Student Guide (With Answers)

Title: ***Synthesis and Stability of Silver Nanoplates***

Introduction:
'Nano' means small, very small; But, why is this special? At the nanometer scale, the properties of matter, such as optical, magnetic and electrical properties, change as function of size and shape. This is a direct consequence of the small size of nanomaterials, physically explained as quantum confinement effects. The consequence is that a material (e.g. a metal) when in the nanometer range can assume properties, which are very different from those when the same material is in a bulk form. For instance, bulk silver is non-toxic, whereas silver nanoparticles (AgNPs) are capable of killing viruses upon contact. Properties like electrical conductivity, color, strength, and weight can change when the nanoscale level is reached.

Silver nanoparticles can take the shape of cubes, spheres, bars, wires, bipyramids, beams, plates, and discs depending upon the seed it forms from. As long as the nanostructure retains its respective shape, it will retain its unique optical properties. If the particle loses shape, or stability, it will lose its exhibited properties.

The distinct properties of nanoscale silver have afforded its use in many everyday items. Silver nanoparticles can be used as biological tags for quantitative detection and in biosensors. It's used as an antibacterial/antifungal agent, and can kill harmful strains of bacteria, which are resistant to antibiotics. Some of these products have been incorporated in apparel, footwear, paints, appliances, cosmetics, food packaging, and plastics. There are several medical uses of silver nanoparticles, which include the dressing of wounds, surgical masks, and surgical instruments. Silver nanoparticles are used to efficiently harvest light and for enhanced optical spectroscopies including metal-enhanced fluorescence (MEF) and surface-enhanced Raman scattering (SERS).

Objective: The purpose of this experiment is to conduct synthesis of silver nanoplates and explore their shape stability that may affect optical property, referred to as localized surface plasmon resonance (LSPR). By completing this activity, you will learn the differences in physical properties and behavior at the nanoscale as compared to the same materials at the macroscale.

Materials:
For Synthesis
- 0.01M Silver nitrate ($AgNO_3$ CAS 7761-88-8)
- 0.001M Poly(vinyl pyrrolidone) (PVP-29K)
- Ethanol (EtOH) (200 proof)
- 20mL vial
- (4) 100mL vials
- Aluminum foil
- Pipettes (1mL, 10mL)
- Conventional oven to perform solvothermal reaction at 80^{o}C

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- Acid digest reaction vessel (used Parr Instrument Co. vessel # 4745)
- Oven mitt
- Googles
- Nitrile/vinyl gloves
- Plastic container to hold vessel

For Stability

- Prepared Ag nanoparticles
- Cuvettes
- 500 mL tap water
- Droppers

For Spectroscopy

- Spectrometer
- Cuvettes
- Ethanol (EtOH) (200 proof)
- Droppers

Procedure:

Part 1. Solvothermal Synthesis of Ag Nanoplates

1. Place 0.3mL of fresh 0.01M $AgNO_3$ into a Teflon liner.
2. Add 12mL of 0.001M Poly(vinyl pyrrolidone) to the same Teflon liner.
3. Use a pipette to mix the solutions in the liner.
4. Seal the liner and place it in the stainless steel reaction vessel.
5. Set the oven to 80^{o}C. Place the stainless steel vessel in the oven and heat for 5 hours.
6. Using an oven mit, remove the vessel after 5 hours have expired.
7. Place the vessel into a plastic container and run water over it to allow the vessel to cool to room temperature for about 15-20 minutes.
8. Remove the solution from the Teflon liner and place in a 20mL vial.

Part 2. Stability of Ag Nanoplates

1. Place 500mL of the Ag nanoplates in a cuvette.
2. Observe the color and record results in the chart below
3. Add 500mL of tap water to the same cuvette.
4. Observe the initial color change of the solution at 0 minutes and record results.
5. Observe the color change of the solution at the 30-minute mark and record the results.
6. Observe the color change of the solution at the 60-minute mark and record the results.

Part 3. Spectroscopy of Ag Nanoplates (Cary 50 UV-Vis Spectrophotometer)

1. Launch the "Scan" program from the desktop or from the "Start" button.
2. The spectrophotometer should make a buzzing noise. Click "Setup" on the left bar and the tab "Cary" should be opened. Under the "X Mode" specify the wavelength (nm) range of the scan.

3. Under "Scan Controls" chose the speed of the scan (Usually "Medium" is preferable).
4. Click on the "Baseline" tab and select "Baseline correction" under the "Correction" section.
5. Click "OK" and the "Baseline" on the left bar. It should give you a window.
6. Prepare a blank sample: retrieve a new cuvette and fill to halfway point with ethanol.
7. Slide open latch to UV-Vis Spectrophotometer and place cuvette in holder. The side with the arrow should face east. Close the latch.
8. Return to the computer and press OK. The spectrophotometer should start scanning the blank sample.
9. Once finished remove the cuvette. Using the pipette withdraw 500mL of the freshly synthesized Ag nanoplates from the 20mL vial and disperse into another cuvette. Use the top of the pipette to mix the sample until homogeneous. Try to refrain from leaving droplets along the side of the cuvette.
10. Place the sample into the spectrophotometer again and close the latch.
11. Click the "Start" button at the top and name your file. Click "Save" and then "OK".
12. The scanning should begin and you will receive your spectrum. Another window will popup asking for a "Sample Name". Record results.
13. Add 500mL of tap water to the cuvette containing 500mL of the Ag nanoplates. To scan the new sample, replace the cuvette and click "OK". Repeat steps 10-12. Record results.
14. Allow the cuvette of 500 mL Ag nanoplates and 500mL of tap water to sit for 2 hours. Rescan the solution following steps 10-12 again. Once you are done, click "Finish". Record results.

Data:

TIME (MIN.)	**COLOR**
0 minutes Ag nanoparticles only	
0 minutes with tap	
30 minutes with tap	
60 minutes with tap	

Spectroscopy

TIME (MIN.)	**WAVELENGTH (NM)**	**ABSORBANCE**
0 minutes Ag nanoparticles only		
0 minutes with tap		
120 minutes with tap		

<u>Analysis</u>

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

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2. Given the observed color changes over the course of 60 minutes, did the Ag nanoparticles experience a blue shift or a red shift change? Does the shift indicate an increase or decrease in the wavelength?

3. If the observed shift trend witnessed in your results continues from where your observations ended, what color do you predict would be observed next?

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