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# ELECTRODEPOSITION OF $CU_2O$ PARTICLES BY USING ELECTROLYTE SOLUTION CONTAINING GLUCOPONE AS SURFACTANT.

**Dahyunir Dahlan**
Physics Department, University of Andalas, Kampus Limau Manih Padang – Indonesia
dahyunir@yahoo.com

**Abstract**

Copper oxide particles were electrodeposited onto indium tin oxide (ITO) coated glass substrates. Electrodeposition was carried out in the electrolyte containing cupric sulphate, boric acid and glucopone. Both continuous and pulse currents methods were used in the process with platinum electrode, saturated calomel electrode (SCE) and ITO electrode as the counter, reference and working electrode respectively. The deposited particles were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). It was found that, using continuous current deposition, the deposited particles were mixture of $Cu_2O$ and CuO particles. By adding glucopone in the electrolyte, particles with spherical shapes were produced. Electrodeposition by using pulse current, uniform cubical shaped $Cu_2O$ particles were produced.

Keyword: Electrodeposition, $Cu_2O$, ITO, surfactant, glucopone

**Abstrak**

Partikel-partikel oksida kuprum diendapkan diatas substrat gelas yang telah dilapisi dengan oksida timah indium (ITO). Elektropengendapan dilakukan terhadap elektrolit yang mengandung kuprik sulfat, asam borik dan glukopon. Arus kontinyu dan arus pulsa digunakan dalam proses ini. Elektroda platinum, elektroda kalomel jenuh (SCE) dan elektroda ITO berturut-turut digunakan sebagai elektroda penghitung, elektroda rujukan dan elektroda kerja. Partikel-partikel yang terendap dikarakterisasi menggunakan difraksi sinar-X (XRD) dan mikroskop pengimbas elektron (SEM). Dengan menggunakan arus berkesinambungan didapatkan pengendapan yang terdiri dari partikel-partikel $Cu_2O$ dan CuO. Penambahan glukopon pada larutan elektrolit menghasilkan partikel-partikel berbentuk bulat pipih. Sedangkan dengan menggunakan arus pulsa dihasilkan pengendapan partikel-partikel $Cu_2O$ dengan bentuk kubik yang seragam.

Kata Kunci: Elektropengendapan, $Cu_2O$, ITO, surfaktan, glukopon

## 1. Introduction

The copper oxide received a great deal of attention nowadays for numerous applications such as solar cells and photovoltaic material, electrochromic coating, catalytic application and high Tc-superconductor. The copper oxide system (Cu-O) has two stable oxides: cupric oxide (CuO) and cuprous oxide ($Cu_2O$). $Cu_2O$ for examples, due to there unique properties such as n- and p-type semiconductor, non toxic nature, low cost production and band gap lie in an acceptable range for solar energy conversion, make them highly sough for the currently existing application. However CuO is also possessing n-type semiconductor [Figueiredo et. al 2008].

CuO and $Cu_2O$ thin films can be prepared by several methods such as chemicall vapor deposition (CVD) [Piszczek et. al 2008], electroless plating [Carvalho et. al 2008] and electrodeposition [Dong et. al 2008]. Recently have been received special attentions for fabrication Cu-O via electrodeposition by using different current shape (pulse and continued) and added of surfactant to electrolyte solution.

In this paper we use electrodeposition technique to prepare $Cu_2O$ particles onto ITO substrate by controlling current shape and added surfactant effect. Variable morphology of $Cu_2O$ particles such as cube, spherical and irregular particle can be prepared. The mechanism for shape control grown of $Cu_2O$ particles will be discussed.

## 2. Experimental

The solution for electrodeposition consisted of 0.01M $CuSO_4$ was obtaining from Sigma, 0.243M $H_3BO_3$ from Merck, and 3% wt glucupone 215 CSUP (a technical grade alkyl polyglucosides) was supplied by Fluka Chemica. The boric acid was used as buffer solution. Glucopon was used as

surfactant to control particles size. Solution was stirring for 30 minutes in beaker glass. All electrochemical deposition was carrying out in a glass compartment comprising three electrodes. A platinum plate of 1 $cm^2$ was employed as counter electrode while saturated calomel electrode (SCE) as reference electrode. Indium tin oxides (ITO) with cutting area 1 $cm^2$ coated glass were used as working electrode. The electrodeposition process was performed by using a galvanostat, Gamry Instrument DC105 at room temperature (25°C) under a constant current density of 0.5mA/$cm^2$. Both continuous and pulse currents methods were used in the process. For continuous current, electrodepositions have done for 120 seconds continuously. However, electrodeposition by using pulse current, deposition process was interrupted every 2 second by on/off current for 2 x 120 seconds. While a part of them were characterization by using X-ray and SEM directly. The present investigation is to clarify the microstructure and growth features of the Cu-O particles by using X-ray diffractometer (XRD) Siemen D500. The morphology of electrodeposited Cu-O was studied by using a scanning electron microscope (SEM) LEO1450 VP.

**3. Results**

Fig. 1 shows electrodeposited of Cu-O particles onto ITO substrate. Particles produced from electrodeposition process by using continuous current of 0.1 M $NiSO_4$ +15 g /l $H_3BO_3$ solution for 120 second is shown on fig 1a. Cubic particle with size about 500 nm deposited onto substrate, and also dispersed of smooth particles can be seen. X-ray analysis for the sample (fig. 2a) confirms the product of cubic is $Cu_2O$ particles, and the other smooth particles are CuO. By comparing by JCPDS confirms that $Cu_2O$ reflected by planes at 29.25° (110), 36.54° (111) and 42.8° (200) of 2θ angel. The crystal shape preferred particles is cubic crystalline. On the other hand, CuO reflected by planes at 36.70° (111), 43.00° (200) with lower intensity. While electrodeposition process by using pulse current, that is on/off current every 2 second for (2 x 120) second, the cubic $Cu_2O$ particles were produced on to substrate (fig. 1b). Distribution of $Cu_2O$ is denser. The peak of X-ray diffraction for the corresponding sample investigates only $Cu_2O$ were produced (fig.2b). When electrolyte solution of $CuSO_4$ was containing 3% wt. of glucopone, the electrodeposition have produced spherical Cu-O particles. Fig. 1c show electrodeposited Cu-O particles synthesize from cupric sulfate by using surfactant glucopon with continous current. The presence of glucopone in the solution has lead to a sphere Cu-O particles. However, while electrodeposition via pulse current, the particle produced sphere particles which were uniformly distributed over the substrate and denser (fig.1d). It is believed that glucopone has controlled morphology growth during electrodeposition process [Peycova et.al]. The addition of glucopone to the solution have produced sphere particles which were uniformly distributed over the substrate surface. Fig. 2 showed x-ray diffraction of electrodeposited Copper oxide onto ITO substrate. In Fig 2a, $Cu_2O$ reflected by angel 20°, 36° and 42° and CuO reflected by angel 36.5° and 43°. In Fig. 2b, $Cu_2O$ phase can be detected without CuO phase. Its mean, electrodeposition by using pulse current produced cubic $Cu_2O$ only. However electrodeposition by using glucopone as surfactant with continuous or pulsed current is produced amorphous $Cu_2O$ and CuO particles phase (Fig 1.c, Fig 1d and Fig 2c, 2d)

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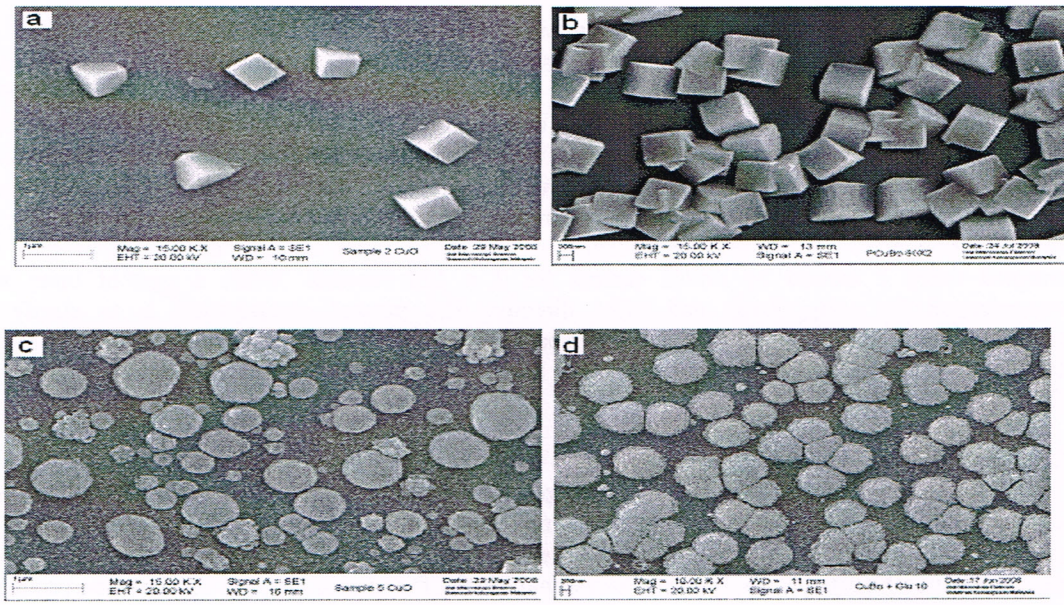


Figure 1. Micrograph electrodeposited copper oxide onto ITO coated glass substrates using solution 0.1M $NiSO_4$ + 0,243M $H_3BO_3$ by: 1a. continuous current for 120sec., 1b. pulsed current (on off current) for 2x120sec., 1c. continuous current for 120 sec. with 3% wt glucopene, 1.d. pulsed current ( on off current) for 2x120sec with 3% wt glucopone.

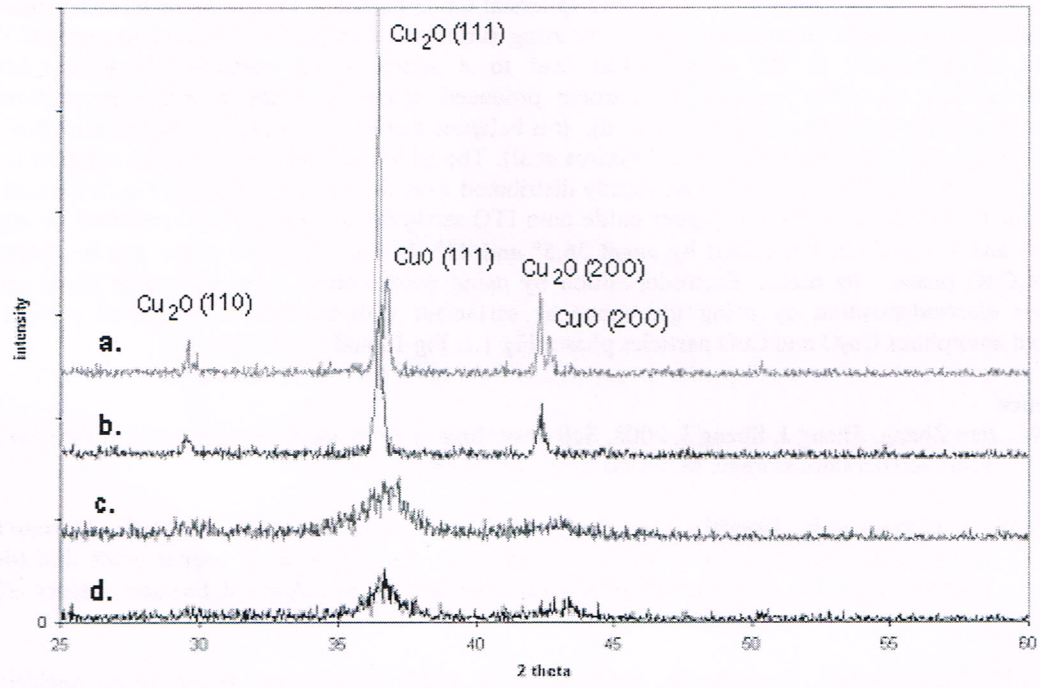


Figure 2. X-ray diffraction of electrodeposited copper oxide onto ITO coated glass substrates using solution 0.1M $NiSO_4$ + 0,243M $H_3BO_3$ by using: 2a. Continue current for 120sec., 2b. Pulse current (on off current) for 2x120sec., 2c. Continue current for 120 sec. with 3% wt glucopone, 2d. Pulse current (on off current) for 2x120sec with 3% wt glucopone.