

LAB MANUAL: EXPERIMENT 7

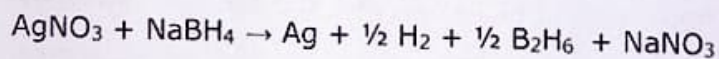
Aim: To synthesize Silver Nanoparticles by NaBH_4 reduction method.

Theory:

Nanomaterials are materials that typically have length scales on the order of 1-100 nm in at least one dimension. Examples include nanoparticles, nanowires, and thin films. The observed properties of materials change as their sizes approach the nano-scale. An easily observable example is the color change between bulk-scale and nano-scale materials. One historically relevant case is the production of gold-stained glass in medieval architecture. Artisans would create red glass by mixing in small amounts of nanoscale gold particles during the manufacturing process, resulting in a ruby color.

Silver nanoparticles (AgNPs) are another widely studied nanomaterial. AgNPs are known for their antibacterial properties, which is of interest in the health industry, textiles, food storage, and several environmental applications. The electrochemical properties of AgNPs are also useful for the development of faster and more sensitive nanoscale sensors. As you will see in this lab, silver nanoparticles can be used to create yellow glass.

This lab analyzes a colloidal silver solution synthesized via chemical reduction using silver nitrate and sodium borohydride, which is given by the following chemical equation:



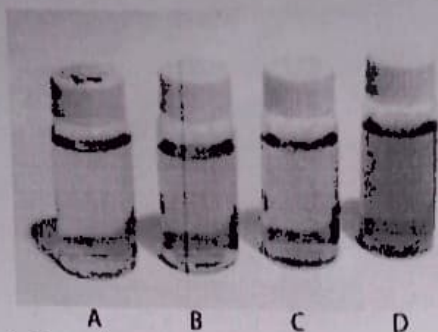


Figure 1. Colloidal silver in various stages of aggregation, (A) clear yellow, (B) dark yellow, (C) violet, and (D) gray solutions, as aggregation proceeds from a colloidal to aggregated solution.

As AgNPs begin to form, the solution turns yellow. As the particles aggregate together to form larger and larger particles, the solution will begin to turn violet and eventually gray (Figure 1). To prevent aggregation, the silver nanoparticles can be stabilized, i.e. kept from precipitating out of solution, by a protective layer of borohydride ions. Particles can also be stabilized with a polymer coating such as polyvinyl pyrrolidone (PVP), EDTA etc.

Requirements:

EDTA ($\text{Na}_2\text{H}_2\text{Y} \cdot 2\text{H}_2\text{O}$), AgNO_3 solution (0.01M), NaOH solution, NaBH_4 solution (0.005M), Pipette, conical flask (100 mL), Beaker (200 mL), measuring cylinder.

Procedure:

1. Take 20 mL of 0.01 M of EDTA solution in a 100 mL Erlenmeyer flask.
2. Add 1 mL 0.01 M AgNO_3 solution.
3. Maintain the pH of the solution at 11 by using NaOH solution dropwise.
4. Add 3 ml 0.005 M NaBH_4 solution.
5. The solution becomes yellow indicating the formation of silver nanopartilces.

6. Measure absorption of the diluted colloidal silver solution over a wavelength range of 300 to 520 nm with a gradual increment of 10-20 nm in the operating wavelength each time.
7. Plot the absorbance vs wavelength and find the wavelength at which maximum absorption is obtained.

| Silver nanoparticle solution | | | |
|------------------------------|------------------|--------------|-------|
| λ nm | A | λ nm | A |
| 300 | 1.041 | 420 | 1.635 |
| 310 | | 430 | |
| 320 | 0.631 | 440 | 1.317 |
| 330 | 0.687 | 450 | |
| 340 | 0.687 | 460 | 1.012 |
| 350 | 1.012 | 470 | |
| 360 | | 480 | 0.584 |
| 370 | | 490 | |
| 380 | 1.352 | 500 | 0.579 |
| 390 | | 510 | |
| 400 | 1.665 | 520 | 0.574 |
| 410 | | | |

Result:

- ❖ The absorbance was recorded with a gradual increment of20..... nm in the operating wavelength each time.
- ❖ The wavelength at which maximum absorption was obtained is found to be.....400.nm.