

6: B- Synthesis of Synthetic Opal (Experiment)

Introduction

Consult the publication [Jiang, P., Bertone, J.F., Whang, K.S., Colving, V.L., Single-Crystal Colloidal Multilayers of Controlled Thickness. *Chem. Mater.*, 1999, 11, 2132-2140.](#) for background and synthetic procedure.

Materials:

- 4 disposable 20 mL screw cap vials (glass)
- 4 glass pipettes (3x1 mL, 1x5 mL)
- 4 micro stir bars
- 4 glass microscopy slides (cut to 1cm x 7.5cm) – **NOT DISPOSABLE**. Clean for re-use.
- disposable cuvette for UV/vis spectroscopy
- Sample holders for electron microscopy digital or cell phone camera (bring own)
- flash drive (bring own);
- Image J program: <http://imagej.nih.gov/ij/download.html>

Chemicals:

- Tetraethoxysilane (TEOS), Alfa Aesar (99+%)
- Absolute (200% proof) ethyl alcohol (Koptec)
- Concentrated ammonium hydroxide solution (EM Science, 28.0~30.0%)
- Deionized water (18 MΩ)



Safety Precautions

Perform experiment on bench top. Measure concentrated ammonia in fume hood. Avoid eye contact with tetraethoxysilane.

Waste disposal

Use the containers provided.

Table 1. The amounts of chemicals used in different samples

Sample Number	1	2	3	4
Volume of TEOS (mL)	0.76	0.76	0.76	0.76
Mass of TEOS (g)	0.71	0.71	0.71	0.71
Volume of ammonium hydroxide (mL)	1.33	1.33	1.33	1.33
Mass of ammonium hydroxide (g)	1.20	1.20	1.20	1.20
Volume of deionized water (mL)	0	0.40	0.75	1.30
Mass of deionized water (g)	0	0.40	0.75	1.30
Volume of ethyl alcohol (mL)	17.91	17.51	17.16	16.61
Mass of ethyl alcohol (g)	14.13	13.82	13.54	13.11

Day 1

Synthesis of colloidal SiO₂ particles. For each experiment the necessary amounts of dehydrated ethanol, ammonium hydroxide, and deionized water (18 MΩ) were first mixed in the reaction vial. Then add the TEOS quickly and with mild stirring. The solution was colorless and transparent. Continue to stir the solution with magnetic stir bar for 1 h. The final appearance of the solution is white milky. The amounts of chemicals used in different samples are shown above in **Table 1**, while the concentrations of TEOS,

NH_3 , and H_2O in the ethanol solution are shown below in **Table 2** (assuming that the total volume is the sum of volumes of each chemicals).

Table 2. The concentrations of TEOS, NH_3 , and H_2O in the ethanol solution

Sample Number	1	2	3	4
Concentration of TEOS (mol/L)	0.17	0.17	0.17	0.17
Concentration of NH_3 (mol/L)	1.0	1.0	1.0	1.0
Concentration of H_2O (mol/L)	2.4	3.5	4.5	6.0

Growth of SiO_2 Colloidal Crystal. Transfer 10 mL SiO_2 nanosphere solution into a new scintillation vial and insert a glass slide (1cm x 7.5cm) in upright position. Let the solvent evaporate naturally in fume hood to produce films on the microslide.

SEM sample preparation. Transfer ~3 mL SiO_2 nanosphere solution into a disposable 15mL centrifuge tube and then centrifuge at 6000 rpm for 5 min. **Be sure to balance your samples before centrifugation.** Discard the colorless supernatant in the waste container. Disperse the white precipitate in ~3 mL ethanol and centrifuge again. Then discard the colorless supernatant and disperse the white precipitate in ~12 mL ethanol. These solutions will be used for UV-Vis measurement later. Pick one sample solution and dilute it tenfold. Place 5 μL onto a clean glass slide and examine it after evaporation. A faintly visible ring signifies that the concentration is appropriate. A clearly visible ring or series of rings indicates the concentration is too high. Transfer 5 μL diluted solution to a silicon wafer. The uncovered silicon wafer with sample is then oven dried for at least one day.

Day 2

SEM measurement. In Chemistry 0011 at 6 pm or 10 am. Please bring your own flash drive. From SEM images the size of SiO_2 nanoparticles is obtained for the picked sample.

UV-Vis spectra for SiO_2 nanosphere solution. The UV-Vis spectra for the SiO_2 nanosphere solutions above are recorded by using the disposable cuvette. Use a cuvette filled with ethanol to record the background.

SiO_2 colloidal crystal: Place the glass slide with the SiO_2 colloidal crystal into the spectrometer to record the transmission spectrum for the range from 350 nm to 1000 nm. Use an uncoated glass slide to record the background. The absorption of the glass slide corresponds to the size of the SiO_2 nanoparticles (Glass slides and SiO_2 do not absorb visible light.)

6: B- Synthesis of Synthetic Opal (Experiment) is shared under a [CC BY-NC-SA 4.0](https://creativecommons.org/licenses/by-nc-sa/4.0/) license and was authored, remixed, and/or curated by LibreTexts.