

Effect of synthesis time on yield and particle growth of silica nanosphere

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Abstract : The yield and particle growth of silica nanosphere were studied with respect to reaction time. It is found that the yield gives a half S type curve with reaction time. A bimodal particle size was observed through out the reaction time. With time, the minor particles were increased up to certain size. The other particles were stable as such. An addition of octadecyltrimethoxy silane was normalizing the particle size. In both the cases, the particle shape did not change with any time from nanosphere.

Keywords : Silica nanosphere, time, yield, particle size.

Introduction

There were several studies on silica nanosphere as they possess peculiar and desirable properties in the wafer polishing process and packing materials. The first silica nanosphere was synthesized by Stober *et al.* from sol-gel method¹. Later Bogush and Zukoski studied the influence of various reaction parameters on particles size distribution². Recently, different authors described the ways to control particle size by varying the reactor type and concentrations of ammonia, water and alcohol³⁻⁶. Kim *et al.* found that the effect of various parameters on the particle size and shape. The size with respect to various synthesis parameters was as follows, reaction temperature < [H₂O/tetraethylorthosilicate (TEOS)] < concentration of ammonia < feed rate of reactant³. They also found that the influence of various reaction methods such as semi-batch reaction and batch reaction on particle size and distribution. A relatively slow rate of hydrolysis for TEOS occurred in semi-batch process along with larger particles and narrow distribution.

Even though, there are various studies on silica nanoparticle synthesis, some of the important properties such as the effect of time on yield and particle size and shape were unattended. Beside, bimodal particle size is a common problem appeared in this synthesis. We have succeeded in normalizing the particle size by adding a promoter such as octadecyltrimethoxy silane.

Experimental

1.06 ml of tetraethylorthosilicate (98%, Aldrich, USA) is mixed with 5 ml of ethanol (98%, Aldrich, USA). To

this mixture 0.215 ml of aqueous ammonia (28–30%, Aldrich, USA) is added. Then 1.08 ml of water was added. Finally 5 ml of ethanol was added. The total mixture (0.5TEOS : 6H₂O : 0.7NH₄OH : 85EtOH at 298 K) is stirred continuously. Similar batches were carried out for different particle growth times from 0.25 to 48 h. Products were centrifuged, washed and dried at 363 K for 12 h.

The particle size and shape were analyzed by a Topcon, SM-300 scanning electron microscope. The copper disc pasted with carbon tape and the sample was dispersed over the tape. The disc was coated with gold in ionization chamber before microscopic analysis.

Results and discussion

The silica nanosphere yield with different time (in hours) intervals were given in Fig. 1. It follows a half S

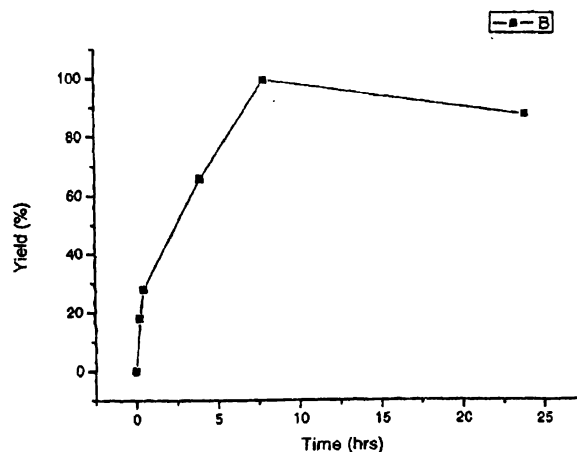


Fig. 1. Time (h) vs yield (%) plot.

type curve. It shows the reaction is first order reaction. The yields were calculated in comparison with the theoretical silica yield from the total silica input. Maximum yield occurs at 8 h.

Table 1. Yield with time

Reaction time (h)	100 nm (%)	300 nm (%)
0.25	92	8
0.50	83	17
1.00	80	20
2.00	78	22
4.00	72	28
8.00	53	47
16.00	42	58
2.00 (ODTS)	-	100 (400 nm)

The morphology and particle size were seen from the scanning electron micrograph of Fig. 2. At 0.25 h, fused spheres occur. Later (0.5 h) a clearly visible sphere with two different particle sizes occur. The lower particle size were in larger (100 nm, 80%) amount and higher par-

and smaller particles were increased up to (200 nm). Addition of octadecyltrimethoxy silane normalizes the particle size and increases the growth rate. Octadecyl group in trimethoxy silane molecule increase the electron density around the silane atom. So the basicity around oxide group was increased and the rate of reaction (particle growth) was increased. In general, tetraethylorthosilicate monomers were hydrolyzed to hydroxides. Hydrolyzed silicates were condensed to form polymeric silica. Ammonia is acting as catalyst^{7,8}.

Conclusions :

Silica nanosphere was prepared from tetraethylorthosilicate, water, aqueous ammonia, ethanol as constituents. The yield, particle size and shape were studied with respect to reaction time. Yield shows a half S type curve and reaches maximum at 8 h. A bimodal particle sizes viz. 100 and 300 nm were observed with spherical shape. The concentration of minor particles were decreased with time, however did not reach the maximum. The particles were normalized on addition of octadecyltrimethoxy silane.

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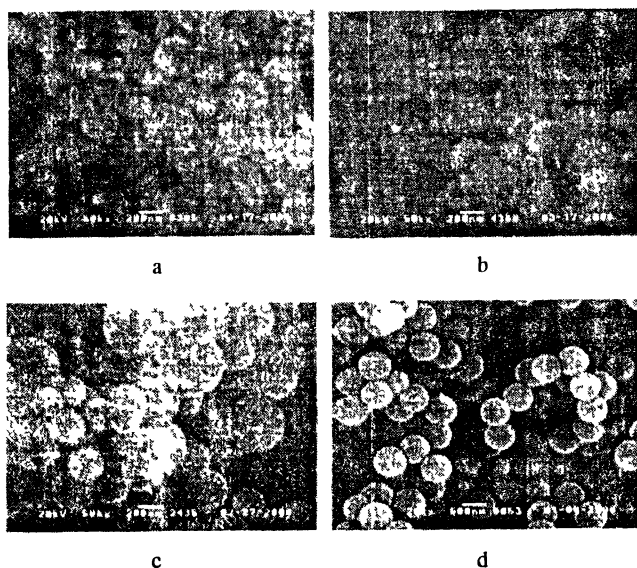


Fig. 2. Scanning electron micrograph of silica nanosphere preparation at (a) 0.5 h, (b) 2 h, (c) 8 h, (d) 2 h (with octadecyltrimethoxy silane).

ticles are lesser (300 nm, 20%). Then with increase in time, the particle size of bigger particles was constant