## ***EXPERIMENT NO 2***

## ***Synthesis of nano powders by Chemical reduction***

### ***Objective:***

To synthesize cuprous oxide nanoparticles (NPs) through chemical reduction method

### ***Basic Theory:***

- Interest in cuprous oxide (Cu2O) nanoparticles with defined size and shape arise from its useful optical and electronic properties.
- Several researchers have synthesized a variety of micro and nanostructures of Cu2O, such as nano cubes, octahedra, nanocages, hollow spheres, nanowires, and other highly symmetrical structures.
- A number of documents and reports on the preparation methods of nano-$Cu_2O$ exist due to its broad application prospects.
- Chemical reduction in aqueous media is a standard synthesis route for the production of silver, copper-silver core-shell or Cu2O nanoparticle.

### ***Raw Materials:***

- $CuSO_4.5H_2O$
- 6g polyethylene glycol (capping agent)
- 0.4g sodium borohydride ($NaBH_4$) (reducing agent)
- 0.9g ascorbic acid (antioxidant)
- 0.4g sodium hydroxide (NaOH) (to adjust pH of the solution)
- Deionized water (500 ml)
- Burette Stand
- Magnetic Stirrer
- Weighing machine

### ***Procedure:***

1. Dissolve 1.25 g of CuSO4, 5H2O in 50 ml deionized water
2. Then 6 g of PEG is dissolved in 50 ml deionized water, and the solution is added in the copper sulfate solution.
3. Next, add
   - ascorbic acid (0.9 g)
   - NaOH (0.4 g)

   in 100 ml deionized water, and the resulting solution is added to the previous solution.
4. Finally, aqueous solution (50 ml) of 0.4g of $NaBH_4$ is dissolved in 50 ml deionized water for reducing agent

5. The dark red solution is left to cool for the whole night. The next day, the solution is centrifuged.
6. Finally, the precipitate is used for further characterization.

***Synthesis mechanism:***

In aqueous solution, the reaction takes place as

$$4Cu^{2+} + BH_4^- + 8OH^- = 4Cu + B(OH)_4^- + 4H_2O$$
$$4Cu + O_2 = 2Cu_2O$$

- Initially, the $Cu^{2+}$ have been compounded with PEG
- Then $Cu^{2+}$ reacted with $BH4^-$ ion, and Cu particles have been generated.
- The solution has been kept under ambient atmosphere and the oxidation has been qualitatively monitored with time by observation its color change.
- Cu oxidized to $Cu_2O$ by oxygen in air and colloidal suspension of $Cu_2O$ nanoparticles form.

***Lab Deliverables:***

1. What are the advantages and disadvantages of wet-chemical methods?

Sol:

Advantages:

- Controllable particle size and morphology: The particle size and morphology of nanoparticles can be controlled by adjusting the reaction conditions, such as temperature, pH, and concentration of reagents.
- High purity: Wet-chemical methods can produce nanoparticles with high purity, which is important for many applications.
- Scalability: Wet-chemical methods can be scaled up to produce large quantities of nanoparticles.

Disadvantages:

- Complexity: Wet-chemical methods can be complex and require careful control of reaction conditions.
- Sensitivity to impurities: Wet-chemical methods can be sensitive to impurities, which can affect the properties of the nanoparticles.
- Environmental impact: Some wet-chemical methods use hazardous chemicals, which can have an environmental impact.

2. List different methods to synthesize nano powders.

Sol:

- Sol-gel: The sol-gel method involves the hydrolysis and condensation of metal alkoxides to form a sol, which is then dried to form a gel. The gel is then calcined to form the nanoparticles.
- Co-precipitation: Co-precipitation involves the simultaneous precipitation of two or more metal salts from solution. The precipitate is then filtered and washed to remove impurities.
- Hydrothermal synthesis: Hydrothermal synthesis involves the reaction of metal salts in a sealed vessel at high temperature and pressure. This method can be used to produce nanoparticles with unique morphologies.
- Aerosol spray pyrolysis: Aerosol spray pyrolysis involves the spraying of a solution of metal salts into a hot flame. The solvent evaporates and the metal salts decompose to form nanoparticles.

3. How to get different particle size through wet chemical methods?

Sol:

- Concentration of reagents: The concentration of reagents can affect the rate of nucleation and growth of nanoparticles.
- Temperature: The temperature can affect the rate of hydrolysis and condensation of metal alkoxides.
- pH: The pH of the solution can affect the stability of colloids.
- Addition of surfactants or additives: Surfactants and additives can be used to control the growth of nanoparticles.

4. Distinguish heterogeneous and homogeneous precipitation.

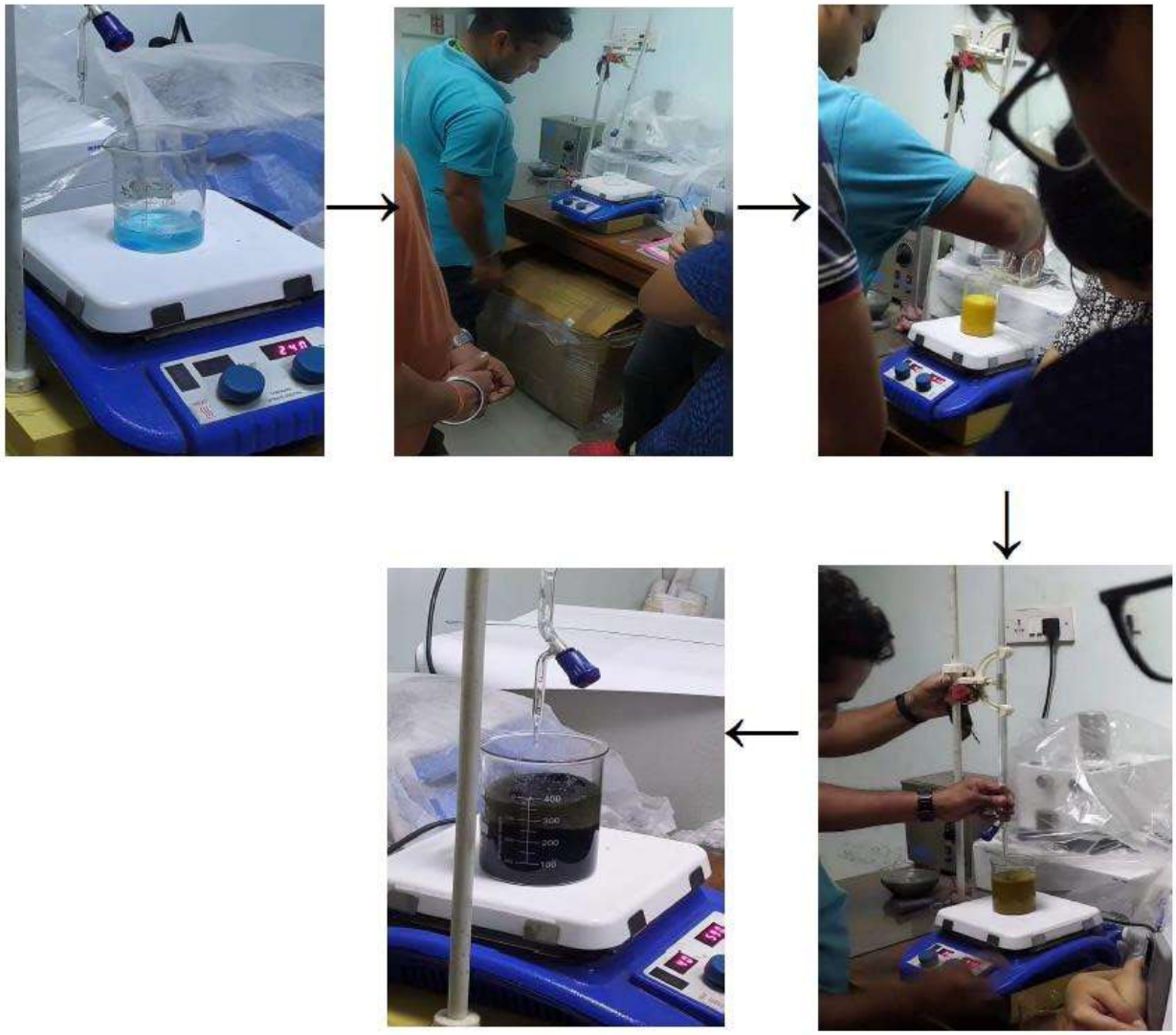
Sol:

- Heterogeneous precipitation involves the reaction of two or more substances that are in different phases, such as a solid and a liquid. For example, the precipitation of silver chloride from a solution of silver nitrate and sodium chloride is a heterogeneous precipitation reaction.
- Homogeneous precipitation involves the reaction of two or more substances that are in the same phase, such as a liquid and a liquid. For example, the precipitation of barium sulfate from a solution of barium chloride and sodium sulfate is a homogeneous precipitation reaction.

| ***Feature*** | ***Heterogeneous Precipitation*** | ***Homogeneous Precipitation*** |
|---|---|---|
| Phase of reactants | Different phases | Same phase |
| Nucleation | Occurs at the interface between the two phases | Occurs throughout the solution |
| Particle size | Typically larger | Typically smaller |

***Observations:***

Color change pattern observed:



The color first changed from blue to clear on addition of polyethylene glycol and then slowly turns yellow to dark greenish black on drop by drop addition of $NaBH_4$ solution

***Conclusion:***

The experiment involves the reaction of copper sulfate, polyethylene glycol, ascorbic acid, sodium hydroxide, and sodium borohydride in aqueous solution to form a dark red solution of cuprous oxide nanoparticles.