Au nanoparticles synthesis

Materials:

- Tetrachloroauric acid ($HAuCl_4$), solution 1 mM;
- Sodium Citrate ($C_6H_5Na_3O_7$), solution 95 mM;
- 10 mL pipette and a rubber bulb;
- Micropipette up to 1000 µL and the corresponding tips;
- Beaker 50 mL, tall form;
- Crystallizer (flat beaker);
- Magnetic stirring bar covered with Teflon;
- Proper supports and arms for the beakers;
- Clock glass;
- Hot plate with stirrer;
- Milli-Q ultrapure water.

Procedure for 10 mL of Au NP [1]:

1. Dilute 1/10 the gold chloride from the stock solution, which in our case is 10mM, in order to obtain 1mM solution.
2. Obtain the 95 mM solution of Sodium Citrate tribasic dehydrate (*SC*) ($C_6H_5Na_3O_7$, MW 294.10 g/mol) in Milli-Q water.
   For example, dilute *x* grams of Sodium Citrate in $\frac{x}{MW*[SC]}$ liters of water.
3. Fill the crystallizer with tap water and heat it up to the boiling temperature on the plate.
4. Pour 9.5 mL of 1mM Tetrachloroauric acid in the 50 mL beaker using the 10 mL pipette.
5. Put the stirring bar in and cover with the clock glass.
6. Dip partially the beaker into the crystallizer filled with boiling tap water and fix it with the proper supports. The beaker must not lie on the bottom of the crystallizer but has to be suspended!
7. Start the stirring of the heat plate. The mixing assures the homogeneity of both the temperature and the concentration of all the volume of the Tetrachloroauric acid.
8. Separately, heat up to 100°C 5 mL of sodium citrate in a 20 mL glass vial as for the acid.
9. When both the acid and the sodium citrate reached the 100 °C, that is when the water inside the crystallizers boils, add rapidly 0.5 mL of sodium citrate to the Tetrachloroauric acid with the micropipette.
10. Keep stirring at this temperature for 15 minutes. Solution should switch from yellow to transparent (reduction of the acid) and then to a dark red color as gold nanoparticles grow.
11. After 15 minutes remove from the beaker from the boiling water and let it cool down to the room temperature.
12. A ratio of 5:1 is obtained between the citrate and the gold with this recipe.

Optical measurements:

1. Dilute to 1/10 the nanoparticles solution in order to obtain 95µM concentration. Use the plastic cuvettes and add 300 µL of fresh nanoparticles solution to 2700 µL ultrapure water.
2. Measure absorbance spectra with the spectrophotometer. Remember that the optical path of the cuvette is 1 cm.

Silanization of substrates and nanoparticle deposition [2]:

1. Clean Silicon/SLG substrates with 3:1 acid and basic piranha in order to clean the substrates and enrich the surface with –OH groups.
2. Prepare 5 mM APTES solution (0.946 g/mL, MW 221.37 g/mol) in absolute ethanol. Here, (3-Aminopropyl) triethoxysilane comes as liquid with concentration => 98%, so stock solution is ~ 4.188 M. For example, for 40 mL of solution around 50 µL of APTES have to be added to the ethanol. (0.04 L * 0.005 M / 4.188 M).
3. Immerse the substrates in APTES solution for 40 min.
4. Rinse the substrates first in ethanol then in ultrapure water.
5. Dry with nitrogen flow.
6. Bake the substrates at 120 degrees for 30 minutes in order to complete the Si-O bond formation.
7. Finally, deposit the gold nanoparticles on the substrates and leave them for 12h. Thus, gold nanoparticles will stick to the substrate and self-assemble in a disordered patter.
8. Rinse the substrates with ultrapure water to remove excess nanoparticles and dry with nitrogen.

References

[1] Kimling, J. *et al.* Turkevich Method for Gold Nanoparticle Synthesis Revisited. *J. Phys. Chem. B* **110,** 15700–15707 (2006).

[2] Shantang Liu et al., Evaporation-induced self-assembly of gold nanoparticles into a highly organized two-dimensional array, *Phys. Chem. Chem. Phys.,* **4**, 6059-6062 (2002)