

Research Article

Synthesis of High Surface Area Mesoporous Silica Powder Using Anionic Surfactant

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Over the past several years, synthesized mesoporous nanosilica (SiO_2) powders have been developed by many different methods. The methods include precipitation and microemulsion, the Stöber method, chemistry, and technology. In this study, mesoporous silica powders are synthesized by the sol-gel method. The sol-gel method obtains a high purity silica powder; however, the process yields a low percentage. Past syntheses of SiO_2 powder precursors are expensive, but this study needs to find a replacement precursor for low cost alternatives. A high surface area was used to form an anion surfactant sodium dodecyl sulfate, which regulates the molar concentration. The particles size variability was changed by the precursor molar ratio of the sodium silicate solution with hydrochloric acid.

1. Introduction

The silica powders are considered competent materials because of their unique characteristics, such as low density, low thermal conductivity, high surface area, high thermal shock resistance, and high specific strength [1, 2]. They also have the potential to be used in a wide range of applications, including catalysts, thermal and electrical insulators, adsorbents, filters, light-weight structural materials, optoelectronic devices, humidity sensors, chemical polishing, and other fine precision equipments [3–6]. According to recent academic and technical reports, various chemical methods have been developed to control the particle shapes, size, and distribution of fine silica particles. In the conventional method, ammonia-catalyzed reactions of tetraethylorthosilicate with water in low-molecular-weight alcohols are synthesized by Stöber method. However, this method has to use a high cost alkoxide or organometallic compound. In this work, we demonstrate a sol-gel method that controls the sphere size, cubic shapes, and mesoporous silica using the low cost sodium silicate solution with surfactant sodium dodecyl sulfate (SDS). The pore size and surface area of the silica varied with pH and aging time.

The characteristics of the silica varied with the change in surface area, pore volume, and particle shapes.

2. Experimental

2.1. Synthesis. The flow chart in Figure 1 shows the experimental steps involved in the rapid processing of cubic silica powders. In a study on the typical synthesis, the water glass solution was prepared in a beaker and the 1 M HCl solution dripped for 30 minutes. The mixture was stirred for 10 hours with a magnetic bar at 400 rpm using a churner [7]. The water glass solution was diluted with the desired weight percentage of silica in the starting material then washed in running water using a filter paper. In the washing process, our group used at least 5 L of deionized water to remove Na^+ ions from the water glass solution, resulting in the formation of silicic acid and got a high purity silica powder. [8] In the next step, the displaced pore water was then removed from the beaker and the organic-gels were dried at ambient pressure in hot air drying equipment at 80°C . This far explained the fundamental synthesis of silica powder. Figure 2 shows the second method of the high surface area of nanoparticle silica.

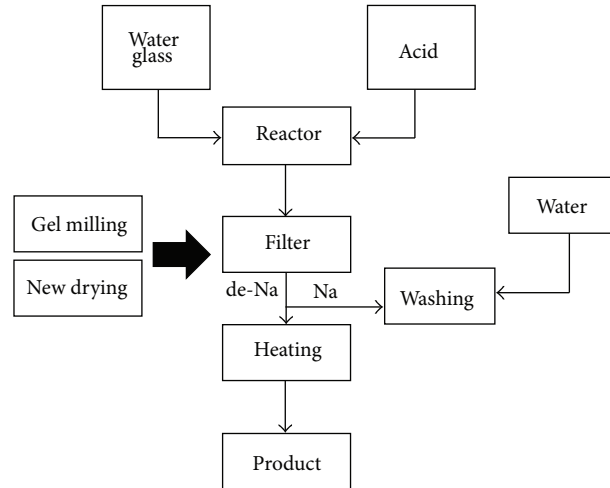


FIGURE 1: Flow chart showing the processing steps for the cubic silica powder.

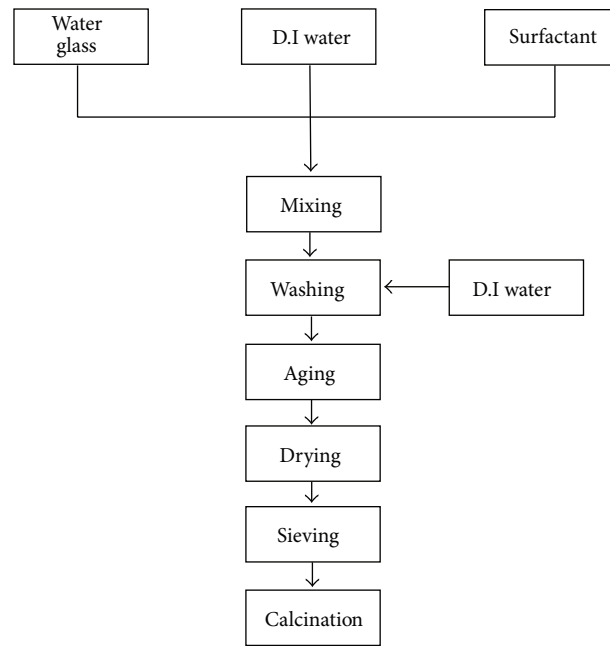


FIGURE 2: Flow chart showing the processing steps for high specific surface area of nanosilica power and spherical shape particles.

The high surface area silica powder manufactured for the experimental methods was based on experimental methods used for pH adjustment and surfactant. The reaction mixtures for the synthesis of high surface silica powder were prepared in beakers. pH 1 to 6 solutions were prepared by adding deionized water dropwise by a pipet. The other synthesis was performed with different proportions of reagents, surfactants, and pH, which are summarized in Table 1. Sodium dodecyl sulfate (SDS) was dissolved by stirring in a mixture of deionized water and concentrated hydrochloric acid (HCl). The stirring was stopped when the mixture was homogenized. The mixture was aged at 50°C in hot air drying equipment under static conditions for 24 hours. Precipitated gelation was filtered off, washed with 5 L deionized water, and dried at

TABLE 1: pH reaction condition (pH 1~pH 6).

Number	WG	SDS	Water	HCl	pH
1	1	0.03	20	0.2	1
2	1	0.03	20	0.2	2
3	1	0.03	20	0.2	3
4	1	0.03	20	0.2	4
5	1	0.03	20	0.2	5
6	1	0.03	20	0.2	6

80°C for 10 hours. The surfactant was removed by calcination at 700°C for 10 hours [9].

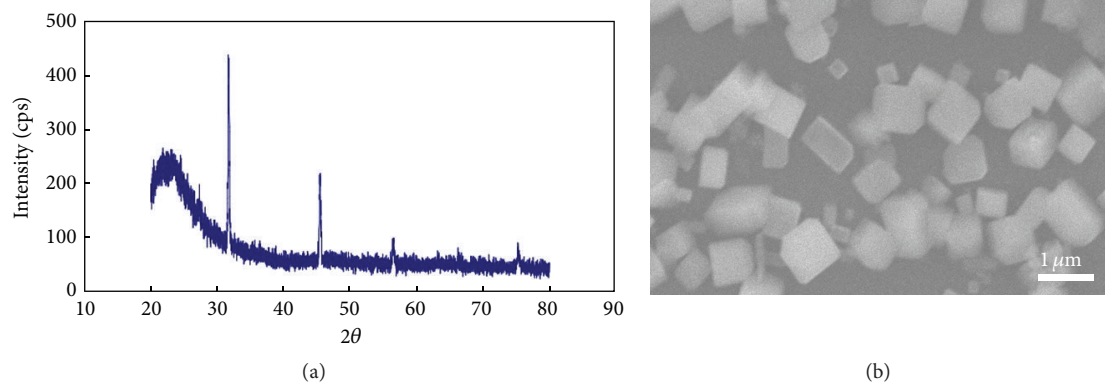


FIGURE 3: Centrifuges, hot air drying machine, XRD graph, and SEM image.

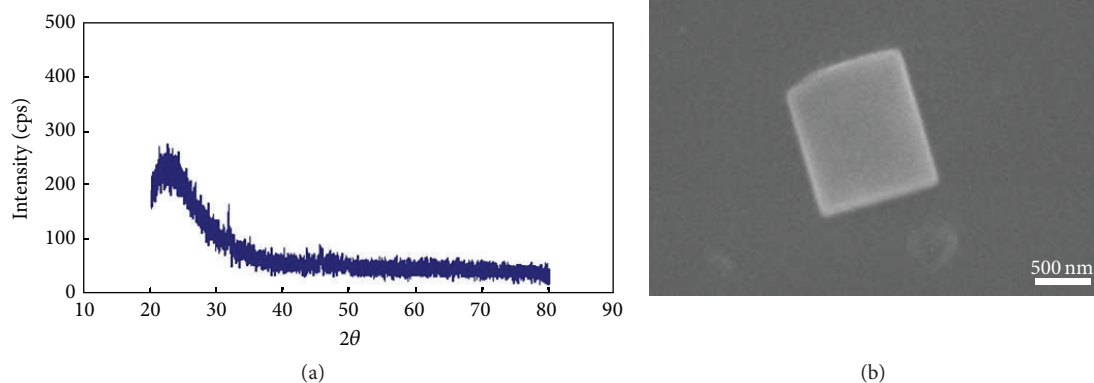


FIGURE 4: Filter paper washed and hot air drying machine.

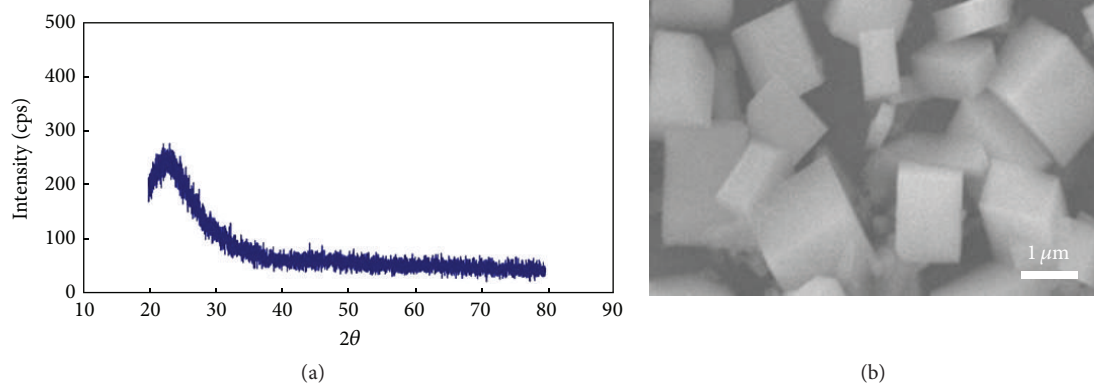


FIGURE 5: Filter paper and drying using a microwave.

2.2. Characterization. Field emission scanning electron microscopy (FE-SEM) was performed with a JEOL 6700F instrument to observe the particle morphology and size. Samples were gold-coated for SEM using a PS-2 coating unit. The crystal structure of the silica powder was determined by X-ray diffraction (XRD; Cu target; $K\alpha$; Philips DY616) and

the operation parameters were scanned in a range of $2\theta = 20\text{--}80^\circ$ and 40 kV. The surfactant effect of the silica powders was investigated by analyzing the surface area properties with standard N_2 gas adsorption using a surface area analyzer. The silica powders were first degassed at 250°C for 3 hours and the adsorption/desorption of the liquid nitrogen was

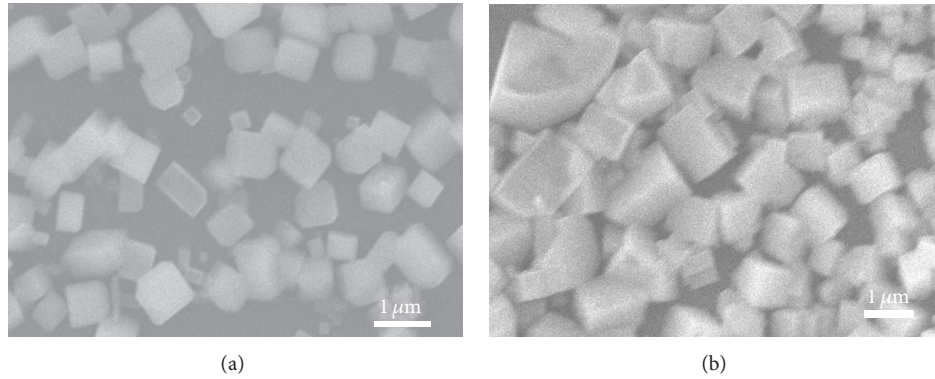


FIGURE 6: SEM image of cubic particles.

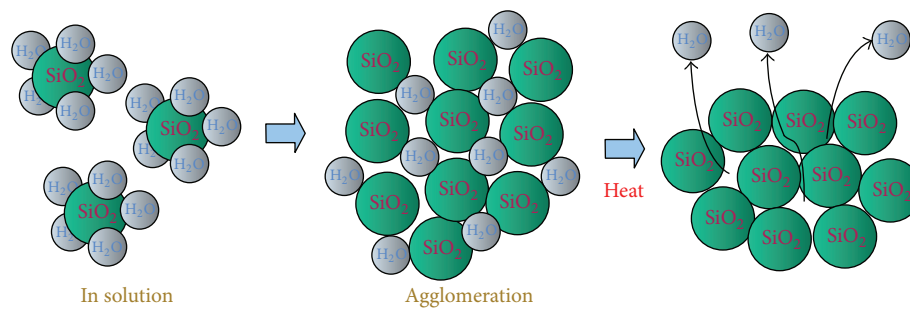


FIGURE 7: Increasing agglomeration of particle size in reverse proportion.

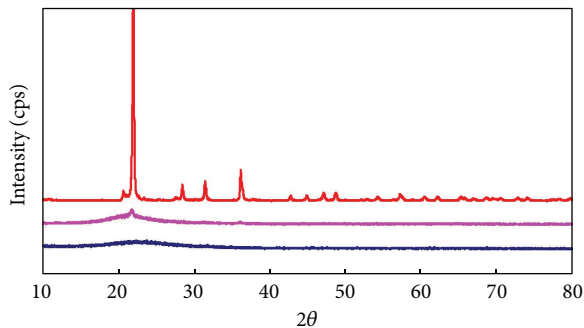
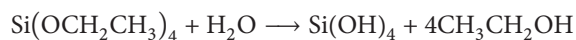


FIGURE 8: Effect of calcination.

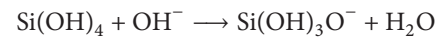
recorded using equilibration times at approximately 5 min per point [10]. The specific surface area of the nanosilica powder was calculated by the Brunauer-Emmett-Teller (BET) method.

3. Results and Discussion

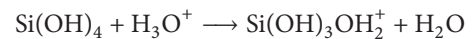
The basic chemical reactions of silica powder synthesis using a sodium silicate silica composite are



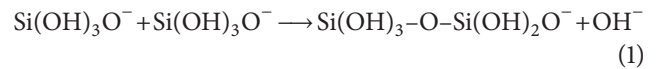
High pH range (alkaline):



Low pH range (acidic):



Polymerization:



For the synthesis of the silica powder, the remaining Na^+ in sodium silicate was removed using sodium silica in the sol-gel process. [11, 12] In the silica sol, the Na^+ ion was breaking the silica network structures as a modifier during silica sol-gel gelation and decreasing the purity of silica powders. To remove the sodium from sodium silicate, sodium silicate was diluted to 10 wt% HCl acid. Silica hydrogel was made by the sol-gel reaction after the sodium ions were removed by washing and the sodium crystallization was precipitated by centrifugation. [13, 14] There are two washing methods: deionized water washed on filter paper and a centrifugation method. Compared to the XRD data, the filter paper was better for removal of Na^+ ions to produce a more high-purity silica powder product; however, the main drawback is the loss of silica. [15–17] The shape of the cubic powder silica was synthesized with 1 M HCl 200 mL in the water glass solution by sol-gel. The silica powder product from each of

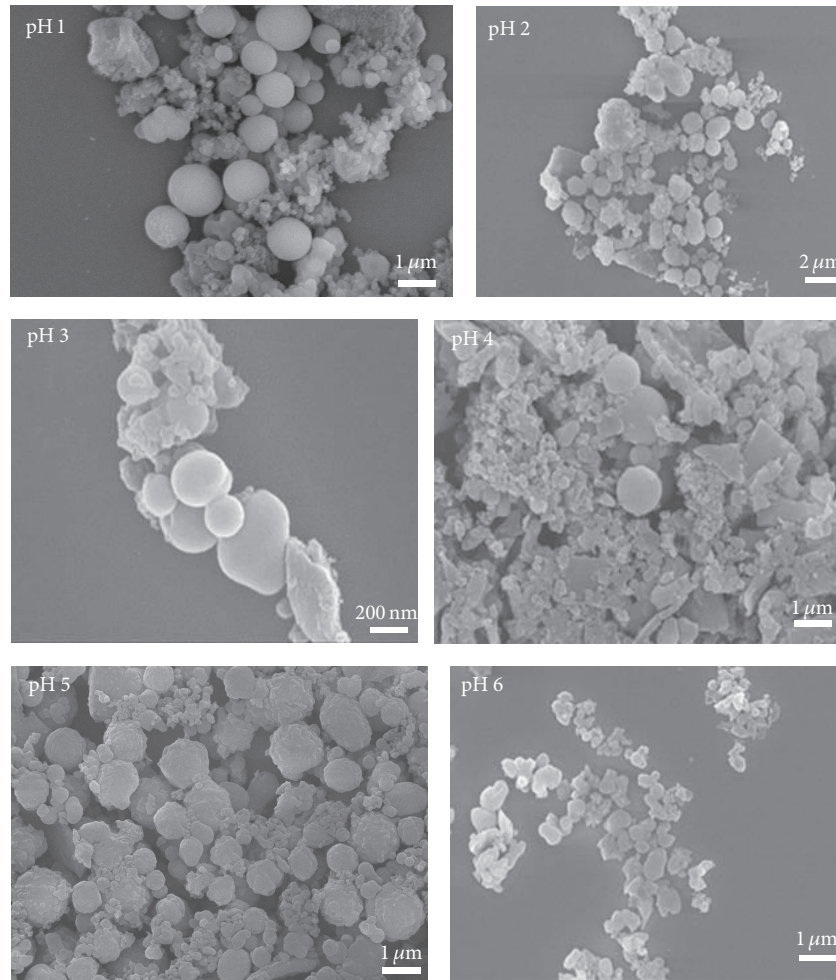


FIGURE 9: pH effect of particles shapes.

the washing and drying methods were confirmed by XRD and the morphology of the particles was examined using scanning electron microscopy (SEM) that is shown in Figures 3, 4, and 5.

In Figure 6, the SEM images of each particle confirm the cubic was approximately 500 nm to 1 μm in diameter. Microwave drying could produce more aggregation than that shown in Figure 7, increasing the particle size in reverse proportion to the specific surface area that was smaller than the BET data and could not be confirmed. As shown in the XRD data results (Figure 8), the transition to full cristobalite occurred at 1200°C. The pH change of the silica composite with reference to Figure 9 was performed. Figure 9 shows the change in the pH 1 to 6 ranges of various particle morphology images observed by SEM. Lower pH values produced particle shapes with more spherical shapes; however, the BET specific surface area data of the particles did not differ with the pH change (Table 2).

4. Conclusions

The nanostructured silica powder particles were obtained over a range of 500 nm to 1 μm particle sizes, with high

TABLE 2: Effect of pH specific surface area (m^2/g).

pH	Specific surface area (m^2/g)
1	857.84
2	1403.24
3	1370.02
4	984.14
5	630.10

specific surface area and cubic and spherical shapes by changing the surfactant pH and drying methods with sol-gel process. The silica powders were characterized in terms of the silica content, specific surface area, particle size, particle shape, and pore size distribution. The synthesis of a 10% water glass solution with 1 M HCl created a mixture reaction that effectively produced cubic particles. Increasing deionized water and HCl molar ratios created smaller size particles. The obtained silica nanoparticle showed high specific surface area.

Conflict of Interests

The authors declare that they have no conflict of interests.

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