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Synthesis of Cuprous Oxide Nano Cubes and Platelets Using Both Electrodes of Copper

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ABSTRACT

Cu_2O (Cuprous Oxide) is usually produced using expensive electrodes such as platinum, graphite, etc. In the present study, it is shown that Cu_2O nanostructures can be synthesized by electrolysis using copper as both anode and cathode. In the present study, the effect of electrolyte pH on the morphology of the Cu_2O nanostructures was studied. For this, the copper electrodes of 99.99% purity with 3x3x0.3 mm dimensions were dipped in the electrolytes of simple distilled and double distilled water and connected with the external power source. The synthesized products were analyzed using SEM (Scanning Electron Microscope), EDS (Energy Dispersive Spectroscope) and UV spectroscope. Results showed that Cu_2O nanostructures of 67 and 150 nm size were developed when the pH of electrolyte was 6.4 and 5.7 respectively, otherwise not. From the course of experiments conducted in this work it is noted that pH play vital role in the production of Cu_2O nanoparticles using simple electrolysis technique.

Key Words: Cuprous Oxide, Nano-Platelets, Nano-Cubes, Electrolysis, Scanning Electron Microcopy.

1. INTRODUCTION

Nanostructures such as nanowires and nanocubes attracts the considerable attention in recent years as they have a lot of superior electronic, electrochemical, catalytic and magnetic properties [1]. They have been utilized for different applications in microelectronics, solar energy, organic contaminants conversion, chemical industry, photovoltaic materials, gas sensors, biosensors, lithium ion batteries, nonmagnetic devices, semiconductors, and as well as in super conductors [2]. Despite to this, these materials can also be used as a catalyst for water splitting under visible light [3-4]. Obviously. there are many advantages of Cu_2O nanostructures too, to be used as a photo catalyst of low toxicity, environmentally friendly, a low cost electrode and easily available in abandoned quantity material [5-7]. Several methods have been reported to synthesize the Cu_2O nanostructures, that is, electrodeposition/ electrolytic, sonochemical, PVD (Physical Vapor Deposition), thermal relaxation, liquid-phase reduction, the CPSA (Complex Precursor Surfactant-Assisted) Route

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and vacuum evaporation. All these approaches, usually employ a complex control over the process as well as they require the expensive materials/chemicals to fabricate Cu_2O nanostructures [2,8].

In the application point of view, Cu_2O is considered as a promising and effective p-type semiconductor ceramics, having direct bandgap of 2.1 Ev [2,5-8], while the bandgap of 1.64 eV could be achieved if the hydrothermal electrochemical deposition technique was used to deposit thin films [9]. Further to this, hallow nanospheres with different outer diameters (100-200 nm) were fabricated by Chang et. al. [11], to claim that the respective band gap (E_g= 2.405-2.170 eV) of different sizes with different colours respectively, of Cu_2O were being discussed for the application of solar energy harvesting devices in the visible range. Rakhshani et. al. [12], reviewed and emphasized along with the size scale, the processing technique parameters also have the effect on the band gap of Cu_2O. In general, it is noted that materials having nanoscale size, possessing quite different properties as compared to the microstructural materials. For instance, Cu_2O nanostructures were also exhibited comparatively useful and enhanced optical and electronic properties, inverse to the bulk [1,11]. Cu_2O nanoparticles can be stabilized by forming a thin layer of copper(II) oxide, also known as cupric oxide (CuO), on the surface of Cu_2O. Pure Cu_2O are not stable in bulk and does not show any passivity under ambient atmosphere and temperature. In contrast, it was worthwhile, the engineered nano size (around 25nm) of Cu_2O was reported more stable than CuO [7-8]. Researchers have also synthesized and applied Cu_2O nanostructure in combination with different materials such as aluminum, graphene, polymers, etc.[12-16].

Despite to use the cheap electrodes made of Cumetal, many workers have been produced Cu_2O nanostructures by utilizing expensive electrodes of Pt and graphite as cathode [5,7]. In this work, the production of Cu_2O nanostructures were obtained through the electrolysis process, where the Cu plates were used for both cathode and anode at the same time. In addition, the distilled water, with different pH values, was used to study the effect of the alkaline and acidic nature of the electrolyte (corrosive medium) on morphologies of nano-oxides (corrosive product) of Cu.

2. EXPERIMENTAL PROCEDURE

2.1 Materials Used

Copper (99.99% pure) plates were purchased and used after rough polishing with alumina (0.2 μm) containing suspension followed by intermediate polishing and thorough cleaning in analytical grade acetone. Distilled water of different pH values was obtained locally.

2.2 Synthesis of Cuprous Nanostructures

In present work, the two pure Cu metal electrodes were placed parallel to each other into the electrolyte. In all experiments, Cu plates of the same dimensions were used, as mentioned in Table 1. Throughout the experiments, cathode to anode ratio was set to 1:1 and distance between them was 1 cm. Whereas, the pH and conductivity of the water used as an electrolyte is shown in Table 1.

The copper plates were supported and mounted vertically downward with the help of polymer lid as shown in Fig. 1. Than anode and cathode were connected to DC power supply by using copper wire (99.9% pure) and finally electrolysis experiments were conducted at different current and volts as shown in Table 2.

2.2 Characterization

The material deposited on either anode or cathode was analyzed using SEM (FEI Quanta 200), and UV-Visible spectrophotometer (22pc spectrum lab).

3. RESULTS AND DISCUSSION

Different nanostructures of Cu_2O, shown in Figs. 2-3, were formed during the course of electrolysis experiments. It can be seen in Figs. 2-3 that when pH value was less than 7, the mixed nanostructures containing nano-cubes and nano-platelets were developed. Analysis of SEM images indicated that dimensions of nano-plates and nano-cubes developed, below pH 7, was roughly 67-100 and 120-132 nm respectively.

In order to ensure that product collected at cathode and anode, when pH was less than and greater than 7, is copper, the EDS analysis and UV visible spectroscopy was carried out. The EDS analysis result shown in Fig. 4 confirmed that product is mainly composed of copper. Similarly, the spectrum of UV visible spectroscope, shown in Fig. 5, further provided the supported evidences.

FIG. 1. EXPERIMENTAL SETUP

TABLE 1. ELECTRODE PLATE (COPPER) DIMENSIONS

Electrode Properties	Cathode Material		99.9% Copper	
	Anode Material		99.9% Copper	
	Electrode Dimensions (cm)		3x3x0.3	
	Electrode Surface Area (cm^2)		20.7	
Electrolyte Test Results	Water (Electrolyte)	Double Distilled	Single Distilled	Single Distilled
	pH	5.704	6.4	7.9
	Conductivity (Micro Siemens per cm)	1.6	16.8	98.7

TABLE 2. OPERATING CONDITION OF EXPERIMENT

Electrolyte			Operating Parameters		
Electrolyte	pH	Conductivity	I (amp)	V (volts)	Time (mins)
1	5.704	1.6	0.001	20	120
2	6.400	16.8	0.001	20	120
3	7.900	98.7	0.001	20	120

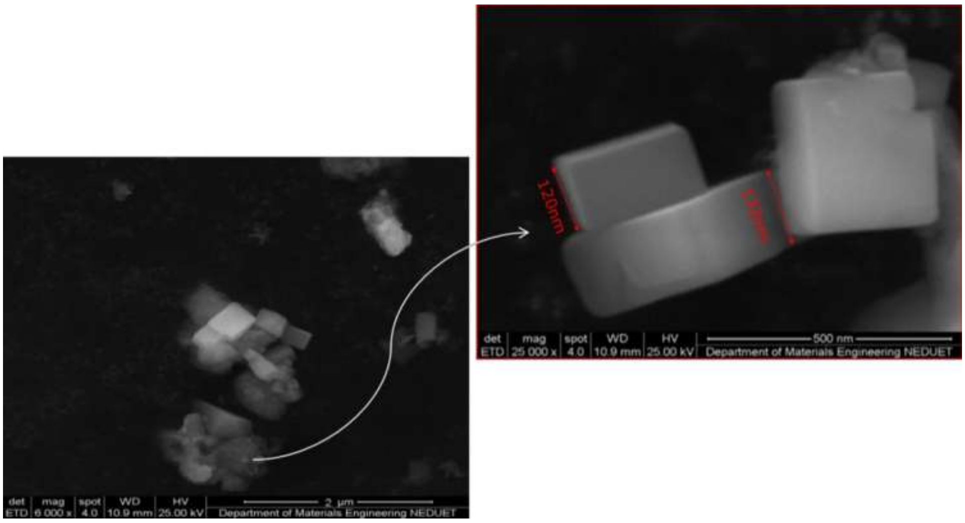

FIG. 2. SEM IMAGE OF NANO-COPPER PARTICLES OBTAINED AT pH 5.7

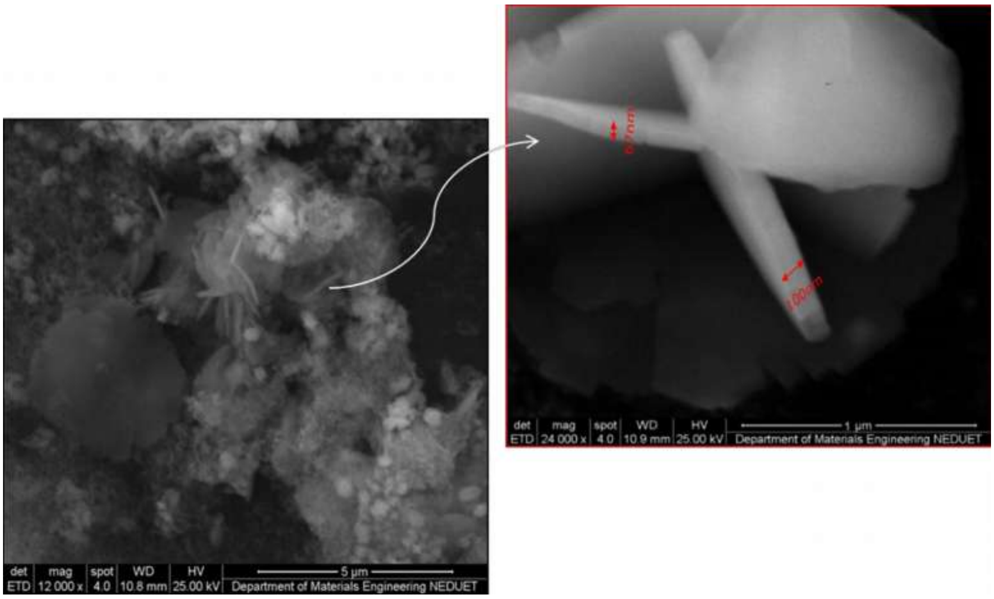

FIG. 3. SEM IMAGE OF NANO-COPPER PARTICLES OBTAINED AT pH 6.4

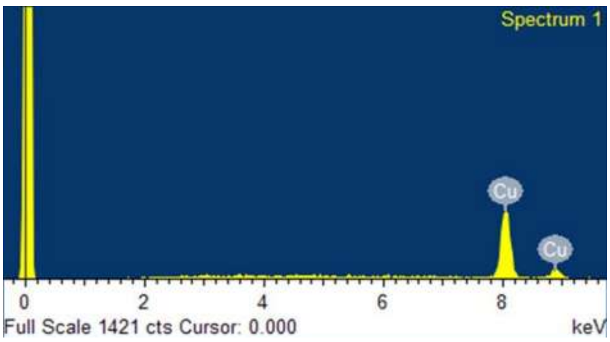

FIG. 4. ENERGY DISPERSIVE X-RAY SPECTRUM OF OBTAINED PRODUCT

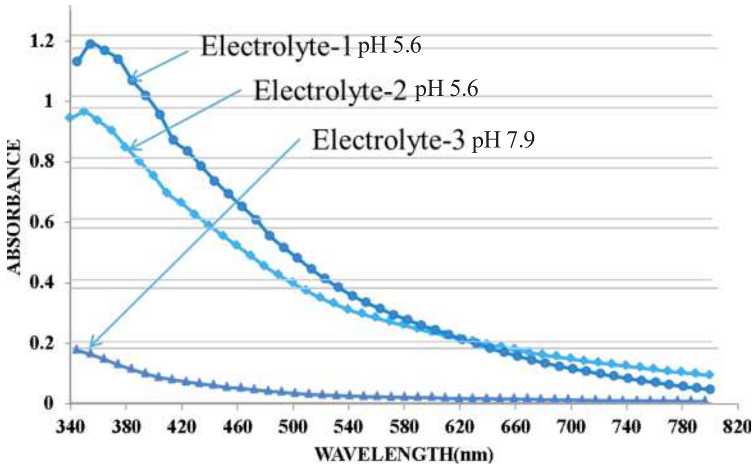

FIG. 5. UV ABSORBANCE OF PRODUCT OBTAINED AT THREE PH VALUES

5. CONCLUSION

Following are the main conclusions of the present work:

(i) pH of the electrolyte plays vital role in the synthesis of the nano-copper plates.

(ii) The shape and size are strongly dependent on the pH of the electrolyte

(iii) Acidic conditions of electrolyte are optimal for synthesis of nano copper

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