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Impact of Varying the Concentration of Tetraethyl-OrthoSilicate on the Average Particle Diameter of Monodisperse Colloidal Silica Spheres

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Abstract

In this study, spherical Silica particles with narrow size distribution were prepared by hydrolysis of Tetraethyl OrthoSilicate (TEOS) in Ethanol and the presence of ammonia based on the Stober method. The average particle diameter and monodispersity were controlled by varying the concentration of TEOS. Microscopic analysis showed that a smaller average particle diameter and good degree of monodispersity can be obtained by decreasing the concentration of TEOS.

Keywords: Silica particles; Size distribution; Average particle diameter

Introduction

Substantial recognition has been attracted by monodisperse inorganic nanoparticles because of their innumerable utilization in the fields of polymer technology, optical devises, catalysis, and so on [1]. The nontoxic nature of Silica and the fact that it can be readily improved with several functional groups makes it useful in biomedical field [1]. For example, modification on Silica surface can be made to allow effective cellular movement, enabling drug delivery [2]. It can also boost the particles' ability to be attached electro-statically to DNA, enabling it to be further applied in drug delivery techniques [2]. In addition, Silica particles have been used to boost the mechanical properties of rubbers and plastics as additives [3]. The key properties that must be controlled in order to obtain Silica nanoparticles with more improved properties are the particles' morphology, size distribution, and phase composition [3].

Several methods have been used to prepare Silica nanoparticles with narrow size distributions. Some of which includes: colloidal and surfactant template, water-in-oil microemulsion [4,5], the continuous microwave hydrothermal synthesis techniques [6,7], stirred bead milling [8], synthesis via 'fumed' Silica nanoparticles [9], reverse micelle and sol-gel processing [10-12]. These techniques can yield nanoparticles which are small in size and have narrow size. One of the most widely used techniques in Silica nanoparticle preparation is the relatively simple Stober method. The Stober method is the most widely used in the preparation of large size Silica particles [13]. However, getting reliable results in the preparation of small size Silica nanoparticles using this method is usually difficult [13].

In this study, we prepared large and small sized Silica particles by controlling the concentration TEOS using the Kolber and Stober method.

Materials and Method

Materials

Tetraethyl orthoSilicate (TEOS 98%), ammonia, absolute ethanol, de-ionized water.

Synthesis of mono-dispersed silica colloidal particles

The Colloidal Silica sphere was synthesized as described by Stober [13].

In a typical synthesis, a solution containing suitable quantities of aqueous ammonia, absolute ethanol and de-ionized water were stirred for 5 min. Thereafter, an appropriate concentration TEOS in absolute ethanol was added to the above mixture. The full solution was stirred at room temperature for 10 hr. Thereafter, the colloidal solution was separated by high-speed centrifuge, and washed with ethanol for three times to remove undesirable particles [14].

Results and Discussion

Figure 1a illustrates the infrared absorption spectra of solid Silica particles with different intensity. The spectrum reveals intense absorption bands at 1065 cm^{-1}, 945 cm^{-1}, 793 cm^{-1} and 547 cm^{-1} wave numbers. These data indicate the formation of a Silica network [15]. The solid Silica sample showed bands emanating from asymmetric vibration of Si–OH (945 cm^{-1}), and symmetric vibration of Si–O (793 cm^{-1}). The absorption peak arising from 547 cm^{-1} can be assigned to rocking motion of oxygen atoms bridging silicon atoms in siloxane bonds (Si–O–Si). The huge absorption peak observed in the Silica spectrum is present at1065 cm^{-1} and is influenced by anti-symmetric motion of silicon atoms in siloxane bonds.

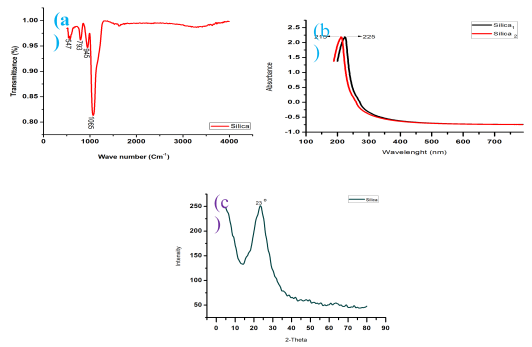

Figure 1: (a) Fourier transform infrared spectroscopy (b) UV-visible absorption spectra (c) XRD pattern of Silica.

Figure 1b shows the UV spectra of as-synthesized Silica 1 and 2 samples. They both showed characteristic maximum absorption near the ultraviolet region at about 210 nm and 225 nm respectively. A red shift of the excitation wavelength was however, observed for Silica 1 compared to Silica 2. This could be because of the observed increase in the average particle diameter of Silica1 compared to Silica 2, as a decrease in particle diameter had in times past been linked with elevation in band gap between the conduction and valence band. This therefore leads to an increase in the energy of excitation and hence a reduction of excitation wavelength [16,17].

Figure 1c shows the XRD patterns of the as-prepared Silica sample. The spectrum revealed a single broad diffraction peak at position 2θ equal to 23°. The typical broad peak indicates that the as-prepared Silica is amorphous with no crystalline phase [18-20]. This could be attributed to the incomplete inner structure of the Silica particles [18].

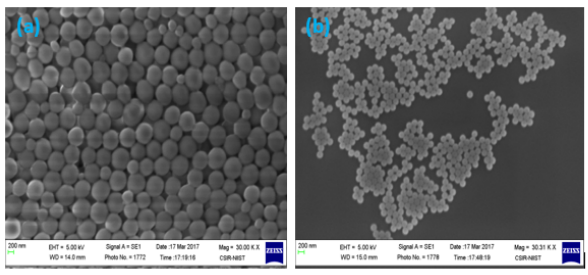

Figure 2: Scanning electron micrographs of (a) Silica1 (b) Silica2.

Silica samples were prepared using two different TEOS concentration of 3 M and 0.8 M while ammonia and water concentrations are fixed at 1.4 M and 6 M respectively.

Figures 2a and 2b shows the scanning electron micrographs of the as-synthesized Silica particles. The SEM images showed that the obtained Silica samples have spherical shaped particles with narrow size distributions. However, Silica 2 shows a slightly wider range of size distribution compared to Silica 1 (Figure 2b). The average particle diameter of the Silica samples is 330 nm and 120 nm respectively. This clearly shows that the average particle diameter of Silica decreased with a decrease in concentration of TEOS. This may be explained as follows: A decrease in the concentration of TEOS from 4 M to 0.8 M leads to a slower rate of hydrolysis and condensation [21]. This in turn decreases the rate of formation of the intermediate. The comparatively slow rate of consumption of the intermediate via condensation reaction at super-saturation point may have increased the nucleation phase [22] and as a result also decrease the final average particle diameter of the Silica particles.

Conclusion

In this study, the impact of TEOS on the resulting average particle diameter of Silica particles was looked upon. Experimental data shows that a reduction in the concentration of TEOS leads to a reduction in the average particle diameter of the as-synthesized Silica samples.

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