

Recipes for Stöber Silica Particles

(Edited by Shan Jiang)

General comments:

Stöber synthesis is something of an art; the most important step is making sure that you are stirring as smoothly and as perfectly as possible during the synthesis.

Recipe 1 (not fluorescently labeled)

Here is the recipe to make monodisperse 460 nm silica particles:

Needed Volume (ml)

TEOS: 5.6

NH₄OH(29%): 9.8

DI water: 10.8

EtOH: 73.8

Procedure: Mix the ammonia, DI water and ethanol first, and add TEOS all at once. React for 12 hours. Clean the colloids by centrifuging and dispersing (need to sonicate) with pure EtOH for 4 times, and centrifuging and dispersing (need to sonicate) with H₂O until the pH reaches ~ 7.

Recipe 2 (fluorescently labeled core shell particles)

Check the particles with SEM every 2-3 additions, and aim for something around 800 nm. In addition, the recipes here are for the pink dye (a rhodamine variant); to use a yellow dye (a fluorescein variant), we will need to scale the weight of dye appropriately to keep the molar ratio of dye:APS constant.

Fw (RITC): 536.1 g/mol

Fw (FITC): 389.4 g/mol

I. 500-1000nm particles

a. Day 1 (stir overnight in small [5-10mL] flask):

- i. 28 mg RITC (rhodamine isothiocyanate) (on weigh paper – add last!)
- ii. 44 mg APS (3-aminopropyltriethoxysilane) (in flask)
- iii. 5 mL 200-proof ethanol
- b. Day 2 (add to 500 mL round-bottom flask and stir for 6h):
 - i. 169.5 mL 200-proof ethanol (133.74 g)
 - ii. 20.4 mL ammonia
 - iii. 3 mL water
 - iv. dye solution from day 1
 - v. 7.2 mL TEOS (tetraethyl orthosilicate)
- c. Additions (first is 6h after day 2 start, then every 2h):
 - i. 1.15 mL water
 - ii. 7.2 mL TEOS
- d. Core shell restart:
 - i. 169.5 mL total 200-proof ethanol (est. amt. in solution and subtract that)
 - ii. 20.4 mL ammonia
 - iii. 3 mL water
 - iv. dyed particles
 - v. 1 mL TEOS
- e. Core shell additions (first after 6h, then every 2h):
 - i. 1.15 mL water
 - ii. 7.2 mL TEOS

II. 100nm particles, based off Zukoski group recipe

- a. Day 1 (stir overnight in small flask):
 - i. 28 mg RITC (on weigh paper – add last!)
 - ii. 44 mg APS (in flask)
 - iii. 5 mL 200-proof ethanol
- b. Day 2 (add to 500 mL round-bottom flask and stir for 6h):
 - i. 176 mL ethanol (138.9 g)
 - ii. 7.7 mL ammonia
 - iii. 4.6 mL water
 - iv. fluorescent cores from day 1
 - v. 7.7 mL TEOS
- c. Additions (every 2h):
 - i. 1.653 mL water
 - ii. 10.185 mL TEOS

III. Modified recipe for dyed particles (500-1000nm)

- a. Note: this recipe can be used with the dye ratio 38 mg RITC/ 44 mg APS for lessdyed particles
- b. Day 1 (stir overnight in small flask):
 - i. 57 mg RITC (on weigh paper – add last!)
 - ii. 44 mg APS (in flask)
 - iii. 5 mL 200-proof ethanol
- c. Day 2 (add to 500 mL round-bottom flask and stir for 6h):
 - i. 68 g 200-proof ethanol
 - ii. 20.27 mL ammonia
 - iii. 37.8 mL water
 - iv. dye solution from day 1
 - v. 5.312 mL TEOS
- d. Additions (first is 6h after day 2 start, then every 2h):
 - i. 1.205 mL water
 - ii. 4.965 mL TEOS
- e. Core shell restart:
 - i. 68 g total 200-proof ethanol (est. amt. in solution and subtract that)
 - ii. 20.27 mL ammonia
 - iii. 37.8 mL water
 - iv. dyed particles
 - v. 1 mL TEOS
- f. Core shell additions (first after 6h, then every 2h)
 - i. 1.205 mL water
 - ii. 4.965 mL TEOS