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Piezoelectric Droplet-Confined Synthesis of Size-Controlled Silica Nanoparticles in Inverse Emulsion Microreactors

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Abstract

A droplet-confined strategy for the synthesis of silica nanoparticles is presented using piezoelectric atomization coupled with an inverse emulsion system. A precursor solution comprising tetraethyl orthosilicate (TEOS), ethanol, water, and ammonium hydroxide was atomized into monodisperse microdroplets and introduced into a continuous oil phase containing light paraffin oil and Span 80. Each droplet functioned as an isolated microreactor, enabling controlled hydrolysis and condensation of TEOS under spatial confinement. Compared to conventional bulk Stöber synthesis, the droplet-based approach resulted in significantly reduced particle sizes and narrower size distributions. The confinement-induced increase in supersaturation promoted rapid and homogeneous nucleation, while the finite precursor content within droplets limited subsequent growth. The influence of droplet size, precursor composition, and surfactant stabilization on particle morphology was systematically analyzed. This method demonstrates a scalable route toward size-tunable silica nanoparticles governed by droplet physics rather than solely reaction kinetics, offering new insights into confined sol–gel processes.

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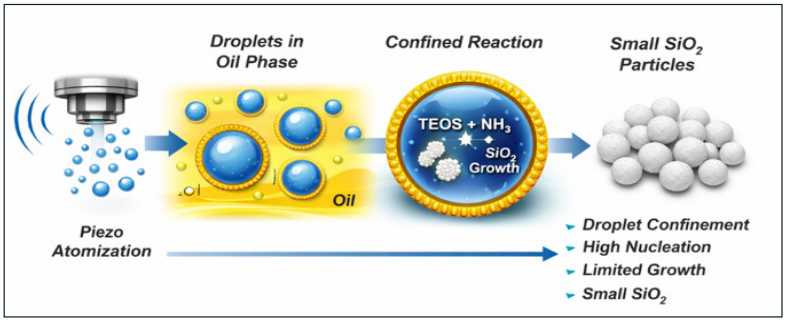

Introduction

Silica nanoparticles synthesized via the classical Stöber process have been extensively studied due to their tunable size, monodispersity, and broad applicability in catalysis, photonics, and biomedical systems. However, conventional bulk synthesis often suffers from limitations such as secondary nucleation, broad particle size distributions, and sensitivity to reaction parameters including solvent composition, ammonia concentration, and temperature [1,2,3]. These challenges arise primarily from the homogeneous and continuous nature of the reaction medium, where nucleation and growth occur simultaneously and are difficult to decouple [4].

Recent advances in microreactor and droplet-based synthesis have provided alternative routes to overcome these limitations by spatially confining reactions within discrete volumes [5,6]. In particular, water-in-oil (W/O) emulsions stabilized by low hydrophilic–lipophilic balance (HLB) surfactants, such as Span 80, offer a platform for compartmentalized sol-gel chemistry [7]. Within such systems, each droplet can act as an independent nanoreactor, enabling precise control over nucleation and growth processes through volume confinement and restricted mass transfer [8].

Piezoelectric atomization presents a versatile method for generating uniform microdroplets with tunable size distributions. Unlike conventional emulsification techniques, piezo-driven droplet formation provides control over droplet size via frequency and voltage modulation, allowing direct coupling between droplet physics and reaction outcomes. Despite its potential, the integration of piezoelectric atomization with inverse emulsion systems for silica nanoparticle synthesis remains underexplored.

Herein, we report a droplet-confined sol–gel approach wherein a TEOS-based precursor solution is atomized into microdroplets and introduced into a Span 80-stabilized paraffin oil phase. The resulting system enables the formation of silica nanoparticles under conditions of spatial confinement, leading to enhanced control over particle size and distribution. The role of droplet size, interfacial stabilization, and precursor composition in governing nucleation and growth is systematically investigated, providing insights into the interplay between fluid dynamics and sol-gel chemistry.

Experimental Section

Materials

Tetraethyl orthosilicate (TEOS), ammonium hydroxide (25–28 wt%), absolute ethanol, and light paraffin oil were used as received. Span 80 (sorbitan monooleate) was employed as the surfactant for stabilizing water-in-oil emulsions. Ultrapure water (18.2 MΩ·cm) was used in all experiments.

Preparation of Precursor Solution

A homogeneous precursor solution was prepared by mixing 10 ml ethanol, 5ml water, and 1ml ammonium hydroxide under stirring. TEOS was then added dropwise to achieve a final concentration of 1mM. The solution was stirred for 5 min prior to atomization to ensure uniform mixing while minimizing premature hydrolysis.

Piezoelectric Droplet Generation

The precursor solution was delivered to a piezoelectric atomizer equipped with a PZT transducer operating at a frequency of 40 kHz. A sinusoidal voltage (5V) was applied using a function generator and amplifier system. The precursor was atomized into microdroplets with diameters in the range of approximately 10–50 μm.

Inverse Emulsion-Based Synthesis

The generated droplets were introduced into a continuous oil phase consisting of light paraffin oil containing Span 80 (1-0.1 wt%). Upon into the oil phase, the droplets formed stable water-in-oil microreactors. Hydrolysis and condensation of TEOS proceeded within these confined droplets under ambient conditions. The reaction mixture was maintained under gentle stirring to prevent droplet coalescence while ensuring uniform dispersion. The system was allowed to react for 2 h to ensure complete silica formation.

Recovery of Silica Nanoparticles

Following synthesis, the emulsion was destabilized via catastrophic phase inversion by the addition of ethanol and excess water. The mixture was centrifuged at 5000 rpm for 15min to separate the aqueous phase containing silica nanoparticles. The particles were washed repeatedly for 5 times with ethanol and water to remove residual surfactant and oil.

Characterization

Particle morphology and size distribution were analyzed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Dynamic light scattering (DLS) measurements were performed to assess hydrodynamic size distributions.

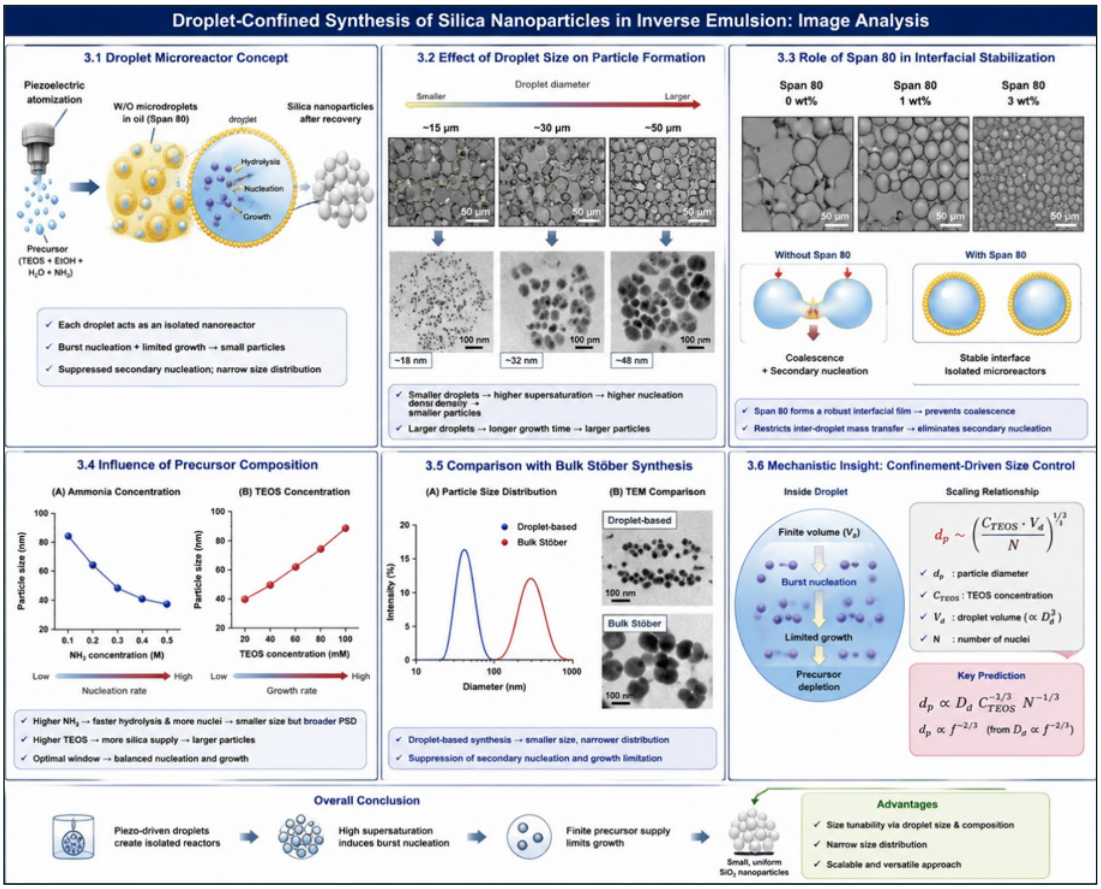

Figure 1: Mechanism explanation.

Results and Discussion

Droplet-Confined Nucleation and Growth

The piezoelectric atomization process generated microdroplets that served as isolated reaction compartments within the oil phase. Unlike bulk systems, where nucleation and growth occur continuously, the confined environment of each droplet imposed a finite limit on reactant availability. This resulted in a burst nucleation event followed by restricted growth, leading to the formation of smaller silica nanoparticles.

Effect of Droplet Size on Particle Formation

Droplet size, governed by the atomization frequency and applied voltage, was found to play a critical role in determining particle size. Smaller droplets exhibited higher supersaturation levels due to rapid hydrolysis and reduced diffusion length scales, resulting in increased nucleation density and smaller final particle sizes. Conversely, larger droplets allowed for extended growth periods, yielding comparatively larger particles.

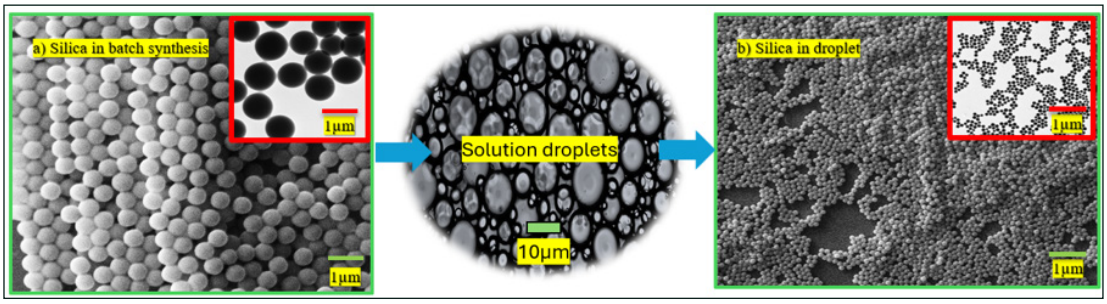

Figure 2: Comparative SEM & TEM analysis of the silica nanoparticles synthesised in (a)batch and (b) droplet.

Role of Span 80 in Interfacial Stabilization

Span 80 stabilized the water-in-oil droplets by forming a robust interfacial film, preventing coalescence and maintaining droplet integrity throughout the reaction. This confinement restricted inter-droplet mass transfer, effectively eliminating secondary nucleation pathways commonly observed in bulk systems. Additionally, the interfacial layer influenced diffusion dynamics, potentially creating radial concentration gradients within droplets that further impacted growth behaviour.

Influence of Precursor Composition

The concentrations of TEOS and ammonium hydroxide significantly affected nucleation kinetics. Higher ammonia concentrations accelerated hydrolysis, increasing nucleation rates but occasionally leading to broader size distributions. In contrast, lower precursor concentrations favored controlled growth and improved monodispersity.

Comparison with Bulk Stöber Synthesis

Compared to conventional bulk synthesis, the droplet-based approach yielded significantly smaller and more uniform silica nanoparticles. This improvement is attributed to the suppression of secondary nucleation and the confinement-induced limitation of growth. The results highlight the importance of spatial confinement as a governing parameter in sol–gel nanoparticle synthesis.

Mechanistic Insight: Confinement-Driven Size Control

The observed size reduction can be rationalized by considering the balance between nucleation and growth under confined conditions. The limited volume of each droplet restricts the total number of TEOS molecules available for condensation, effectively capping particle growth. Simultaneously, rapid supersaturation promotes the formation of multiple nuclei, distributing the available precursor among a larger number of particles and further reducing their size.

Reaction Kinetics in Confined Droplets

Hydrolysis and Condensation

$$Si(OC_2H_5)_4 + 4H_2O \xrightarrow{NH_3} Si(OH)_4 + 4EtOH$$

$$Si(OH)_4 \rightarrow SiO_2 + 2H_2O$$

Supersaturation-driven nucleation

$$S = \frac{C}{C_{eq}}$$

- Smaller droplets → faster increase in S
- Leads to burst nucleation

Scaling Law for Particle Size

Core idea: mass-limited growth

Total silica per droplet:

$$m_{SiO_2} \propto C_{TEOS} \cdot V_d$$

If Nnuclei form:

$$d_p \sim \left(\frac{C_{TEOS} \cdot V_d}{N}\right)^{1/3}$$

Droplet volume scaling

$$V_d \sim D_d^3$$

So:

$$d_p \sim D_d \cdot \left(\frac{C_{TEOS}}{N}\right)^{1/3}$$

Final scaling relationship

$$d_p \propto D_d \cdot C_{TEOS}^{1/3} \cdot N^{-1/3}$$

3.9 Linking Droplet Physics to Particle Size

Droplet diameter vs frequency

For piezo atomization:

$$D_d \sim \left(\frac{8\pi\gamma}{\rho f^2}\right)^{1/3}$$

Substituting into particle size:

$$d_p \propto f^{-2/3}$$

Key prediction

- Higher frequency → smaller droplets → smaller nanoparticles

Dimensionless Analysis

Ohnesorge number (droplet stability)

$$Oh = \frac{\mu}{\sqrt{\rho\gamma D_d}}$$

- Controls droplet formation regime
- Higher Oh → stable droplets, fewer satellites

Weber number (droplet breakup)

$$We = \frac{\rho v^2 D_d}{\gamma}$$

- Determines droplet deformation in oil phase
- Low We → stable spherical droplets

Damköhler number (reaction vs diffusion)

$$Da = \frac{k_{reaction}}{D/L^2}$$

- $Da > 1$: reaction-controlled (fast nucleation)
- $Da < 1$: diffusion-controlled growth

Your system operates near $Da \sim 1–10$ → optimal for uniform nucleation

Conclusion

The integration of piezoelectric atomization with inverse emulsion chemistry provides a powerful platform for the controlled synthesis of silica nanoparticles. By leveraging droplet confinement, this approach enables precise control over particle size and distribution, offering a scalable and tunable alternative to conventional sol–gel methods.

No conflict of interest

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