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Synthesis and Characterization of Electrochemically Deposited Copper Nanoparticles and their application for nano-fluids

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ABSTRACT

Nanotechnology deals with the various structures of matter having dimensions of the order of a billionth of a meter. In the present work, we have prepared copper nanoparticles by using electro-deposition method. The structural, optical and morphological properties of the prepared Cu nanoparticles were characterized by using XRD, UV-Visible Spectroscopy and scanning electron microscopy, respectively. The XRD spectra reveal that the prepared Cu particle shows crystalline and cubic structure. In present investigation, we also studied the effect of the different parameters such as temperature, applied potential, time, stirring rate on properties of the Cu nanoparticles. Here it was observed that the freshness of an electrolyte also matters which is simply prepared from the ethanol, anhydrous $CuSO_4$ and HCl having specific quantity. The present work also investigated the effect of Cu nanoparticles on electrical conductivity of base fluid (DDW). The electrical properties of the base fluid get altered on the dispersion of nanoparticles. The effect of illumination under different source on electrical conductivity of the Cu nanoparticle with base fluid was also discussed.

Keywords: Nanotechnology, Nanoparticles, electro-deposition, electrical conductivity, copper etc.

INTRODUCTION

The nanoparticles are the particles which having the dimensions from 1nm to 100nm. Materials with structure at the nanoscale often have unique optical, electronic, or mechanical properties. Furthermore, the optical and electronic properties of nanomaterials which depend on their size and shape can be tuned via synthetic techniques [1-2]. Nanoparticles are of great scientific interest as they are effectively a bridge between bulk materials and atomic or molecular structures. A bulk material should have constant physical properties regardless of its size, but at the nano-scale this is often not the case. Nanoparticles exhibit a number of special properties relative to bulk material [3-4]. For example, the bending of bulk copper (wire, ribbon, etc.) occurs with movement of copper atoms/clusters at about the 50 nm scale. Copper nanoparticles smaller than 50 nm are considered super hard materials that do not exhibit the same malleability and ductility as bulk copper. Nanoparticles can be used in different fields like in agricultural, industrial, biological, medical, defence, [5-8]. The synthesized Cu nanoparticles are acts as an anti-biotic, anti-microbial, and anti-fungal agent when added to plastics, coatings, and textiles. Cu nanoparticles also for heat sinks and in highly thermal conductive materials. It also an efficient catalyst used in chemical reactions and for the synthesis of methanol and glycol. Sometime it is used as a in sintering additives and capacitor materials. There are several chemical methods for preparation of nanoparticles like Sol –gel method, Colloidal method, Co-precipitation method, Electro deposition methods etc. [9-12]. Among these electrodeposition method which is cheap and easy. Here we have synthesized copper nanoparticles from this method by applying certain potential difference with constant current supply and at the room temperature. This method of synthesis of nanoparticles is cheaper & easier. Multideposition of nanoparticles can be take place and easily portable method. The preparation of the nanoparticle can possible at room temperature in the laboratory by using electrodeposition method. The structural and morphological characterization of film has been carried out by X- ray diffraction (XRD) and Scanning electron microscopy (SEM) respectively and their optical and compositional properties have been studied by UV-vis-NIR spectrophotometer and Energy dispersive spectroscopy (EDS) technique respectively.

In the present work we successfully synthesized the Cu nanoparticles by using electrodeposition method and study its conducting as well other characteristics by using characterization technique.

METHODOLOGY

1. Electrodeposition technique:
It is very useful technique to deposit the metal layer on a conducting substance. The various parameters of the this technique which effect on the properties of the synthesized nanoparticles such as size and shape of electrodes, distance between the electrodes, concentration of the sample, applied voltage, electrolyte etc. In the present synthesis of Cu nanoparticles, 0.1 M $CuSO_4$ (0.80 gm) solution used as a cationic precursor.

Fig 1. Electro-deposition Setup in the laboratory

This solution added in the 50 ml ethanol in beaker. Stir the solution (180 rpm) properly on magnetic stirrer and add 20 drops of 50 % diluted HCl. The voltage given to the electrodes should be 3 V and 4.5 V. The pH of the electrolyte is one. The deposition time is 10 minute. The formation of Cu-NPs follows a series of color changes due to change in applied potential, which may result in variation of particle size. The copper nanoparticles dispersed in ethanol by ultrasonication and then kept at room temperature (27 °C) for 3 months to determine their stability. The X-ray diffraction studies of Cu nanoparticles was carried out using X ray diffractometer (Bruker D8), Scanning electron microscope (JEOL JSM-6360) was used for the surface morphological study. The optical absorption

spectra were recorded in the range 300-1200 nm at room temperature by JASCO spectrophotometer. The electrical conductivity of Cu nanoparticles in aqueous solution also measured.

2. Measurement of electrical conductivity:

The electrical conductivity of any fluid is a measure of its ability to carry an electric current. The electrical conductivity can be expressed as mhos (Reciprocal of ohms) or as siemens. In most fluids, the conductivity is very low, so millisiemens or microsiemens are used as units for conductivity. Chemical composition of fluid determines its conductivity. Since the charge on ions in solution facilitates the conductance of electrical current, the conductivity of a solution is proportional to its ion concentration and their mobility [13]. The ions in water act as electrolytes and conduct the electricity. Most conductivity measurements are made in aqueous solutions, and the ions responsible for the conductivity come from electrolytes dissolved in the water. Although water itself is not an electrolyte, it does have a very small conductivity, implying that at least some ions are present. The ions are hydrogen and hydroxide, and they originate from the dissociation of molecular water. The nano-fluids used in experiment were prepared using a two-step method. First, an appropriate amount of Cu nanoparticles has been weighed with the analytical balance and then mixed with water make conducting aqueous solution. . Then, samples were stirred for 30 min using magnetic stirrer. The electrical conductivity found to be a significant dependence on the dispersed particles and its concen-tration. Thus, the second step of preparation of samples was sonication in ultrasonic bath in order to break up the agglomerates remaining after the mechanical stirring. The time for sonication was 20 min. An ultrasound that we used has a power of about 350 W, and is equipped in ultra-wave generators with frequency about 34 kHz.

RESULTS AND DISCUSSION

1. Structural Analysis:

The crystallographic analysis was done by utilizing XRD. XRD analysis of the prepared sample of copper nanoparticles was taken for the range of 10 to 80 degrees as shown in Fig.2. The FCC Cu phase structure (JCPDS Card No. 04- 0836) is evidenced by the peaks appearing at 2θ =44, 49.89, 75.60, corresponds to (111), (200) and (220) respectively. The XRD spectra reveal that the sample demonstrated a high crystallinity level and the resultant particles are (FCC) Copper nanoparticles. The XRD peak of CuO and Cu_2O are not present in the XRD spectra. This result indicates that the pure nanoparticles were formed. The peak broadening suggested that a high surface area, and surface area to volume ratio of the nano-crystals.

X (2Θ)	hkl	d (Å)
44	(111)	0.2055
49.8969072	(200)	0.1825

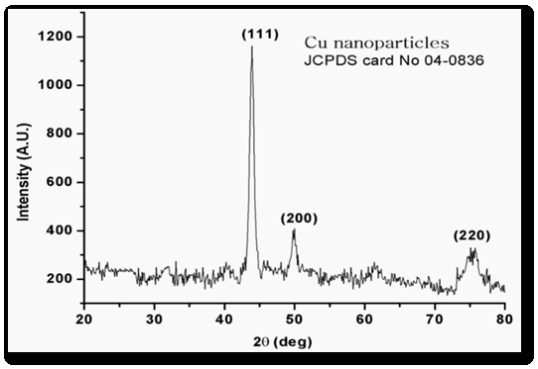

Fig 2. XRD spectra of synthesized Cu nanoparticles

2. Optical Analysis:

The optical analysis of the synthesized nanoparticle was carried out by using UV-Visible spectroscopy in the range of wavelength 300-800 nm. The NPs as observed in Figure 3 which showed a single absorption peak at 577 nm, which indicates formation of Cu-NPs . Since the 577nm wavelength lies between 400nm to 780nm that is in visible region, Cu nanoparticles absorbs visible light.

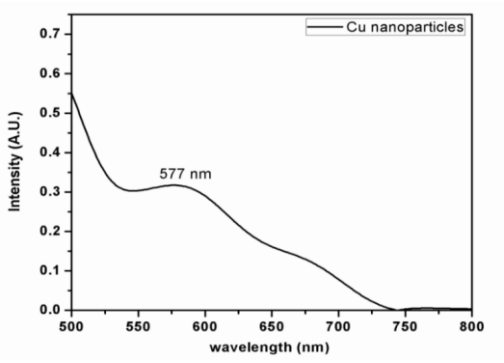

Fig 3: The UV visible spectrogram of synthesized Cu nanoparticles

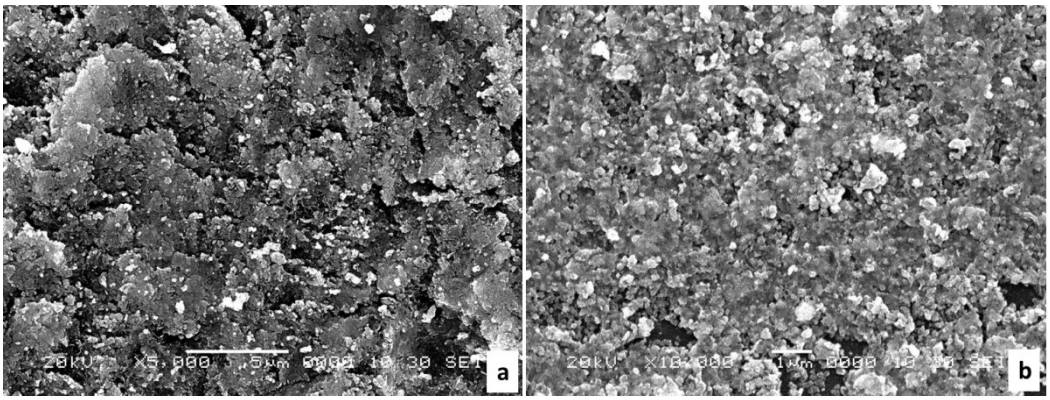

Fig 3: SEM images of electro-deposited Cu nanoparticles prepared at 3V potential

3. Microstructural Properties:

Surface morphology of Cu nanoparticles was characterized using scanning electron microscope. Fig. shows SEM micrographs of Cu nanoparticles. It is observed that the image showed micrometer-sized randomly distributed crystal aggregates, in the range of 2–20 μm. The higher magnification imaging (Fig. 3 b) revealed that the observed crystal aggregates consist of many smaller sized Cu nanoparticles. It happens because nanoparticles have a tendency to agglomerate into bigger aggregates, during continuous electro-deposition.

4. Electrical conductivity:

In the present study the electrical conductivity was measured using electrical conductivity meter. The fluid is subjected to illumination under different illuminating source in order to observe the effect of electromagnetic radiations on the electrical conductivity of prepared sample. The results are shown below as a effect of change in distance between illuminating source and sample.

Above tables shows the electrical conductivity values of water-based nano-fluids with Cu nanoparticles and various source of illumination compared with sun light. From the results, it is observed that the electrical conductivity for Cu nanoparticles prepared at 3V potential is more as compare to that prepared at 4.5V. This may be due to different particle size. It is also observed that the electrical conductivity gradually decreases as the distance between illuminating source and sample increases. It also increases in a nonlinear fashion for different illuminating sources. From all over study of above data we observed that we can obtain maximum electric conductivity in presence of sunlight. The main cause for the enhancement in electrical conductivity of base fluid is the formation of surface charges by polarisation of Cu nanoparticles when dispersed in the base fluid of water which is polar [14]. Thus from the above discussion, it is clear that polarisation is responsible for the current flux across the interface of the particles surface.

Distance(cm)	**Electrical conductivity (μS)**	
Tungsten lamp:		
	Cu(3V)	Cu(4.5V)
10	191.4	158.9
20	184.6	156.5
30	186.5	157.3
40	184.1	156.7
50	182.2	157.0
Sodium vapour lamp:		
10	185.9	168.4
20	183.2	163.3
30	181.7	162.7
40	180.5	162.0
50	179.3	161.1
IR lamp		
10	188.8	166.5
20	185.8	167.8
30	186.3	166.1
40	184.3	164.7
50	184.7	165. 5
Sunlight		
10	0.998	0.999
20	0.998	0.999
30	0.999	0.999
40	0.999	0.999
50	0.999	0.999

CONCLUSION

This study reports the synthesis of Cu nanoparticles by using electro deposition method. The preparation method used is quite cheaper & beneficial. From the XRD result, the synthesized Cu nanoparticle shows well defined peaks and nanocrystalline structure. The UV spectra reveal that the Cu nanoparticles shows maximum absorption in the visible region of the spectra. Copper nanoparticles can be used as conductive inks for flexible electronics. The main purpose of this study is to show that the copper nanoparticles can be use in solar cell because in sunlight absorbs more light. These Cu nanoparticles can be used in different devices such as photovoltaic devices, bio-sensing materials, and in antibiotic activity.

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