Remote Access Laboratory Guide

Colloidal Gold Nanoparticle Synthesis and Characterization

In this exercise, you will:

- Investigate the synthesis of the colloidal nanoparticle solutions.
- Gain experience in nanoscale characterization.
- Learn how nanostructures interact with light and how the interaction is size dependent

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RAIN

Background

What color is gold? If that question seems to be on the same par with, "Who is buried in Grant's tomb?", then you may be in for quite a surprise during the course of this investigation. The color of any element – the gold of gold, the silver of silver, the copper of copper – with which you are familiar, is representative of what is known as a bulk property of matter. As the name implies, this is a property that results from the accumulation of a reasonable mass of the material, i.e. from the presence of a large collection of atoms. The chemical synthesis you will conduct in this experiment will introduce you to a new realm of materials, one where the properties reflect the influence of just a small collection of atoms. Such is the domain of the field known as *nanoscience*, which is studying and manipulating only a few atoms or individual molecules. What makes this field so fascinating is that the characteristics of matter we discover when we get down to this scale may be remarkably – and, some-times, mysteriously – different from those we experience in our macroscopic world.

Background

You will find this to be the case as you prepare nanoparticles (generally defined as particles with a diameter ≤ 100 nanometers) of gold by a relatively straightforward chemical reaction between the compound hydrogen tetra-chloroaurate [**$HAuCl_4$**; although the name is intimidating, the compound can be simply thought of as an H^{+1} and an Au^{+3} balancing four Cl^{-1}] and sodium citrate which is derived from the very familiar citric acid. Your initial objective will be to simply observe the color of the resulting gold nanoparticle system to note – as mentioned above – how different the properties of materials on this scale are from those bulk properties we are so accustomed to experiencing. You will investigate how your samples interact with light and additionally, with the assistance of a few microscopes that allow us to peer into the nano realm – an Atomic Force Microscope or AFM and a Field Emission Scanning Electron Microscope or FE-SEM. A group of scientists who work in this field will help you determine the size of the nanoparticles via an internet connection. They will also help you understand how their size affects the way these gold particles absorb visible light.

For this lab exercise, you will need the following :

- Flinn Scientific Ruby-Red Colloidal Gold Nanotechnology Demonstration Kit (Cat #AP7117 can be obtained from here).
- 100mL Beaker
- Hotplate
- Heat Protection Gloves & Safety Glasses
- Silicon Wafer or Glass Microscope Slides & Tweezers
- 100mL (or larger) Storage/Waste Bottle
- AFM sample mounting supplies (optional)
- FESEM sample mounting supplies (optional)
- Shipping Supplies

Procedure

I. Synthesize nanoparticles at your institution

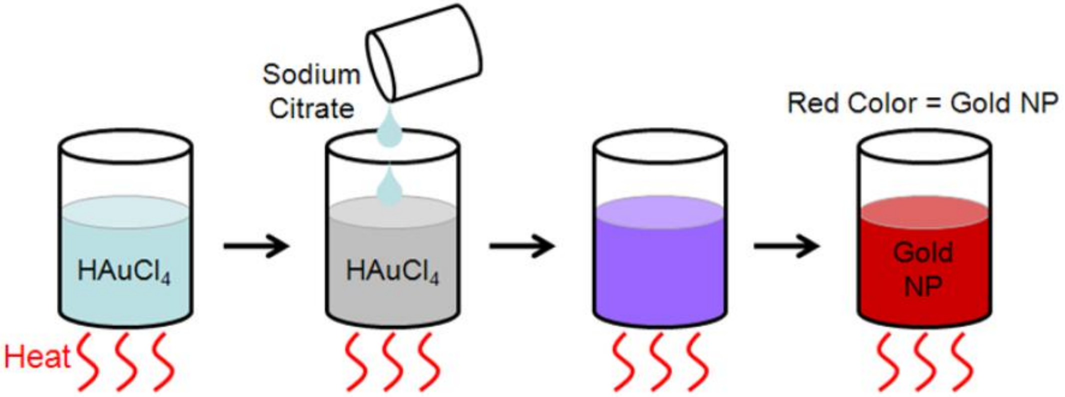
a) Bring 50mL DI water and 5mL of Hydrogen Tetrachloraurate to a boil in a 100mL beaker on a hotplate.

b) Add 0.5 mL Sodium Citrate to the boiling solution. Wait until the reaction occurs denoted by a color change. The solution will turn from colorless to red. Once the solution has turned red, remove the beaker from the hotplate and allow the solution to cool. Steps of the synthesis can also be seen here: http://education.mrsec.wisc.edu/277.htm.

II. Send samples to Remote Access centers or choose to use our in-house samples

a) You can ship the nanoparticle solution to Remote Access centers and we will prepare the samples for characterization or follow the steps in the Section III below.

b) The mailing address and contact info can be found in our web site: http://nano4me.org/.



Procedure

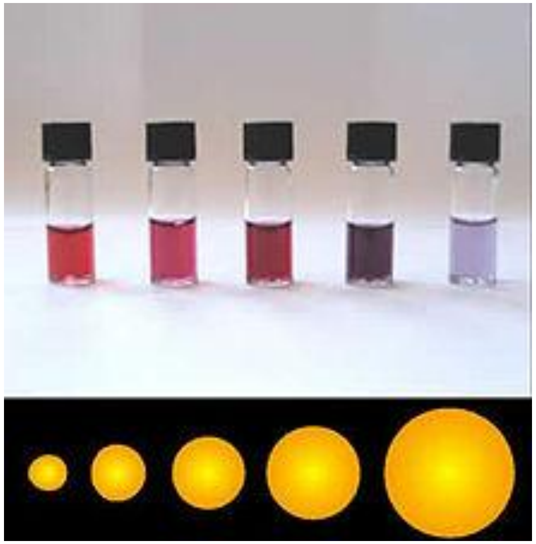
III. Prepare samples for AFM and FE-SEM analysis

a) Select a substrate material. You can adhere the nanoparticles to mica, a silicon wafer, or a microscope slide. Silicon is the best choice for FE-SEM viewing, if available.

b) Poly-L-lysine is a solution you can add prior to the nanoparticle solution to promote adhesion.

c) Simply spin coat the clean substrate (or purchase pre-coated slides) with poly-L-lysine solution.

d) Then add one microliter of the nanoparticle solution to the substrate and allow it to dry. The samples are now ready for characterization or packaging for shipping.



Procedure

IV. Perform Remote UV-Vis Spectrophotometer Analysis (Figure 1)

a) This will be performed remotely with the assistance of the Remote Access center staff.

b) The absorption peak will be determined for the colloidal samples.

V. Perform Remote AFM Characterization (Figure 2)

a) This will be performed remotely with the assistance of the Remote Access center staff.

b) Particle size and distribution will be determined.

VI. Perform Remote FE-SEM Analysis (Figure 3)

a) This will be performed remotely with the assistance of the Remote Access center staff.

b) Particle size and distribution will be determined.

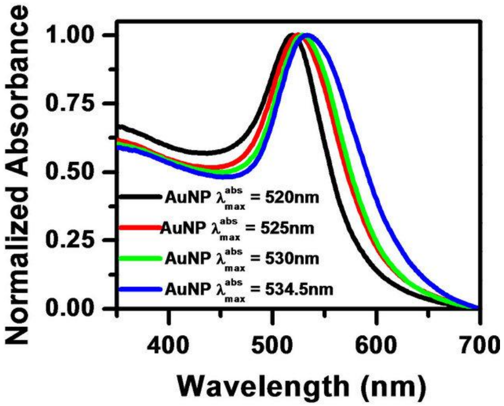


Fig. 1

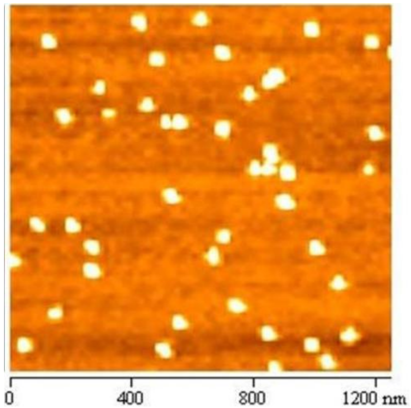


Fig. 2

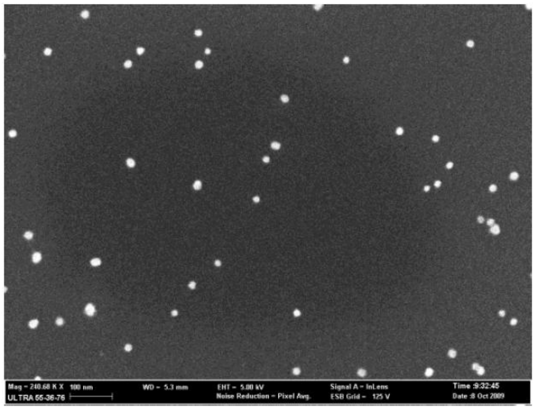


Fig. 3

Remote Access Connection Instructions

What makes these labs different and unique from other classroom experiments is that we have incorporated a section in each activity to remotely characterize your nanoscale samples from your classroom. Remote access to a variety of characterization tools can enhance the visualization of nano-related concepts by allowing students to see the effects of their work first hand. You can choose to mail your samples to our facility to be analyzed at a later date or you can use our samples that have been processed using the same procedure. Please use the following steps to successfully complete a remote session.

I. Request a remote lab session specifying pertinent information such as: the day, the time, and the instrument you are interested in using by visiting our web site http://nano4me.org/ Go to the *Educator's* tab and select the *Remote Access* tab in that section. You will see the list of partners with the instruments provided to chose from.

II. You will be contacted by a Remote Access staff member to set up a test run to ensure you are set up properly and have the required infrastructure.

III. Send samples or verify the in-house sample you would like us to prepare and load for characterization. Send your samples to the Remote Access center that you chose on your request.

IV. There are two communications soft-ware packages, that will allow us to communicate instructions and answer questions during the session.

    I. Zoom: You can obtain a free download at: https://www.zoom.us/

    II. TeamViewer: You can obtain a free download at: https://www.teamviewer.com/en/index.aspx

Remote Access Connection Instructions

V. You will need:

a) Computer with administrator access to install plug-ins and software

b) An internet connection

c) Speakers

d) Microphone

e) Projector connected to the same computer

f) Web browser (Firefox preferred)

VI. During the test run you can refer to this guide to perform the following steps, but it's very important that you only proceed with these steps during your scheduled times. You may interfere with other remote sessions and potentially damage equipment if you log in at other times.

a) Open and logon to your Zoom/Team-viewer account. You will be given the access code to enter at the time of your test and then again during the remote session.

- If you are using the Zoom software, Remote Access staff will give you the access code.
- If you are using the Team-viewer software, Remote Access staff will give you the ID & password.

b) You should soon see the Remote Access desktop and at this point you can interact with the icons on the screen as if it were your desktop.

c) Switch to full screen mode by selecting the maximize screen option in the top right corner of the screen.

d) Upon completion of the session, move your mouse to the top right corner of the screen, and click on the X to disconnect the remote session. It will ask if you want to end the remote session. Click Yes.

References and Supplemental Material

- A.D. McFarland, C.L. Haynes, C.A. Mirkin, R.P. Van Duyne, H.A. Godwin, *J. Chem. Educ. 81 (***2004)** 544A.

The Nanotechnology Applications and Career Knowledge (NACK) Center was established at the Penn State College of Engineering in September 2008 through the National Science Foundation (NSF) Advanced Technological Education program.

Please contact a NACK representative today to assist you in increasing the awareness of nanotechnology and education related opportunities across the nation. Visit our website for an expanded contact list.

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Nanotechnology Applications and
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NACK NETWORK Building College-University Partnerships for Nanotechnology Workforce Development

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