Surfactant:oil:water (51:34:15)

**Surfactant**
- Brij S10

**Oil**
- Cyclohexene (use in hood only, check MSDS for proper handling)

**Water**
- H2O + HAuCl4 salt in various concentrations

7/15/11: Prepared 1.5:1 stock solution of surfactant:oil. 15.054 g Brij S10, 10.036 g cyclohexene (actual: 10.034 g). Heated to 75°C for 20 min to dissolve waxy Brij S10.

Prepared 85:15 solution of Stock:H2O. 0.424 g stock, 0.0748 g H2O (actual: 0.071 g). Heated to 75°C for 20 min to dissolve. Inverted and then vortex solution. Gel formed, but thin layer of water at top. Heated tube again in hot plate block at 75°C for 20 minutes and vortexed. Gel fully solidified.

Prepared another gel

Surfactant/oil stock:water (0.564 g Brij/0.0995 g water (actual: 0.095 g))

Added water to surfactant/oil stock, heated at 80°C for 15 minutes before vortexing. Gel formed completely.

Prepared 3 mesophases: 4 mM, 40 mM, and 400 mM HAuCl4. All three mesophases solidified into gel after vortexing.

7/18/11: Visualized UV-VIS of 4 mM, 40 mM, and 400 mM. Absorbance showed that only 400 mM contained gold particles, but not nanorods (only one peak ~560 nm).
Prepared another three samples of 4 mM, 40 mM, and 400 mM. Mixtures turned deep purple once tubes were placed in hot plate block at 80°C for 30 seconds. Mesophases formed in 4 mM and 40 mM samples, while 400 mM remained a liquid. Floating gold particles visible in 4 mL and 40 mL HAuCl4 stock solutions, so these two stock solutions were remade. Next step: vortex mesophase right after water is added to surfactant/oil. Only heat if necessary.

Prepared another two samples of 4 mM and 40 mM using new stock solution. Vortexed mesophase immediately after water was added, but still formed gel on bottom of the tube and liquid on top. Observed tiny purple specks suspended in gel. To form homogenous mixture, heated in hot plate block at 80°C for 15 minutes. Top reformed into liquid but bottom remained gel. After heating and vortexing, gels formed but color changed drastically. Mesophases became deeper purple color.

7/19/11:

Prepared two test samples of Brij S10/cyclohexene/H2O to test mixing technique.

Sample 1

Combine in order:
1. 0.549 g Brij/cyclohexene
2. 0.0968 g H2O (actual: 0.093 g)
Vortex immediately along length of tube.

**Sample 2**

Combine in order:

1. 0.100 g H2O
2. 0.566 g Brij/cyclohexene (actual: 0.564 g)

Vortexed immediately along length of tube.

Results: The lower layer of Sample 1 formed a gel, however the top remained liquid. Sample 2 formed a complete gel. To mix more thoroughly, samples were sonicated for 30 minutes. After sonication, gel in Sample 1 had very thin layer of liquid on top of solidified gel. Sample 2 was completely homogenized and remained as a solidified gel.

Prepared 4 mM and 40 mM sample by adding H2O+HAuCl4 first.

**4 mM**

1. 0.101 g 4 mM HAuCl4
2. 0.572 g Brij S10/cyclohexene (actual: 0.577 g)
3. Vortexed immediately along length of tube. Continued for ~5 minutes until gel solidified. No heating necessary.

Mesophase remained clear/faint yellow hue, did not turn purple.

**40 mM**

1. 0.101 g 40 mM HAuCl4
2. 0.572 g Brij S10/cyclohexene (actual: 0.572 g)
3. Vortexed immediately along length of tube, gel formed immediately. No heating necessary.

Mesophase remained light yellow hue, did not turn purple.

7/20/11:

4 mM had dark purple particles in mesophase. 40 mM became opaque and rust colored.

UV-VIS: 4 mM showed one peak ~550 nm, indicating no nanorods. 40 mM had no peak.

Tested mixing technique again:

Prepared 4 mM and 40 mM, vortexed side and bottom of each tube immediately after adding H2O+HAuCl4 to Brij/cyclohexene for ~8 minutes. Digested each mesophase with 1 mL ethanol, centrifuged for 5 then 10 additional minutes at 14,500 rpm. Removed supernatant, resuspended sediment in water and took UV-VIS. 4 mM showed slight peak between 800-900 nm. 40 mM showed no presence of gold.
2 hours later: remaining mesophase turned dark purple in both 4 mM and 40 mM. Digested remaining gel in 1.5 mL ethanol, centrifuged for 10 minutes at 14,500 rpm. Removed supernatant, resuspended sediment in water, and took UV-VIS.

7/22/11:

Adjustment: Make 4 mM and 40 mM HAuCl4 solutions using 0.001M HCl (pH 3) and 0.001 M NaOH (pH 12).

4 mM HAuCl4 in 0.001 M HCl
- 1.056 g Brij/Cyclohexene
- 0.186 g 0.001 M HCl (actual: 0.184 g)

4 mM HAuCl4 in 0.01 M NaOH
- 1.054 g Brij/Cyclohexene
- 0.186 g 0.01 M NaOH (actual: 0.165 g)

40 mM HAuCl4 in 0.001 M HCl
- 1.049 g Brij/Cyclohexene
- 0.185 g 0.001 M HCl (actual: 0.183 g)

40 mM HAuCl4 in 0.01 M NaOH
- 1.045 g Brij/Cyclohexene
- 0.184 g 0.01 M NaOH (actual: 0.183 g)

Results: Gels formed immediately for all but 40 mM/HCl. After vortexing ~15 sec, 40 mM/HCl solidified into gel.

Heated all samples at 40 C for 20 min, gels each had a thin liquid layer on top. Vortexed all for 5 min, let stand at room temp. 4 mM/NaOH turned faint purple. Measured UV-VIS of samples; no significant peak observed except for 4 mM/NaOH (@ 525 nm). Kept all samples in 40 C hot plate block for 1.5 hours. 4 mM/NaOH and 4 mM/HCl turned deep purple color. Measured UV-VIS of samples; 4 mM/NaOH and 4 mM/HCl had very high absorbances (3.8 and 2.5, respectively @ 550 nm), 40 mM/NaOH showed small peak (0.95 @ 535 nm).

Digested all gels with 1 mL ethanol and 1 drop CTAB, centrifuged for 10 min at 14,500 rpm, removed supernatant and resuspended sediment in 1.5 mL ethanol. Measured UV-VIS of re-suspended sediment; 40 mM/NaOH showed three peaks (-0.2, -0.2, and -0.19 @ 470, 650, and 805 nm, respectively). 40 mM/HCl showed declining absorbance from -0.21 to -0.30 from 900 nm to 730 nm. There were three small peaks (-0.289, -0.295, and -0.309 @ 680 600, and 480 nm). 4 mM/HCl showed two peaks (-0.260 @ 875 nm) and (-0.258 @ 750 nm).

Next steps: Take SEM images of resuspended sediment