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Research Article

Spectroscopic Study of Synthesized Copper Nanoparticles by Chemical Reduction Method

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A B S T R A C T

Well-dispersed and uniform-size preparation of Copper nanoparticles and its X-ray diffraction studies are reported in this paper. A simple and cheapest chemical reduction method with copper sulfate pentahydrate as a main precursor is used for the synthesis of Copper nanoparticles. A wide range of experimental conditions has been adopted in this process and its X-Ray diffraction characterizations have been studied. UV-visible spectroscopic analysis showed the maximum absorption at 295 nm. Transmission electron microscopy reveals the spherical morphology of the synthesized particles. The results confirm copper nanopowder with an average particle size in the range of 30-60 nm.

Keywords: Copper nanopowder; X-ray Diffraction; UV-visible spectroscopy; Scanning Electron Microscopy.

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INTRODUCTION:

Recently researchers have focused their intention on the preparation of small-size particles in the range of 1-100 nm. Due to the reduction in size of particles, the ratio of surface area to volume increases. More surface area becomes available to interact with the surrounding medium in which the particle is suspended. Optical, electronic, magnetic, and catalytic properties change drastically which is depends on their size, shape, and chemical surroundings [1,2]. There are several methods for nanoparticle preparation, including chemical synthesis, physical methods, and biological methods. Chemical synthesis methods involve the use of chemical reactions to produce nanoparticles. This can include methods such as sol-gel synthesis, co-precipitation, and hydrothermal synthesis. These methods allow for precise control over the size and shape of the nanoparticles by adjusting reaction conditions such as temperature, pH, and concentration of reactants. Physical methods for nanoparticle preparation include techniques such as milling, grinding, and spray drying. These methods involve mechanical or physical forces to break down larger particles into nanoparticles. While these methods may not offer as much control over particle size and shape as chemical synthesis methods, they can be useful for producing large quantities of nanoparticles quickly and efficiently. Biological methods for nanoparticle preparation involve the use of biological organisms such as bacteria, fungi, or plants to produce nanoparticles. This can be achieved through processes such as biomineralization, where organisms produce nanoparticles as part of their natural metabolic processes. These methods can be environmentally friendly and offer unique opportunities for controlling nanoparticle morphology.Overall, controlling the particle size, shape, and morphology in nanoparticle preparation is critical for ensuring the desired properties and applications of the nanoparticles. Different methods offer varying degrees of control and may be

chosen based on the specific requirements of the nanoparticles being produced. [3]. The most important spectroscopic techniques used for the characterization of synthesized nanomaterials are X-ray Diffactrometry (XRD), Fourier Transform Infrared spectroscopy (FTIR), and Scanning Electron Microscopy(SEM). The synthesis of Copper Nanoparticles by the Chemical Reduction Method is discussed in the paper. The preparation of uniformed Copper nanopowder size of less than 50 nm, in a normal room temperature, is important of this study. Its XRD, UV- Visible spectroscopy, and SEM analysis confirm the result [4].

Nanosized metal particles have gained significant attention in the field of science due to their unique physical and chemical properties, which differ from those of bulk materials [5]. Various techniques have been employed to produce nanoparticles in either powder or colloidal form, including physical, chemical, biological, and hybrid methods [6-9]. Methods such as lithographic techniques and vacuum deposition of metals can generate nanoparticles with uniform size, shape, and spacing, but they tend to be expensive and suitable for a limited range of materials systems. On the other hand, electrolytic cathode deposition is a simple, cost-effective method that can be applied to a wide range of materials.

METHODS AND MATERIALS

Chemicals

All chemicals used in the experiment are analytic reagent grade. Copper sulfate pentahydrate (CuSO4· 5H2O) of 98% purity is used. Deionized water was used throughout the experiment as a solvent. Sodium borohydride (NaBH4) is used as a reducing agent in the reaction. Sodium hydroxide (NaOH) is used to adjust pH. Ascorbic acid (C6H8O6) is used as an antioxidant agent for colloidal copper nanoparticles.

Synthesis

The copper nanoparticles were synthesized by a chemical reduction process using copper sulfate pentahydrate (CuSO4· 5H2O) as a precursor. The preparation method starts with the addition of 0.01 M copper sulfate pentahydrate solution into 0.02M solution of ascorbic acid under continuous magnetic stirring for 30 min. In the second step, 1M solution of NaOH in de-ionized water was added to adjust pH. After stirring for 30 minutes at room temperature, 0.1M solution of NaBH4 in de-ionized water was added under continuous stirring. The stirring was continued for 15 minutes in an ambient atmosphere to complete the reaction. Lastly, the blue color of the initial reaction mixture turned red-brown color. After the completion of the reaction, the solution was taken from the heat and allowed to settle overnight and the supernatant solution was then discarded cautiously. The precipitates were separated from the solution by filtration and washed with deionized water and ethanol three times to take out the excessive reagents bound with the nanoparticles. Finally, the precipitates obtained are dried at room temperature. After drying, nanoparticles were stored in a glass vial for further analysis.

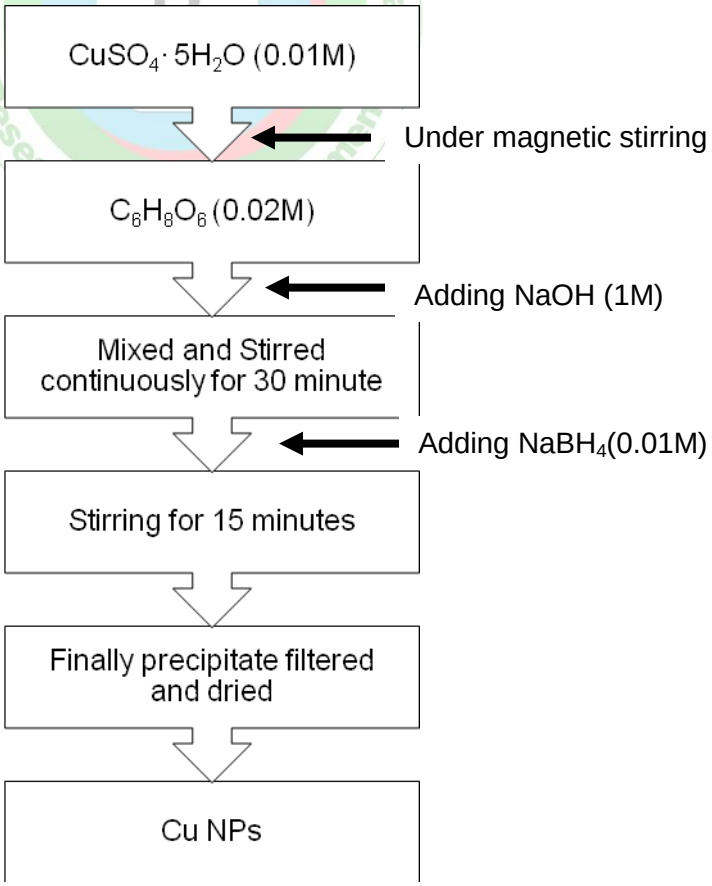


Figure1: Flow chart of synthesis of Cu Nanoparticles

Figure 2: Prapared sampleof Cu nanoparticles

Characterization

The powder X-ray diffraction (XRD) was performed using Philips Holland, XRD system PW 1710 with nickel filtered CuKα (λ = 1.5405 Å) radiation. The average crystallite size (t) has been calculated from the line broadening using Scherrer's relation: $t = 0.9\lambda / B\cos\theta$, where λ is the wavelength of X-ray and B is the full width of half maximum (FWHM). The morphology of Cu nanoparticles was studied using a scanning electron microscope (JEOL JSM 5600). The transmission electron microscopy (TEM) was performed with Tecnai 20 G 2 under 200 KV. Samples are prepared by dispersing drops of colloid on a copper grid, covered with the carbon film and the solvent is evaporated.

X-Ray Diffraction: X-ray Diffraction (XRD) is one of the most important and powerful primary techniques of characterization used by mineralogists and solid-state chemists to examine the physicochemical makeup of unknown materials. XRD is an easy tool to determine the size and shape of the unit cell for any compound. Powder Diffraction Methods are useful for Qualitative analysis (Phase Identification), Quantitative analysis (Lattice parameter determination and phase fraction analysis), etc. Diffraction pattern gives information on translational symmetry - size and shape of the unit cell from Peak Positions and information on electron density inside the unit cell, namely where the atoms are located from Peak Intensities. It also gives information on deviations from a perfect particle, if the size is less than roughly 100 – 200nm, extended defects, and microstrain from Peak Shapes and widths Peak Indexing Indexing is the process of determining the unit cell dimensions from the peak positions. It is the first step in diffraction pattern analysis. To index a powder diffraction pattern it is necessary to assign Miller Indices (h k l) to each peak. Unfortunately, it is not just the simple reverse of calculating peak positions from the unit cell dimensions and wavelength [10].

RESULTS AND DISCUSSION

XRD study

The XRD pattern of as prepared CuO nanoparticles is shown in Figure 1. It gives a single phase with a monoclinic structure. Lattice parameters are a = 4.84 Å, b = 3.47 Å, c = 5.33 Å. The intensities and positions of peaks are in good agreement with the reported values (JCPDS file No. 05-661). No peaks of impurities are found in the XRD pattern. The peaks are broad due to the nano-size Effect. The average crystallite size of Cu nanoparticles is found to be 30-60 nm using the Scherrer formula.

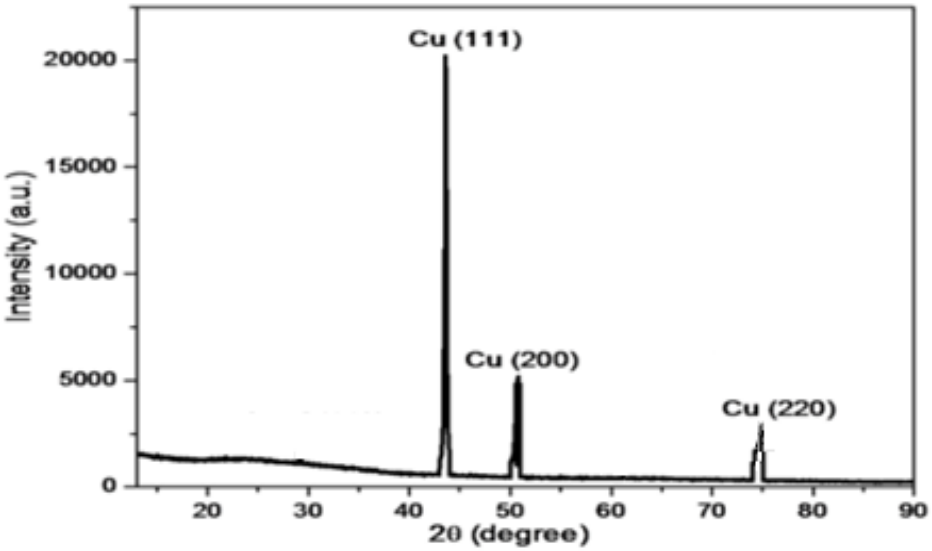


Figure 3: XRD pattern of Cu Nanoparticles

Scanning Electron Microscopy:

Figure 4 shows the SEM image of as prepared Cu nanoparticles. It shows that the Cu nanoparticles are spherical. The size of the particle observed in SEM image is in the range of 30-60 nm which is in good agreement with calculated by Scherrer formula using XRD. It shows that the particles are well crystallized.

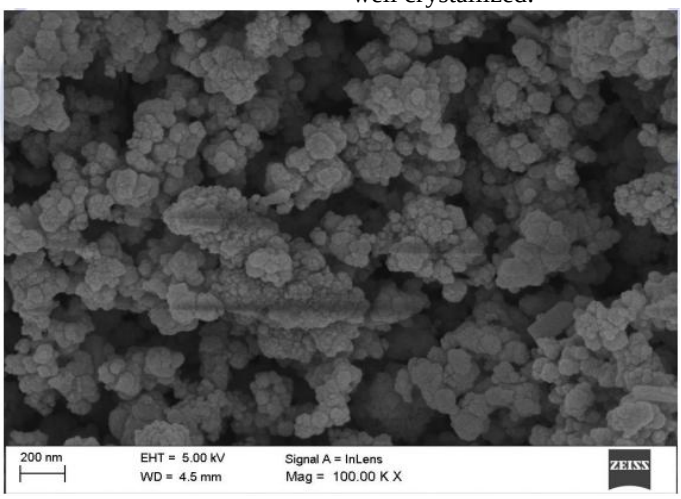
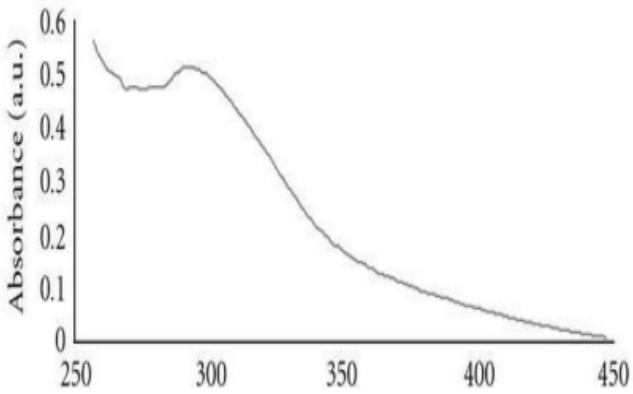


Figure 4: Scanning Electron Microscopy of Cu Nanoparticles

UV-visible spectra of copper nanoparticles:

The absorption of Cu NPs was measured by UV-visible spectroscopy. The absorption band of copper nanoparticles has been reported. UV-visible absorption spectra of Cu NPs by chemical reduction method are shown in Figure 5. The figure shows the absorption peaks at 293 nm respectively, which proves the formation of the copper nanoparticles in the solution [11]. The initial blue-green color turned red-brown, the shifting in color is due to the surface plasmon resonance (SPR). Metals possess SPR in the visible region due to free electrons, which give such intense colors. These properties observed in Cu, Ag, and Au due to the presence of free electrons.



CONCLUSION

In this work, the synthesis of copper nanoparticles (Cu NPs) has been investigated by the chemical reduction method. The average size of copper nanoparticles is found in the range of 30-60 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 to the growth along different crystallographic planes.

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