Student Guide

Spectrophotometry with Metal Nanoparticles

Safety

Students should exercise care when handling the glass cuvettes, when working around hotplates and using chemicals.

Introduction

You have a summer job as an intern at a company that is leading the research for creative uses of new materials. Your boss gives you a small jar of something she calls 'metal nanoparticles' suspended in water. After providing a little background on what they are, she asks you to use your creativity and investigate some properties of these particles.

You decide to investigate the optical properties of the nanoparticles. First, you will synthesis a sample of either gold or silver nanoparticles. You will follow this up with an investigation of the optical absorption properties of the different nanoparticle samples using a spectrophotometer.

Materials:

- Two transparencies with thin film of either gold or silver nanoparticles between them, or 2 microscope slides with thin film of nanoparticles between them
- LED Flashlights of red, green, blue, and UV
- Spectrophotometer
- Absorption curves of a range of both gold and silver nanoparticles
- Calculator
- Chemicals and associated materials to synthesize gold or silver nanoparticles. Your teacher will provide a list of what you need depending on which metal youwill be working with.

Procedure:

Part 1: Thin Film of Gold and Silver Nanoparticles - Teacher Demonstration with Student Observation & Response. Put all responses in Table 1.

1. Your teacher will show you a thin film of nanoparticles. Make observations about this film, including size, shape, color, etc.
2. Shine white light on top of the film at an angle. Note any observations.
3. Shine white light through the thin film (transmission). Note observations.
4. Repeat steps 2 & 3 for lights of various colors, including the UV light.

Development and distribution funded by the **National Science Foundation**

5. Explain your observations with your classmates.

Part 2: Synthesis of Gold and Silver Nanoparticles

Silver Nanoparticles (from https://education.mrsec.wisc.edu/synthesis-of-silver-nanoparticles-nabh4/

1. Put on goggles and wear gloves.

2. Add 30 mL of 0.002M sodium borohydride ($NaBH_4$) to an Erlenmeyer flask. Add a magnetic stir bar and place the flask in an ice bath on a stir plate. Stir and cool the liquid for about 20 minutes.

3. Carefully pour 2 mL of 0.001M silver nitrate ($AgNO_3$) into a graduated cylinder. Then, squeeze the bulb of the plastic pipette and insert the tip into the graduated cylinder. Slowly release the bulb to draw the silver nitrate into the pipette. Slowly drip this solution into the stirring $NaBH_4$ solution **at approximately 1 drop per second**. Stop stirring as soon as all of the $AgNO_3$ is added.

4. Test the presence of a colloidal suspension (silver nanoparticles) by shining a laser beam into the solution. A reflected beam indicates the presence of the nanoparticles.

5. Carefully pour your nanoparticle solution into the provided container. Label your container with: Silver Nanoparticles, your names, and the date.

6. Rinse out your glassware.

Gold Nanoparticles (from https://education.mrsec.wisc.edu/citrate-synthesis-of-gold-nanoparticles/)

1. Put on goggles and gloves.
2. Add 20 mL of 1.0mM $HAuCl_4$ to a 50 mL beaker or Erlenmeyer flask on a stirring hot plate. Add a magnetic stir bar and bring the solution just to a boil.
3. To the boiling solution, add 2 mL of a 1% solution of trisodium citrate dihydrate, $Na_3C_6H_5O_7 \cdot 2H_2O$. The gold solution gradually forms as the citrate reduces the gold (III). Use forceps to remove from heat when the solution has turned deep red (about 10 minutes).
4. Test the presence of a colloidal suspension (gold nanoparticles) by shining a laser beam into the solution. A reflected beam indicates the presence of the nanoparticles.
5. Carefully pour your nanoparticle solution into the provided container. Label your container with: Gold Nanoparticles, your names, and the date.
6. Rinse out your glassware.

Development and distribution funded by the **National Science Foundation**

Part 3: Spectrophotometry of Gold and Silver Nanoparticles

1. Turn on your spectrophotometer at least 20 minutes before beginning this experiment.
2. On the top line of Data Table 2, indicate which type of nanoparticles you are analyzing.
3. If you are analyzing SILVER nanoparticles, set your first wavelength to 350 nm using the wavelength control knob. If you are analyzing GOLD nanoparticles, set your first wavelength to 450 nm using the wavelength control knob. Adjust the amplifier control knob to produce 0% transmittance.
4. In a CLEAN test tube (no fingerprints!) add about 3 mL of distilled water. Place the test tube in the sample holder and close the cover. Adjust the light control knob until the spectrophotometer reads 100 %T. Remove this test tube from the holder.
5. In another CLEAN test tube, add about 3 mL of your nanoparticle solution. Insert this test tube into the holder and record the %T. Remove this test tube from the holder.
6. Adjust the wavelength to 20 nm greater than your previous one. Use the amplifier control knob to adjust the transmittance to 0%T.
7. Place the water-filled test tube into the holder and adjust the light control knob to produce 100%T. Remove this test tube from the holder.
8. Place your nanoparticle sample into the holder and record the transmittance. Remove this test tube from the holder.
9. Repeat steps 6 – 8 (set the new wavelength, adjust to 0%T with nothing in the holder, place the water in the holder and adjust to 100%T, remove the water and place the nanoparticle sample in the holder and record the %T) until you have collected a total of 16 values.
10. When finished, return your nanoparticles to your storage bottle.

Cleanup: There should be minimal cleanup for this activity. All test tubes containing silver and gold nanoparticles should be emptied into the appropriate storage container provided by your instructor. In the event of a spill or breakage of a cuvette containing gold/silver nanoparticles, refer to the MSDS for cleanup and disposal.

Development and distribution funded by the **National Science Foundation**

Record your Observations:

Table 1. Observations of Thin Film Nanoparticles

Observations of the Thin Film		
Color of Light	**Light shined on top of film**	**Light shined from behind film (transmission)**
White		
UV		
Red		
Green		
Blue		

Table 2. Spectrophotometry of Gold/Silver Nanoparticles

	Quantum Dot 1		**Quantum Dot 2**		**Mixed Quantum Dots**	
Wavelength, nm	**A**	**%T**	**A**	**%T**	**A**	**%T**
450						
470						
490						
510						
530						
550						
570						
590						
610						
630						
650						
670						

Development and distribution funded by the **National Science Foundation**

Analyze the Results:

1. Create a nice graph of %T vs. wavelength. Use Excel or Google Sheets. Be sure to put a title and appropriate labels on your graphs.

2. Use the relationship below to create a graph of absorbance vs. wavelength.

$$A = 2 - \log(\%T)$$

3. At what wavelength is your peak %T?__________________
   At what wavelength is your peak A?___________________
4. Absorption spectrum graphs from three silver standards of silver and three gold standards are provided on the next page. Each of these represents a distinct size of nanoparticle. Compare your graph to these standards to estimate the size of nanoparticles you made. You may have to interpolate or extrapolate.

5. Record your estimate here: ____________________

6. Perform some research on the Internet to find three applications of the nanoparticles you created. Be sure to explain how they are used in these applications.

Optional: Your teacher may request that you answer the extension questions:

1. What, if any, is the significance of the *width* of the peak absorption graph?
2. How does this activity relate to the photoelectric effect?
3. Suppose a scientist were to transmit blue light through a sample of quantum dots that emitted both red and green light. What would be produced? *Combining blue, red and green produces white light.*
4. Given the (correct) results of the previous question and your knowledge of light, what are some creative uses of quantum dots, gold and silver nanoparticles? Research these. *Creative uses include imaging, electronic displays (TV's, tablets, phones, computer monitors), solar cells, etc.*

Development and distribution funded by the **National Science Foundation**