Student Guide

Part 1: Silver Nanoparticle Synthesis and Spectroscopy

Introduction: In this lab you will synthesize silver nanoparticles one of the most commonly used nanoparticles found in consumer products. Nanoparticles are made of thousands of atoms of an element and are extremely small, ranging from1 to 100 nanometers (nm) in size. A nanometer is 1 billionth of a meter or $1x10^{-9}$. Nanoparticles often have different properties than those associated with the element at the macroscale. For example, silver, gold, and copper interact with light differently at the nanoscale which in turn affects the color. Nanoscale silver has been used as an antibacterial agent, and can even kill harmful strains of bacteria which are resistant to antibiotics. Silver nanoparticles have been used in the dressing of wounds, surgical masks, food packaging, water treatment, and even socks which prevent the growth of bacteria that cause foul odor.

Silver Nanoparticle Activity:

Materials:

For Synthesis:

- 0.1M $AgNO_3$ ($AgNO_3$ CAS 7761-88-8)
- 0.1M α-D-glucose (α-D-glucose CAS 50-99-7)
- 0.2% wt. soluble starch (soluble starch CAS 9005-84-9)
- 200 µL pipet
- 1000 µL pipet
- 10 mL pipet or graduated cylinder
- Hot plate
- Small Erlenmeyer flask or beaker
- Glass vials

For Spectroscopy:

- Droppers
- Spectrophotometer
- Cuvettes
- Kimwipes
- 0.2% wt. soluble starch for blank

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Procedure

Part 1. Synthesis of Ag Nanoparticles:

1. Place 200 µL of 0.1M $AgNO_3$ into a small Erlenmeyer flask or beaker.
2. Add 500 µL of 0.1M glucose, making sure that it comes into contact with the $AgNO_3$.
3. Invert the starch solution several times. Add 10mL of the starch solution.
4. Heat the solution on a hot plate on a high setting until it is boiling vigorously. **Do not stir the solution!**
5. Boil the solution for 10 minutes. The solution should turn yellow.
6. Remove the sample from the hot plate, and let it cool.

Part 2. Spectrophotometric Analysis of Ag Nanoparticles

1. Turn on the spectrophotometer and allow it to warm up for the recommended length of time.
2. Put the instrument in Transmittance mode. With the sample compartment empty, set the dark current to zero.
3. Invert the starch solution several times. Fill a clean cuvette with the starch solution. Wipe the outside with a Kimwipe, then insert it into the sample compartment.
4. Set the wavelength at 350nm and zero the absorbance.
5. Insert a clean cuvette filled with the AgNP solution, and record the absorbance in the data section in Table 1.
6. Repeat steps 4 and 5 for different wavelengths; increasing the wavelength by 25nm each time until a reading is obtained for 650nm. Remember to zero the machine each time you change the wavelength. Record the data in **Table 1** of the data section.
7. Identify the wavelength from Table 1 with the highest absorbance. Use the spectrophotometer to collect additional absorbance readings by varying the wavelengths in that specific area of the spectrum to determine which gives the wavelength of maximum absorbance (λ_{max}). Record these observations in **Table 2** of the data section. (For example, if the maximum absorbance in Table 1 is at 500 nm, then test the absorbance of the AgNPs at 490, 495, 500, 505, and 510 nm, looking for the wavelength that gives the maximum absorbance).
8. Divide the absorbance at the λ_{max} by 2 and record in **Table 3** of the data section as absorbance at ½ max.
9. Graph the data from Table 2 on a separate sheet of graph paper or in Excel.
10. Using the graph created in step 9, mark the maximum absorbance on the y-axis. Mark the ½ max. absorbance on the y-axis and find the corresponding wavelength on the x-axis.
11. Zero the spectrophotometer at the interpolated $\lambda_{1/2max}$ from step 10. Compare the absorbance of the AgNPs at the interpolated $\lambda_{1/2max}$ to the absorbance calculated in step 8. Adjust the wavelength on the spectrophotometer and record the absorbance of the AgNPs until a value close to the calculated value of the absorbance at $\lambda_{1/2max}$ from step 8 is obtained. Record this wavelength in **Table 3** as wavelength at ½ max.

Calculations:

1. To determine the Peak Width at Half Max (PWHM), first subtract the λ_{max} from the $\lambda_{1/2max}$. The difference is half the peak width. Multiply by 2 to get the PWHM.

$$PWHM = (\lambda_{1/2max} - \lambda_{max})*2$$

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2. How many silver atoms are in one nanoparticle?
   Determine the average number of atoms per nanoparticle using the following formula:

$$N = \frac{\pi \rho D^3}{6 \; M} N_A$$

N = number of atoms per nanoparticle
ρ = density of face centered cubic (fcc) silver = 10.5g/cm^3 (convert to g/nm^3)
D = average diameter of nanoparticles = 11nm
M = atomic mass of silver
N_A = number of atoms per mole
Note: This equation is taken from Liu, Atwater, Wang, and Huo (see References.) It assumes that the nanoparticles have a spherical shape and a uniform fcc crystaline structure. **Be sure your units are consistent!**

3. What is the concentration of your silver nanoparticle solution?
   Determine the molar concentration of the nanoparticle solution using the following formula:

$$C = \frac{N_T}{N V N_A}$$

C = molar concentration of nanoparticle solution
N_T = Total number of silver atoms added in $AgNO_3$
N = number of atoms per nanoparticle (determined in Calculation 2)
V = volume of the reaction solution in L
N_A = number of nanoparticles per mole

4. What effect might a large variance in particle size have on the width of the absorbance peak in the visible spectrum?

5. Name a physical property of silver that changes at the nanoscale.

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Data:

Table 1

Wavelength (λ) (nm)	Absorbance
350	
375	
400	
425	
450	
475	
500	
525	
550	
575	
600	
625	
650	

Table 2

Wavelength (λ) (nm)	Absorbance
412	
418	
422	
428	
480	
483	
481	

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Table 3

	Wavelength (nm)	Absorbance
λ_{max}	425	
$\lambda_{1/2max}$	480	

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