SYNTHESIS OF COPPER NANOPARTICLES BY CHEMICAL REDUCTION METHOD

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ABSTRACT: *Copper nanoparticles have been synthesized by using chemical reduction method using de-ionized water as solvent. The surface morphology is observed by Atomic Force Microscope (AFM). The formation of copper nanoparticles was confirmed by UV-Visible spectrophotometer (UV-Vis), X-ray diffraction (XRD) and fourier transform infrared spectroscopy (FTIR). Copper nanoparticles fabricated by chemical reduction method have diameter in the range 14 nm to 55 nm. Structural analysis revealed the face centered cubic (fcc) crystal structure of copper nanoparticles.*

Keywords: Chemical Reduction, Copper, UV-Visible Spectrophotometer, AFM, FTIR.

INTRODUCTION

Nanoparticles are fascinating materials that find many applications in fields of basic and applied research. Copper (Cu) nanoparticles (NPs) with high fraction of surface atoms and high specific surface area have been widely studied. The Cu NPs have special physical and chemical characteristics which include catalytic activity, optical properties, anti-microbial activity and electronic properties [1]. Copper nanoparticles can be fabricated by using different physical and chemical techniques such as chemical reduction [2, 3], laser ablation [4, 5], electrochemical [3], thermal decomposition [6] and polyol method [7]. Among all these methods chemical reduction is convenient method for the fabrication of nanoparticles. High yield of metallic nanoparticles has been attained by chemical reduction method (CRM). This method is economical, simple, faster and can have better size distribution of nanoparticles by controlling the experimental parameters [8]. Currently, noble metal nanoparticles have been extensively studied for many applications. For nanoparticles synthesis the noble metals such as silver and gold are being used, despite their cost [9-11]. In this context, copper are good alternative material because they are more economical than silver and gold. L. A. Figueroa *et al* have reported that chemically synthesized copper nanoparticles having diameter of 25 nm [12].

In the current research, the attention have been focused on the fabrication of Cu NPs. Nanoparticles are synthesized by chemical reduction method (CRM).The Cu NPs were characterized by using different techniques such as UV-Visible Spectrometry, Atomic force microscopy (AFM), X-ray diffractometer (XRD) and Fourier transform infrared spectra (FTIR).

Materials and method

Materials

All of chemicals used in experiment are of analytical grade and used as purchased without any purification. Copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), of 98% purity is used. De-ionized water used as a solvents. Sodium borohydride ($NaBH_4$) is used as reducing agent, while sodium hydroxide (NaOH) is used to adjust the pH. Ascorbic acid is used as the antioxidant for colloidal Cu NPs.

METHOD

The flowchart of experimental procedure is shown in figure 1. Ascorbic acid solution (0.02 M) was prepared in de-ionized water. A 0.01 M solution of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) separately prepared in de-ionized water and this was added to ascorbic acid solution under continuous magnetic stirring. To adjust the pH, 1 M solution of NaOH in de-ionized water was added. After stirring for 30 minutes at room temperature, 0.1 M solution of $NaBH_4$ in de-ionized water was added under continuous stirring. The stirring was continued for 15 minutes in ambient atmosphere to complete the reaction. The blue color of initial reaction mixture turned red-brown color as shown in figure 2.

$Cu^{2+} + 2BH_4^- \longrightarrow Cu + H_2 + B_2H_6$

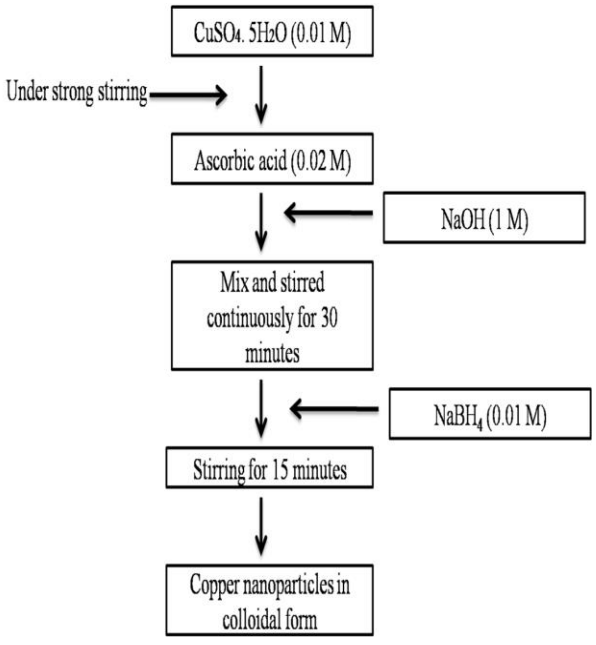

Figure 1: Flow chart of experimental process.

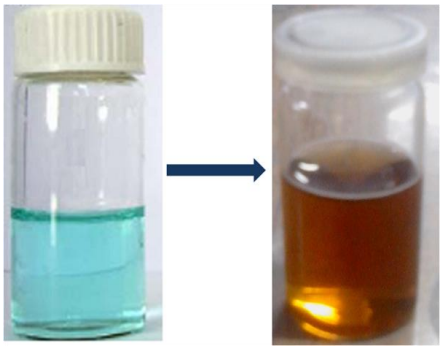

Figure 2: Colloidal solution of copper nanoparticles, initial blue color turned red-brown.

RESULTS AND DISCUSSIONS

To study the stability of Cu colloidal solution in air, the absorption of Cu NPs was measured by UV-visible spectroscopy. The absorption band of copper nanoparticles has been reported in the range of 500-600nm [10, 13]. UV-visible absorption spectra of Cu NPs by chemical reduction method (CRM) is shown in figure 3. This spectrum is recorded immediately after the synthesis of particles. The figure show the absorption peaks at 588 nm respectively, which proves the formation of the copper nanoparticles in the solution [14]. The initial blue green color turned red-brown, the shifting in color is due to the surface plasmon resonance (SPR). Metals possess SPR in visible region due to free electrons, which give such intense colors. These properties observed in Cu, Ag and Au due to presence of free electrons [12].

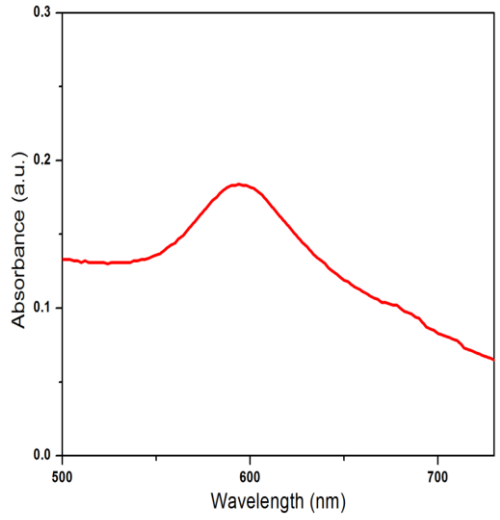

Figure 3: UV-visible spectra of copper nanoparticles fabricated by chemical reduction.

AFM is an important technique for study the morphology of nanoparticles. Tapping mode AFM imaging is applied to study copper nanoparticles. Figure 4 shows an AFM image (2 μm ×2 μm) of copper nanoparticles (3D) having particle size range 14 nm to 55 nm.

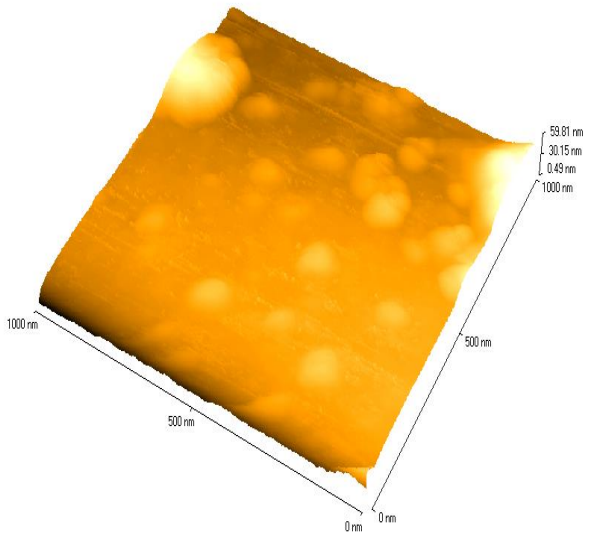

Figure 4: 3D topographical view of copper nanoparticles, having particle size 14 nm to 55 nm.

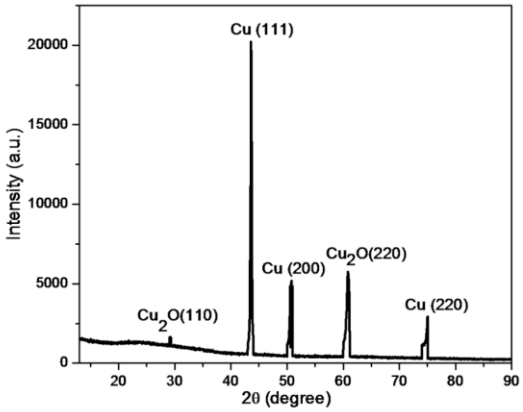

Figure 5: XRD pattern of the copper nanoparticles.

The crystal structure and phase composition of synthesized copper nanoparticles is analyzed by XRD, as shown in figure 5. The diffraction data exhibits that the copper nanoparticles have face centered cubic structure (FCC) with characteristic diffraction peaks (111), (200) and (220) at 2-theta value of 43.4º, 51.1º and 74.9º respectively. On the other hand, two diffraction peaks were indexed to cuprous oxide (Cu_2O) having corresponding peaks to (110) and (220) at 2-theta value of 29.1º and 60.5º respectively. The presence of Cu_2O indicates the partial oxidation of copper nanoparticles with dissolved oxygen in the solution [14, 15].

For copper nanoparticles, the oxygen present in ambient atmosphere rapidly forms an oxide layer on the particle surface when exposed to air. This means that copper nanoparticles could be synthesized in atmospheric environment, at atmospheric pressure and at room temperature by using de-ionized water as solvent in the case of present work. It is not necessary to perform the chemical

reaction in an inert atmosphere. All the parameters or variables reduce the cost of the process [15, 16].

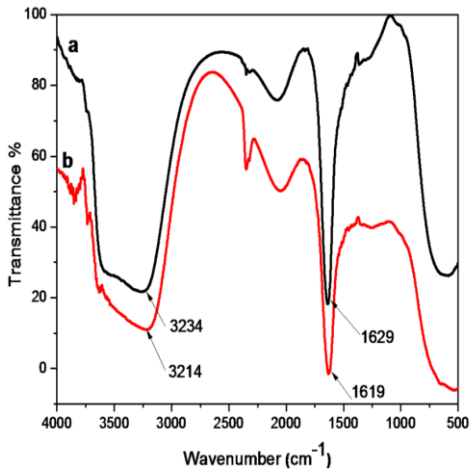

Figure 6: FTIR spectra (a) de-ionized water (b) copper nanoparticles.

FTIR spectra of de-ionized water and copper nanoparticles are shown in Figure 6. In the 3500–3000 cm^{-1} region, a broad absorption of hydroxyl group (O-H) of de-ionized water appear at 3234 cm^{-1} and 3214 cm^{-1} before and after nanoparticles formation, respectively, showing 20 units red shift of this polar group. Intermolecular and intramolecular hydrogen bonds are considered to be responsible for the broadening of the –OH band in the FTIR spectra. This decrease in wave number may occur due to interaction of copper nanoparticles with–OH group [17]. The bands are in the region 2000-1500 cm^{-1} are due to C=C bond. This bond appears at 1629 cm^{-1} before nanoparticles formation and shifted at 1619 cm^{-1} after nanoparticles formation. The shifting in wave numbers was due to the C=C stretching and shows the co-ordination with copper nanoparticles [18, 19].

CONCLUSION

In this work, synthesis of copper nanoparticles (Cu NPs) has been investigated by chemical reduction method (CRM). The average size of copper nanoparticles prepared by CRM is 14 to 55 nm. The absorption peak appeared at 591 nm which confirms the formation of copper nanoparticles. The observed fcc XRD peaks for copper nanoparticles are ascribed the growth along different crystallographic planes. Another phase coprous oxide (Cu_2O), also observed which shows the partial oxidation of copper nanoparticles with dissolved oxygen in the solution.

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