

RESEARCH PAPER

## Optimization of Silica Synthesis via Stöber Method: Effects of CTAC, Temperature, and Reaction Time on Spherical Particle Size

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### ABSTRACT

Monodisperse ORMOSIL particles with a size of 0.4-7.5  $\mu\text{m}$  were synthesized in a single-step, low-temperature homogeneous solution reaction using tetraethyl orthosilicate (TEOS) as the precursor, base catalyst (triethylamine), and cetyltrimethylammonium chloride (CTAC) surfactant. In the current study, the influence of three significant parameters (CTAC surfactant content, reaction duration, and synthesis temperature) was systematically investigated to have precise control over particle size and shape. The result showed that increased concentration of the CTAC reduced particle size, whereas increased temperature and longer reaction duration favored larger particle size and increased sphericity. In addition, the ORMOSIL particles synthesized were thermally treated at 600  $^{\circ}\text{C}$  to remove organic species and yield highly pure silica particles with a monodisperse particle size. We achieved precise control over particle size and morphology by varying CTAC concentration, temperature, and reaction time. The strategy offers advantages in scalability and ease and allows for precise tuning of particle properties for different applications in drug delivery, catalysis, electronics, and nanocomposites. The findings of this study form a foundation for the synthesis design of multidimensional silica particles with tunable properties, which would provide exciting opportunities for future industrial and applied research applications.

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## 1. INTRODUCTION

Researchers have shown a great interest in the past few years to learn about ways of synthesizing silica particles. Owing to its ability of creating very controlled size, distribution and structure of particles, the process can be widely used in various

scientific and industrial fields [1]. Stöber process fast stands out as a thoroughly admired approach within this field, which has been designed to produce silica particles that have wonderful properties such as high surface area, appropriate pore volume and homogenous particle size [2]. In the synthesis, it is a common practice in solutions

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with water, and because of its wide applicability, it stands out to be of great value to industries such as pharmaceutical, chemistry and environmental technologies [3]. Its ability to control the structural and physical characteristics of silica nanoparticles is what makes Stöber process so relevant to nanoparticle synthesis. Using this technique, particles are synthesized by chemical reactions and the combination of tetraethyl orthosilicate (TEOS) in aqueous solution leads to the formation of silica particles. The potencies of silica particles produced via the Stöber process may be adjusted by changing factors like temperature, pH, and reactant ratio. Such changes in size, morphology, and internal structure could have significant implications regarding the differences in particles' efficiency and functional potential (especially in environmental and pharmaceutical use) [4] [5]. The role of the pH during the synthesis process in making silica nanoparticles by the Stöber technique is emphasized in this study. The acidity and alkalinity of the solution plays a huge role in how fast silica molecules are going to hydrolyze and condense. Research has shown that the alterations of pH can change the silica nanoparticle physical structure [6]. Beside pH, the temperature of the reaction has become a vital factor in the creation of the particles' properties. Temperature changes affect the reaction kinetics causing a shift in the particle size and the evenness distribution. Higher temperatures cause chemical reactions to speed up and this may lead to the creation of larger and more nonuniform particles. On the other end of the temperature scale, the particles produced are usually smaller with better homogeneity [7]. Complementing pH and temperature, the selection of the catalyst type, along with its concentration, may have a significant effect on the yield of Stöber process-produced particles. The choice of catalysts, such as ammonium hydroxide or amines, influences greatly the kinetics of nanoparticle synthesis as well as their structural characteristics [11]. The products of the Stöber process have properties that qualify them as potential candidates for a

wide range of applications in many areas. The specific surface area of these particles is notably high, thus, their adsorption and reactivity in chemical processes. Chemical, pharmaceutical, and environmental industries value the unique properties of the silica nanoparticles [9]. Silica nanoparticles are involved in the pharmaceutical industry as carriers for the drug substances. Their large specific surface area and the mesoporous structure, allow silica nanoparticles to deliver drugs exactly and in a controllable manner to targeted zones inside the body. Besides, such nanoparticles are also used in water and air purification systems due to their ability to adsorb pollutants. The nanoparticles of silica have aroused interest with regard to their use in environmental applications for their ability to remove the pollutants that affect both water and soil. Their mesoporous characteristics allow the silica nanoparticles to attach with such pollutants like heavy metals and organic compounds thus aiding their removal from environment [12]. Although the Stöber process possesses numerous strengths in synthesizing silica nanoparticles, there is still a need for the continuous improvement for its performance. Under varied reaction conditions, nanoparticles are commonly sized in unique ways that will clump unevenly and negatively affects them in being useful for different applications. Simultaneously, surfactants used in the nanoparticle synthesis may lead to such issues as the generation of unwanted contaminants or the inflation of expenditure [13] [12]. In more recent days, there has been a trend towards the development of methods to use in silica nanoparticle production that eliminates the use of surfactant. Using surfactant-free approaches not only reduces cost and environmental hazards but also promotes the production of nanoparticles with desired optimal and stable properties. The majority of recent improvements have focused on achieving greater refinement of synthesis protocols and fine tuning the molar stoichiometry of precursor materials in order to synthesize silica nanoparticles with ideal mesoporous attributes [14][15]. A detailed review of the existing

literature on silica nanoparticle synthesis in the last few years presents five key approaches to the surfactant-free routes. Kolodziejczak-Radzimska and Pota studied enzyme stabilization and the use of humic compounds with silica nanoparticles prepared using Stöber methods, allowing for the synthesis free of the surfactants suitable for biological applications. Han and Mayer replaced ammonia with amine-based organic catalysts such as DETA and ethanolamine, which facilitated the size limitations (11-942 nm) associated with the classical Stöber method [5][11]. Third, Sun showed that alterations to surfaces with amine and carboxyl groups revealed 61.6% reduction in cytotoxicity [12][15]. Yamamoto and Galland Barra work included the Stöber method and emulsion polymerization in the manufacture of multifunctional core-shell nanocomposites [8][14]. Following the results of these studies, it becomes clear that four critical strategies for the surfactant removal are crucial: Improving process efficiency as optimised pH (at 10), appropriate temperature, and the ratio of molar quantities for TEOS-H<sub>2</sub>O-EtOH systems (1:4:20), use of alternative organic catalyst. The data demonstrate that surfactant-free approaches reduced production costs substantially (up to 30-40%) and enhanced the efficiency in catalysis processes, drug transport and filtration of water through accurate control of pores (3-10 nm) and the concept of surface functionalities (APTES, PEG). However, issues such as void creation in mesoporous forms and issues relating to scaling up of production beyond 100 g/day require further discussions. This thorough review describes critical strategies to enhance the development of environmentally friendly synthesis of silica particles. In the light of the advances that have been made with synthesizing

silica nanoparticles using the Stöber method, it is clear that this approach is extremely efficient and financially viable to produce nanoparticles with a customisable set of properties. However, due to the fact that these nanoparticles are used in many industries, especially in the areas such as environmental management and pharmaceuticals, these nanoparticles are of significant value. In the future, scientific achievements may allow for the better synthesizing technologies, overcome present-day limitations, and enable the industrial manufacturing of more effective and exactly programmable nanoparticles.

In sum, the primary objective of this work was to develop an uncomplicated, one-step, low-temperature synthesis pathway for the synthesis of monodisperse ORMOSIL particles with controlled sizes (0.4–7.5  $\mu\text{m}$ ) by systematically studying the impact of three key parameters: CTAC surfactant concentration, reaction temperature, and the reaction time. By optimizing these parameters, the research aimed to achieve precise control over particle size and shape.

## 2. MATERIALS AND METHODS

### 2.1. All materials used

Tetraethyl orthosilicate (TEOS, 98%, Coupsyl) was used with further purification (TEOS was pre-treated in an oven at 90°C for 1 hour to evaporate residual ethanol and water prior to use), triethylamine (TEA, 99.0 wt.%, Neutron) was used as a condensation catalyst and cetyltrimethylammonium chloride (CTAC, 99.0%, Behbood Shimi Hamoon) as the surfactant.

### 2.2. Synthesis method

The experimental workflow involved:



The synthesis procedure was developed in this study. A mixture of 100 mL DI water and different amounts of CTAC (0 g – 2 g – 3 g- 5 g) for each experiment were transferred to a 250-mL flask and stirred at 40 °C (as an example). Then, 0.3 mL TEA was added, followed by dropwise addition of 10 mL TEOS under continuous stirring at 500 rpm. Also, the pH of the solution was adjusted to 10 using triethylamine (TEA) as a base catalyst.

The formed particles were collected and rinsed several times with DI water after different times for the various experiments. Then the particles were dried in an oven at 110 °C for 2 h, and the reaction variables of the above procedure (for example, the temperature, time and the dosage of CTAC used in the experiments) were changed over many runs to get the reaction conditions of monodisperse ORMOSIL particles with different sizes. For the conversion of TEOS-based ORMOSIL particles into silica particles, the particles were sintered at a heating rate of 10 °C/min for 5 h at 600 °C.

formation of silica, as clearly evidenced by the characteristic absorption peaks observed in the spectrum. The most prominent feature is the strong and broad absorption band centered at 1045.6 cm<sup>-1</sup>, which corresponds to the asymmetric stretching vibrations of Si-O-Si bonds - the fundamental structural component of silica. This peak serves as the primary indicator of silica formation. Additionally, the spectrum shows a distinct peak at 474.91 cm<sup>-1</sup>, attributed to the bending vibration modes of the Si-O-Si network, further confirming the silica structure. The absence of significant peaks corresponding to organic contaminants or other phases indicates the successful formation of relatively pure silica. These spectral features, particularly the dominant Si-O-Si stretching and bending vibrations, are in excellent agreement with well-established reference spectra for the silica materials, providing conclusive evidence of silica formation in the analyzed sample. The results demonstrate that the sample consists primarily of silica with its characteristic tetrahedral SiO<sub>4</sub> network structure.

### 3.RESULTS AND DISCUSSION

The FTIR analysis in Figure 1 confirms the

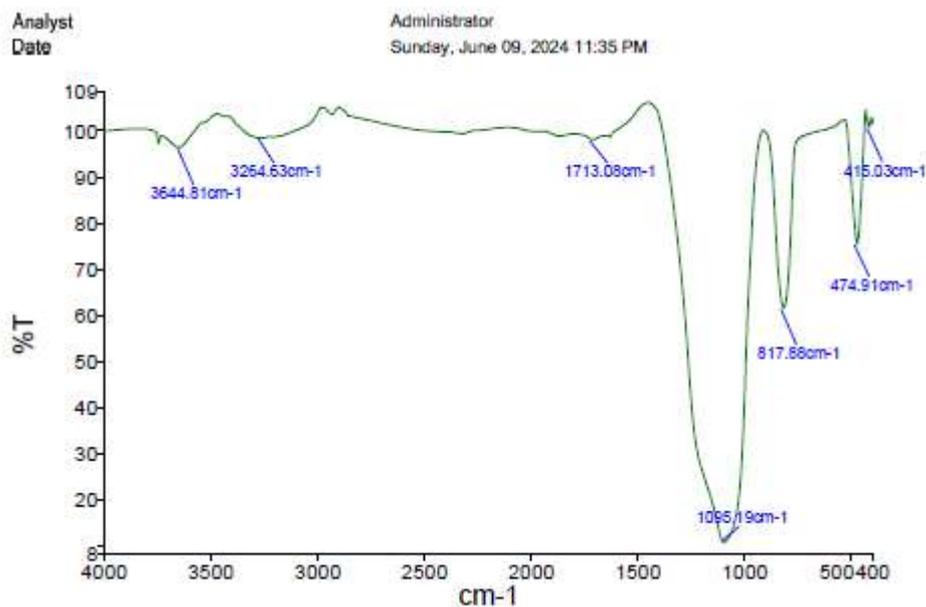


Figure 1. The FTIR analysis of silica particles

The XRD pattern in Figure 2 shows a characteristic broad peak centered at 22° 2θ, confirming the amorphous nature of the synthesized silica. The absence of sharp diffraction peaks indicates that no crystalline impurities are

present. These results demonstrate the successful preparation of pure amorphous silica, consistent with standard references for this material. The broad diffraction feature is typical of amorphous silica's short-range ordered structure.

Sample Identification:	SILICA
Measurement Date / Time:	2025/04/30 07:18:00 P.U
Start Position [°2Th.]:	5.0100
End Position [°2Th.]:	79.9900
Step Size [°2Th.]:	0.0200
Anode Material:	Cu
K-Alpha1 [Å]:	1.54060
Generator Settings:	40 mA, 40 kV
Diffractometer Type:	XPert MPD

### Graphics

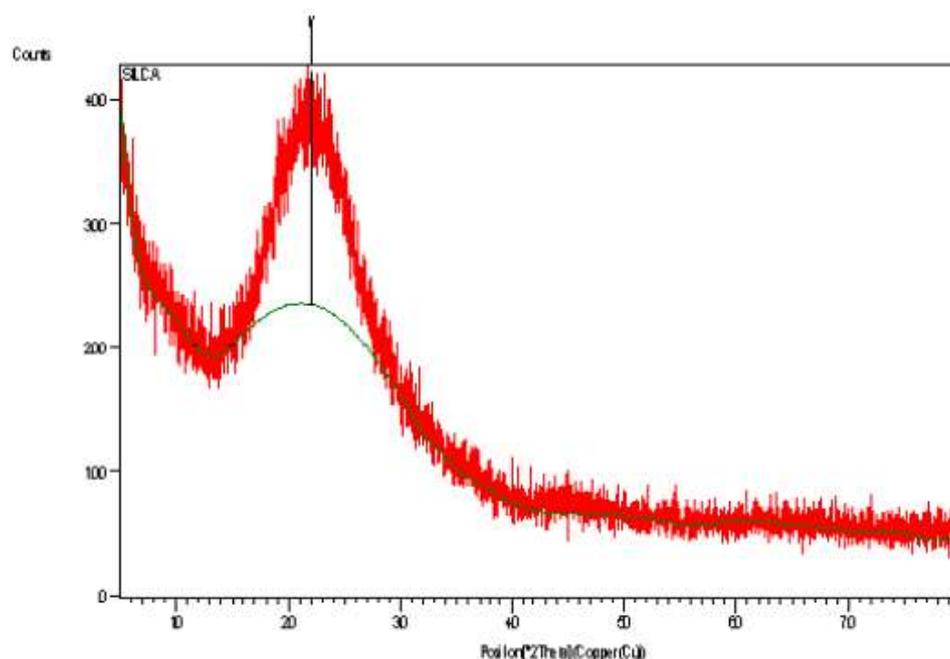


Figure 2. The XRD analysis of Silica Particles

In the following sections, we summarize the effect of each of the reaction parameters on the morphology of the ORMOSIL particles (Figs. 1–9). Also, each experiment was repeated three times.

#### Methods for Particle Characterization:

##### 1. Sphericity Measurement:

The degree of particle sphericity was quantified

by analyzing the optical microscopy images. A square grid was overlaid on each image, and the percentage of spherical particles relative to the total number of particles within the grid was calculated.

##### 2. Particle Size Measurement:

Particle size distribution was determined using Field Emission Scanning Electron Microscopy

(FESEM). The diameters of individual particles were measured from FESEM micrographs, and the statistical analysis was performed to report the average size and uniformity.

### 3.1. Exploring the effects of CTAC on ORMOSIL particles

The initial question queried how particle properties are affected by the concentration of CTAC surfactant. Figure 3 is quantitative to show how varying the amount of CTAC alters the extent of sphericity, with higher levels of CTAC significantly increasing roundness of the particles. Figure 4 rounds off this view by demonstrating the corresponding particle size determinations, with the reverse effect of CTAC concentration going down and the particle diameter going up. Figure 5 provides visual evidence through FESEM images, comparing particles synthesized without CTAC (irregular shape) with those with 2g CTAC (intermediate improvement) and 5g CTAC (highly spherical). These figures cumulatively establish CTAC's dual role as both size-controlling and shape-directing agent through micelle formation and the surface stabilization mechanisms.

In the absence of CTAC, the particle formation occurs through the uncontrolled hydrolysis and condensation of TEOS to form non-spherical aggregates with broad size distribution. Surfactant stabilizes nuclei and regulates spherical growth through micelle templating. In the absence of CTAC, the lack of interfacial stabilization results in polydisperse, non-spherical particles, as evident from FESEM images (Figure 5a). This agrees with classical Stöber synthesis, in which surfactants are optional but necessary for the monodispersity.

CTAC is a cationic surfactant which is used a lot for the silica nanoparticle synthesis. For example, in Stöber synthesis, CTAC is used as a template to grow spherical silica by controlling the growth and aggregation of silica precursors. The effectiveness in the stabilization of silica particles for such uniform growth into a spherical form leads to its presence in the synthesis solution [16][17]. Thus, CTAC is an important ingredient for the spherical

silica particle synthesis as it serves as a template for their formation. It plays a critical role due to its ability to form micelles in solution by self-assembly. These micelles become templates for the silica precursor aggregation and condensation, leading to the formation of spherical silica particles [18]. The following are some important factors that make CTAC's role significant: 1. Template Formation: CTAC molecules self-assemble into micelles, which provide a structured framework to which silica precursor molecules may diffuse and dissolve, thus promoting growth. The template ensures that silica nanoparticles will grow into a spherical shape. 2. Controlled Growth: CTAC micelles control the rate at which silica particles grow and join. This control is needed to achieve the uniformity in size and shape of the particles that is generally desirable to satisfy most applications. 3. Stabilization: CTAC molecules are responsible for the coverage of growing silica nanoparticles at their surfaces and the prevention of aggregation. This stabilization effect is important to maintain the integrity and dispersibility of the nanoparticles. 4. The manipulation of CTAC concentration with other reaction conditions i.e. temperature, allows tuning of size, shape, and surface properties of the silica produced. Such versatility is required for broad applications ranging from catalysis, drug delivery to engineered nanocomposite materials [18][19]. In short, as can easily be deduced from the observations made in the bar charts (Figs. 3 and 4), CTAC act as a templating agent in silica synthesis and whose influences haunt the forms adopted, properties, and even the size of the particles obtained during the synthesis, making CTAC an integral part in developing spherical silica particles. It is seen that different additions of CTAC in different experiments have shown a huge decrease of spheroidization of particles to almost around 650 cm<sup>2</sup> (from 700 cm<sup>2</sup> to 50 cm<sup>2</sup>). Furthermore, particle size is reduced gradually by about 1µm after the addition of 2 gr of CTAC and afterward significantly reduced with increased addition of 3 gr of CTAC to 4µm. Finally, in the last experiment, with 5 gr of CTAC, the particle size

drops a little over 2µm.

Higher CTAC concentrations (5 g/L) reduced particle size to ~2 µm by stabilizing smaller

nuclei through micelle formation, while lower concentrations (2 g/L) resulted in larger particles due to limited surface stabilization.

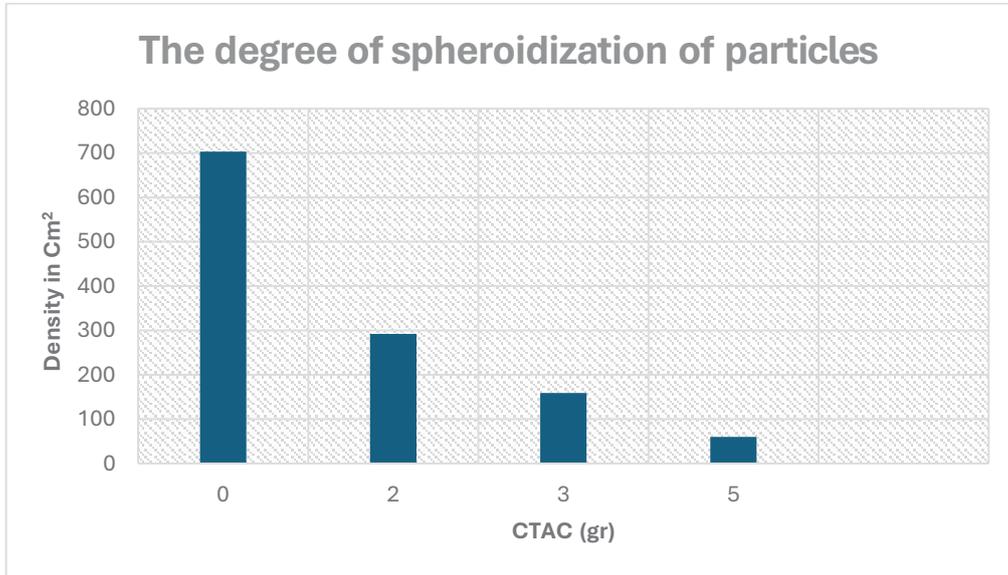


Figure 3. The spheroidization degree of particles through changes in addition of CTAC

Density in cm<sup>2</sup> : Surface Density = (Number of Particles in Image) / (Total Analyzed Area)  
By considering a square grid overlay on optical

microscopy images of the samples, the percentage of spherical particles within each square unit was calculated.

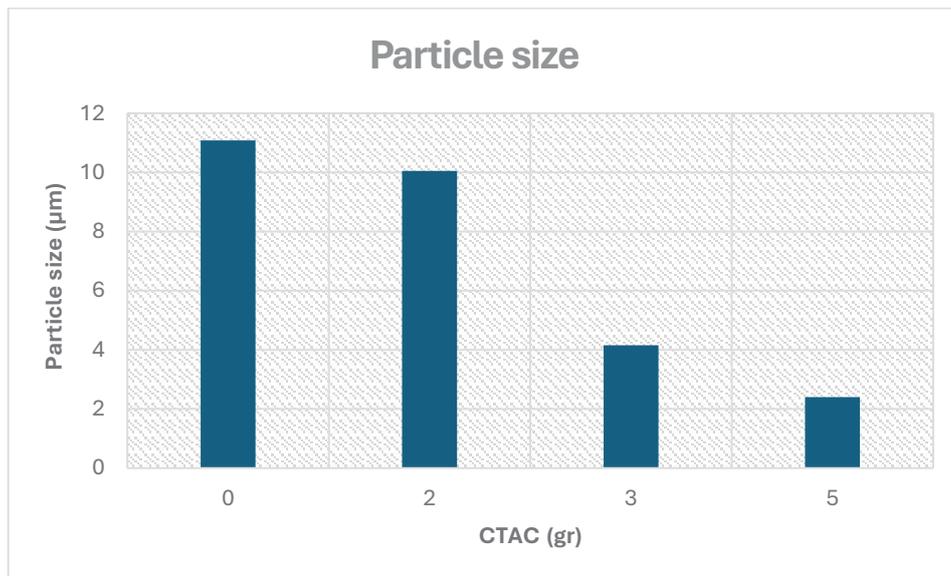
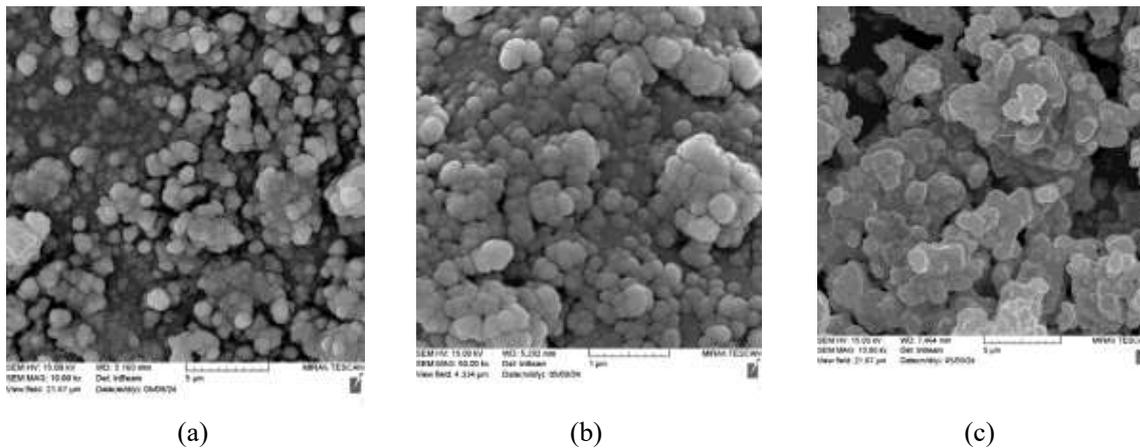


Figure 4. Particle size through changes in addition of CTAC



**Figure 5.** FESEM images of ORMOSIL particles prepared at different addition of CTAC: (a) 0 g, (b) 2 g (c) 5 g (conditions for all experiments, reaction time: 8 hr, temperature: 40 °C)

### 3.2. Exploring the effects of temperature on ORMOSIL particles

Temperature effects are explored beginning with Figure 6 sphericity measurements as a function of reaction temperature and showing increasing particle uniformity with higher temperatures. Figure 7 size distribution values show a pattern of growth with temperature, with larger particles produced at higher temperatures. Figure 8 FESEM images visually document this progression from small, less-defined particles at 25°C to larger, more perfect spheres at 40°C. These results collectively describe how the temperature controls the reaction kinetics, with optimal conditions being a balance between growth rate and morphological perfection. The results show that thermal energy prefers precursor reactivity and surface reorganization to more low-energy (more spherical) states.

Temperature plays a crucial role in silica synthesis because it affects the kinetics and thermodynamics of the reaction [20]. Higher temperatures generally increase the rate of reaction, allowing for faster silica formation. Additionally, temperature can influence the crystallinity, particle size and morphology of the synthesized silica [21]. Different synthesis methods require specific temperature conditions to control these factors and produce silica with desired properties [22].

In the Stöber method, increasing the temperature typically leads to an increase in the particle size as it is clear in Figure 8. Furthermore, it enhances the spheroidization of particles. This is primarily due to the increased rate of hydrolysis and condensation reactions at higher temperatures [22]. When the temperature is elevated, the hydrolysis of silica precursors (such as TEOS) occurs more rapidly, resulting in the formation of larger silica particles. Additionally, the condensation reactions, where the hydrolyzed silica species polymerize to form the silica particles, are favored at higher temperatures, promoting the growth of particles. Moreover, the enhanced mobility of the reactant molecules at higher temperatures facilitates the diffusion-controlled growth process, leading to the formation of more spherical particles [22][23]. As a result, increasing the temperature in the Stöber method generally leads to larger and more uniformly spherical silica particles. From a general perspective, it is evident that the degree of spheroidization of particles is increased by approximately around 120 cm<sup>2</sup> (from 152 cm<sup>2</sup> to 297 cm<sup>2</sup>) during different temperatures in different experiments (Fig. 6).

In addition, particle size increased incrementally with temperature, from 1.5 μm at 25 °C to 3.2 μm at 40 °C, due to the accelerated hydrolysis and condensation rates.

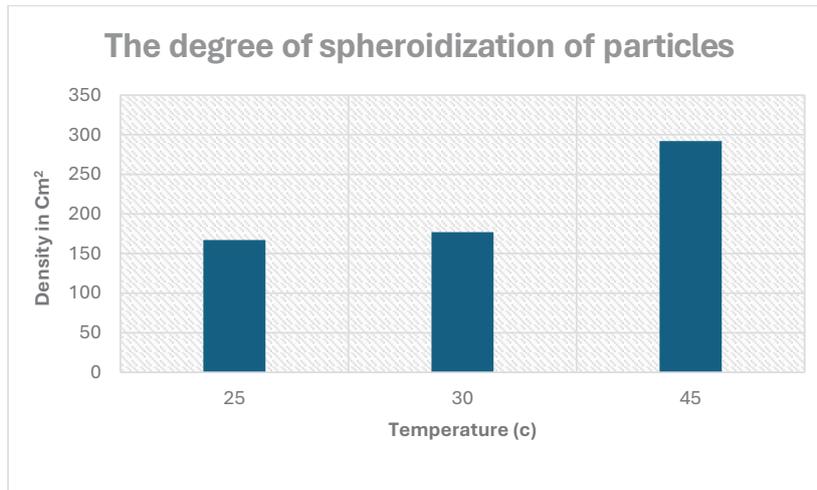


Figure 6. The degree of spheroidization of particles through changes in temperature

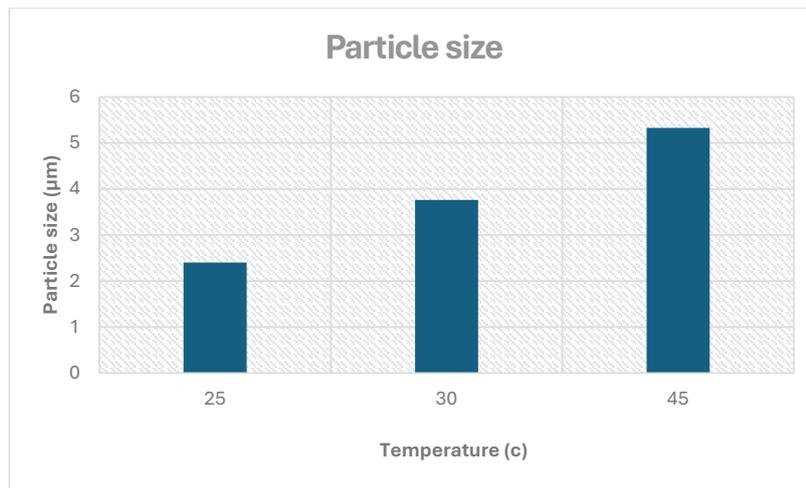


Figure 7. Particle size through changes in temperature

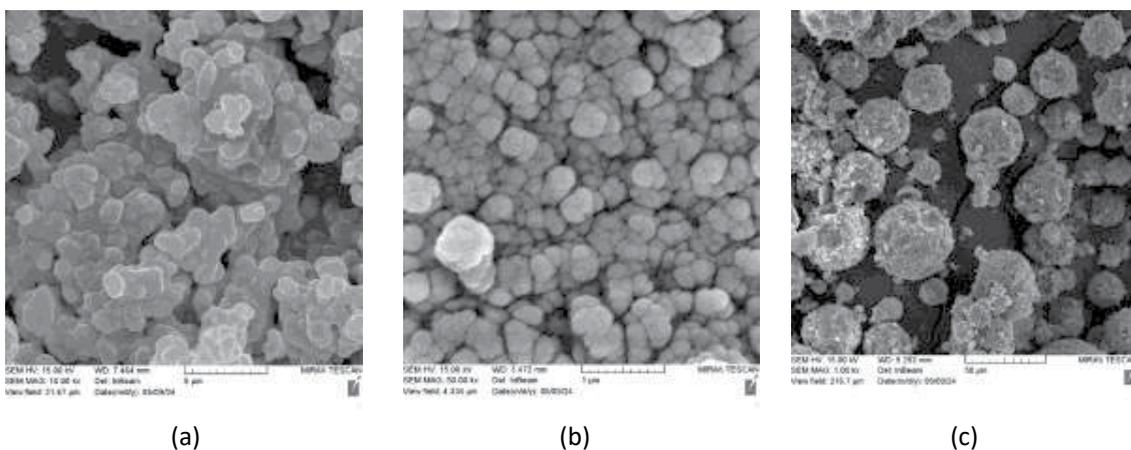


Figure 8. FESEM images of ORMOSIL particles prepared at different temperatures: (a) 25 °C, (b) 30 °C, (C) 45 °C (conditions for all experiments, reaction time: 8 hr, CTAC: 0 g)

### 3.3. Exploring the effects of reaction time on ORMOSIL particles

Time development of particle formation is characterized by Figure 9 measurement of sphericity, which shows how perfection of particle shape develops over time. Figure 10 graphs the corresponding size increases, with distinct growth stages before reaching a plateau. Figure 11 shows how time-series FESEM images provide dramatic visual proof of this maturation process, from the initial nucleation (1 hour) to maximum growth (5 hours) to potential overgrowth (8 hours). This temporal analysis defines the window of critical time for achieving maximum uniformity without undesirable secondary processes like aggregation or Ostwald ripening at longer times.

Longer reaction time generally results in larger particle size in silica synthesis. Longer reaction times allow for more extensive nucleation and growth of the silica particles. This gives the particles more time to come together and form larger structures. Moreover, prolonging the reaction time permits the diffusion of reactants enough to form more crystalline phase [24][25]. On the other hand, very long reaction times can

also result in undesired side reactions or the formation of agglomerates with the potential to affect negatively the desired properties of the resulting silica material and the consequent degree of spheroidization (Figures 9 and 10) [25].

Unexpected size reduction at prolonged reaction times:

While particle size increased steadily with reaction time up to 5 hours (Figure 10), a slight decrease was observed at 8 hours. This deviation from the classical growth models can be explained by:

1. Aggregation-Fragmentation Dynamics: Prolonged stirring may induce weak aggregation, which breaks apart during the centrifugation or FESEM sample preparation, artificially reducing the measured size.

2. Secondary Nucleation: Residual precursors could form new small particles at late stages, skewing the size distribution.

These effects align with literature reports on silica synthesis [23], where excessive reaction times lead to polydispersity. Our data suggest that 5 hours is the critical threshold for achieving optimal monodispersed in this system.

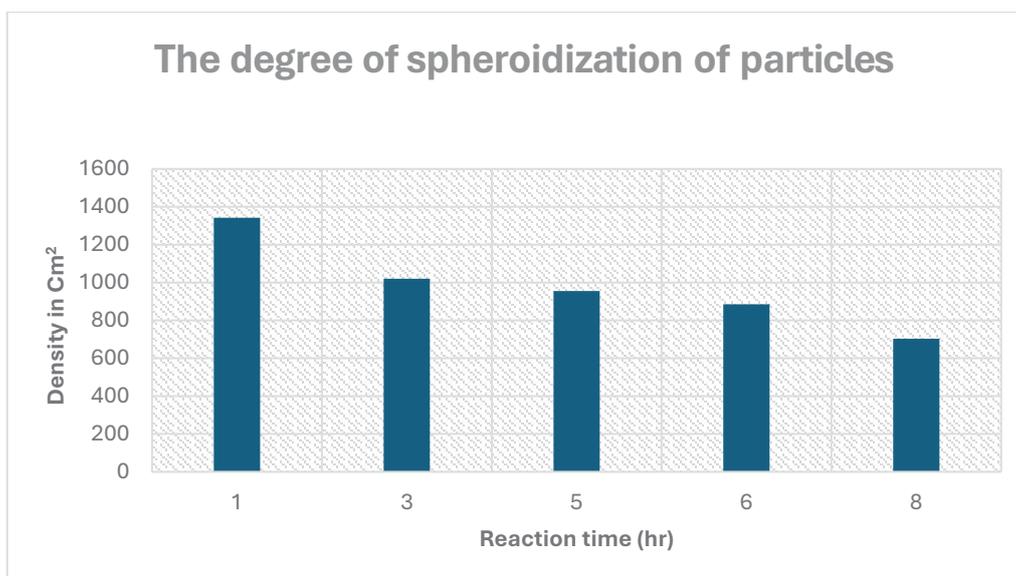


Figure 9. The degree of spheroidization of particles through changes in reaction time

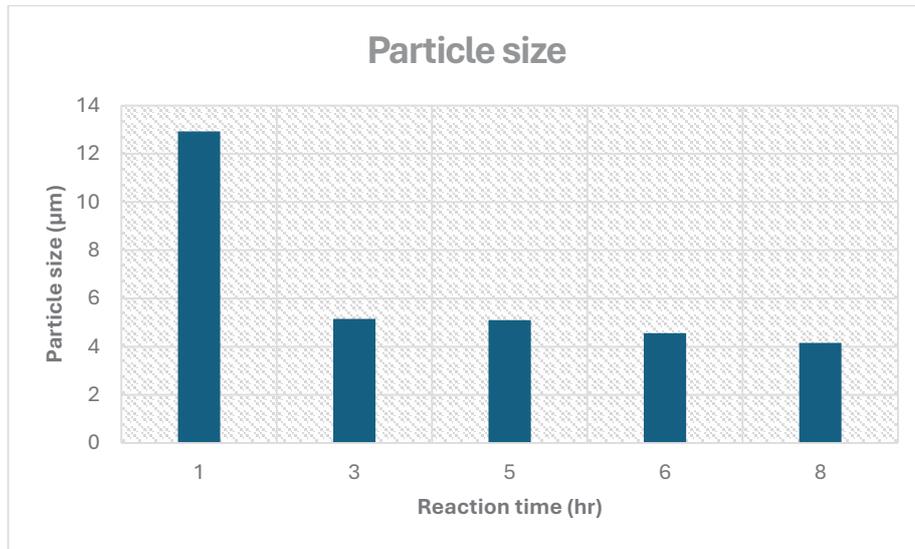


Figure 10. Particle size through changes in reaction time

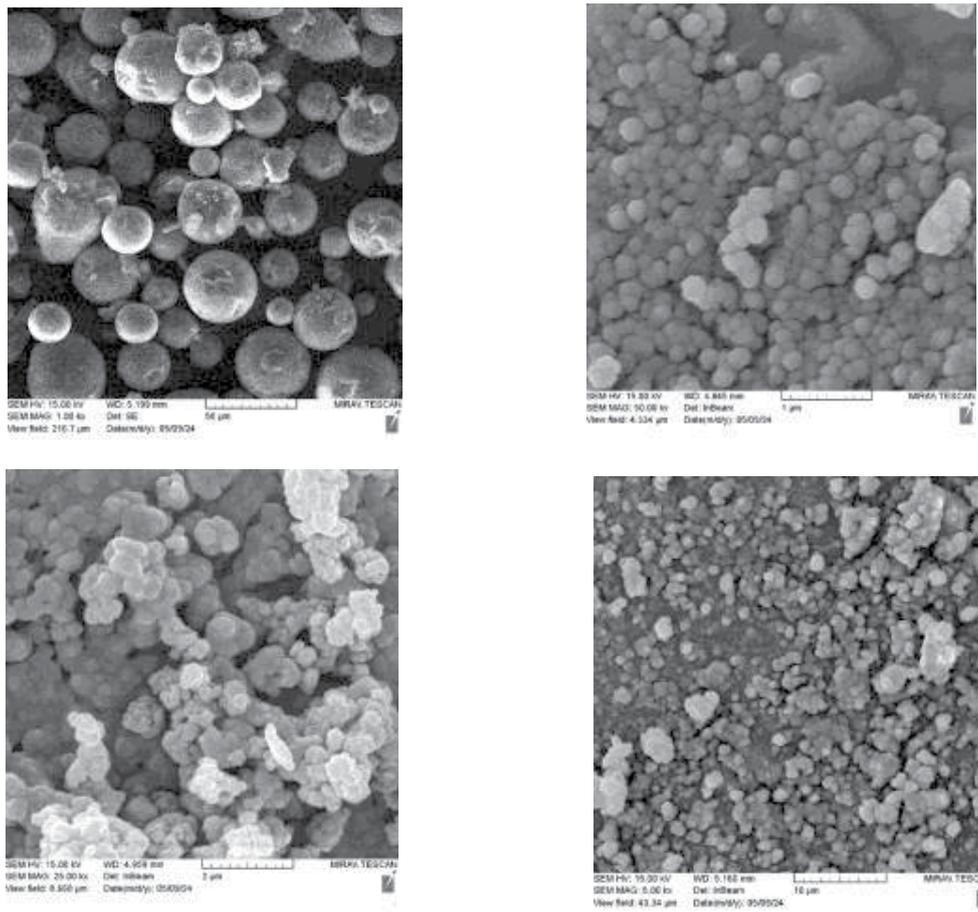


Figure 11. FESEM images of ORMOSIL particles prepared at different reaction times: (a) 1 hr (b) 3 hr (c) 5 hr (d) 8 hr (conditions for all experiments, temperature: 40 °C, CTAC: 0 g)

#### 4. CONCLUSION

The research reported here successfully introduced a one-pot low-temperature self-emulsification process to prepare monodisperse ORMOSIL particles (0.4-7.5  $\mu\text{m}$ ) with tunable size and morphology by the controlled regulation of CTAC surfactant concentration, temperature, and time through systematic optimization. The templating role of CTAC in regulating spherical growth was explained, and the calcination at 600°C successfully converted these particles into high-purity silica without disturbing structural homogeneity, as verified by FESEM and elemental analysis. The demonstrated ability of the particle feature control through facility control of the parameter modification indicates the applicability of the approach to numerous material engineering applications.

The main innovation of this research is the development of a simple, one-step, low-temperature synthesis method for the production of monodisperse ORMOSIL particles with tunable sizes (in the range 0.4-7.5  $\mu\text{m}$ ), through the simultaneous and methodical investigation of the three significant parameters: CTAC concentration, reaction temperature, and the reaction time. While previous studies have typically been aimed at optimizing one or two parameters, or have required multi-step synthesis procedures with limited scalability, this work presents a focused and scalable approach that enables precise control over particle size and morphology. Compared to earlier methods (e.g., seeded growth or two-step acid–base catalysis) this base-catalyzed pathway not only simplifies the process but also enhances the reproducibility, making it more suitable for industrial-scale applications in drug delivery, catalysis, and nanocomposites.

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## بهینه‌سازی سنتز سیلیکا به روش اشتوبر: بررسی اثرات CTAC، دما و زمان واکنش بر شکل و اندازه ذرات کروی

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