolysis typically occurs in the presence of mineral acids (HCl) or bases (NaOH, NH_4 OH) as catalysts. The well-known Stöber process has served as a foundation for synthesizing silica nanoparticles through the hydrolysis of TEOS with ammonia as a catalyst. We investigated how process temperature, initial reactant ratios, and catalyst type affect the properties of the synthesized silica sols. The size and shape of the silicon dioxide particles can be regulated by varying the conditions and ratios of synthesis, enabling the production of porous particles.[18-19] Generally, the silica particles produced by the Stöber synthesis are in the size range of 20 to 800 nm. A distinctive characteristic of this method is the ability to separate the hydrolysis and condensation stages of silica particle formation, which facilitates more precise control over both particle size and morphology, including pore size. These particles are frequently utilized in drug delivery due to their well-developed surfaces and the ability to modify them by attaching various functional groups. Moreover, silicon dioxide nanoparticles are extensively employed as biosensors, catalysts, and in the creation of hierarchical superhydrophobic surfaces. Previous studies have shown that existing techniques for controlling nanoparticle size include varying temperature, TEOS, ammonia, and water concentrations, as well as adjusting reaction duration. Another approach involves altering the dielectric constants of the reaction medium using different water-miscible alcohols, which results in smaller particle sizes as the polarity of the medium decreases.[20] On the other hand, the sol-gel method utilizing alkoxysilanes produces a highly pure product and allows for control over particle size during synthesis. In this study, we investigate the fabrication of silica nanoparticles using the sol-gel method and analyze the impact of TEOS precursor concentration on particle size, morphology, and surface characteristics.[21] By systematically varying the TEOS concentration and studying its effect on the synthesized nanoparticles, we aim to establish a correlation between precursor concentration and nanoparticle properties.[22-23] The findings of this study will contribute to the optimization of silica nanoparticle synthesis for potential applications in nanotechnology, biomedical engineering, and materials science.

Applications of Silica Nanoparticles

Application	Description
Drug Delivery	Used in controlled release of drugs, improving bioavailability and targeting specific cells or tissues.
Cosmetics	Incorporated into skincare and makeup products for its light-reflecting properties, as well as in anti-aging formulations.
Biomedical Imaging	Used as contrast agents in imaging techniques like MRI and CT scans to improve the resolution.
Catalysis	Acts as a support material in heterogeneous catalysts, enhancing reaction efficiency and selectivity.
Sensors	Employed in sensors for detecting gases, biological agents, and other chemicals due to their high surface area and reactivity.
Food Industry	Used as food additives (anti-caking agents) and in packaging to extend shelf life by absorbing

	moisture.
Water Treatment	Applied in water filtration systems for removing contaminants and improving water quality.
Environmental Remediation	Utilized in removing pollutants like heavy metals and organic toxins from water or soil.
Electronics	Used in the production of advanced materials such as semiconductors, photonic devices, and in the fabrication of microelectronics.
Textiles	Incorporated into fabrics for water and stain resistance, improving durability and functionality.
Solar Cells	Used in enhancing the efficiency of solar cells by improving light absorption and energy conversion.
Paints and Coatings	Used to enhance the durability and scratch resistance of paints and coatings, as well as providing anti-corrosive properties.

II. Material And Methods

The synthesis of silica nanoparticles was carried out by using tetraethylorthosilicte (TEOS) (99%) as a precursor, Ammonium hydroxide (NH_4 OH)**,** Ethanol (C_2 H_5 OH), and Milli pore water (H_2 O). (Sigma-Aldrich, Chemtrade pvt. Limited, Ujjain MP, India), All chemicals were used without further purification.

Synthesis of Silica Nanoparticles via Sol-Gel Stöber Method-

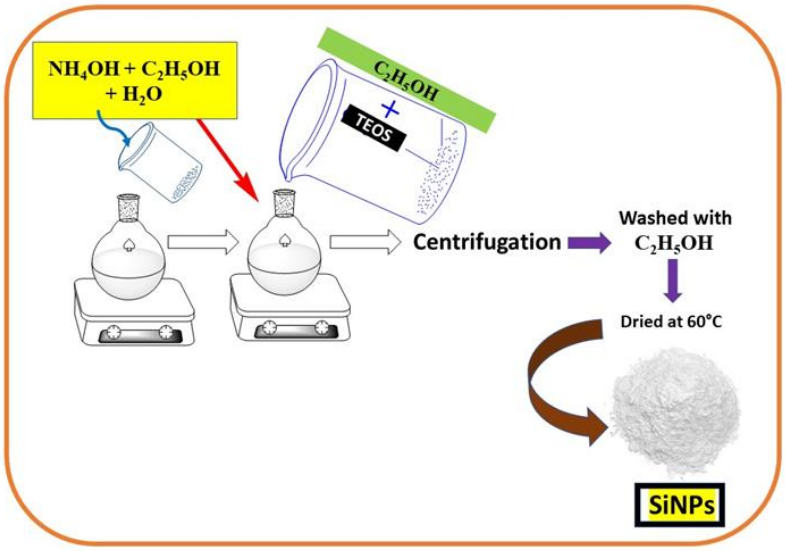

Figure-1 Synthesis of SiNPs

The synthesis of silica nanoparticles was performed using the sol-gel Stöber method, employing tetraethyl orthosilicate (TEOS) as the silica precursor, ammonium hydroxide as the catalyst, and milli water as the solvent. The detailed procedure is as follows: A reaction solution was prepared by mixing 5 mL ethanol and 5 mL millipore water in a beaker under continuous stirring at room temperature. The volume ratio of ethanol to water was maintained,after few minutes the Ammonium hydroxide was added dropwise to the solution to maintain an alkaline pH (around 10-11, as measured using a pH meter). After 1 hour 2 mL Tetraethyl orthosilicate (TEOS) was added dropwise to the reaction mixture while maintaining vigorous stirring . The solution was stirred at 10000 rpm for 5 hours in water bath (30°C). During this process, TEOS undergoes hydrolysis and condensation reactions, leading to the formation of silica nanoparticles. The reaction mixture was allowed to age at room temperature for 24 hours**,** facilitating the growth of silica nanoparticles. The resultant solution was centrifuged at 10,000 rpm for 10 minutes to separate the silica nanoparticles from the supernatant. The nanoparticles were washed multiple times with ethanol and millipore water to remove unreacted precursors and impurities. The washed silica nanoparticles were dried in an oven at 60°C to obtain a fine powder.

III. Results

Effect of TEOS Concentration on Silica Nanoparticle Size:

The size and morphology of silica nanoparticles (SiNPs) synthesized using the sol-gel method were analyzed using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), X-Ray Diffraction Spectroscopy (XRD) and Dynamic Light Scattering (DLS). The results indicate that the concentration of TEOS plays a crucial role in determining particle size, uniformity, and agglomeration. As the TEOS concentration increased, the particle size also increased due to enhanced silica network growth.

Structural and Morphological Analysis:

FTIR spectra confirmed the formation of Si–O–Si bonds, indicating successful silica formation. XRD analysis showed the presence of amorphous silica, consistent with the sol-gel-derived nanoparticles. DLS results revealed the size of nanoparticles, SEM images revealed the morphology of SiNPs.

Particle Size Distribution and Stability:

DLS measurements showed a gradual increase in hydrodynamic diameter with increasing TEOS concentration. The Zeta potential values indicate good colloidal stability at lower TEOS concentrations but a decline as aggregation increased at higher concentrations.

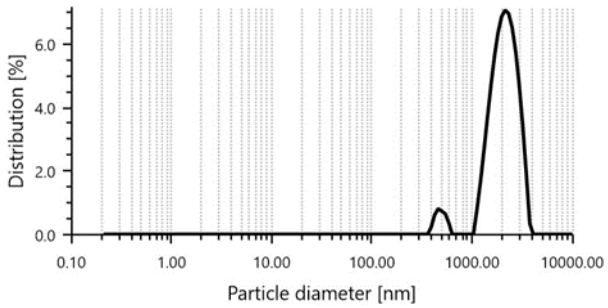

Figure-2 DLS Spectra of SiNPs

Table 1: Effect of TEOS Concentration on Silica Nanoparticle Properties

TEOS Concentration (M)	Particle Size (DLS) (nm)	SEM Morphology
0.05 M	48 ± 5	dispersed
0.10 M	72 ± 7	Uniform, slight agglomeration
0.15 M	95 ± 10	Partially aggregated spheres
0.20 M	125 ± 12	Increased aggregation
0.25 M	160 ± 15	Large clusters, high agglomeration

FTIR and XRD Analysis:

The FTIR spectra of SiNPs was recorded to analyze the presence of functional groups, this spectrum was recorded in the ATR mode.

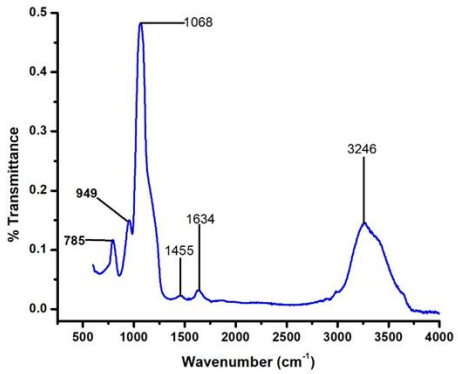

Figure-3 FTIR Spectra of 0.5 M SiNPs

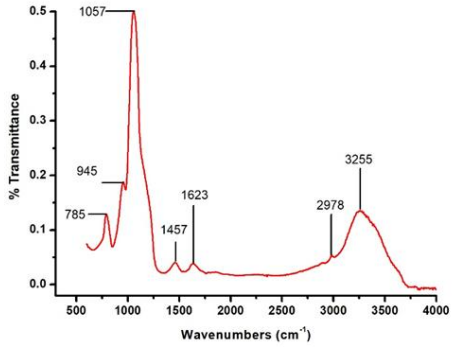

Figure-4 FTIR Spectra of 0.10 M SiNPs

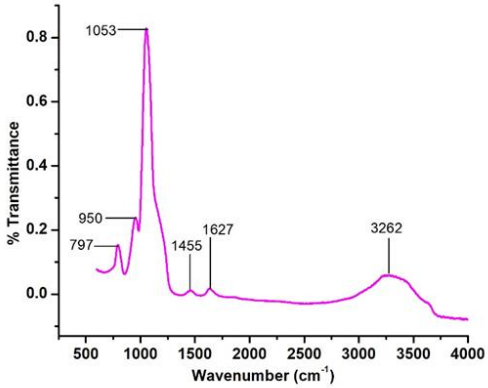

Figure-5 FTIR Spectra of 0.15 M SiNPs

FTIR spectra displayed characteristic peaks at **~1068 cm⁻ ¹** (Si–O–Si asymmetric stretching) and **~800 cm⁻ ¹** (Si–O bending), confirming silica formation. XRD patterns showed a broad peak at **~32° (2θ)**, indicative of crystalline silica

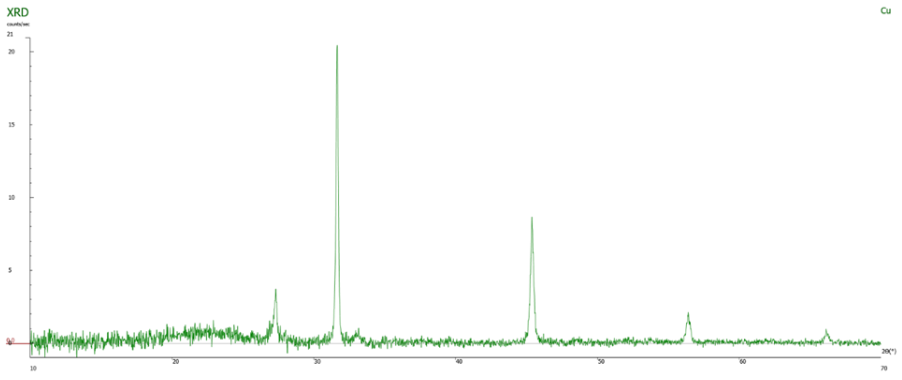

Figure-6 XRD Spectra of SiNPs

Microscopic Characterization

SEM images revealed that lower TEOS concentrations resulted in small, well-dispersed particles, while higher concentrations led to increased agglomeration.

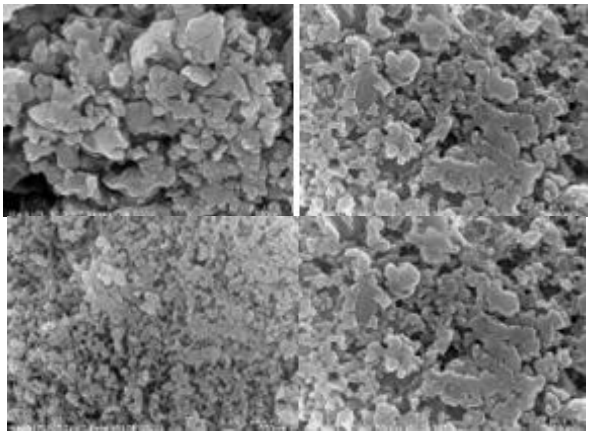

Figure-7 SEM images of SiNPs

The results indicate that optimizing TEOS concentration is essential for controlling the size, stability, and morphology of silica nanoparticles. Lower TEOS concentrations yielded well-dispersed nanoparticles, while higher concentrations promoted aggregation due to excessive silica network formation.

IV. Discussion

The sol-gel method is a widely used technique for synthesizing silica nanoparticles (SiNPs) due to its versatility, cost-effectiveness, and ability to control particle size. In this study, we investigated the effect of TEOS concentration on the structural, morphological, and colloidal properties of SiNPs. Our findings indicate that TEOS concentration plays a significant role in controlling particle size, dispersibility, and surface charge, which are critical factors for applications in catalysis, sensing, and biomedicine.

Effect of TEOS Concentration on Nanoparticle Size and Morphology:

One of the primary findings of this study is the strong correlation between TEOS concentration and nanoparticle size. As an increase in TEOS concentration resulted in larger particle sizes. This phenomenon can be attributed to the higher availability of silica precursor molecules in the reaction medium, leading to enhanced hydrolysis and condensation reactions. At lower TEOS concentrations (0.05 M - 0.10 M), the nanoparticles formed were relatively smaller (48–72 nm), well-dispersed, and spherical. However, as the TEOS concentration increased, larger nanoparticles (up to ≥160 nm at 0.25 M TEOS or more than 0.25 M concentration) with increasing agglomeration were observed. This aggregation is likely due to the uncontrolled growth of silica networks, which promotes particle clustering. Thus, TEOS concentrations in the range of 0.05 M to 0.10 M are ideal for achieving well-dispersed and stable silica nanoparticles.

FTIR and XRD Analysis: Structural Confirmation:

The FTIR spectra confirmed the successful formation of silica nanoparticles by displaying characteristic peaks of Si–O–Si bonds. The peak at ~1068 cm^{-1} corresponds to Si–O–Si asymmetric stretching, while the peak at ~800 cm^{-1} is associated with Si–O bending vibrations. The absence of peaks corresponding to organic residues or unreacted precursors suggests the formation of high-purity silica nanoparticles.

The XRD analysis further supports these findings, revealing a broad peak around 32° (2θ), which is characteristic of crystalline silica. The crystalline nature of the synthesized nanoparticles is a desirable feature, particularly for applications where high surface area, porosity, and reactivity are essential, such as catalysis and adsorption-based technologies.

Comparison with Literature Studies:

Our findings are consistent with previous studies on sol-gel synthesized silica nanoparticles, where TEOS concentration has been identified as a key factor in controlling particle size and dispersibility. Similar trends have been observed in studies where silica nanoparticles synthesized with lower TEOS concentrations (≤0.10 M) exhibited better dispersion and stability, whereas higher concentrations led to increased aggregation and polydispersity. The ability to tune particle size by adjusting TEOS concentration provides a scalable approach for synthesizing silica nanoparticles tailored to specific applications.

Implications for Practical Applications:

The ability to control silica nanoparticle size and stability by adjusting TEOS concentration has significant implications for various applications:

- ❖Biomedical Applications: Smaller, well-dispersed silica nanoparticles (50–70 nm) are highly desirable for drug delivery, bioimaging, and biosensing due to their ability to penetrate biological systems efficiently.
- ❖Catalysis and Adsorption: Larger particles with controlled porosity are beneficial for catalytic applications and adsorption-based separations.
- ❖Optical and Electronic Applications: The amorphous nature of the synthesized silica nanoparticles makes them ideal for optical coatings, thin films, and electronic devices.

Limitations and Future Work:

Although this study provides valuable insights into the role of TEOS concentration in silica nanoparticle synthesis, several aspects require further investigation:

1. Long-Term Stability Studies: Future work should examine the aging effects on nanoparticle dispersion and stability over extended periods.
2. Porosity and Surface Area Analysis: A BET (Brunauer–Emmett–Teller) surface area analysis can provide a deeper understanding of surface properties and pore structures.
3. Functionalization Studies: Surface modification with organic ligands or biomolecules can enhance the applicability of silica nanoparticles in biosensing and drug delivery.
4. Synthesis of Zeolitic imidazolate framework with the help of silica nanoparticles.

V. Conclusion

This study demonstrates that TEOS concentration significantly influences the size, stability, and morphology of silica nanoparticles synthesized via the sol-gel method. Lower TEOS concentrations (0.05 M – 0.10 M) resulted in smaller, well-dispersed nanoparticles, whereas higher concentrations led to increased aggregation and reduced stability. The findings provide a foundation for the controlled synthesis of silica nanoparticles tailored for nanotechnology, catalysis, and biomedical applications. Future work should explore surface functionalization, porosity control, and application-specific modifications to further enhance the potential of sol-gel-derived silica nanoparticles. TEOS serves as the silica precursor in the sol-gel process, where it hydrolyzes and condenses to form silica nanoparticles. By increasing the quantity of TEOS, more silica is available for the reaction, which results in a higher overall yield of silica nanoparticles. As TEOS is added in larger quantities, the reaction might proceed further, leading to a greater number of silica particles, but the process should still be controlled to prevent issues like over-condensation, which can lead to the formation of larger aggregates or a more viscous solution that might reduce the effective yield. The size of silica nanoparticles typically increases as the quantity of TEOS is increased. This happens because more silica precursor is available to form larger particles during the hydrolysis and condensation steps. Larger amounts of TEOS result in more silica molecules being available for the growth of the particles, leading to larger nanoparticle sizes. The actual size also depends on factors like solvent choice, reaction temperature, and the presence of catalysts. If these parameters are optimized alongside the increase in TEOS, the growth of nanoparticles can be controlled to achieve a desired size range.

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