Synthesis and characterization of copper nanoparticles by using electrochemical method

S. L. Kadam

Abstract - In the present work copper nanoparticles were prepared by electrochemical method. Copper sulphate and distilled water were used initially to prepare nanoparticles. All the procedure was done at room temperature by applying voltage 2V and passing current of 1.5A. The time required for the deposition of nanoparticles is very less around half an hour. The characterization was performed by X-Ray diffraction, scanning electron microscope and ultraviolet visible spectroscopy. XRD confirms formation of FCC structure and nanoparticle. SEM confirms crystalline nature with spherical shape. UV-Visible spectroscopy shows that band gap is 2.11 eV.

Index Terms— X-ray diffraction**,** SEM, UV-VIS, Copper nanopowder, Debye- Scherrer.

1 Introduction

In recent research, preparation and study of nanoparticles becomes very important. Copper nanoparticles are of great interest because of its many applications such as medical items, cleaning, antiseptics, textiles, paintings, intrahospital coatings etc. [1]. The antibacterial effect of copper nanoparticles has been reported by Yoon et.al [2] and Cioffi et. al [3]). In preparation of nanoparticles the size requirement is not enough, it is necessary a procedure that assures a tight distribution of size particles, a controlled morphology and composition and identical crystal structure [1]. Nanosized metal particles are attracting the attention of present science field because of their physical and chemical properties, which are quite dissimilar from those of bulk materials [4]. Various techniques have been adopted to produce nanoparticles on solid surfaces, including diverse lithographic techniques, vacuum deposition of metal, controlled nanoparticle growth by diffusion, electrophoretic deposition of a metal colloid, chemical and electrochemical deposition of metal nanoparticles, etc. [5], [6], [7], [8].

Most of the existing synthesizing methods of metal nanoparticles are complicated, require specific equipments and produce only small amount of nonmaterial [9]. Metallic nanoparticles are traditionally synthesized by wet chemical methods where the chemicals used are often toxic and flammable [9]. Discussion about easy, simple, fast and low cost preparation i.e. electrochemical method and the characterizations X-Ray diffractions, Scanning electron microscope and ultraviolet visible spectroscopy are studied in this research paper.

• Post Graduate Department of Physics, New Arts, Commerce and Science College, Parner,(Savitribai Phule Pune University), Dist. Ahmednagar, Maharashtra. India-414302.
Corresponding author's Email: mr.sukadeo@rediffmail.com

This method can be used to prepare wide range of materials. From literature the Cu nanoparticles are synthesized from thermal decomposition, electrochemical reduction, vapour deposition, radiolysis reduction and chemical reduction of copper metal salt. The synthesis of metal nanoparticles using natural extracts is greener from a chemical hazard standpoint and advantageous from an economical point of view [10].

2 Preparation

In a clean glass vessel, copper sulphate was kept. Distilled water was poured in the vessel and a homogenous aqueous copper sulphate solution was prepared. Anode and cathode copper electrodes were kept inside this vessel. Then 2V voltage was applied and current of 1.5 A was passed through anode and cathode and electrolysis of solution was done. Within half an hour nanoparticles were deposited on cathode surface. All the preparation was done at room temperature..

3 Experimental Details

Electrolysis is a chemical effect of electric current. It passes and conducts electric current through the electric solution. It is dissociation process of an electrolyte and the dissociated ions that appears at the two electrodes. Electrolytes conduct electricity due to drifting of ions (positive cations and negative anions) [9]. At the end of electrolyzing process, a layer of copper deposition on the cathode surface was observed. Copper layer was removed from the cathode surface and collected as copper nanopwder. Similar method was adopted initially by Theivasanthi and Alagar [11].The uumber of atoms, ions or molecules take part in nucleation process and Cu^{2+} ions are migrated towards cathode. In the nucleation process number of atoms or ions comes in contact and a cluster of these atoms or ions are formed on the surface of cathode.

XRD analysis was done using a Philips Model PW-1710 with CuKα radiation (λ=1.5418 A^0) and data was taken for the 2θ range of 10 to 80^0 with a step of 0.04^0. The surface mor-

phology was analyzed by using SEM model S-3000H of HITACHI. The absorbance was recorded at different stages of synthesis using UV-visible spectrometer (model Shimadzu UV-2450), in the wavelength range 400 to 800 nm.

4 Results and Discussion

Fig. 1 shows the X-ray diffractogram of the copper nanoparticles synthesized by electrolysis method. There are three main characteristic diffraction peaks for Cu. The peaks were observed at 2θ = 43.64, 50.80 and 74.42 deg. which corresponds to (1 1 1), (2 0 0) and (2 2 0) crystallographic planes of face-centered cubic Cu phase (JCPDS No. 04- 0836 and ASTM 03-1005-standard powder diffraction card) [12]. A small peak is also observed at around 36^0 indicates that a small amount of copper is oxidized and converted into copper oxide. The XRD study confirms that the resultant particles are FCC copper nanopowder. The experimental diffraction angle (2θ) and standard diffraction angle (2θ) are in good agreement and also confirmed by other researchers [9], [13].

The intensity of peaks reflects the high degree of crystallinity of the copper nanoparticles. However, the diffraction peaks are broad which indicates that the crystallite size is very small [9], [14].The size of the Cu nanoparticles estimated from Debye-Scherrer formula D = $(0.89\lambda) / (\beta \cos\theta)$ is around 24 nm, where λ is wavelength of X-Ray (0.1541 nm), β is FWHM (full width at half maximum), θ is the diffraction angle and D is the particle diameter size. The FCC crystal structure of copper has unit cell edge a=3.62 A^0 and this value is calculated theoretically by the formula, $a=4/\sqrt{2}\, r$, for copper r=128 pm. The experimental lattice constant a, calculated from most intense peak (1 1 1) of the XRD pattern is 3.59 A^0. Both theoretical and experimental lattice constant 'a' are in good agreement. The details of 'a' value of all peaks have been produced in Table 1. The value of 'd' (the interplaner spacing between the atoms) is calculated using Bragg's law, $2d \sin\theta = n\lambda$.

Fig. 2 shows the SEM of the copper nanoparticles. It is clear that crystalline nanoparticles are obtained. They are spherical in shape. There is nearly monodispersed distribution of particle sizes. The observation of some larger nanoparticles is composed of Van der Waals clusters of smaller entities. From geometry, it is clear that the sizes of small individual particles are less than 100 nm in diameter [9].

Fig. 3 shows the UV-Vis spectra of copper nanoparticles. It is clear that an absorption peak at around 587 nm is observed. This peak can be assigned to the absorption of copper nanoparticles. The broadness of the absorption peak probably stems from the wide size distribution of nanoparticles. The band gap is found using the formula $E= h\nu$ or $E=hc/\lambda_{max}$. $E= (6.626 * 10^{-34} * 3 * 10^8) / (587*10^{-9}) = 0.03386*10^{-17}$ J or $E= (0.03386*10^{-17})/ (1.6*10^{-19})$ or E= 2.11 eV i.e. band gap is 2.11 eV. Copper nanoparticles have already been studied extensively due to their potential technological applications in various fields like catalysis, lubricants, electronics etc.[11], [15].

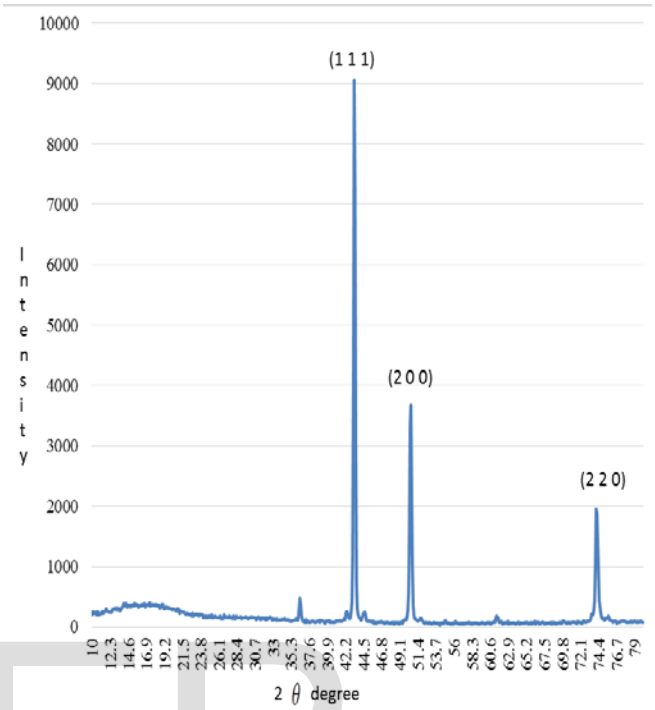

Fig. 1 XRD Showing Peak Indices

TABLE 1
The different parameters of copper nanopowder

2θ of the intense peak(deg)	(h k l) Miller Indices	θ of the intense peak (deg)	FWHM of intense peak (β) radians
43.64	(111)	21.82	0.0059
50.80	(200)	25.40	0.0066
74.42	(220)	37.21	0.0070

Size of the particle (D) nm	d-spacing nm	Standard value of 2θ (JCPDS)	Lattice parameter (a) A^0
25.32	0.2070	43.297	3.5905
23.26	0.1796	50.433	3.5920
24.88	0.1274	74.130	3.6034

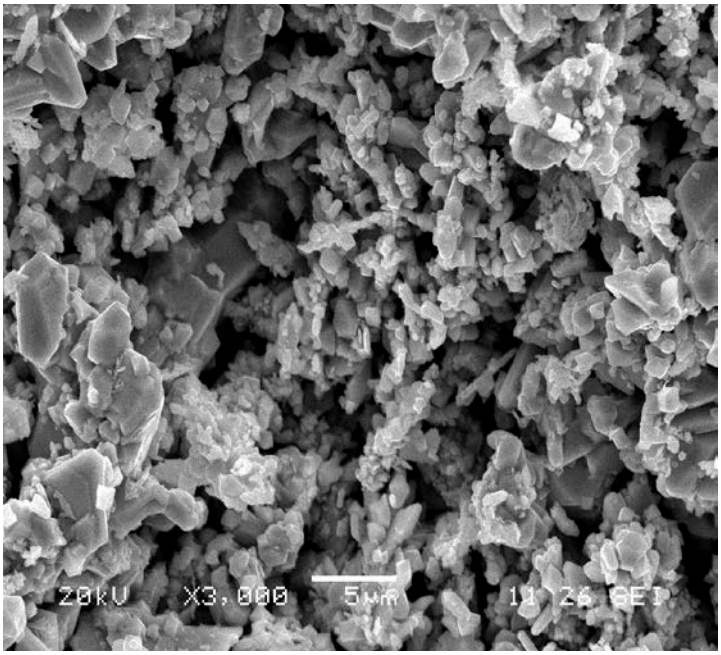

Fig. 2 SEM of the copper nanoparticles

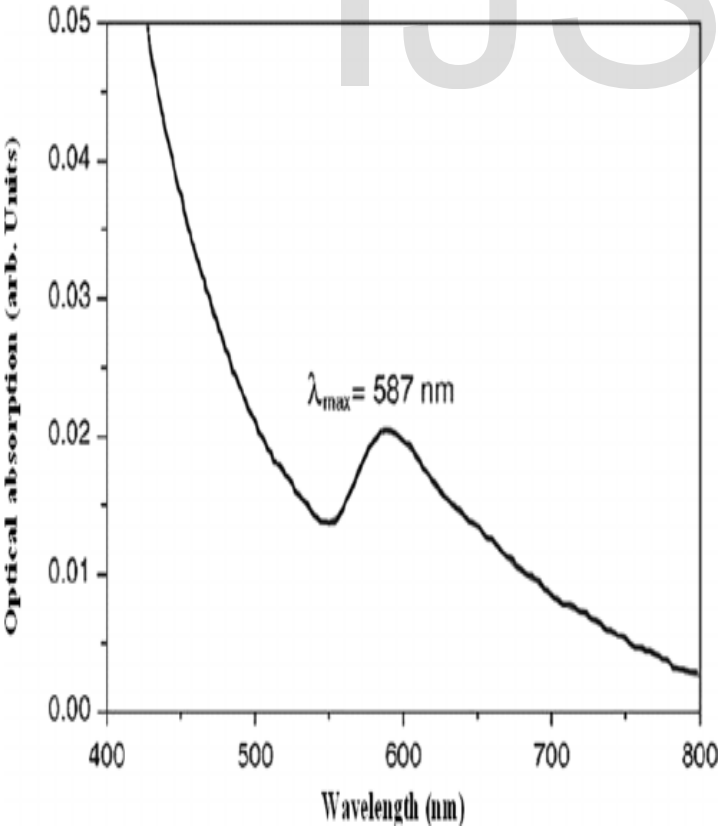

Fig. 3 UV-VIS spectra of copper nanoparticles

5. Conclusion

Electrochemical method is a simple, fast and economical to synthesize copper nanoparticles. It provides a clean, nontoxic and eco-friendly route for synthesis of nanoparticles at room temperature. Some other nanopowder may be prepared with the basis of this study. The instruments for this method are easily available. The synthesized copper nanoparticles are spherical in shape with particle size around 24 nm. Their characterization has been successfully done using XRD, SEM and UV-VIS spectroscopic techniques.

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