Eurasian Journal of Analytical Chemistry
ISSN: 1306-3057 OPEN ACCESS 2018 13 (03): 1292-1294

Characterization of Electrodeposited Copper Nanoparticles

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Abstract
In the present study Copper Nanoparticles (Cu NPs) were prepared on the aluminium substrate by the electrodeposition technique from an aqueous electrolyte prepared from aqueous solution of copper sulphate and citric acid at room temperature. The morphology, chemical composition and crystal structure of the prepared (Cu NPs) were characterized using scanning electron microscope (SEM), energy dispersive X-ray analysis (EDX) and X-ray diffraction (XRD) techniques. The SEM images confirmed the deposition of uniform and highly dense spherical copper nanoparticles on the aluminium substrate. The EDX spectra confirmed the presence of copper on the substrate with no impurities. The crystallite size of the electrodeposited (Cu NPs) was calculated using Debye–Scherrer's formula and found to be around 65 nm. By varying the deposition time and the concentration of electrolyte the growth of Cu NPs on the substrate can be controlled.

Key Words: Copper Nanoparticles, Electrodeposition, Electrolyte, Morphology

1. Introduction

Metallic nanoparticles are of great importance due to their extraordinary chemical physical and optical properties, such as high surface to volume ratio, high electrical conductivity and high heat transfer. Among them copper and copper-based nanoparticle are of great importance low cost and easy availability compared to silver and gold [1]. Copper nanoparticles (Cu NPs) have variety of applications in cooling fluids [2] conductive inks [3] optical switches, photochromic glasses [4], sensors [5] and catalyst [6]. Recently Cu NPs have been known to exhibit antimicrobial properties which can have an enormous impact on the application of antimicrobial surface coatings [7,8]. The main problem lies in their preparation and preservation as they get oxidized immediately when exposed to air. To overcome this problem an inert atmosphere is being used in many experimental arrangements such as Argon, Nitrogen [9-11] and also for the reduction of copper salt many reducing, capping or protecting agents are used. A large number of synthesis methodologies by reduction with hydrazine in ethylene glycol under microwave irradiation [12], pulse electrodeposition [13], seed-mediated growth [14], electrochemical deposition [15,16] have been reported. Among all these well-developed techniques, electrochemical deposition is one of the most promising techniques for preparing the Cu NPs. This technique is non-toxic and easy to use.
In the present work, a simple electrodeposition technique is used to synthesise the spherical Cu NPs through an electrochemical deposition on aluminium substrate. This method is inexpensive, technically simple in material synthesis and can be used as an alternative method for the uniform distribution of nanoparticles on the substrate. The synthesized Cu NPs can be used effectively in variety of applications such as plasmon enhancement of light harvesting in photovoltaic cells, catalysis, biosensors, fuel cells, electronic nanodevices etc. The electrodeposited Cu NPs were analysed using X-ray diffraction patterns for structural properties and morphological properties were studied by SEM micrographs.

2. Experimental

Electrodeposition of Cu NPs was carried out by immersing Aluminium substrate in an aqueous electrolyte prepared from aqueous solution of 0.1 M copper sulphate ($Cu\,SO_4.5H_2O$) and 1 M citric acid at room temperature. Aluminium substrate was cleaned with acetone to remove possible contaminants. All the chemicals were purchased from Thomas baker. The electrodeposition was performed with a two electrode system. The Aluminium substrate was used as the working electrode and graphite rod as a counter electrode. The electrodeposition was carried out at 5V using a supply for the deposition time of 40 minutes at room temperature. After deposition, the samples were dried out in the air.
The morphology of the as prepared Cu NPs was characterized with a JEOL-JSM 6360-A scanning electron microscopy (SEM). To know the Chemical composition of the prepared Cu NPs energy dispersive X-Ray analysis (EDAX) was performed. The X-ray diffraction (XRD) spectra was recorded on a Rigaku D/max-2400, CuKα = 0.154nm X-ray diffractometer.

3. Results and Discussion

3.1. SEM Analysis

Figure 3.1 represents the beautiful SEM images of as synthesised Cu NPs at room temperature with different magnifications. Highly dense Spherical Cu NPs were uniformly distributed on the aluminium substrate. The nanoparticles are large and well defined with some agglomeration. This may be due to, as the deposition time increases the density of nanoparticles increases but the size of the particle decreases [16]. It is possible to control size and shape of the nanoparticles by varying the deposition time, applied potential and the concentration of the copper sulphate. The energy dispersive X-ray analysis (EDX) was performed to know the elemental composition of the sample. The EDX spectrum confirms the presence of Cu, Al and O without any impurities. The atomic percentage of Cu on the substrate is 14.74%.

3.2 XRD Analysis

The XRD analysis was performed to confirm the crystal structure and crystalline quality of the as synthesized Cu NPs. The recorded XRD spectra of the prepared Cu NPs is shown in figure 2. The prominent diffraction peaks at 43.60^0, 50.86^0 and 74.63^0 can be indexed to (111), (200) and (220) planes respectively perfectly. XRD patterns showed the presence of pure crystalline Cu with face centred cubic (FCC) structure (JCPDS data file 04-0836). The additional diffraction peaks at 45.15^0 and 65.48^0 may be due to aluminium substrate. The average crystalline size of the as synthesised Cu NPs was calculated to be around 65nm respectively according to the half width of the (111) diffraction peak using Debye–Sherrer formula.

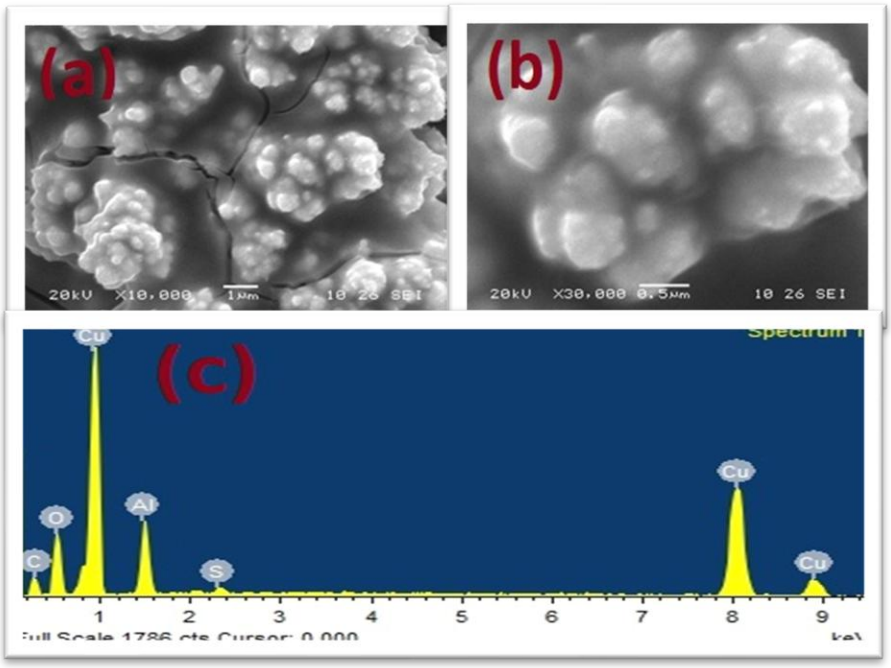

Fig. 1 (a, b) Low and high magnification SEM micrographs of electrodeposited Cu NPs. (d) Corresponding EDX spectra.

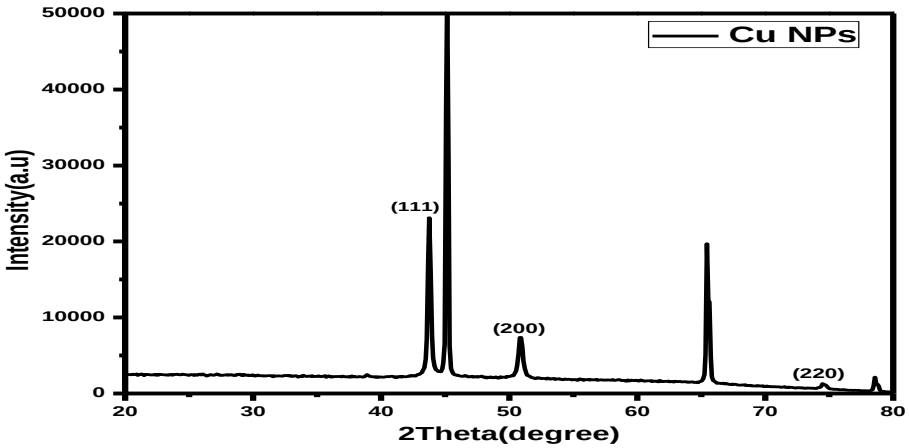

Fig. 2 XRD Spectra of the electrodeposited Cu NPs.

4. Conclusion

A very simple and cost effective synthesis of Copper nanoparticles was performed using electrodeposition technique. Highly dense spherical Cu NPs of 65 nm crystallite sizes were uniformly distributed on the aluminium substrate. By varying the deposition time and the concentration of electrolyte the growth of the Cu NPs can be controlled. The synthesized Cu NPs can be used effectively in variety of applications such as plasmon enhancement of light harvesting in photovoltaic cells, catalysis, biosensors, fuel cells, electronic nanodevices etc.

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