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COPPER NANOPARTICLES (CuNPs): SYNTHESIS AND CHARACTERIZATION

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ABSTRACT

Nanotechnology is one of the most important and emerging technology these days which deals with understanding and control of matter at nano-scale. As, in the recent years, metal nanoparticles were highly used in diverse areas like in the fields of biology, chemistry and medicine, due to their unique physical, biological and chemical properties. So, now days a number of metal nanoparticles have been synthesised and characterized. There are several methods to make metal nanoparticles, the major techniques being used were chemical methods like chemical reduction, micro-emulsion, electrochemical and biological synthesis. In this context, the present paper, here, discuss, in detail the synthesis of copper nanoparticles by chemical reduction of copper sulphate with sodium hypophosphite in ethylene glycol, in the presence of a polymer surfactant polyvinylpyrrolidone (PVP). PVP was being included to prevent congregation and give dispersion stability to the resulting colloidal nanoparticles. The characterization then has been performed by using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) techniques.

Keywords- Copper Nanoparticles (CuNPs), Synthesis, Characterization, Scanning Electron Microscopy Technique, X-ray Diffraction

[1] INTRODUCTION

A keen interest has been developed in the past few years on the synthesis of metal nanoparticles due to their wide applications in diverse fields like biology, chemistry and medicine, as they have unique physical, biological and chemical properties [1-7]. Various methods are now known and available which enable one to prepare these nanoparticles with specific size and shape, are thermal decomposition, mechanical attrition, metal vapour deposition, electrochemical reduction, radiolytic reduction, and chemical reduction methods. Among these methods, the chemical reduction method is one of most used and simple method for the preparation of metal nanoparticles [8].

Copper is now days, one of the most widely used materials all over the world. It has a great importance in industries, majorly in the electrical sector due to its cheap cost. Copper nanoparticles have been synthesized and characterized using various methods. Stability and

reactivity are also being two most important factors that impede the use and development of the metal cluster in a new and smart generation of nano-electronic devices [9-11].

The present paper describes and discusses the synthesis of copper nanoparticles by chemical reduction method *via* solution chemistry. Chemical reduction of copper sulphate has been done using sodium hypophosphite in ethylene glycol, in the presence of a polymer surfactant polyvinylpyrrolidone (PVP). PVP was used to prevent congregation and give dispersion stability to the resulting colloidal nanoparticles. The characterization then has been done by using techniques like X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

[2]EXPERIMENTAL WORK

Material:
Polyvinylpyrrolidone (PVP, K-30), sodium hypophosphite monohydrate ($NaH_2PO_2 \cdot H_2O$), copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), ethylene glycol, and 2-(2-butoxyethoxy) ethanol were all analytical grade and purchased from Merck, India.

Synthesis:
Copper nanoparticles were synthesised by chemical reduction of copper sulphate with sodium hypophosphite in ethylene glycol within the presence of a polymer surfactant polyvinylpyrrolidone (PVP). PVP was used to prevent congregation and give dispersion stability to the resulting colloidal nanoparticles. 0.28 kg PVP and 100 g sodium hypophosphite were mixed with 1 litre ethylene glycol inside a round-bottom flask while vigorously stirring at room temperature under ambient atmosphere. The mixture was heated till 90^0C at a rate of 5^0C per min. 0.25 litre of a mole solution of copper sulphate in ethylene glycol at 90^0C was then rapidly added into the PVP/sodium hypophosphite solution, stirring vigorously. As reduction occurred, the colour of the suspension turned from green to henna colour within 2–3 minutes, indicating the formation of copper nanoparticles. The reaction was quenched and the suspension was rapidly cooled by adding chilled deionised (DI) water. The copper nanoparticles were then separated and washed with deionised water. The resulting precipitates were dried under vacuum at 40^0C for 2–3 hours.

[3]CHARACTERIZATION

Structural Characterization of Copper Nanoparticles:
The dried powder of copper nanoparticles was analysed under X'Pert Pro X-ray diffractometer operated at a voltage of 40 kV and a current of 40 mA with Cu Kα radiation. All the data were collected in reflection mode. Technai G^2 S-Twin 200 Kev (MRC, MNIT, Jaipur) transmission electron microscope (TEM) has been used to get micrograph of Cu nanoparticles. Size and shape of Cu nanoparticles are conformed by TEM micrograph analysis.

[4]THEORY

Crystalline domain size was calculated from the width of the X-Ray Diffraction (XRD) peaks using the Debye-Scherrer's formula i.e.

$$D = 0.94\lambda/\beta \cos\theta \quad (1)$$

Where, D is the average crystalline domain size perpendicular to the reflecting planes, λ is the X-ray wavelength (1.54Å), β is the full width at half maximum (FWHM) and θ is the diffraction angle.

[5]RESULT AND DISCUSSION

The crystal structure and size of the nanoparticles were verified by XRD analysis. Figure 1 exhibits the XRD pattern of the synthesised nanoparticles. Peaks observed at 2θ values of 43.40^0, 50.43^0 and 73.65^0 corresponds to (111), (200) and (220) planes of metallic copper (Cu) nanoparticles. These three peaks were quite consistent with those of the standard JCPDS Card No. 04-0836 for the standard spectrum of the pure face centred cubic(FCC) metallic copper nanoparticles. Besides the metallic copper peaks, several other diffraction peaks appeared at 29.63^0, 36.55^0, 42.44^0, 61.55^0, 73.65^0 and 77.49^0 corresponding to (110), (111), (200), (220), (311) and (222) planes of cuprite, respectively, indicating the formation of cubic copper (I) oxide nanocrystals. X-Ray Diffraction (XRD) peaks observed for cuprite matched well with the standard powder diffraction card of body centered cubic (BCC) cuprite (JCPDS No. 05-667).

The XRD diffraction pattern showed the coexistence of two crystalline phases, i.e., metallic copper (Cu) and cuprous oxide (Cu_2O). This obviously illustrates that the zero-valent copper nanoparticles formed in the chemical reduction stage go through decomposition due to limited stability of copper nanoparticles and Cu_2O might be formed by oxidation [12]. All the nanocubes were indeed Cu and Cu_2O; no other phase of copper oxide (CuO) was present. The peak broadening in the XRD pattern indicates the presence of small nanocrystals [13]. The mean size of the crystalline Cu and Cu_2O nanoparticles calculated from the major diffractions peaks' using the Debye-Scherrer's formula was about 28.73nm and 25.19 nm, respectively.

Formation and shape of Cu nanoparticles was confirmed by TEM micrograph analysis. Shape of nanoparticles was found spherical and particles were agglomerated as shown in figure2. Figure 3 shows the SEM analysis which was used to study the surface morphology of synthesized nanoparticles. SEM image showed that the prepared Cu and Cu_2O nanoparticles were spherical in shape.

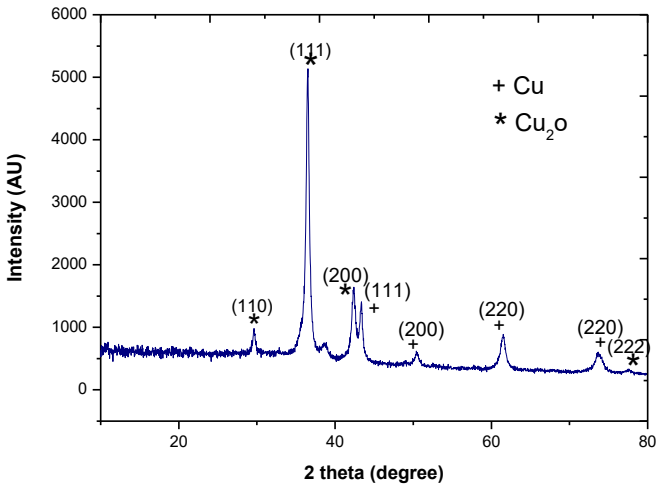
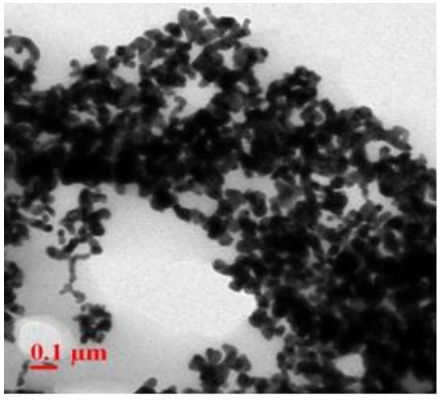


Fig-1: XRD pattern of Cu nanoparticles



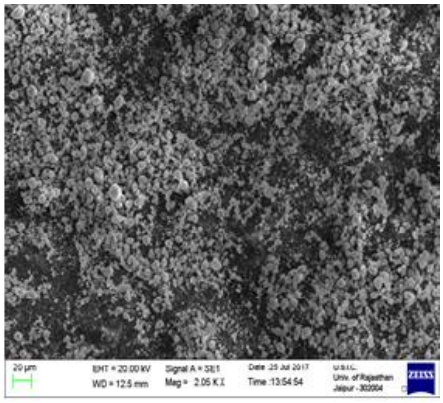


Fig.-2: TEM micrograph of Cu nanoparticles **Fig.-3:** SEM micrograph of Cu nanoparticles

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