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Preparation of thin layer CuO from Cu_2O using the Spin Coating Method at Various Annealing Temperature and Number of Dripping for Photoelectrochemical Water Splitting

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Abstract

A thin layer preparation of CuO from Cu_2O powder using Fehling's solution for photoelectrochemical applications has been performed. The research was focused on studying the effect of annealing temperature and the number of drops on the performance of CuO thin layer semiconductors from Cu_2O powder prepared by spin coating with a rotation rate of 500 rpm for 15 seconds. The thin layers were treated with annealing with temperature variations of 300°C, 400°C, and 500°C for 1 hour and variations in the number of drops of 10, 20, and 50 drops. The CuO thin layer was tested in a photoelectrochemical process as a photocathode to split water with a simulated light of 1.5 AM (100 mW/cm²). The process of splitting water as a method of producing hydrogen energy by photoelectrochemistry is assisted by semiconductors, such as CuO, in an electrolyte solution to capture photons and drive the water-splitting reactions. Copper (II) Oxide (CuO) is a p-type semiconductor with a band gap of 1.2–2.5 eV, which can be used as a photocathode. The optimum photoelectrochemical measurement results were obtained at an annealing temperature of 400°C and 50 drops with a current density of 0.584 mA/cm² at a potential of 0.2 V versus the Reversible Hydrogen Electrode (RHE). The results of the Scanning Electron Microscope (SEM) analysis show that the morphology of the oxide is spherical. Energy dispersive X-ray (EDX) analysis displays that the sample contained 51.46% and 48.54% of Cu and O, respectively. The X-ray diffraction pattern (XRD) analysis shows that the oxide grain size is 44.137 nm.

1. Introduction

Hydrogen energy has great potential to be used as a renewable energy source. Hydrogen can be obtained through the photoelectrochemical (PEC) water splitting process. In PEC, solar energy is absorbed by the semiconductor material and produces electron-hole pairs, followed by a chemical reaction at the electrode-electrolyte interface to produce hydrogen [1]. To reduce the cost of converting light energy to hydrogen, light-absorbing semiconductor materials need to be made at a low cost but have good photoelectrochemical properties and stability [2].

Semiconductors used in the photoelectrochemical water splitting process must have a band gap value of 1.5–2.5 eV [3]. Copper oxide is an interesting semiconductor material to develop, because of its abundance in the earth's crust, non-toxic, easy to synthesize, high absorption ability in the visible light region, low production costs, and includes p-type semiconductors, so it is suitable for use as photocathodes in hydrogen production [4, 5, 6]. The two most common forms of copper oxide are copper (I) oxide (Cu_2O) and copper (II) oxide (CuO). Cu_2O is a semiconductor with a band gap value of 2–2.5 eV [5], while CuO has a lower band gap of 1.2–2.5 eV, which makes it more ideal for absorbing solar energy [7].

Annealing Cu_2O in the air at temperatures above 300°C can change the Cu_2O phase to the CuO phase because the Cu (II) component is more dominant than Cu (I) [2].

The synthesis of Cu_2O powder is generally carried out through oxidation of pure copper [8, 9] and reduction of Cu^{2+}. The most common method is Cu^{2+} reduction using Fehling's solution and glucose. The Fehling method is used because it can produce Cu_2O in a simple nanometer-size [10]. CuO thin layers can be prepared using the spin coating method with variations in the annealing temperature. Spin coating was chosen because this method produces a uniform thin layer with a nanometer thickness [11, 12].

The light absorption ability of the semiconductor affects the performance of the PEC cell. The process of light absorption by semiconductors can be affected by the thickness, grain size of the crystal, and the thin layer crystallinity. Annealing treatment was carried out to change the structure of Cu_2O to CuO and study its effect as a photocathode [7]. In this research, a thin layer semiconductor CuO was prepared from Cu_2O powder using a spin coating deposition. The variation of annealing temperature and the number of drops were studied to determine their effect on the photocathode performance for photoelectrochemical water splitting.

2. Methodology

The research was carried out in 4 stages, including 1) preparation of tools and materials, 2) synthesis of Cu_2O powder, 3) manufacturing of CuO thin layers with spin coating, and 4) photoelectrochemical measurements.

2.1. Materials and equipment

The materials used include Fluorine doped Tin Oxide (FTO) (Sigma Aldrich) glass, $CuSO_4 \cdot 5H_2O$ (Merck), $KNaC_4H_4O_6 \cdot 4H_2O$ (Merck), NaOH (Merck), Na_2SO_4 (Merck), glucose (Merck), tea tree oil, and distilled water. While the equipment used was spin coater, potentiostat (CorrTest CS 150), furnace, oven (Kirin), ultrasonic bath (Alpha), X-Ray Diffraction (XRD) instrument (Shimadzu XRD-7000), and SEM-EDX instrument (JEOL JSM-6510LA).

2.2. Cu_2O Powder Synthesis

Cu_2O powder synthesis was carried out by mixing the same volume of Fehling A (0.027 mol $CuSO_4.5H_2O$) and Fehling B (0.12 mol $KNaC_4H_4O_6 \cdot 4H_2O$ solution with 0.3 mol NaOH) and stirring for 15 minutes. A total of 0.027 moles of glucose as a reducing agent was added to the solution, then heated at 60°C with stirring until a brick-red precipitate formed. The precipitate was then washed with distilled water and ethanol, then dried in an oven. The powder obtained was characterized by XRD and SEM.

2.3. CuO Thin Layer Preparation by Spin Coating

The CuO thin layers were prepared using the spin coating method. FTO glass measuring 2 x 1 cm^2 was previously cleaned with HCl, ethanol, and distilled water to remove impurities. A total of 0.1 gram of synthesized Cu_2O powder was dispersed in 2.5 mL of tea tree oil and stirred for 1 hour. The solution was then sonicated for 10 minutes. In the next step, the FTO glass was placed on a spin coater, then the Cu_2O solution in tea tree oil was dripped on the FTO and was rotated at a speed of 500 rpm, followed by heating with a hot plate at a temperature of 110°C for 5 minutes. In this process, the drop is carried out at 10, 20, and 50 drops. After obtaining a thin layer, the annealing process was carried out with annealing temperature variations at 300°C, 400°C, and 500°C for 1 hour to obtain a CuO thin layer. The thin layers were further characterized using XRD and SEM-EDX.

2.4. Photoelectrochemical Measurements

The photocathode photoelectrochemical properties were measured using a potentiostat with three electrodes in linear sweep voltammetry (LSV) mode. A thin layer of CuO with various annealing temperatures was used as a working electrode, platinum as the anode, and Ag/AgCl as a reference electrode in a 0.1 M Na_2SO_4 electrolyte solution pH 9.0. Measurements were made at a scanning rate of 10 mV/s with and without irradiation using simulated AM 1.5 light every 5 seconds. The photoelectrochemical properties measurements were current density, onset potential, and Applied Bias Photon-to-Current Efficiency (ABPE). The potential in the PEC measurement, which refers to the Ag/AgCl ($E_{Ag/AgCl}$) electrode, is converted to a Reversible Hydrogen Electrode (RHE) using equation (1) [13]:

$$E_{RHE} = E_{Ag/AgCl} + (0{,}059 \times pH) + 0{,}199 \quad (1)$$

Current density measurement was obtained from the J-V curve (current vs potential density). The maximum current density value was determined under irradiation conditions at a specific potential. The measurement of onset potential was carried out by squaring each photocathode's resulting current density and presented in the form of a squared current density (J^2) versus potential (V) curve. The intersection of the two dotted lines was the value of the onset potential [14]. ABPE describes irradiated PEC cells' efficiency at AM 1.5, taking into account the effect of bias applied between the working electrode and the counter electrode [14]. Equation (2) is used to calculate ABPE value.

$$ABPE\ [\%] = J \times V \times 100/P_{AM1,5} \quad (2)$$

3. Results and Discussion

3.1. Cu_2O Powder Synthesis

Synthesis of Cu_2O is carried out by reducing Cu^{2+} by reacting Fehling A and Fehling B solutions with glucose. Fehling's solution contains tartrate ions, which act as a complexing agent to keep Cu^{2+} ions in solution [15] and prevent the precipitation of $Cu(OH)_2$ [16]. The Cu^{2+} ion in the complex in the presence of glucose is reduced to the Cu^+ ion, which results in a brick-red Cu_2O [15]. The brick-red deposit that is formed indicates that Cu_2O has been produced. The precipitate is then washed using distilled water and ethanol to remove impurities. The Cu_2O powder obtained was 1.9052 grams, with a yield of 98.628%.

3.2. CuO Thin layer prepared by Spin Coating

The spin coating process was carried out at a rotating speed of 500 rpm to get a thin layer. The number of drops of 10, 20, and 50 drops was varied to obtain a thin layer with the highest current density value. The thin layer was then annealed with an annealing temperature of 300°C, 400°C, and 500°C for 1 hour to change the structure of Cu_2O to CuO and find out its effect on the performance of CuO semiconductors as a photoelectrochemical in water splitting. The annealing process caused the thin layer to change color, from brick red to black. This shows that in the annealing treatment, the Cu_2O thin layer reacted with oxygen from free air, which oxidized Cu_2O to CuO [17]. The reaction of Cu_2O oxidation with oxygen is as follows:

$$2\ Cu_2O_{(s)} + O_{2(g)} \rightarrow 4\ CuO_{(s)} \quad (3)$$

3.3. Photoelectrochemical Measurements

Photoelectrochemical measurements with simulated light aim to determine the maximum current density produced by the photocathode when irradiated. The first potential of the current appears at the time of exposure (onset potential) and ABPE. The results of current density measurements with variations annealing temperature and the number of drops are shown in Figure 1.

Figure 1 (a) shows that the thin layer without annealing treatment does not indicate a current even though it has been irradiated. This happens because the thin layer is covered by tea tree oil so that the absorption of light by the semiconductor is disrupted. The annealing treatment causes the organic compounds in the tea tree oil to break down so that there is contact between the thin layer of CuO and the FTO substrate and shows a current. The more drops that are superimposed on the thin layer, the higher the current density. At 50 drops, the resulting current density is the largest because the highest amount of Cu_2O is deposited in the thin layer to absorb more light by the thin layer semiconductor. Based on these results, this 50-drop treatment was subsequently used for the measurement of other photoelectrochemical properties.

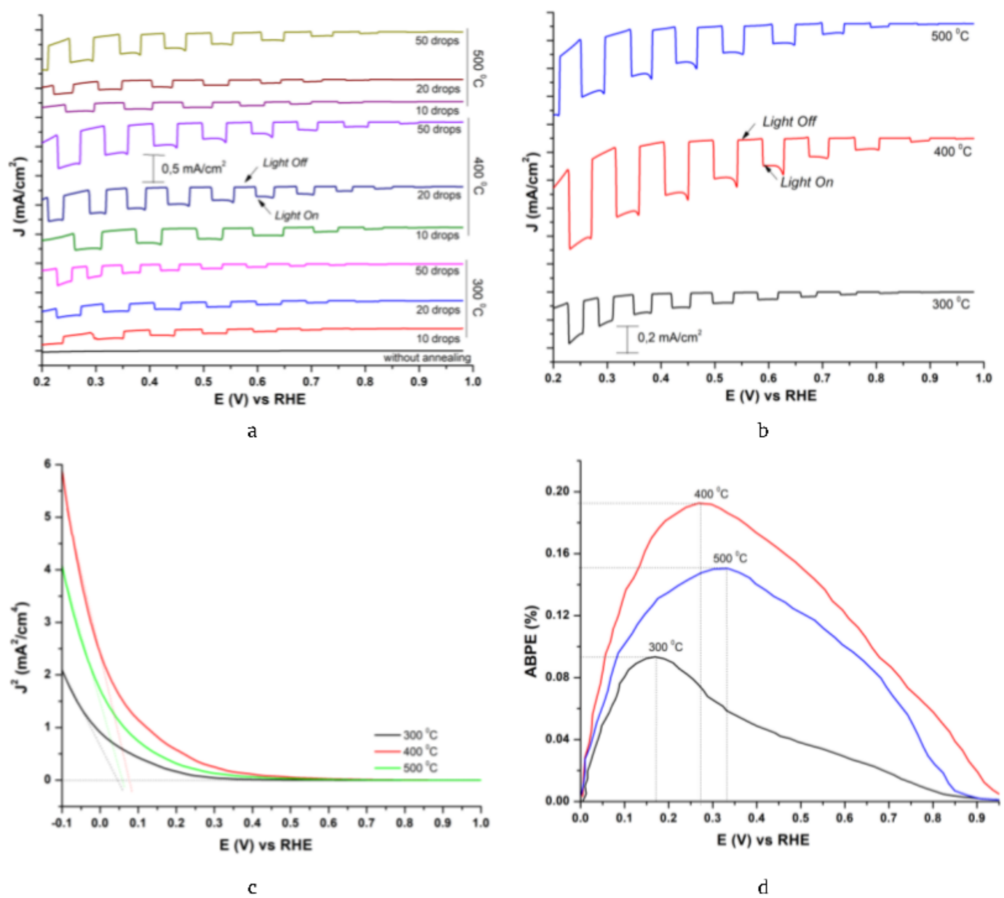


Figure 1. (a) current density of thin layers with variations in the number of drops and annealing temperature, (b) current density of thin layers with variations in annealing temperature with 50 drops, (c) potential onset value, and (d) ABPE value measured at 0.1 M Na_2SO_4 pH 9

The annealing temperature affects the photoelectrochemical properties of the photocathode. At an annealing temperature of 400°C, the maximum current density, onset potential, and the highest ABPE were 0.584 mA/cm² at 0.2 V vs. RHE; 0.08 V vs. RHE; and 0.193%, respectively. Compared with the results of Kharismawati [18] which obtained a current of 0.413 mA/cm² at 0.2 V vs. RHE, the electrochemical properties in this study increased. The annealing temperature of 400°C produced the best crystallinity compared to different temperatures (presented in the XRD diffractogram). The emergence of current density indicates that the semiconductor as a PEC photocathode is successfully used to reduce water to hydrogen gas. The high current density indicates that the light absorption process by the semiconductor is working optimally. The decrease in current density value at an annealing temperature of 500°C was due to the decreasing crystallinity of the CuO layer, indicated by the intensity and the crystal size on the XRD diffractogram, which was lower than the annealing temperature of 400°C. Also, the higher the annealing temperature, the resistivity of the FTO substrate increases, resulting in a decreased ability to flow electrons [19]. A more positive onset potential and an immense ABPE value indicate the CuO semiconductor performance in responding to photon energy. The greater the ABPE value, the higher the semiconductor efficiency.

3.4. Characterization of CuO Thin Layer

XRD characterization was used to determine the crystal's average grain size and identify the crystal structure of the synthesized Cu_2O powder and CuO thin layer semiconductor with variations in annealing temperature. The results of the XRD analysis of the four samples are presented in Figure 2.

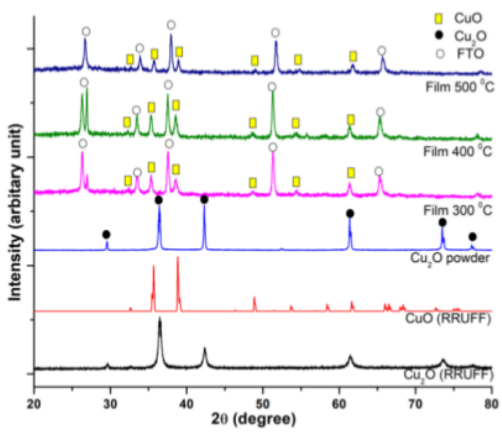


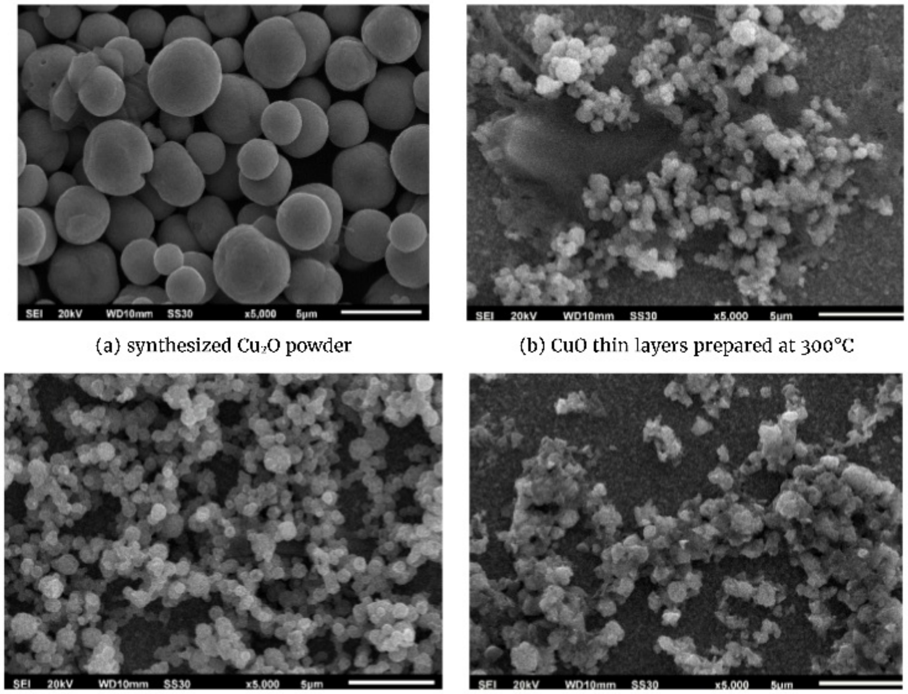
Figure 2. XRD diffractogram of synthesized Cu_2O powder and CuO thin layers with variations in annealing temperature

Figure 2 shows the diffraction peaks of the synthesized Cu_2O powder sample with a value of 2θ of 29.59°; 36.44°; 42.32°; 61.39°; 73.52° and 77.38°. The peaks that appear correspond to the standard Cu_2O peaks (RRUFF no R050384) at 2 of 29.60°; 36.47°; 42.34°; 61.39°; 73.50° and 77.40°. These results indicate that Cu_2O has been successfully formed. In all thin-layer samples with variations in annealing temperature, there was a CuO peak that was in accordance with the CuO standard (RRUFF no R120076), namely at 2θ of 32.48°; 35.48°; 38.64°; 48.88°; 53.35°; 61.65° which indicated that the annealing temperature treatment caused Cu_2O to be oxidized to CuO [17]. At annealing temperature of 300°C, CuO peaks appeared at 2θ of 32.42°; 35.36°; 38.58°; 48.73°; 53.40° and 61.72°. The thin layers prepared at 400°C showed a CuO peak at 2θ of 32.35°; 35.35°; 38.56°; 48.79°; 53.26° and 61.56° with a higher intensity than other temperature variations. Meanwhile, the thin layer prepared at 500°C appeared CuO peaks at 2θ of 32.72°; 35.73°; 38.48°; 48.83°; 53.46° and 61.74°.

The average grain size of crystals in the samples of synthesized Cu_2O powder, CuO thin layers prepared with an annealing temperature of 300°C, 400°C, and 500°C calculated using the Debye-Scherrer formula were 59.345; 33,977; 47,459 and 43,343 nm. The crystal size decreases when it is made into thin layers. This is because the sonication process causes the particles to experience splitting of intermolecular interactions due to vibrations from ultrasonic waves, which result in a decrease in particle size [20]. The crystal size has increased from an annealing temperature of 300°C to 400°C. This may be due to the incorporation of smaller grains and an increase in the CuO diffractogram intensity, which increases its crystallinity [21].

On the other hand, at an annealing temperature of 500°C, the grain size and decreasing the intensity of the CuO diffractogram caused a decrease in crystallinity. These results are in accordance with research conducted by Armouzi *et al.* [21]. As crystallinity increases, charge separation and charge transfer is easier because less resistance occurs, so the resulting current density is higher [2, 14].

SEM analysis was used to determine the surface morphology of the synthesized powder samples and a thin layer of CuO with variations in annealing temperature. Figure 3 shows that all samples show a spherical morphology. The annealing treatment causes agglomeration (merging) of the smaller grains and makes the spherical shape unclear due to grain boundaries changes. CuO thin layer prepared at an annealing temperature of 400°C showed homogeneous surface morphology and evenly distributed grain compared to other annealing temperature variations. A more even and homogeneous surface increases the light absorption process, which causes the resulting high current density value, and the photoelectrochemical water splitting process runs more efficiently.



(a) synthesized Cu_2O powder (b) CuO thin layers prepared at 300°C

(c) CuO thin layers prepared at 400°C (d) CuO thin layers prepared at 500°C

Figure 3. Surface morphology of synthesized Cu_2O powder samples and CuO thin layers with variations in annealing temperature

EDX analyzed the composition of each element contained in the sample. The quantitative composition of the elements Cu and O is presented in Table 1.

Table 1. Elemental composition (%) in samples from EDX analysis

Semiconductors	Elements (Mass%)	
	Cu	O
synthesized Cu2O powder	84.57	15.43
CuO thin layers prepared at 300°C	31.12	68.88
CuO thin layers prepared at 300°C	51.46	48.54
CuO thin layers prepared at 300°C	28.02	71.98

The EDX results show that the sample contains Cu and O elements, which indicates that the synthesis of Cu_2O powder and the preparation of CuO layers has been successful. The treatment of annealing temperature variations causes a change in the amount of Cu in the sample. In the synthesized Cu_2O powder, the Cu element is more than the element O. This corresponds to the mole ratio. At 500°C, there is more O element than Cu. This is due to FTO interference as a substrate containing O. At 400°C, the mass ratio of the elements Cu and O is not too different. So, it can be said that the formation of CuO with various annealing temperatures has been successfully carried out. This result strengthens the previous XRD analysis, which proves the presence of CuO peaks on the diffractogram, and the results are following photoelectrochemical measurements.

4. Conclusion

Synthesis of Cu_2O can be carried out by reducing Cu^{2+} ions using the Fehling method. The variation of annealing temperature in the manufacture of CuO thin layers from Cu_2O affects the thin layer's photoelectrochemical properties. The results of photoelectrochemical measurements at annealing temperature variations of 300°C, 400°C, and 500°C showed optimum results at 400°C with a current density of 0.584 mA/cm^2 at 0.2 RHE and the resulting onset potential of 0.08 V with an efficiency of 0.151 % at 0.332 RHE.

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Preparation of thin layer CuO from Cu20 using the Spin Coating Method at Various Annealing Temperature and Number of Dripping for Photoelectrochemical Water Splitting

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Instructor

PAGE 1

PAGE 2

PAGE 3

PAGE 4

PAGE 5

PAGE 6