



Investigation of Structural Characteristics of Single-Phase Cuprous Oxide (Cu₂O) Powder Prepared By Chemical Method

¹Ramatu Jummai Umar, ²Tijjani Hassan Darma, ³Kazeem Suleiman

¹Demonstration Secondary School, Ahmadu Bello University, Zaria,

²Department of Physics, Bayero University, Kano

³Department of Science Education, Abubakar Tafawa Balewa University, Bauchi

ABSTRACT

Energy is essential to sustain human society and continues development of planet Earth but Humans have relied more often than not on fossil fuel for energy since the start of the civilized world. The problem is that the reserves of fossil fuels are limited, and the fossil fuel will be depleted shortly if the usage of this primary energy supply remains at its current level, hence the need for an alternative source of energy such as solar cell which is made from silicon. Therefore, it is paramount to understand the structural characteristics of cuprous oxide. Most of the deposition methods employed in the preparation of copper oxides result in the formation of combined phases of Cu₂O and CuO. This study reports a simple and efficient technique used in synthesizing a single-phase Cu₂O powder via the chemical method. XRD characterization of the powder sample using Empyrean diffractometer DY 674 (2010) reveals polycrystalline Cu₂O phase, no CuO phase was observed. The average grain size evaluated using Scherer's formula was found to be in the range of 56 – 80nm.

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INTRODUCTION

The growing population of the world, industrialization and the desire for comfort has led to the demand for an alternative reliable, less costly, and friendly source of energy. This has resulted in the development of many new technologies. The most promising technologies are based on the utilization of solar radiation to either generate heat or electricity and window glazing that provides comfort at homes and offices. These include solar concentrators, window coatings, and solar cells. Solar concentrators require a receiver which absorbs the radiation and convert it to another form of energy. A combination of solar cell and photovoltaic (PV) concentrator uses optics to focus sunlight on a small receiving solar cell; thus, the cell area in the focus of the concentrator can be reduced by the concentration ratio. At the same time, the light intensity on the cell is increased by the same ratio. In other words, cell surface is replaced by lens or mirror surface in PV concentrators and the efficiency and price of both determine the optimum configuration (Luque and

Andreev, 2007). In solar cells, light from solar radiation is converted into electricity using semiconducting materials. One of the important design criteria in the development of an effective solar cell is to maximize its efficiency in converting sunlight to electricity. Window coatings are applied on any glass window or door for decoration, privacy and to filter out solar radiation. Window coating is done using a window film to coat window most of which are UV rated, which means that they can filter out more than 90% of UV ray from the sun.

Solar energy is by far the renewable energy source with the greatest potential. It can meet the world's energy demand several thousand times. Solar energy is readily available in abundance particularly in tropical regions like Africa. Today's political considerations to move the world's energy consumption away from fossil fuels such as oil, coal and gas has increased the focus on renewable energy. Renewable energy is perceived as a sustainable solution to ensure future energy supply as well as being

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environmentally friendly, free of carbon-dioxide emissions.

One of the alternative materials is cuprous oxide due to the abundance, less cost and low toxicity of the starting material (copper), and simple methods of preparation among others. Musa et al, (1998) produced cuprous oxide by thermal oxidation and studied its physical and electrical properties. During this process, the copper foils were heated in the range of 200 to 1050 °C. It was noted that in the temperature range of 1040-1050°C, only one phase i.e. cuprous oxide existed while in the temperature range of 200-950 °C, the mixture of cuprous and cupric oxide was observed. (Abdul, 2008)

Wang, (2006) synthesized cuprous oxide from a 0.4 M solution of copper sulphate mixed with a 3.0M lactic acid. The lactic acid was to stabilize Cu (II) ions at a bath pH higher than 7.0. The bath pH could be controlled between 5.8 and 12 by addition of 4.0M NaOH. The effect of deposition condition on the properties of cuprous oxide was studied. It was shown that the growth rate was a function of deposition time at a constant temperature of 60 °C and a constant pH of 9.0, 10.0 and 12.5 with an increased thickness of the deposited film. With the increase in time, the growth rate was decelerated in contrast to that in a physical vapour deposition method. The reason behind this phenomenon was that with increasing film thickness, the effect on conductivity in the cell by indium tin oxide (ITO) was replaced by a less conductive material i.e. cuprous oxide. The effect of bath temperature and pH was also studied and was shown that by increasing both of these parameters, the film thickness was increased. The most interesting feature of this method was that both n-type and p-type cuprous oxide was synthesized. This has helped in the formation of a homojunction. This homo junction results in the increased efficiency of the solar cell. Before the research work performed by Wang, only a heterojunction formation was possible which had a lower efficiency (Abdul, 2008).

Nair et al, (1999) deposited cuprous oxide on glass substrate using the chemical technique. The glass slide was dipped first in a 1-Molar aqueous solution of NaOH kept at 50-90°C for 20 s and then in a 1-Molar aqueous solution of copper complex. After three immersion cycles, a

30 nm thick film having a bright silvery colour appeared (Abdul, 2008).

Most of the deposition techniques used for growing copper oxide results in a mixture of Cu₂O and CuO phases. Phase purity and stability Cu₂O has been an important issue affecting its application in solar cell and other devices. Since the cubic Cu₂O phase is more symmetric compared to the monoclinic CuO phase, Cu₂O tends to be more stable at nano dimensions (Darma, 2011). It is therefore essential to study in details, properties of the single-phase Cu₂O for solar cell application. Currently, cuprous oxide (Cu₂O) has attracted attention for solar cell applications due to its direct bandgap around 2 eV. Therefore, the need for detail investigation of the structural properties of the Cu₂O powder becomes paramount. A single-phase Cu₂O powder was prepared using a chemical method. The study focuses on the chemical synthesis of single-phase cuprous oxide (Cu₂O) and its structural characteristics.

MATERIALS AND METHOD

Experimental Procedure

The experimental procedures employed are as follows:

- A. Preparation
 - i. Preparation of reagent
 - ii. Sample preparation
 - iii. Heating of solution
 - iv. Structural Characterization

Preparation Procedures

All chemicals used in this experiment were of analytical grade and were used without further purification. The nanoparticles were obtained with the same preparation conditions given by Huang et al. (2008). The following reagents were used for the study namely: Glucose powder, CuSO₄, NaOH, Distill water and Ethanol.

Preparation of Reagent

The amount of concentration needed to form a certain concentration of any compound can be easily calculated by using dilution formula, i.e.

$$\text{Concentration in mol/dm}^3 = \frac{\text{no. of moles} \times 1000}{\text{Volume in cm}^3}$$

Where,

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$$\text{no. of moles} = \frac{\text{mass}}{\text{molar mass}}$$

Therefore,

$$\text{mass in grams} = \frac{\text{conc.} \times \text{molar mass} \times \text{vol.}}{1000}$$

Therefore, 0.1mol/L concentration of CuSO_4 aqueous solution and 0.3mol/L concentration of NaOH aqueous solution was obtained using this formula.

From the dilution formula, to have 0.1M CuSO_4 , 3.990g was dissolved into 250cm^3 of distilled water. In the preparation of 0.3M NaOH solution, 1.20g was dissolved into 100cm^3 of distilled water separately.

Sample Preparation

3.4g of glucose powder was directly added to 100mL of CuSO_4 aqueous solution with the concentration of 0.1mol/L as prepared above and was allowed to dissolve completely in the solution. A blue precipitate was observed. Then 100mL of NaOH aqueous solution of 0.3mol/L concentration dropped to the solution after the glucose had dissolved completely. A sky blue precipitate was obtained.

Heating of Solution

Heating of solution was done using a furnace with controlled temperature. The obtained suspended solution was immediately heated at 60°C in the furnace for 30 min, a red precipitate was then obtained. The red precipitate obtained was allowed to settle, it was then filtered, washed with distilled water and ethanol and further dried at 60°C for 4h in the furnace. A dry reddish powder thought to be Cu_2O sample was finally obtained.

Structural Characterization

X-ray powder diffraction (XRD) pattern of the as-prepared cuprous oxide powder was recorded on an Empyrean diffractometer DY 674 (2010) with a copper anode material manufactured by Panalytical, Holland with $\text{CuK}\alpha$ radiation of wavelength 1.5418\AA . The sample was analyzed at the National geosciences research laboratory Kaduna. X-ray powder diffraction is most widely used for the identification of unknown crystalline materials (e.g. minerals, inorganic compounds), determination of unknown

solids that are difficult to determine optically, determination of unit cell dimensions, as well as measurement of sample purity. With specialized techniques, XRD can be used to determine crystal structures. (DY 674 manual 2010)

These X-rays are collimated and directed onto the sample. As the sample and detector are rotated, the intensity of the reflected X-rays is recorded. When the geometry of the incident X-rays impinging the sample satisfies the Bragg Equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or a computer monitor.

The value of d (the interplanar spacing between the atoms) is obtained using the Bragg equation: $2d\sin\theta = n\lambda$. Where n is an integer, λ is the wavelength of a beam of x-rays incident on a crystal with lattice planes separated by distance d , and θ is the Bragg angle.

The goniometer forms the central part of the Empyrean diffraction system. It contains the basic axes in X-ray diffractometry, the omega and 2θ axes. The sample was analyzed using the reflection-transmission spinner stage using Theta-Theta settings. The two-Theta starting position was 5.5131 and ends at 75.000 with a two-theta step of 0.026 at 3.57 seconds per step. Tube current was 40mA and the power was 45VA. A Fixed Divergent Slit size of 1° was used and the goniometer radius was 240mm. The intensity of diffracted X-rays is continuously recorded as the sample and detector rotate through their respective angles. Peak intensity occurs when the mineral contains lattice planes with d -spacing appropriate to diffract X-rays at that value of θ . Although each peak consists of two separate reflections ($\text{K}\alpha_1$ and $\text{K}\alpha_2$), at small values of 2θ the peak locations overlap with $\text{K}\alpha_2$ appearing as a hump on the side of $\text{K}\alpha_1$. Greater separation occurs at higher values of θ . Typically these combined peaks are treated as one. The 2λ position of the diffraction peak is typically measured as the centre of the peak at 80% peak height. (DY 674 manual 2010)

The software used is the Data Collector which connect the XRD machine with the work station, Xpert High score Plus which is used to analyse and interpret the data to produce the

*Corresponding author: Kazeem, S. alhassan77077@gmail.com Department of Science Education, Abubakar Tafawa University, Bauchi. © 2021 Faculty of Technology Education, ATBU Bauchi. All rights reserved

resulting diffractogram and the ICDD PDF 2 (2010) database for easy searching and matching

of the diffractogram to identify the minerals and compounds.



Plate 1: Empyrean diffractometer DY 674

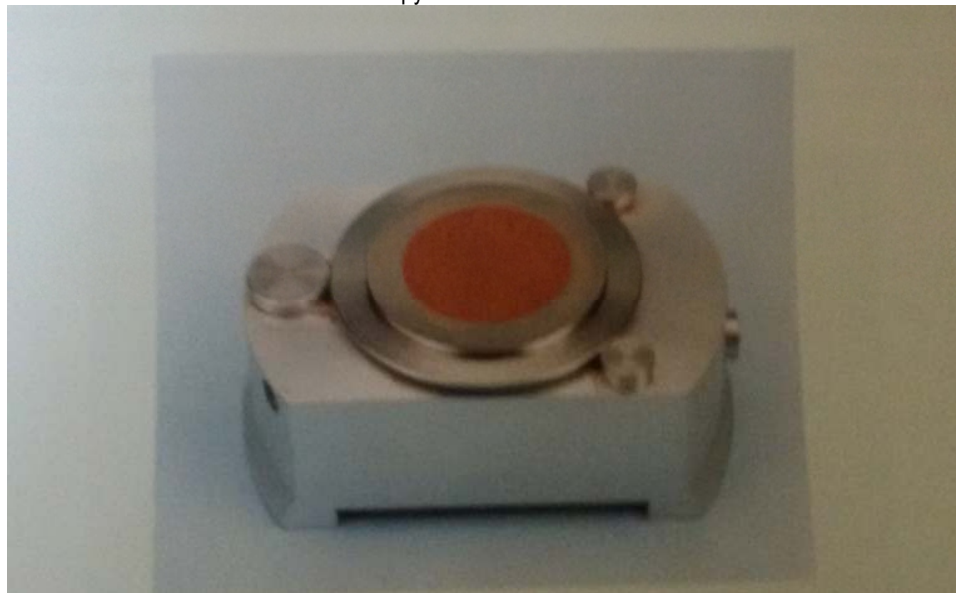


Plate 2: sample holder ring with sample on it removed from the preparation table

Determination of grain size of the Cu_2O

The grain size of the Cu_2O powder can be calculated according to the X-ray line-broadening method using Scherrer's equation:

$$L = \frac{0.99\lambda}{\beta \cos \theta}$$

Where L is the average grain size; λ is the X-ray wavelength; β is the full width at half

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maximum FWHM of the diffraction peak, and θ corresponds to the peak position or angular spacing.

RESULT AND DISCUSSION

This section present detail explanations of the result obtained from the X-ray powder diffraction (XRD) Empyrean diffractometer DY 674 (2010) was used to obtain the structural and phase purity of the as-synthesized Cu_2O powders. The average grain size was also determined using

Scherrer's equation.

XRD Sample Analysis

The structural and phase purity of as-synthesized Cu_2O powders studied by X-ray powder diffraction technique is shown in Figure1. Diffraction peaks of the samples could be assigned to the crystalline cuprous oxide in a cubic structure in fig. 2

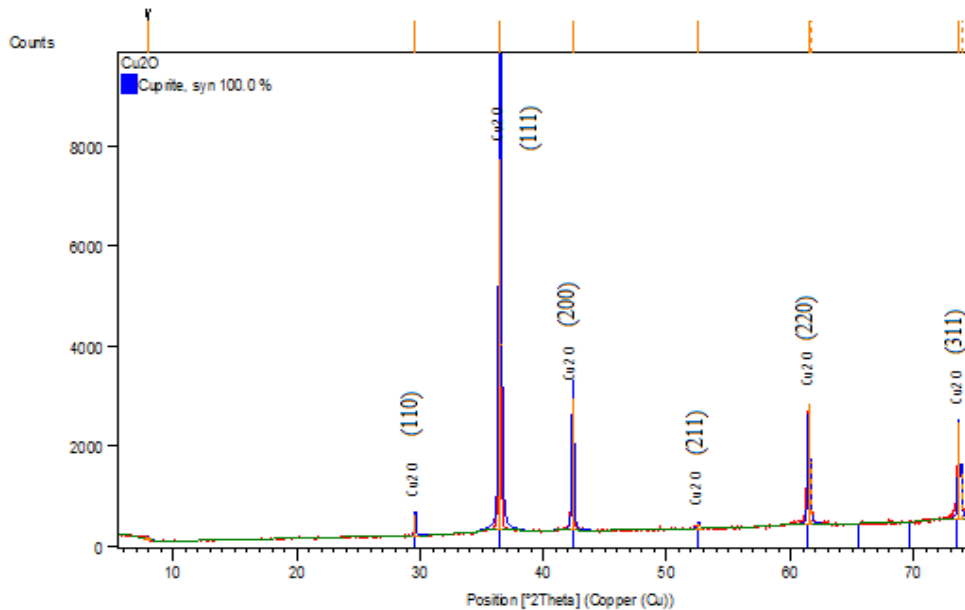


Fig. 1: XRD spectra of as-synthesized Cu_2O powders

No impurity phase such as CuO could be detected in this pattern, indicating that a pure phase of cuprous oxide can be readily synthesized. The diffraction spectra depicts small peak at $2\theta = 29.6^\circ$ ($d = 3.02\text{\AA}$) corresponding to the (110) plane of Cu_2O , a strong peak at $2\theta = 36.5^\circ$ ($d = 2.46\text{\AA}$) corresponding to the (111) plane of Cu_2O , a medium peak at $2\theta = 42.4^\circ$ ($d = 2.13\text{\AA}$) corresponding to the (200) plane of Cu_2O , a weak peak at $2\theta = 52.5^\circ$ ($d = 1.74\text{\AA}$) corresponding to the (211) plane of Cu_2O , another

medium peak at $2\theta = 61.5^\circ$ ($d = 1.50\text{\AA}$) corresponding to the (220) plane of Cu_2O , and another medium peak at $2\theta = 73.6^\circ$ ($d = 1.3\text{\AA}$) corresponding to the (311) plane of Cu_2O . All the peaks from the XRD pattern matched well with the literature standard values (standard ICDD PDF 2, 2010) which give a clear confirmation of Cu_2O cubic crystal structure in fig 4.2 with a space group Pn-3m lattice parameters of $a = 4.269000\text{\AA}$, $b = 4.269000\text{\AA}$ and $c = 4.269000\text{\AA}$ with $\alpha = 90^\circ$ $\beta = 90^\circ$ and $\gamma = 90^\circ$ respectively.

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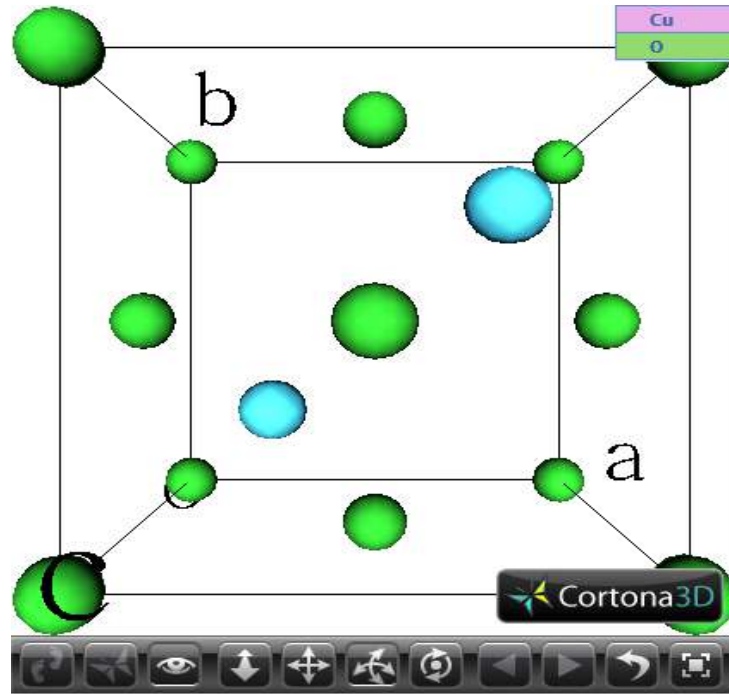


Fig. 2: XRD Crystal Structure of Cu₂O

The Average Grain Size

The average grain size was calculated using the Scherrer's formula. The average grain sizes evaluated for each peak level are tabulated in table 1.

$$L = \frac{0.99\lambda}{\beta \cos \theta}$$

Where x-ray wavelength (λ) is 1.5418Å°

The average grain size was found to be high but within the reported values in the literature. (Huang et al, 2008). The high grain size is to expect because of the chemical method employed compared to those obtained by sputtering and chemical vapour deposition (CVD) methods.

Table 1: Evaluated grain sizes

S/NO	FWHM(β) in radian	Position(2θ)	(2θ)/2	L (nm)
1	0.00179	29.6142	14.8071	80.18
2	0.00357	36.4699	18.2350	40.92
3	0.00268	42.3633	21.1817	55.53
4	0.00218	61.4735	30.7373	74.05
5	0.00272	73.6582	36.8291	63.73

CONCLUSION

Cuprous oxide (Cu₂O) nanoparticle powder was prepared via chemical technique. The characterization methods employed in the investigation of the structural properties of the cuprous oxide was the Empyrean diffractometer DY 674 (2010) for X-ray powder diffraction (XRD) analysis which gives the structural and phase

purity of the as-synthesized Cu₂O powders. The average grain size was also determined using Scherrer's equation.

The Cu₂O nanoparticles were found to be polycrystalline with a simple cubic structure. The average grain sizes of the reflection planes were determined and found to be high compared to reports in the literature. This is attributed to the

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heating and or drying process adopted.

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*Corresponding author: Kazeem, S. [✉ alhassan77077@gmail.com](mailto:alhassan77077@gmail.com) [✉](mailto:alhassan77077@gmail.com) Department of Science Education, Abubakar Tafawa University, Bauchi. © 2021 Faculty of Technology Education, ATBU Bauchi. All rights reserved



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