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Synthesis of silica powders by pressured carbonation[☆]

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ABSTRACT

A method was proposed for the preparation of silica powders using inexpensive material of sodium silicate (Na_2SiO_3) and carbon dioxide (CO_2) by pressured carbonation, in which carbon dioxide acted as a precipitating reagent. Microstructure and size analyses of the precipitated silica powders were carried out using transmission electron microscopy and dynamic light scattering. The average particle size, size distribution and yield of silica powders were affected by reaction time, temperature and concentrations of surfactant and sodium silicate solutions. The particle size of silica powders increased with reaction temperature and concentration of sodium silicate, and the yield of silica powders increased with increasing reaction time. The size distribution of silica powders was affected by concentration of surfactant PEG. The optimal preparation conditions were experimentally determined for obtaining the silica powders with nanometer size, narrow size distribution, spherical shape and high purity without sodium carbonate and surfactant.

1. Introduction

Ultrafine metal and oxide particles find wide applications because they exhibit novel physical and chemical properties that markedly differ from those of bulk materials [1–4].

Oxide powders which are well defined physically and chemically have many industrial applications. They are used for the production of a variety of technical ceramics, glasses and composites with special mechanical, electrical, magnetic or chemical properties [5–7].

Ultrafine silica (SiO_2) powders have considerable advantage for a wide range of applications including catalysts support, adsorbents, light-weight structural materials, humidity sensors, colloidal damper in the field of mechanical engineering, and other fine precision equipment [8–10].

Various methods have been reported for preparing silica materials, such as plasma synthesis [11], chemical vapor deposition [12], sol–gel processing [13,14], microemulsion processing [15], combustion synthesis [16,17] and hydrothermal technique.

Currently, fumed silica is used in the production of thermal insulation materials because of the low densities and high surface area. Thermal methods of making fumed silica involve the hydrolysis of silane compounds in an oxygen–hydrogen gas flame, but these methods can sometimes be undesirable because of the extremely high temperatures required for processing (approximately 2000 °C) [18], high risk of reaction process and large investment.

Sol–gel processing was examined in this work as a method of producing powders with controlled characteristics which could be matched to those of fumed silica. The sol–gel processing using metal alkoxides, such as tetraethyl orthosilicate (TEOS), has been widely used for the synthesis of silica based materials [19–21]. This technique for making silica particles involves hydrolysis and subsequent condensation of silicon alkoxide using short-chain alcohols as solvents. Strober et al. [19] prepared silica powders by hydrolysis of TEOS in a basic solution of ethanol and ammonium hydroxide and produced particles of diameters 0.2–1.5 μm. TEOS is hydrolysed according to the following general reaction:

$$Si(OR)_4 + xH_2O \rightarrow (HO)_xSi(OR)_{4-x} + xROH, \quad \text{where } R = CH_3CH_2$$

Silica powders produced by the above methods have good properties such as fine particle size, large surface area and good dispersibility. However, the main disadvantage of this technique is that an aging time of several hours (2–240 h) [22,23] is required for complete processing of particles and particle growth. Moreover, high production costs and heavy pollution have limited the large-scale

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commercial applications. Development of alternative cheaper and environmental-benign routes to produce ultrafine silica powders with desirable characteristics is therefore of considerable interest.

In recent years, one of the principal world concerns is the ‘greenhouse’ effect, especially related to carbon dioxide (CO_2). In the last 10 years, several papers have reported the utilization of CO_2 gas or supercritical (SC) CO_2 to synthesize silica powders. For example, Chattopadhyay and Gupta [18] prepared silica particles by bubbling supercritical CO_2 through a W/O microemulsion of an aqueous sodium silicate solution having a dispersion of silica nanoparticles. In this method, supercritical CO_2 acts both as an antisolvent and as a reactant. Zhang et al. [24] proposed a simple acid gelation route to synthesize silica particles using SC CO_2 as a special reactant. By adding SC CO_2 into the sodium silicate aqueous solutions, porous silica microflowers were produced. Hu et al. [25] prepared high reinforcing SiO_2 from gas mixture containing CO_2 and sodium silicate by CO_2 precipitation in a builder.

The classical industrial method for the preparation of silica powders generally employs sulphuric acid. In order to reduce the cost of inorganic acid and protect environment, clearly an alternative and more efficient route to such silica powders would be of considerable commercial interest. Recently, carbonation methods have been considered. Therefore, we can use carbon dioxide instead of organic [26] or inorganic acid to prepare silica powders, which is environmentally friendly and economical.

In the present investigation, we proposed the pressured carbonation technique for the synthesis of ultrafine silica powders. The silica powders were prepared by precipitation from sodium silicate using carbon dioxide as latent acid reagent.

$$2NaOH{\cdot}SiO_2 + CO_2 \rightarrow Na_2CO_3 + 2SiO_2\downarrow + H_2O \qquad (1)$$

The advantages of the proposed technique over the conventional ones for producing silica powders include inexpensive starting materials, environmental benign, high purity, comparatively narrow size distribution, spherical particle shape and large-scale production capability.

2. Experimental

2.1. Materials

All chemicals were analytical grade and used without further purification. Sodium silicate ($Na_2O{\cdot}SiO_2{\cdot}9H_2O$) and polyethylene glycol 6000 (PEG 6000) were obtained from Wuxi Chemical Co., Ltd. CO_2 was of chemical grade (at a purity of 99%). Ethanol (C_2H_5OH) and other materials were all analytical grade. In all experiments, deionized water was used.

2.2. Apparatus

A schematic representation of the apparatus used for the precipitation of ultrafine silica powders is shown in Fig. 1. The main component of the apparatus is a high-pressure reactor (R) approximately 40 cm^3 in volume. The pressure inside the reactor is generated by the high pressure of carbon dioxide in the cylinder (C). To maintain the constant temperature of the reactor, the temperature inside the precipitation reactor is controlled by a temperature controller (T). An aqueous sodium silicate solution (N) is injected inside the precipitation reactor via a solution injector (S). The outlet port (O) is located on the bottom of the precipitation reactor; carbon dioxide is fed into the reactor through a gas inlet port (G) located at the top of the reactor. The carbon dioxide and aqueous sodium silicate solution are blend by an agitator (A). Valve V3 is used to control the flow of carbon dioxide into the high-pressure reactor. The pressure inside the reactor is measured using a pressure gauge (P3). The pressure difference across carbon dioxide cylinders and the solution injector is measured using the pressure gauges P2 and P1.

2.3. Preparation of ultrafine silica powders

$Na_2O{\cdot}SiO_2{\cdot}9H_2O$ was used for the synthesis of ultrafine silica powders. Carbon dioxide and PEG 6000 were employed as a precipitating agent and nonionic dispersant, respectively. The sodium silicate was dissolved in 2000 mL deionized water to be water glass. PEG 6000 was added to water glass solution in a given mole ratio. The aqueous sodium silicate solution with an appropriate concentration was then loaded into the “solution injector” and fed into high-pressure reactor under stirring at a certain temperature. Carbon dioxide was injected into the reactor at a pressure of 0.2 MPa. After the reaction, the resulting slurry was kept aging for 2 h at 50 °C, and then was filtered using a pump. The precipitate was washed using deionized water and ethanol several times, and then dried in vacuum at 50 °C for 24 h.

2.4. Single factor test

The main affecting factors influencing the average particle size are concentration of Na_2SiO_3 and reaction temperature, and the major factors influencing the yield and particle size distribution are reaction time and concentration of surfactant PEG [27,28].

To find the effects of reaction time and temperature on the yield of production and average particle size, the reaction time was varied from 30 to 180 min and the temperature was varied from 60 to

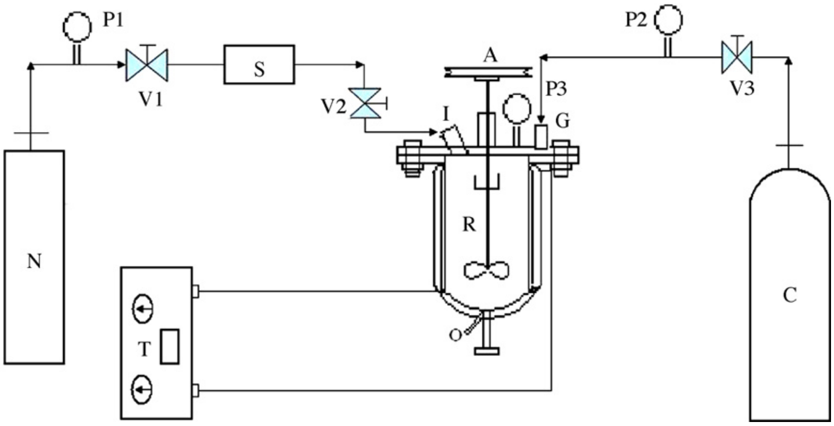


Fig. 1. Apparatus for the formation of ultrafine silica powders using carbon dioxide. (R, high-pressure reactor; C, carbon dioxide cylinders; T, temperature controller; I, solution inlet; O, production outlet; G, gas inlet; A, agitator; P1, P2, P3, pressure gauges; V1, V2, V3, control values; S, solution injection device)

90 °C while fixing other parameters. To find the effects of the PEG and Na_2SiO_3 concentration on the average particle size and size distribution, the reaction was performed at 70 °C for 1 h varying the concentrations at a range of 0.25–1.0 mol/L of Na_2SiO_3 and 0–15% of PEG while fixing other conditions.

2.5. Orthogonal tests ($L3^4$)

A group of orthogonal tests ($L3^4$) was arranged to investigate the effects of reaction temperature, reaction time, Na_2SiO_3 and PEG concentrations on the average particle size and yield of silica, while reaction pressure was set as constant 0.2 MPa, which is shown in Table 1. A series of experiments were carried out in sequence, as shown in Table 2. Finally, the average particle size and yield of samples 1–9 were obtained.

2.6. Characterization

Fourier transform infrared (FT-IR) spectra were obtained using Nicolet FT-IR Avatar 360 (Nicolet, USA) with the KBr method. X-ray diffraction (XRD) measurements were carried out with D/Max-IIIC (Rigaku, Japan), using Cu-Kα radiation. The sizes of silica powders were tested by a transmission electron microscope (TEM, Hitachi H-600-II, Japan). A Hitachi S-4700 scanning electron microscope (SEM) was used to study the morphology of the silica powders. Specific surface areas of silica powders were determined by N_2 adsorption (BET) using ASAP 2020M+C instrument (Micrometrics Instrument Co.). The size distribution of silica powders in water was obtained using Malvern HPPS5001 laser particle size analyzer. The element analysis of the powders was conducted using a Hitachi S-4700 scanning electron microscope appurtenance (SEM-EDS).

Table 1
Factors of orthogonal tests ($L3^4$).

No.	Temperature (°C)	Na_2SiO_3 (mol/L)	PEG (%)	Reaction time (h)	Pressure (MPa)
1	80	1	5	3	0.2
2	70	0.5	10	2	0.2
3	60	0.25	15	1	0.2

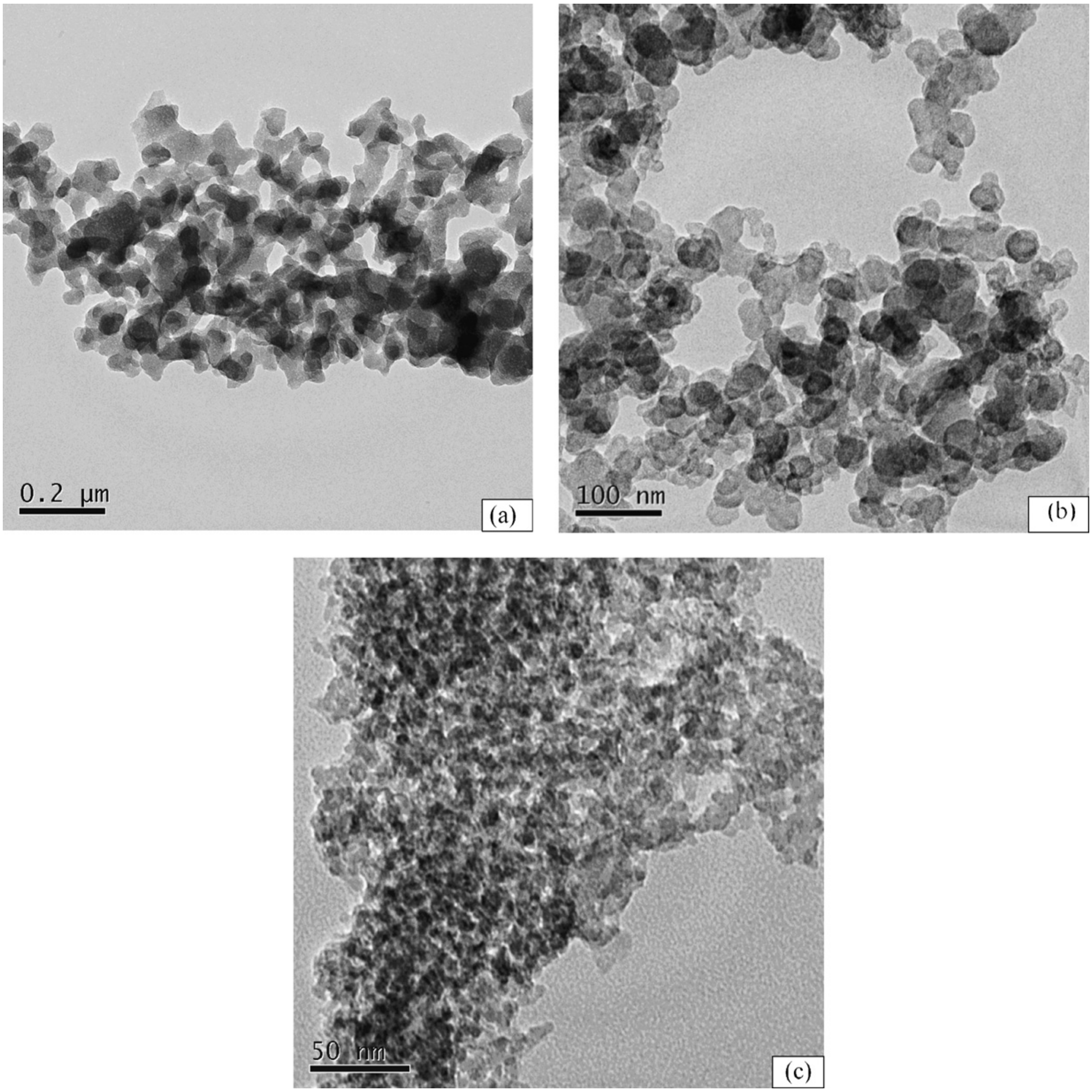


Fig. 2. TEM micrographs of silica powders prepared at 70 °C for 1 h 5% PEG with various concentrations of Na_2SiO_3: (a) 1.0 mol/L, (b) 0.5 mol/L, and (c) 0.25 mol/L.

Table 2
Results of orthogonal tests ($L3^4$).

No.	Temperature (°C)	Na_2SiO_3 (mol/L)	PEG (%)	Reaction time (h)	Average size (nm)	Yield (%)
1	80	1.0	5.0	3	54	86.4
2	70	1.0	10	2	52	84.5
3	60	1.0	15	1	46	75.2
4	80	0.50	15	2	36	84.7
5	70	0.50	5.0	1	33	82.3
6	60	0.50	10	3	36	88.6
7	80	0.25	10	1	19	85.3
8	70	0.25	15	3	17	86.3
9	60	0.25	5.0	2	15	86.8

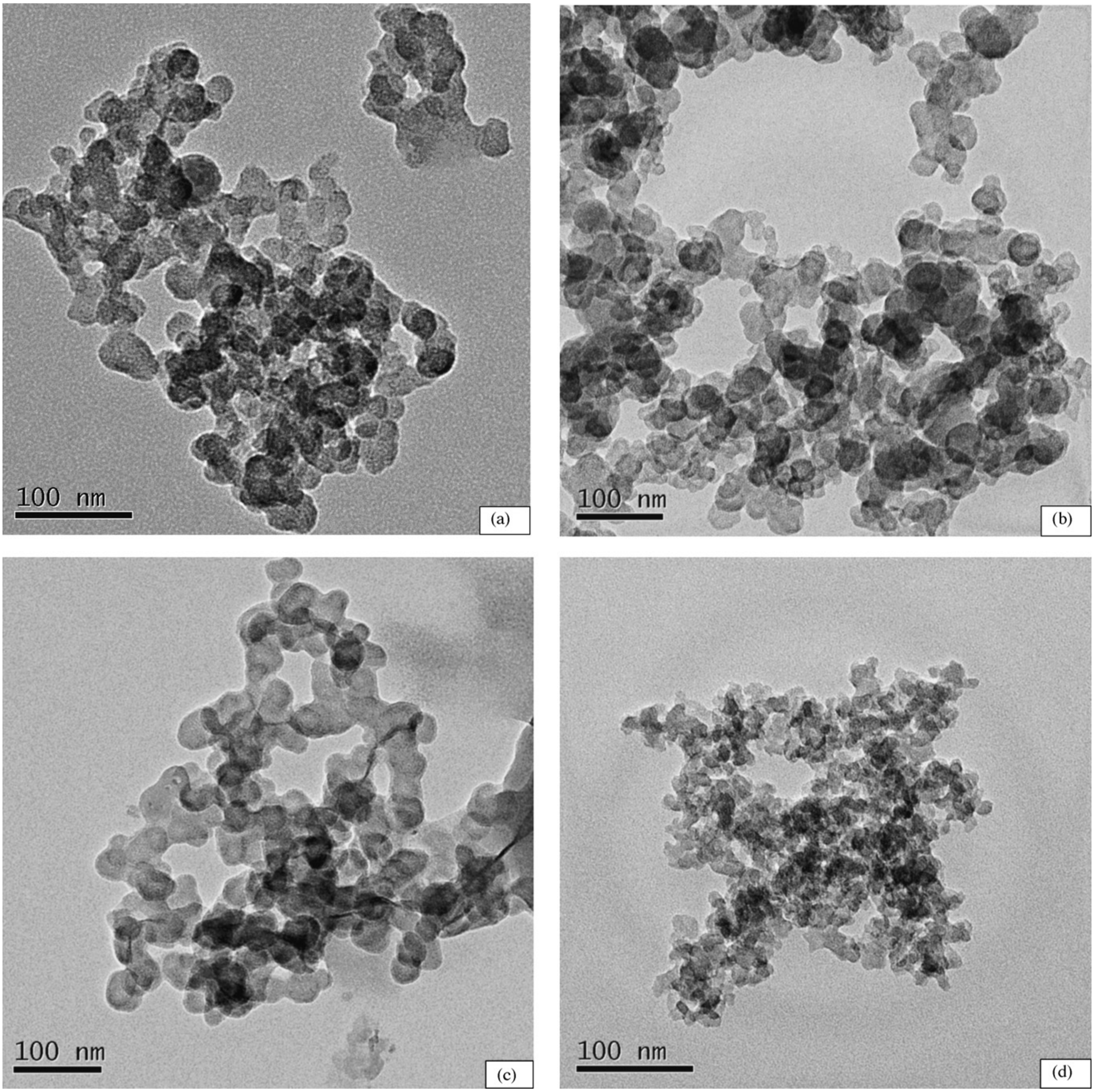


Fig. 3. TEM micrographs of silica powders prepared at 70 °C for 1 h 0.5 mol/L Na_2SiO_3 with various concentrations of surfactant PEG: (a) 0% PEG, (b) 5% PEG, (c) 7.5% PEG, and (d) 15% PEG.

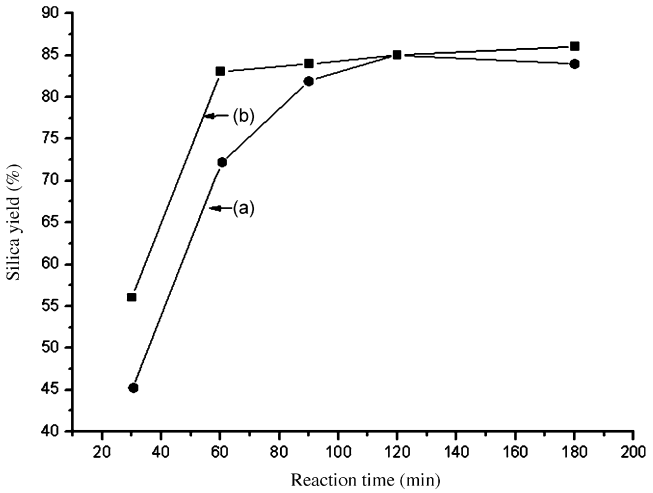


Fig. 4. Effect of reaction time on yield of silica powders prepared at 70 °C, 5% PEG for different reaction times with (a) 1.0 mol/L Na_2SiO_3 and (b) 0.5 mol/L Na_2SiO_3.

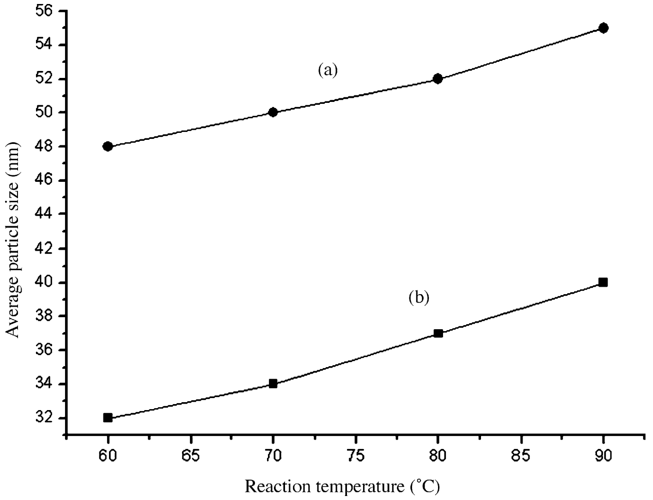


Fig. 5. Effect of reaction temperature on average particle size of silica powders prepared at different reaction temperature for 1 h, 5% PEG with (a) 1.0 mol/L Na_2SiO_3 and (b) 0.5 mol/L Na_2SiO_3.

3. Results and discussion

3.1. Single factor test analysis

3.1.1. Effects of reactant concentration and surfactant PEG

To find the effect of the reactant Na_2SiO_3 concentration on average particle size, the reaction was performed at 70 °C for 1 h varying the concentration at a range of 0.25–1.0 mol/L of Na_2SiO_3 while fixing the pressure of reaction at 0.2 MPa and the rate of agitation at 100 rev/min. The resultant powders were confirmed to be silica by X-ray diffraction and FT-IR examinations. The average particle size of the powders increased with increasing concentration of the reactant, possibly due to the rapid growth of crystal nucleus at higher concentration of Na_2SiO_3. Increasing the Na_2SiO_3 concentration results in an increase of the viscosity of reaction solution, which is unfavorable to the carbon dioxide delivery, thus increasing the nuclei coagulation and leads to the formation of larger particles. Fig. 2 shows TEM micrographs of the samples obtained at various concentrations of Na_2SiO_3 in a range of 0.25–1.0 mol/L.

To find the effect of surfactant PEG on the particle size distribution, the reaction was preformed at 70 °C for 1 h with various concentrations of surfactant at the range of 0–15% while fixing the reactant concentration at 0.5 mol/L. The influence of surfactant on particle size distribution was studied. The nonionic surfactant PEG plays a certain dimensional hindrance effect. There are two kinds of hydrophilic groups (hydroxyl group and ether linkage) in PEG molecule, therefore, the PEG is a good water soluble surfactant. In aqueous solution, the PEG chain might look like a snake, so it can easily be adsorbed onto the surface of the particles to form a macromolecule protection layer. Due to the dimensional hindrance of PEG, silica particles with smaller size and better dispersibility can be obtained. It was reported that with the assistance of surfactant, the size distribution of particles prepared in many systems are controllable, however, further investigation is required to study the influence of surfactant on particle size distribution. Fig. 3 shows TEM micrographs of the samples obtained at various concentrations of surfactant PEG in a range of 0–15%.

3.1.2. Effects of reaction time and temperature

To find the effect of reaction time on silica yield, the reaction time was varied from 30 to 180 min with the Na_2SiO_3 concentration of 0.5 mol/L and temperature at 70 °C while fixing other factors. The silica yield increases with increasing reaction time, but after a relatively long time, a plateau is reached, as shown in Fig. 4. The same tendency is observed for both samples with 0.5 and 1.0 mol/L Na_2SiO_3 except that the reaction time is longer for sample with 1.0 mol/L Na_2SiO_3. However, no significant increase of silica yield was observed at reaction time longer than 60 min for 0.5 mol/L Na_2SiO_3 (Fig. 4a) and 90 min for 1 mol/L Na_2SiO_3 (Fig. 4b). The influence of reaction time on silica yield might be explained by the fact that hydrolysis of sodium silicate is the key step of reaction process, which needs some time to reach the plateau. No significant difference in silica yield was observed after longer reaction time.

To find the effect of reaction temperature on average particle size, the temperature was varied from 60 to 90 °C with 0.5 and 1.0 mol/L Na_2SiO_3 while fixing other factors. The particle size increased with increasing reaction temperature, as shown in Fig. 5. The same tendency is observed in particle size reduction with reaction time for both samples with 0.5 mol/L (Fig. 5a) and 1.0 mol/L Na_2SiO_3 (Fig. 5b) except that the particle size is smaller for sample with 0.5 mol/L Na_2SiO_3. The influence of reaction temperature on particle size might be explained by the fact that in the case of higher temperature, the rate of nuclei growth is more rapid than that of nucleation, resulting in larger particle size. Furthermore, it has been found that with the temperature increasing, the vaporized water is increased, leading to serious gelation of reaction system and

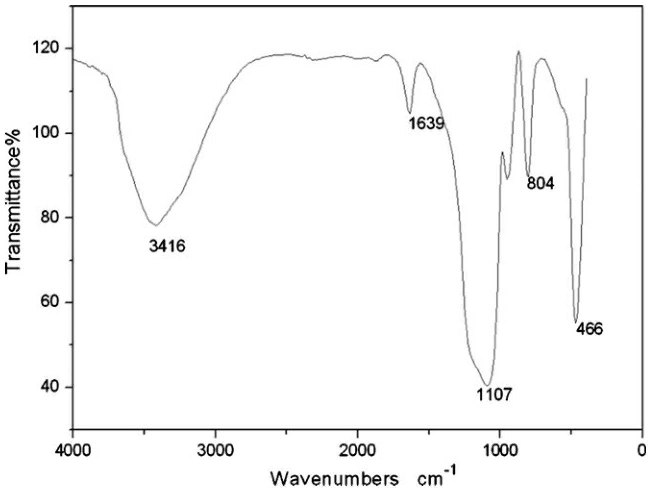
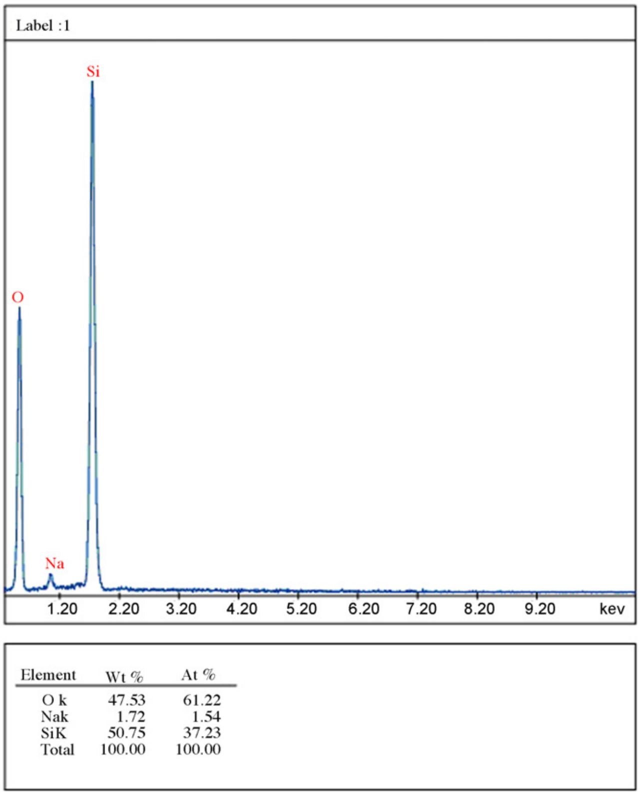


Fig. 6. FT-IR spectra of silica powders dried in vacuum at 50 °C for 24 h.



Element	Wt %	At %
O k	47.53	61.22
Nak	1.72	1.54
SiK	50.75	37.23
Total	100.00	100.00

Fig. 7. SEM-EDS analysis of silica powders dried in vacuum at 50 °C for 24 h.

difficulty of immersion cleaning. Therefore, high temperature is not recommended for the reaction system.

3.2. Orthogonal tests ($L3^4$) analysis

The optimum preparation conditions were obtained from the orthogonal tests: reaction pressure 0.2 MPa, reaction temperature 70 °C, reaction time 1 h, Na_2SiO_3 concentration of 0.5 mol/L and PEG concentration of 5%. Silica powders prepared at the optimum condition have average particle sizes and BET specific surface areas about 30 nm and 175 m^2/g, respectively. The powders obtained at the optimum condition have been chosen for study hereafter.

The resultant powders are confirmed to be silica by FT-IR examination in Fig. 6, SEM-EDS (energy dispersive X-ray spectrometer) in Fig. 7, and X-ray diffraction in Fig. 8. The absorptions in Fig. 6 agree well with the peaks (466, 804, 945 and 1107 cm^{-1}) of silica reported by Chen et al. [29]. Fig. 6 also shows absorption bands at 3416 and 1639 cm^{-1}. These bands are due to the H–O–H stretching and bending modes of the adsorbed water, respectively. There are no C–H absorption bands at 2910 and 1500–1300 cm^{-1}. Therefore, the FT-IR confirms that the powder is silica without the contamination of PEG.

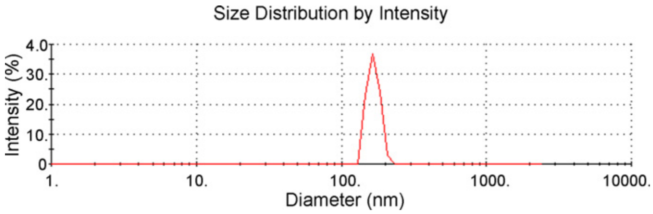


Fig. 9. Particle size distribution of silica powders prepared at 0.2 MPa in optimum condition of reaction time for 1 h with 70 °C, 0.5 mol/L Na_2SiO_3, 5% PEG.

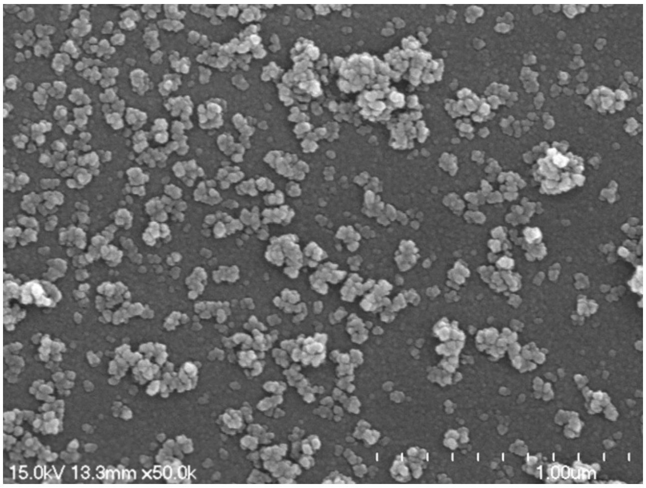


Fig. 10. SEM micrographs of silica powders prepared at 0.2 MPa in optimum condition of reaction time for 1 h with 70 °C, 0.5 mol/L Na_2SiO_3, 5% PEG.

SEM-EDS can be used for the analysis of the element contents of powders. Fig. 7 shows a typical SEM-EDS elemental analysis of silica powders, and it is found that the content of Si element reaches to 50.75%, O element to 47.53%, Na element only to 1.72%, and no other impurity elements. The EDS analysis in Fig. 7 confirms that the powders consist of high purity silica, 98.0% without Na_2CO_3 and PEG impurity, which is reasonably matched with FT-IR spectra in Fig. 6. It is believed that the silica powders were obtained by the precipitation reaction, as shown in Eq. (1).

The Na_2CO_3 and PEG in the precipitate can be washed out easily due to the high solubility of Na_2CO_3 and PEG, which reflects that the powders consist of silica without Na_2CO_3 and PEG.

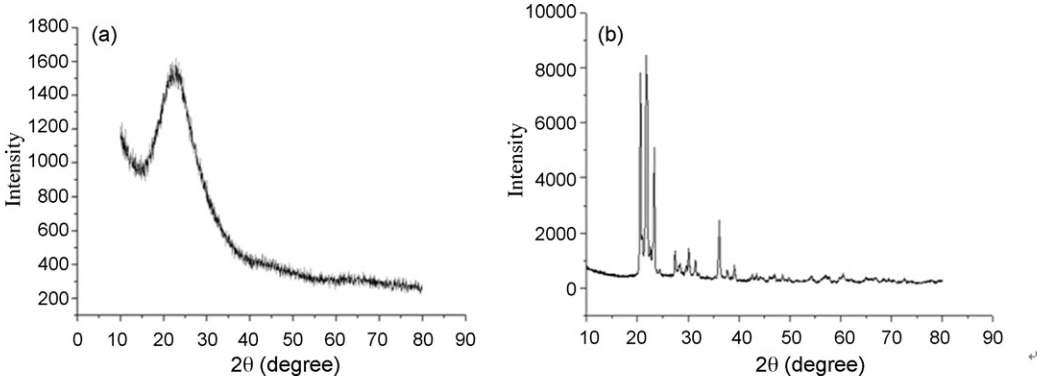


Fig. 8. XRD patterns of the silica powders (a) without heat-treatment and (b) with heat-treatment at 1000 °C for 1 h (Preparation condition: pressure, 0.2 MPa; reaction time, 1 h; reaction temperature, 70 °C; Na_2SiO_3 concentration, 0.5 mol/L; PEG concentration, 5%.).

The X-ray patterns in Fig. 8 confirm the powders to be silica, showing amorphous and crystalline silica at 70 and 1200 °C, respectively.

The size distribution of silica particles was measured by laser light scattering, showing a major peak, as illustrated in Fig. 9. The size of aggregated particles obtained by dynamic light scattering was about 160 nm. Fig. 10 shows SEM micrographs of the sample obtained at optimum condition. It was seen that the particles were spherical in shape with some aggregates, which was reasonably matched with laser light scattering in Fig. 9.

4. Conclusions

Ultrafine silica powders could be prepared at low cost using inexpensive materials of sodium silicate (Na_2SiO_3) and CO_2 to absorb the greenhouse gas of carbon dioxide and to enhance the continuous growth of economy. Particle size and particle size distribution can be controlled by the factors of the reaction time and temperature, and concentrations of surfactant and sodium silicate solutions. The optimum preparation condition was determined for obtaining the silica powders with nanometer, narrow size distribution, sphere in shape, and high purity without contamination of Na_2CO_3 and PEG. It was found that the desired powders could be synthesized at 0.2 MPa in the optimum condition of the reaction time 1 h, reaction temperature 70 °C, surfactant PEG 5%, Na_2SiO_3 0.5 mol/L using pressured carbonation method.

Acknowledgments

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