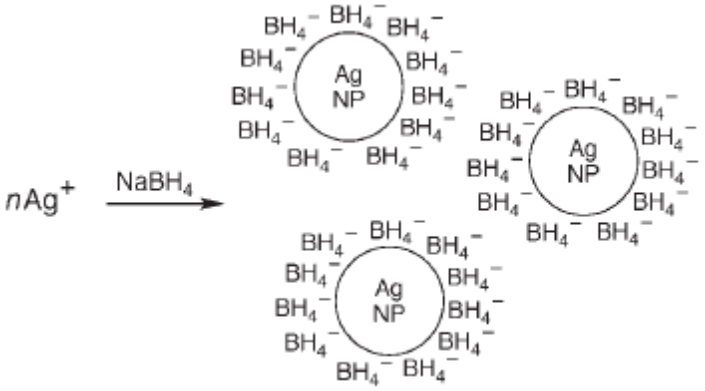
SYNTHESIS OF SILVER NANOPARTICLES (Ag NPs)

(Experiment)

Introduction:

Silver nanoparticle (Ag NP) is the first nanomaterial to be exploited commercially for various applications. It can be synthesized under laboratory conditions by sodium borohydride reduction method. In this method, AgNPs are formed by reduction of precursor $AgNO_3$ using sodium borohydride as a reducing agent and SDS as stabilizing agent. The chemical reaction that takes place is mentioned below:

$$AgNO_3 + NaBH_4 \rightarrow Ag + \tfrac{1}{2}H_2 + \tfrac{1}{2}B_2H_6 + NaNO_3$$



(Source: Journal of Chemical Education. Vol.84, No.2, 2007)

The repulsive forces between like charges prevent the formed nanoparticles from aggregation.

Chemicals required:

Silver Nitrate ($AgNO_3$), sodium borohydride ($NaBH_4$), sodium dodecyl sulfate (SDS), distilled water.

Synthesis of AgNPs by $NaBH_4$ reduction method:

1. Take 1mL distilled water in a micro centrifuge tube.

2. Add $AgNO_3$ (0.1M) to 1.5 mL micro-centrifuge tube.

3. Immediately add freshly prepared (2mg/mL) $NaBH_4$ solution to it.

4. Then add SDS solution (0.02%) to the above solution.

5. Agitate the micro-centrifuge tube containing all the components vigorously.

6. Appearance of yellow color is indicative of the formation of silver nanoparticles.

7. Check the presence of Surface Plasmon Resonance (SPR) band by UV- visible Spectroscopy, stability of the formed nanoparticles by measuring the Zeta Potential and the size can be determined by Transmission Electron Microscope (TEM) analysis.

The schematic representation of the entire procedure is shown in figure1.

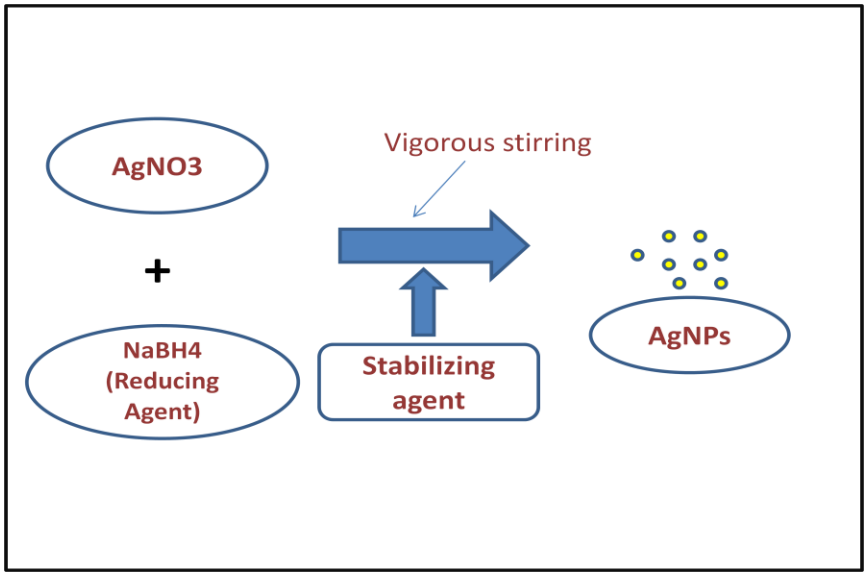


Figure1: Schematic representation of synthesis of silver nanoparticles.

Characterization of AgNPs:

1.UV-Vis Spectroscopy:

Double click the "UV-VIS Analyst" software (Lasany UV-VIS LI-2800 double beam spectrophotometer, Germany).

⇩

Set scanning range for the sample as 200-600 nm at a scan speed of 480 nm/min.

⇩

Add 1mL distilled water to each cuvette and place inside chamber for blank calibration.

⇩

Go to UV Photometer icon. Select "Calibrate system baseline".

⇩

Select "Automatic blank calibration".

⇩

Replace one of the blank reference cuvette with 1ml Silver nanoparticle solution.

⇩

Click on the "red color" start icon to record absorbance.

⇩

Silver Nanoparticles show a characteristic absorbance between 380-430nm.

Particle Size and Spectral Features of Ag Nanoparticles

Particle Size/nm	λ_{max}/nm
10–14[a]	395–405
35–50[b]	420
60–80[c]	438

Source: Journal of Chemical Education. Vol.84, No.2, 2007

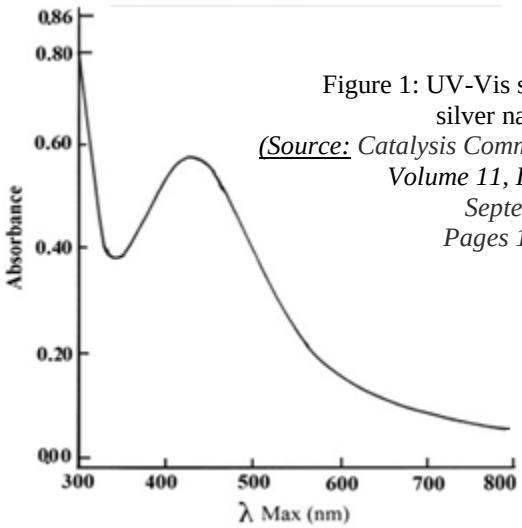


Figure 1: UV-Vis spectrum of silver nanoparticles *(Source: Catalysis Communications Volume 11, Issue 15, 25 September 2010, Pages 1233–1237)*

2. Zeta Potential:

The zeta potential of the silver nanoparticles can be measured according to the following procedure:

Double click the "DTS NANO Software" (Zetasizer nano-ZS90 series, Malvern Instruments Pvt. Ltd., Germany).

⇩

Add 1 mL of silver nanoparticle solution in a clear disposable zeta cell and place it inside the chamber at 25^0C.

⇩

Select "Zeta potential" and "Manual measurement" mode in the software.

⇩

Select "File" and click on "Save as" to save measurement file.

⇩

Click Measure and set parameters- '3' rounds of measurements; '0' equilibration time.

⇩

Press 'Green color' start button to measure zeta potential.

⇩

Zeta potential value of silver nanoparticles close to -30.0mV indicates good stability.

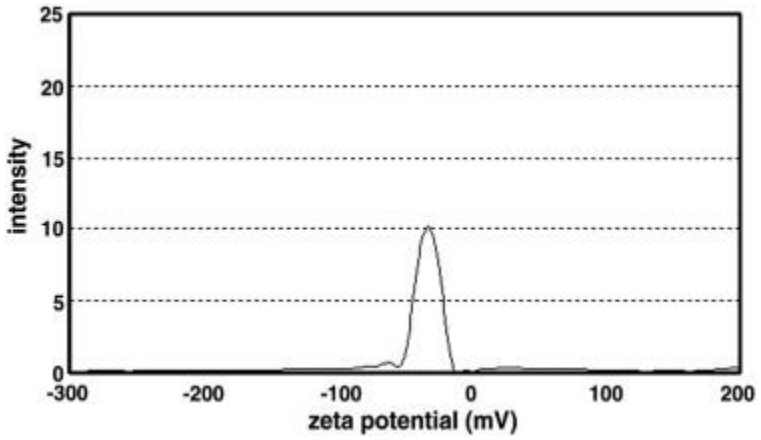


Figure 2: Surface zeta potential of Ag NPs. The maximum potential peaks of Ag NPs were measured at −32.76 mV. *(Source: Nanomedicine: Nanotechnology, Biology and Medicine Volume 6,Issue 4, August 2010, Pages 570–574)*

3. X-ray Diffraction (XRD) Analysis:

Silver nanoparticles can be characterized based on the diffraction pattern obtained by XRD analysis.

Procedure: For Liquid Samples

1. Take 1x1mm glass slide.
2. Put few drops of the nanoparticle solution on the glass slide and keep it for drying in air.
3. Repeat step 2 for 10-15 times.
4. Analyze the sample using THIN FILM XRD.

*Liquid samples can be dried to form powder by rotary evaporation under reduced pressure or by freeze drying method.

Procedure: For Powder Samples

1. Take small amount of finely grounded powder(sample)
2. Place it on the sample holder and analyze it using POWDER XRD.

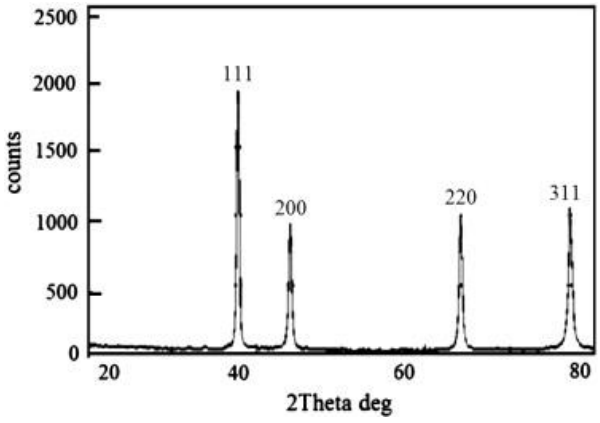


Figure 3: XRD pattern of Ag NPs (*Source: Catalysis Communications Volume 11,Issue 15, 25, 2010, Pages 1233–1237)*

4. Scanning Electron Microscopy (SEM) Analysis:

The morphology of the formed silver nanoparticles can be determined using SEM.

Procedure:

1. Place a small volume of the sample on a glass slide.
2. Stick a double sided conducting tape to the bottom of glass slide and place the entire arrangement on the aluminum stub.
3. Then subject it to gold sputtering and view it under the electron microscope.

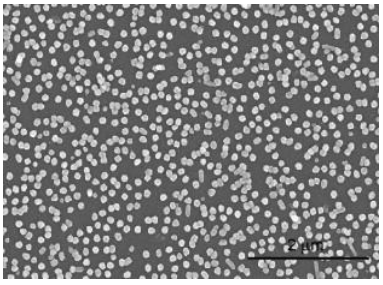


Figure 4: SEM image of AgNPs *(Source: Thin Solid Films Volume 516, Issue 6,2008,Pages 953–956)*

5. Transmission Electron Microscopy (TEM) Analysis:

The average particle size of the Ag crystallite can be determined by TEM.

Procedure:

1. Place a small volume of the diluted sample on the non-shinning side of the carbon coated copper grid.
2. Air dry the sample.
3. Observe the dried sample under TEM microscope.

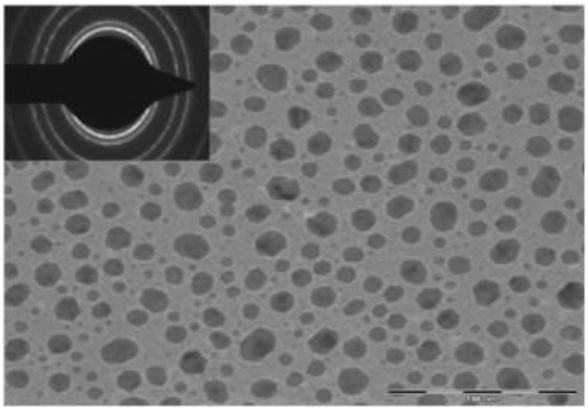


Figure 5: TEM image of Ag NPs. *(Source: Catalysis Communications Volume 11, Issue 15,25,2010, Pages 1233–1237)*

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