# Synthesis of Various Silica Nanoparticles for Foam Stability

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## 1. Introduction

The synthesis of monodispersive silica nanoparticles with various sizes, shapes, and structures is a major challenge in advanced materials science owing to their many emerging applications such as catalytic supports or as highly efficient packing materials for highresolution separations in high-performance liquid chromatography (HPLC) and electro-chromatography [1]. Although the synthesis of the non-porous silica nanoparticles with uniform sizes has been reported through the Stöber method [2], the synthesis of mesoporous silica nanoparticles with a specific morphology such as core-shell, rod-like, and hexagonal shapes is not so common. As a synthetic strategy for controlling the particle size, shape, and porosity, the synthesis of core-shell silicas with mesoporous shells formed on silica particle cores through the self-assembly of silica precursor and organic templates or spherical mesoporous silicas using modified Stöber method was also reported [3,4].

Recently, in an effort to reduce the amount of radioactive waste and enhance the decontamination efficiency during the decontamination process of nuclear facilities contaminated with radionuclides, a few research for the preparation of the decontamination foam containing solid nanoparticles has been reported [5].

In this work, the silica nanoparticles with various sizes, shapes, and structures were synthesized based on the previous literatures. The resulting silica nanoparticles were used to investigate the effect of the nanoparticles on the foam stability. In a study on the foam stability using various silica nanoparticles, the results showed that the foam volume and liquid volume in foam was enhanced when using a smaller size and lower density of the silica nanoparticles.

### 2. Methods and Results

2.1 Synthesis of the  $SiO_2$  NPs with various sizes, shapes, and structures: non-porous, mesoporous core-shell, and mesoporous NPs

The monodispersive  $SiO_2$  NPs were synthesized according to the previous literatures. In the typical procedures, the  $SiO_2$  seed NPs were synthesized adding the  $SiO_2$  precursor (TEOS) into the reaction mixture consisting of absolute ethanol, deionized water, and aqueous ammonia, and stirring for 24 h at ambient temperature. The SiO<sub>2</sub> seed solution (5 ml) was then suspended in a mixture solution of absolute ethanol (500 ml), deionized water (40 ml), and 28 wt.% aqueous ammonia (20 ml). After stirring for 30 min, the TEOS (10 ml) was added into the above reaction mixture and the mixture was stirred for 6 h under the same condition. Finally, the TEOS (65 ml) was added again and stirred for 6 h more.

To synthesize the mesoporous core-shell SiO<sub>2</sub> NPs, 10 ml of the suspended silica NPs in a mother liquor was dispersed in 20 ml of the deionized water containing 0.1 ml of 28 wt.% aqueous ammonia. The surfactant solution (CTABr, 0.24 g) dissolved in a 2:1 mixture of deionized water and ethanol was added into the reaction mixture solution. After stirring for 1 h, the TEOS (0.43 ml) was added to the above reaction mixture and stirred for 10 min more, and the reaction mixture was then aged for 15 h at 343 K. The assynthesized samples were retrieved by centrifugation, and then dried at 343 K overnight. The as-synthesized samples were calcined at 823 K for 5 h under an air atmosphere.



Fig. 1. Representative SEM and TEM images of (a) non-porous silica NPs and (b) mesoporous core-shell silica NPs.

To synthesize mesoporous silica NPs with various sizes and shapes, a mixture solution consisting of CTABr (0.06-0.12 g), NH<sub>4</sub>OH (0.25-2.0 ml), and DI  $H_2O$  (30 ml) was vigorously stirred for 30 min until reaching a clear solution. The TEOS (0.43 ml) was then added into the above mixture solution and stirred for 60 min. The reaction mixture was then aged at 343 K overnight. The as-synthesized samples were retrieved by centrifugation, and dried at 343 K overnight. The as-synthesized samples were calcined at 823 K for 5 h under an air atmosphere.



Fig. 2. Representative TEM images of the mesoporous silica NPs with different sizes synthesized by varying the amount of ammonia solution.  $NH_4OH/CTABr = (a) 0.25 \text{ ml}/0.08 \text{ g}$  and (b) 1.00 ml/0.08 g, respectively.

#### 2.2 SEM and TEM images analysis

Fig. 1 shows the representative SEM image of the non-porous silica NPs with a size of ca. 150 nm in diameter and a TEM image of the mesoporous coreshell silica NPs with a size of ca. 185 nm in diameter and a mesoporous shell of ca. 21 nm in thickness, respectively.

Low- and high-magnification TEM images of the mesoporous silica NPs are shown in Fig. 2. The pore structures of the mesoporous silica NPs exhibited the existence of an ordered hexagonal array from the result of the TEM analysis. Also, the resulting materials showed the spherical (Fig. 2a) and hexagonal (Fig. 2b) shapes by varying the amount of the ammonia solution. The particle sizes of the mesoporous silicas increased with an increase in the amount of the ammonia solution and surfactant.

# 2.3 Preparation of the foam and investigation of the foam stability using various $SiO_2$ NPs

In the preparation of the foam, a decyl glucoside (Elotant<sup>TM</sup> Milcoside 440N; EM 440N) supplied by LG Household & Health Care, which contains an average of 10 alkyl chains was used as a surfactant. Various silica NPs (1 wt.%) synthesized in this work were added to the EM 440N surfactant solution (1 wt.%) to investigate the effect of silica NPs on the foam stability. The foaming properties (i.e., foam formation and foam stability) were determined using a Foamscan instrument (Teclis/IT Concept, Logessaigne, France) to determine the foam stability (i.e., the decay of foam volume as a function of time) and foam drainage (i.e., the change of the liquid volume in the foam as a function of time).

From the results of the investigation of the foam stability, the foam volume and liquid volume in the addition of various silica NPs was higher than that with only EM 440N surfactant only. Also, the use of the silica NPs with a smaller size and lower density in the preparation of the foam showed an enhancement of the foam stability (foam volume and liquid volume in the foam) owing to their structural difference such as high specific surface area, large pore volume, and porosity.

#### **3.** Conclusions

Silica nanoparticles with various sizes, shapes, and structures such as a non-porous, mesoporous core-shell, and mesoporous silica were synthesized to investigate the effect of the foam stability. The sizes and structural properties of the silica nanoparticles were easily controlled by varying the amount of silica precursor, surfactant, and ammonia solution as a basic catalyst. The foam prepared using various silica nanoparticles showed that foam the volume and liquid volume in the foam were enhanced when using a smaller size and lower density of the silica nanoparticles.

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