

Procedures for the Synthesis of Fluorescent Silica Nanoparticles. Updated November 24, 2013.

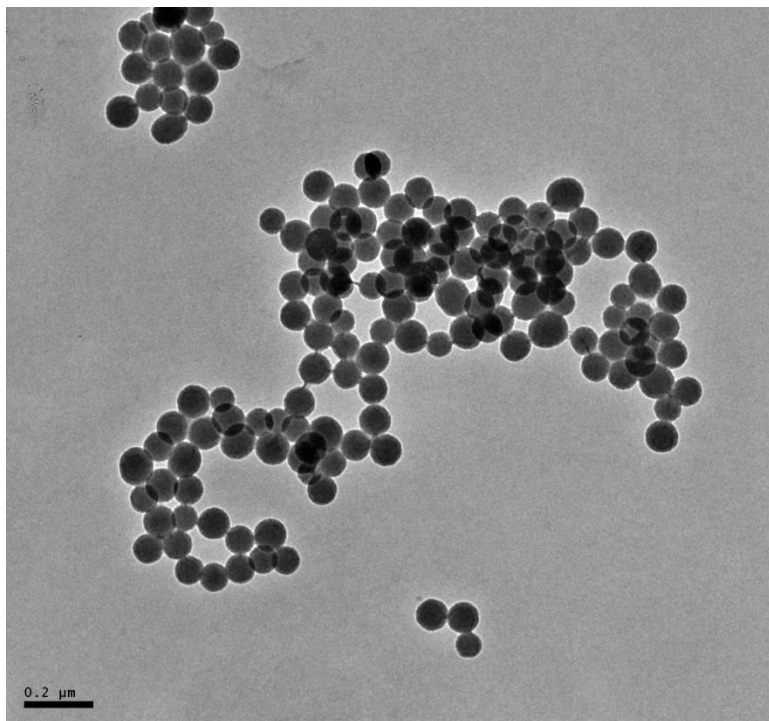
WFSNP - Synthesis of silica nanoparticles with amino groups, FITC, and phosphate

Synthesis of FITC-APTS conjugate

1. Added 69 mg (73 μ L) APTS and 5.25 mg FITC in 1 mL ethanol in a 20 mL round-bottom flask. under dry nitrogen atmosphere.
2. Stirred magnetically for 12 hours.
 - a. The FITC-APTS conjugate solution is protected from light during reaction and storage to prevent photo bleaching. The conjugate is later used as the fluorescent silane reagent.

Synthesis of FSNPs

1. Added 7.7 mL cyclohexane (oil), 1.65 mL Triton X-100, 1.6 mL n-hexanol, 0.34 mL DI water to a 20 mL round-bottom flask.
2. Stirred for 15 minutes.
3. With an interval of 10 min between two successive additions, Added 50 μ L FITC-APTS conjugate, 100 μ L TEOS and 100 μ L 30% aqueous NH_4OH .
4. After 10 minutes of stirring, step 3 was repeated.
5. Stirred for 30 minutes.
6. Added 15 μ L THPMP.
7. Stirred for 24 hours at room temperature.
8. The micro-emulsion system was destabilized by adding \sim 10 mL denatured ethanol.
9. Centrifuged at 7,000 rpm for 10 minutes. Supernatant decanted and then nanoparticles redispersed in 10 mL ethanol using sonication and shaking. Repeated centrifugation and redispersion 4 more times.
10. Centrifuged at 7,000 rpm for 10 minutes. Supernatant decanted and nanoparticles redispersed in 10 mL DI water. Repeated centrifugation and redispersion 2 more times.
11. Stored in 10 mL DI water and protected from light to prevent photo bleaching.



TEM Image: WFSNP

WFSNPC - Synthesis of FSNP without THPMP and then carboxylated

Synthesis of FITC-APTS conjugate

1. Added 69 mg (73 μ L) APTS and 5.25 mg FITC in 1 mL ethanol in a 20 mL round-bottom flask under dry nitrogen atmosphere.
2. Stirred magnetically for 12 hours.
 - a. The FITC-APTS conjugate solution is protected from light during reaction and storage to prevent photo bleaching. The conjugate is later used as the fluorescent silane reagent.

Synthesis of FSNPs

1. Added 7.7mL cyclohexane, 1.65mL Triton X-100, 1.6mL n-hexanol, and 0.34mL DI water to a 20mL round-bottom flask.
2. Stirred for 15 minutes.
3. Added 50 μ L FITC-APTS conjugate, 100 μ L TEOS, and 100 μ L NH₄OH.
4. After 10 minutes of stirring, step 3 was repeated.
5. Stirred for 24 hours at room temperature.
6. The micro-emulsion system was destabilized by adding ~10 mL denatured ethanol.
7. Centrifuged at 7000 rpm for 10 minutes, redispersed in 1.5mL ethanol per vial. Repeated 4 more times.
8. Centrifuged at 7000 rpm for 10 minutes, redispersed in 1.5mL DMF per vial. Repeated 2 more times.
9. Added 2.5mL succinic anhydride solution (0.1g succinic anhydride in 5mL DMF) to flask with SNPs.
10. Stir for 1 hour.
11. Centrifuged at 7000 rpm for 10 minutes, redispersed in 1.5mL DMF per vial. Repeated 3 more times.
12. Stored in 10mL DMF and protected from light to prevent photo bleaching.

WFSPC - Synthesis of Silica Nanoparticles Functionalized with FITC, Phosphate, and Carboxylated Amines

Synthesis of FITC-APTS conjugate

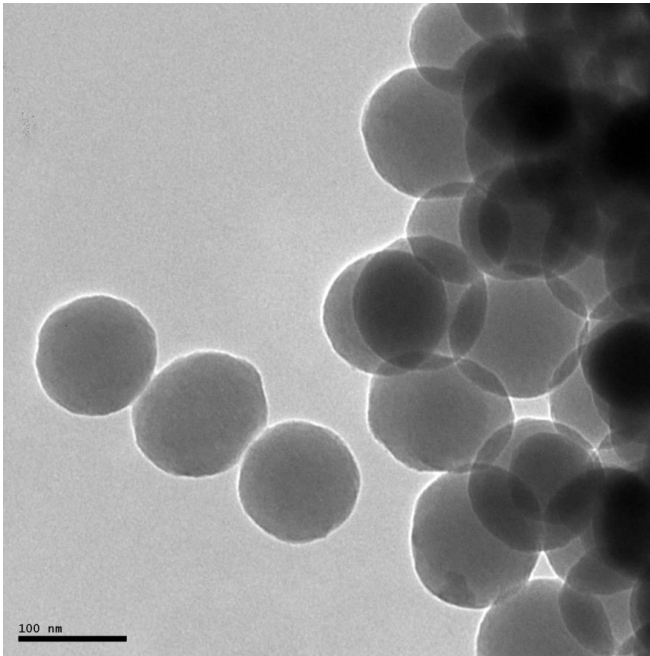
1. Added 69 mg (73 μ L) APTS and 5.25 mg FITC in 1 mL ethanol in a 2 mL reaction vial under dry nitrogen atmosphere.
2. Stirred magnetically for 12 hours.
 - a. The FITC-APTS conjugate solution is protected from light during reaction and storage to prevent photo bleaching. The conjugate is later used as the fluorescent silane reagent.

Synthesis of FSNPs

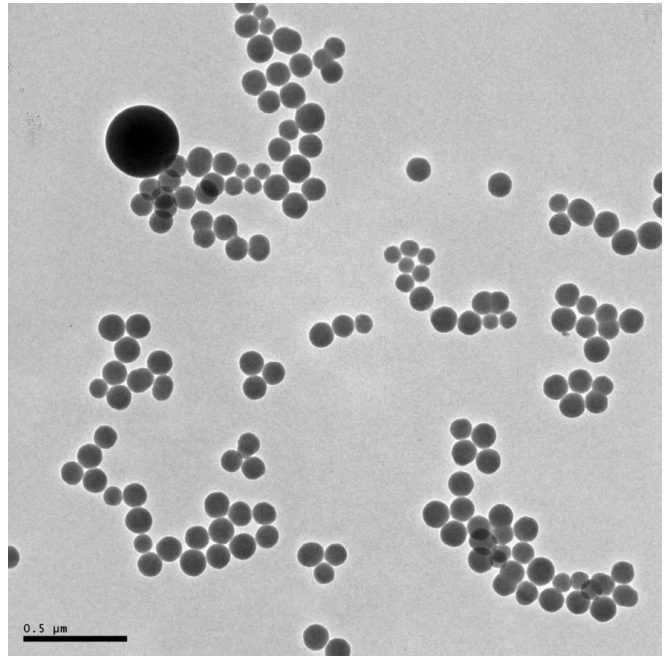
1. Added 7.7 mL cyclohexane (oil), 1.65 mL Triton X-100, 1.6 mL n-hexanol, 0.34 mL DI water to a 20 mL round-bottom flask.
2. Stirred for 15 minutes.
3. Added 50 μ L FITC-APTS conjugate, 100 μ L TEOS and 100 μ L 30% aqueous NH₄OH.
4. After 10 minutes of stirring, step c. was repeated.
5. Stirred for 30 minutes.
6. Added 15 μ L THPMP.
7. Stirred for 24 hours at room temperature.
8. The micro-emulsion system was destabilized by adding ~10 mL denatured ethanol.
9. Centrifuged at 7,000 rpm for 10 minutes. Supernatant decanted and then nanoparticles redispersed in 10 mL ethanol using sonication and shaking. Repeated centrifugation and redispersion 4 more times.
10. Centrifuged at 7,000 rpm for 10 minutes. Supernatant decanted and nanoparticles redispersed in 10 mL DI water. Repeated centrifugation and redispersion 2 more times.
11. Stored in 10 mL DI water and protected from light to prevent photo bleaching.

Carboxylation

1. Redispersed SNPs in dimethylformamide (DMF).
 - a. 1.5 mL SN2 centrifuged at 7000 rpm for 10 minutes.
 - b. Supernatant decanted.
 - c. Redispersed in 1.5 mL DMF.
 - d. Repeated 2 more times.
2. Added 2.5 mL succinic anhydride solution (0.1 g succinic anhydride in 5 mL DMF).
3. Stirred for 1 hour.
4. Centrifuged at 7000 rpm for 10 minutes. Supernatant decanted and then nanoparticles redispersed in 1.5 mL DMF using sonication and shaking. Repeated centrifugation and redispersion 2 more times.
5. Stored in 1.5 mL DMF.



TEM Image: WSFPC



TEM Image: WSFPC

WFSN - Synthesis of Silica Nanoparticles with FITC and Amino Groups (without Phosphate)

Synthesis of FITC-APTS conjugate

1. Added 69 mg (73 μL) APTS and 5.25 mg FITC in 1 mL ethanol in a 2 mL reaction vial. under dry nitrogen atmosphere.
2. Stirred magnetically for 12 hours.
 - a. The FITC-APTS conjugate solution is protected from light during reaction and storage to prevent photo bleaching. The conjugate is later used as the fluorescent silane reagent.

Synthesis of FSNPs

1. Added 7.7 mL cyclohexane (oil), 1.65 mL Triton X-100, 1.6 mL n-hexanol, 0.34 mL DI water to a 20 mL round-bottom flask.
2. Stirred for 15 minutes.
3. Added 50 μL FITC-APTS conjugate, 100 μL TEOS and 100 μL 30% aqueous NH₄OH.
4. After 10 minutes of stirring, step 3 was repeated.
5. Stirred for 24 hours at room temperature.
6. The micro-emulsion system was destabilized by adding ~10 mL denatured ethanol.

7. Centrifuged at 7,000 rpm for 10 minutes. Supernatant decanted and then nanoparticles redispersed in 10 mL ethanol using sonication and shaking. Repeated centrifugation and redispersion 4 more times.
8. Centrifuged at 7,000 rpm for 10 minutes. Supernatant decanted and nanoparticles redispersed in 10 mL DI water. Repeated centrifugation and redispersion 2 more times.
9. Stored in 10 mL DI water and protected from light to prevent photo bleaching.

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