Gold Nanoparticle Synthesis and Characterization

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Introduction: Nanoparticles are used in all kinds of things you interact with on a daily basis including sunscreens, antibiotic treatments, solar cells, and cosmetics. CHANL invites you and your students to synthesize gold nanoparticles in your lab classroom and characterize those nanoparticles with a variety of analytical instrumentation in CHANL.

Target audience: Grades 9-12; community college

NC standards:

[1] We will prepare hydrogen tetrachloroaurate(III) and fructose solutions. However, what is a solution? What will the result of this reaction give us? An Au nanoparticle solution? A colloid? A suspension?

PSc.2.1.1 Classify matter as: homogeneous or heterogeneous; pure substance or mixture; element or compound; metals, nonmetals or metalloids; solution, colloid or suspension.

[2] This lab will also teach students how to prepare a solution of a certain concentration.

Chm.3.2.3 Infer the quantitative nature of a solution (molarity, dilution, and titration with a 1:1 molar ratio).

Format: Laboratory (≤50 min) followed by a remote workshop or a UNC campus visit

Objective(s):
The students will:

1. Synthesize gold nanoparticles.
2. Interpret and draw conclusions from qualitative and quantitative data.
3. Understand characterization techniques including transmission electron microscopy (TEM), energy dispersive spectroscopy (EDS), and ultra-violet spectroscopy (UV-vis).
4. Be able to develop gold nanoparticle synthesis using other reducing agents.

Materials:

1. Hydrogen tetrachloroaurate(III) trihydrate ($HAuCl_4 \cdot 3H_2O$)
   Work within a chemical hood to prevent exposure to this material, and wear protective gloves, a lab coat, and safety glasses. (Please see attached Material Safety Data Sheet)

   Available from: Alfa.com (1g) for $98.10 + shipping
   (about 250ml 10^{-2}M solution = 2500 single use)

2. Fructose
   Available from: Alfa.com (250g) for $24.14 + shipping
   (about 1250 single use)
3. Scintillation vial
4. Balance
5. Graduated cylinder (5 mL)
6. Autopipette that can deliver 100 µL (plus tips)
7. Glass stir rod or stir bar and stir plate

Lab procedure: (in fume hood and wear proper personal protective equipment)

1. In a scintillation vial, add 0.2 g of fructose and 3.9 mL of water followed by 100 µl (10^{-2} M) of hydrogen tetrachloroaurate(III) solution.
2. Stir the solution either with a glass stirring rod or magnetic stir bar + stir plate.
3. Heat the solution in a water bath at 70–75°C; stir the solution every once in a while.
4. Wait until the solution turns pink (in minutes), which is indicative of the formation of metal nanoparticles.
5. Let the solution cool on the bench to room temperature.

Possible variations:

1. **Varying fructose concentration:** Instead of 0.2g of fructose in 3.9ml water, 0.02g of Fructose in 3.9ml water, and 0.02g of Fructose in 39ml water can also be investigated.
2. **Varying reducing agent:** Glucose, sucrose, or other reducing agent can be used.
3. **Varying reaction time**: Try increasing and decreasing the amount of time the reaction spends in the water bath.
4. **Varying reaction temperature**: Try the experiment at room temperature, 30ºC, and 50ºC.

Measurements (to be done at CHANL):

1. UV-vis for quantitation of product made
2. TEM for particle size measurement
3. EDS for particle chemical composition

References:

[1] Panigrahi, S., Kundu, S., Ghosh, S. et al. Journal of Nanoparticle Research (2004) 6: 411. doi:10.1007/s11051-004-6575-2

[2] Abdelhalim MAK, Mady MM, Ghannam MM (2012) Physical Properties of Different Gold Nanoparticles: Ultraviolet-Visible and Fluorescence Measurements. J Nanomed Nanotechol 3:133. doi: 10.4172/2157-7439.1000133

[3] http://www.cytodiagnostics.com/store/pc/Gold-Nanoparticle-Properties-d2.htm

Sample results:

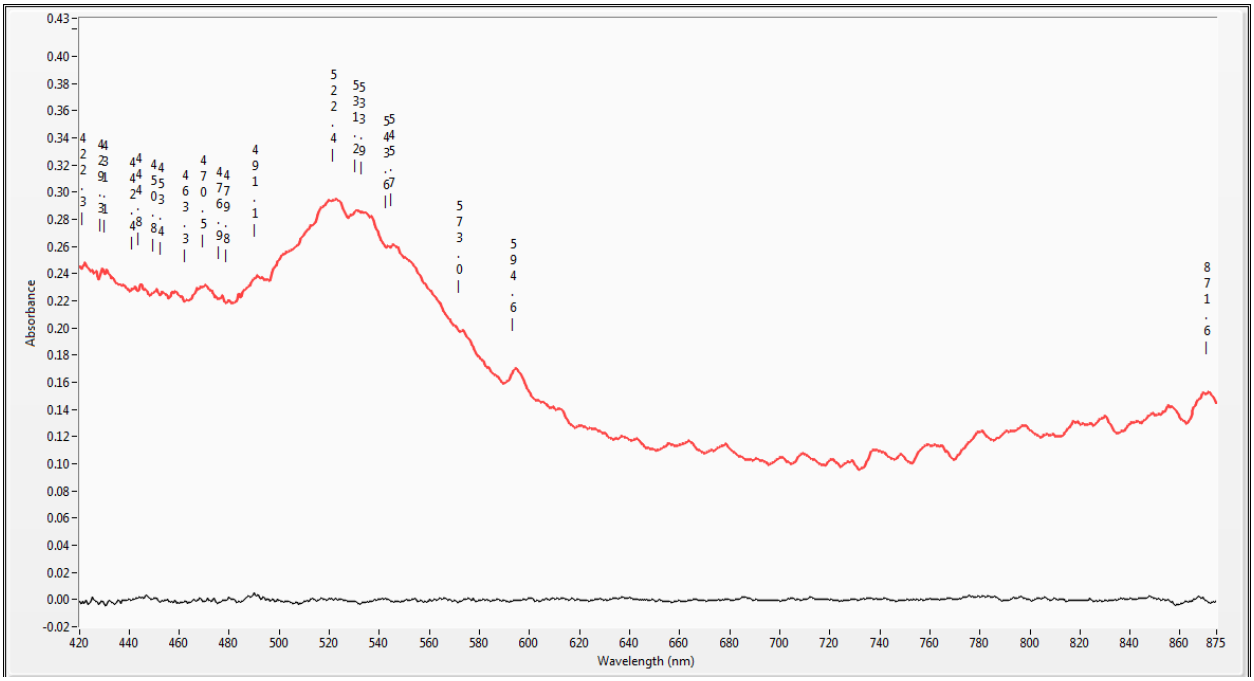

Figure 1. UV-vis spectrum for example solution. Peaks around 522 nm are indicative of metal nanoparticle formation.

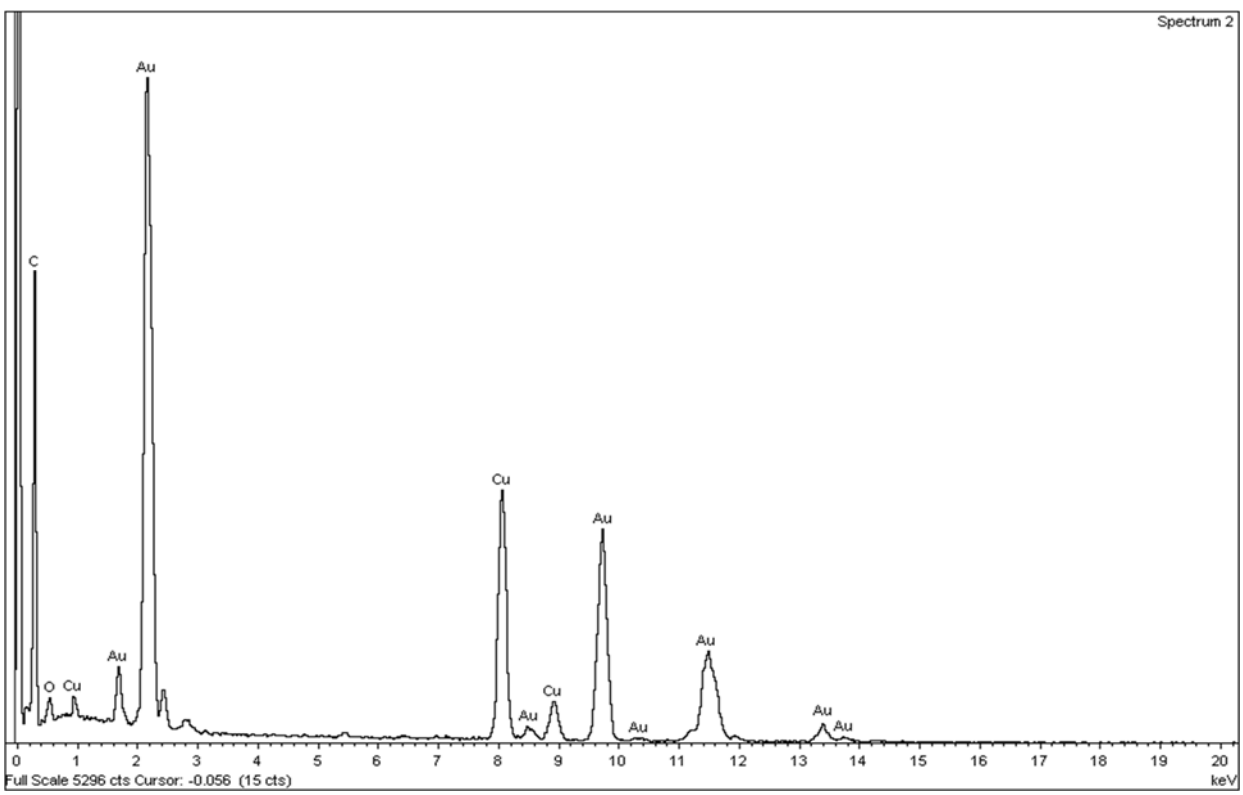

Figure 2. EDS data for example solution and indicates the presence of gold, copper, oxygen, and carbon.

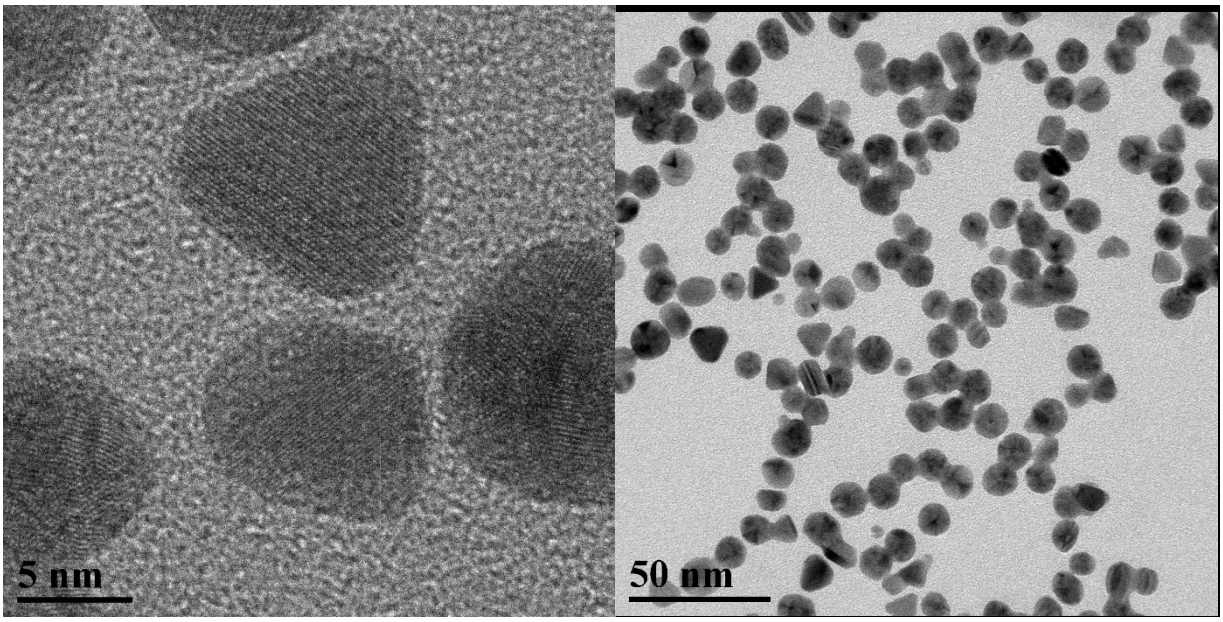

Figure 3. TEM images indicate that particles are around 10 nm in diameter.