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LETTER

Preparation of nanostructured cuprous oxide (Cu_2O) absorber layer for photovoltaic application

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Abstract

In this investigation, a nanostructured Cu_2O thin film absorber layer is electrodeposited, exploring the impact of varying negative applied voltages and deposition time. Notably, the Cu_2O thin film demonstrated optimal absorbance at -0.95 V, contrasting sharply with a minimum at -0.97 V. The authors' findings underscore that the peak absorbance was achieved at -0.95 V, coinciding with the lowest transmittance observed after 80 min of deposition, aligning with a maximal absorption coefficient of $21 \times 10^3 \text{ cm}^{-1}$. At a deposition time of 5 min, the Cu_2O thin film exhibited a noteworthy maximum Urbach energy of 2.00 eV and a minimum steepness parameter of 0.013. In contrast, the lowest Urbach energy was recorded at 0.34 eV, with the highest steepness parameter occurring at an applied voltage of 0.93 V. Furthermore, this study revealed a gradual increase in the refractive index with higher applied voltages, reaching its pinnacle at -1.5 V. These results collectively emphasize the nuanced interplay between applied voltage, deposition time and the optical properties of the nanostructured Cu_2O thin film. The observed trends hold significant implications for optimizing the performance of thin film absorber layers, particularly in the context of enhancing absorbance and tailoring optical characteristics for specific applications.

1 | INTRODUCTION

Nanoparticles have garnered significant interest and found diverse applications across multiple domains, including health-care, electronics, energy and environmental studies [1–3]. In environmental science, for instance, scientists employ gold nanoparticles incorporated within porous manganese oxide as a catalyst to facilitate the degradation of volatile organic contaminants in the atmosphere at ambient conditions. Similarly, iron nanoparticles are utilized to remediate carbon tetrachloride emissions from groundwater sources. Furthermore, researchers have demonstrated that the focused application of sunlight on nanoparticles can yield remarkably efficient steam generation [4, 5].

Reference [6] investigated and analysed various synthesis methods for nanostructured Cu_2O , including chemical vapor deposition (CVD), sol-gel, electrochemical deposition and other bottom-up approaches. Reference [7] evaluated the influence of synthesis parameters such as temperature, precursor concen-

tration and reaction time on the morphology and crystalline structure of Cu_2O nanostructures. Reference [8] investigated the optical and electronic properties of nanostructured Cu_2O , including its bandgap, carrier mobility and absorption coefficient. Reference [9] explored the impact of these properties on the performance of Cu_2O -based photovoltaic devices.

Cuprous oxide (Cu_2O), a commonly studied p-type semiconductor, has been the subject of extensive research in recent decades [10–13]. This is due to its numerous advantages, such as affordability, non-toxicity, abundance and ease of synthesis. Additionally, researchers have shown significant interest in tailoring the structures of Cu_2O crystals to leverage their unique physicochemical properties for a wide range of applications [14–17]. Notably, remarkable advancements in energy conversion, catalyst development, sensor technology and chemical templates involving Cu_2O have driven the rapid progress in manipulating the size, shape, facets, defects, dopants and heterostructures of Cu_2O . The authors [18] reported that the introduction of Cu into ZnO nanostructures changed

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the morphology, resulting in uniformly distributed clusters of elongated nanorods. Reference [19] fabricated top-contact pentacene-based organic thin-film transistors (OTFTs) with bilayer WO_3/Au electrodes.

Solar steam devices are designed for deployment in under-developed regions, lacking access to electricity. They can be employed, for instance, to cleanse water or sterilize dental tools [20–23]. Reference [24] researched the fabrication of $\text{Cu}/\text{Cu}_2\text{O}/\text{CuO}/\text{ZnO}/\text{Al-ZnO}/\text{Ag}$ hetero junction solar cell structure on glass substrates utilizing sputtering technique is demonstrated. In [25], the researcher reviewed the effect of Cu_2O thin film thickness prepared by oxidation techniques on efficiency, which was analysed; afterward, past related work was systematically studied to investigate structural and morphological possessions of Cu_2O cover.

The challenge lies in the fact that finite fossil fuel reserves are depleting rapidly and if we persist in relying on this primary energy source as we do now, they will soon be completely depleted. In addition, burning fossil fuels produces carbon dioxide emissions that hurt the environment [26]. Carbon dioxide is recognized as both an air pollutant and a contributor to the greenhouse effect [27, 28]. In 2021, a study focused on Bangladesh, delving into the present situation and future potential of renewable and sustainable energy sources [29]. In [30], the various properties of fluorine-doped and un-doped CdO thin films prepared by varying the dopant concentrations are described. The authors examined the influence of adding more Sn-element to Sb–Se thin films to improve the films' compositional structure and the films' linear and non-linear optical and optoelectrical capabilities [31]. Reference [32] synthesized thin CuInSnS_4 films or allocated to studying the optical properties and parameters of these film samples.

This research aimed to explore their integration into numerous public and private initiatives designed to address the nation's growing electricity needs [33–36]. So far, the initial iteration of solar cells, which rely on crystalline semiconductors like silicon and gallium arsenide, introduced in the 1970s, remains the top-performing technology [37]. Copper oxide (Cu_2O), on the other hand, was one of the first semiconductors that was investigated for use in solar applications, predating the development of silicon (Si) and germanium (Ge) devices [38]. Moreover, when electrodeposited Cu_2O thin films are employed in solar cells, their effectiveness has frequently been variable as well as inconsistent [39]. Through the adjustment of the electrodeposition parameter, the main goal of this study is to improve the properties of the electrodeposited p-type Cu_2O film for its use in heterojunction solar cells [40].

Reference [19] studied the deposition of undoped ZnO , Al-doped ZnO and Cu-doped ZnO nanostructured films via room temperature radiofrequency (RF) sputtering. We focus on the preparation of nanostructured cuprous oxide for use as an absorber layer in photovoltaic applications. Reference [41] fabricated the top contact pentacene-based OTFT by inserting a thin layer of TiO_2 as a hole injection layer between the Au electrodes and organic layer. We address a broader range of factors in the preparation of nanostructured Cuprous Oxide for photovoltaic applications. Reference [42] investigated the super-

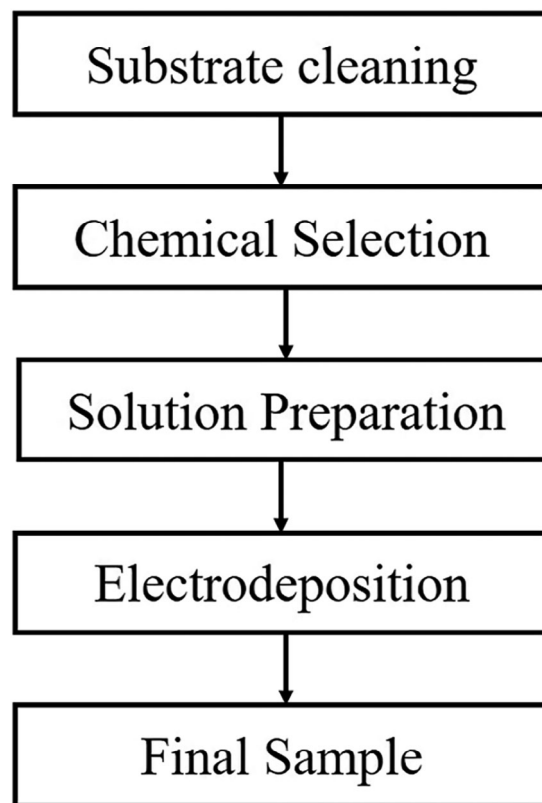


FIGURE 1 Block diagram for electrodeposition of Cu_2O thin film on fluorine-doped tin oxide (FTO) substrate.

capacitive performance of MgCo_2O_4 and coated the surface of the electrode with ZnS to enhance the electrochemical performance at higher cycles. We have a broader scope, considering various aspects for the preparation of a material for photovoltaic applications.

The novelty in the preparation of nanostructured cuprous oxide as an absorber layer for photovoltaic applications lies in the synergistic combination of tailored nanostructure design, advanced deposition techniques, enhanced charge carrier dynamics, novel materials synthesis approaches, bandgap engineering, robust stability and potential integration with tandem or hybrid architectures. This pioneering method paves the way for more efficient and sustainable solar energy conversion technologies. Accomplishing this objective will lead to a more comprehensive comprehension of the Cu_2O electrodeposition process and, consequently, result in enhanced efficiency for oxide solar cells based on Cu_2O electrodeposition.

2 | MATERIAL AND METHODS

2.1 | Electro deposition process

Figure 1 illustrates the schematic representation of the process involved in depositing a thin film of cuprous oxide (Cu_2O) onto a fluorine-doped tin oxide (FTO) substrate. The

electrodeposition of Cu_2O thin film on an FTO substrate comprises the following sequential stages.

2.2 | Cleaning process

One of the most important steps in the thin film deposition process is cleaning the glass substrate. Fluorine-doped tin oxide (FTO) is used as the substrate in this experiment. The adhesion of the thin film relies on the quality of the glass substrate. The glass onto which the solution was applied underwent a cleaning process using the Ultrasonic Cleaner after being scrubbed with a mixture of methanol and DI water. The cleaning process in the ultrasonic bath took place in four steps, they are: soaking in methanol, in acetone and then in DI water, where each step continued for 15 min. This meticulous cleaning procedure ensures the substrate is thoroughly cleaned and prepared for the subsequent thin film deposition.

2.3 | Solution preparation

A typical Cu-lactate system is used to conduct the electrodeposition of Cu_2O . This section employs three different volumetric flux values. The electrolyte composition contains 3 M lactic acid, 0.2 M CuSO_4 and KOH pellets to get the pH to 9.5. The temperature used for all deposition experiments is 65°C . A pH meter was used to monitor the pH value and a potassium hydroxide (KOH) solution was used to carefully adjust the pH of the solution to the desired level.

2.4 | Thin film deposition on FTO substrate

Fluorine-doped tin oxide (FTO) was used as the working electrode to deposit Cu_2O films, with FTO glass being a commercially available product. These depositions of cuprous oxide thin films were conducted with different applied potentials and deposition times and the counter electrode was a rod made of graphite. Following the deposition process, the samples were subjected to rinsing with DI water and subsequent air drying.

3 | CHARACTERIZATION TECHNIQUES

Ultraviolet-visible spectroscopy, abbreviated as UV-vis spectroscopy, is a technique employed to measure how a sample absorbs electromagnetic radiation at various wavelengths. By identifying the wavelengths at which a thin film exhibits the highest absorption, we can deduce important details about the film's valence and conduction bands, as well as estimate its band gap. UV-vis spectrophotometer (UV-2600, Shimadzu Co., Ltd. Japan) was used to do optical measurements. XRD (X'pert PRO X-ray diffractometer (PW3040), Philips Pan Analytical Co., Ltd., Netherlands) was used to analyse the physical structure and

phase of the powder samples, using $\text{Cu } K_\alpha$ source of X-ray wavelength of 0.15406 nm. Additionally, a filament source was used to drive high-energy electrons at the sample surface and the scattered electrons provided important surface topography information. Under various experimental circumstances, the grain size of the electrodeposited thin films was measured using a scanning electron microscope (SEM, Leo 1530, Zeiss Co., Ltd., Switzerland). These techniques will be used to evaluate the effects of experimental electrodeposition and heat treatment settings [43].

4 | RESULTS AND DISCUSSION

4.1 | Absorbance measurements

Figure 2 presents the UV-vis absorption spectra of films within the wavelength range of 500–1100 nm, with a constant deposition time of 40 min. The graph illustrates the absorption behaviour of copper oxide thin films under different voltage conditions (-0.93 , -0.95 , -0.97 , -1.0 V). In the wavelength plot, it becomes evident that the maximum absorption within the spectral range for copper oxide thin films occurs at various voltages (-0.93 , -0.95 V) with absorbance values of (12.00, 17.20, 10.90 and 11.60 a.u., -0.97 and -1.07 V). The figure demonstrates that absorbance decreases as wavelength increases across all cases [44]. This indicates that copper oxide thin films deposited at voltages of -0.95 and -0.93 V are suitable for use as absorption layers in the production of solar cells. Consequently, these copper oxide thin films with voltages of -0.95 and -0.93 V can be employed effectively as absorption layers. The absorbance of cuprous oxide thin films in the visible spectrum is critical for harvesting sunlight. Higher absorbance ensures that a larger portion of the incident solar radiation is absorbed, leading to increased energy conversion efficiency in the solar cell.

4.2 | Transmittance measurements

Figure 3 illustrates the variation in transmittance as a function of wavelength (λ , nm) within the wavelength range of 500–1400 nm for four distinct samples of cuprous oxide (Cu_2O) thin films. These samples, denoted as S1, S2, S3 and S4, were prepared under different applied voltages. Among these, the maximum transmittance values in the spectrum were recorded as 34.00, 41.00, 41.00 and 45.00 for cuprous oxide thin films deposited at applied voltages of -0.93 , -0.97 , -0.95 and -1.0 V, respectively. Notably, the minimum transmittance values remained consistent across all samples. It is evident from the data that transmittance generally increases with the wavelength for all samples.

Additionally, it was observed that cuprous oxide thin films deposited at an applied voltage of -0.93 V exhibited lower transmittance. This aligns with findings from previous research, which anticipated low transmittance in cuprous oxide thin films [45]. In the context of cuprous oxide thin films, balancing high

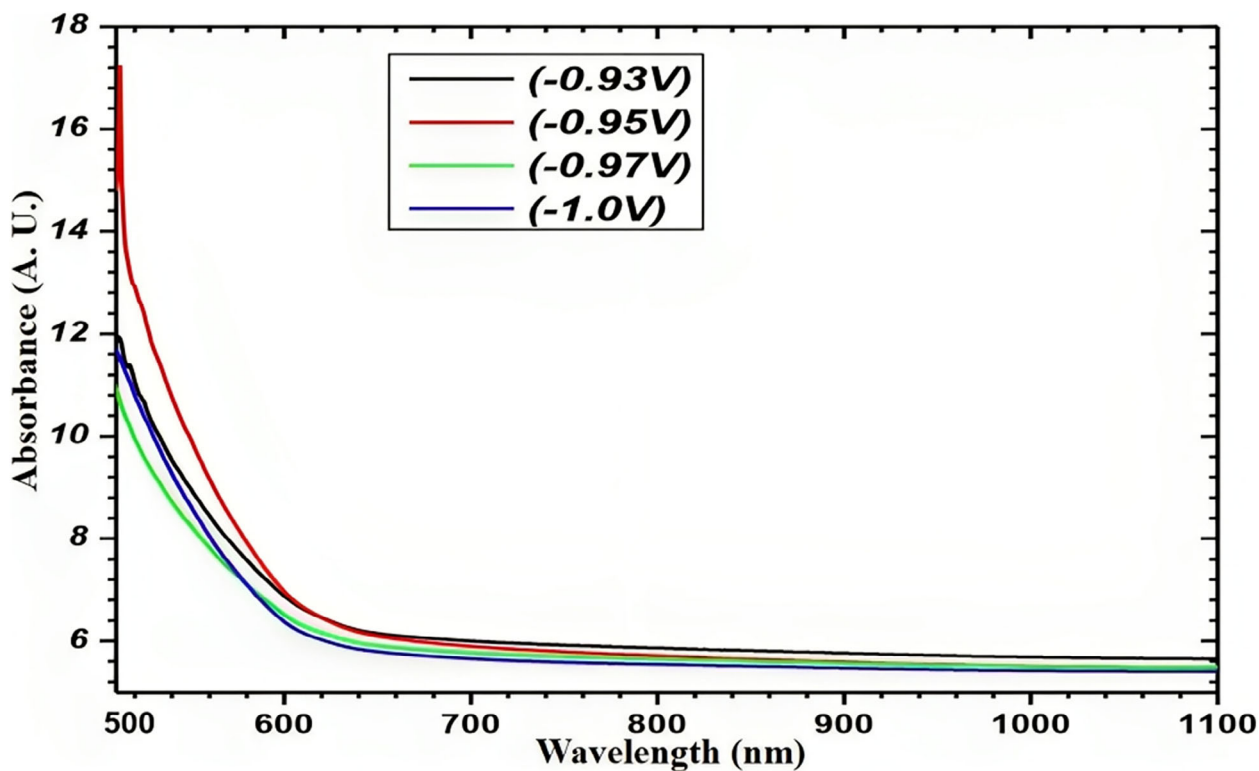


FIGURE 2 Absorbance versus wavelength plots for cuprous oxide thin films (for constant time i.e. 40 min).

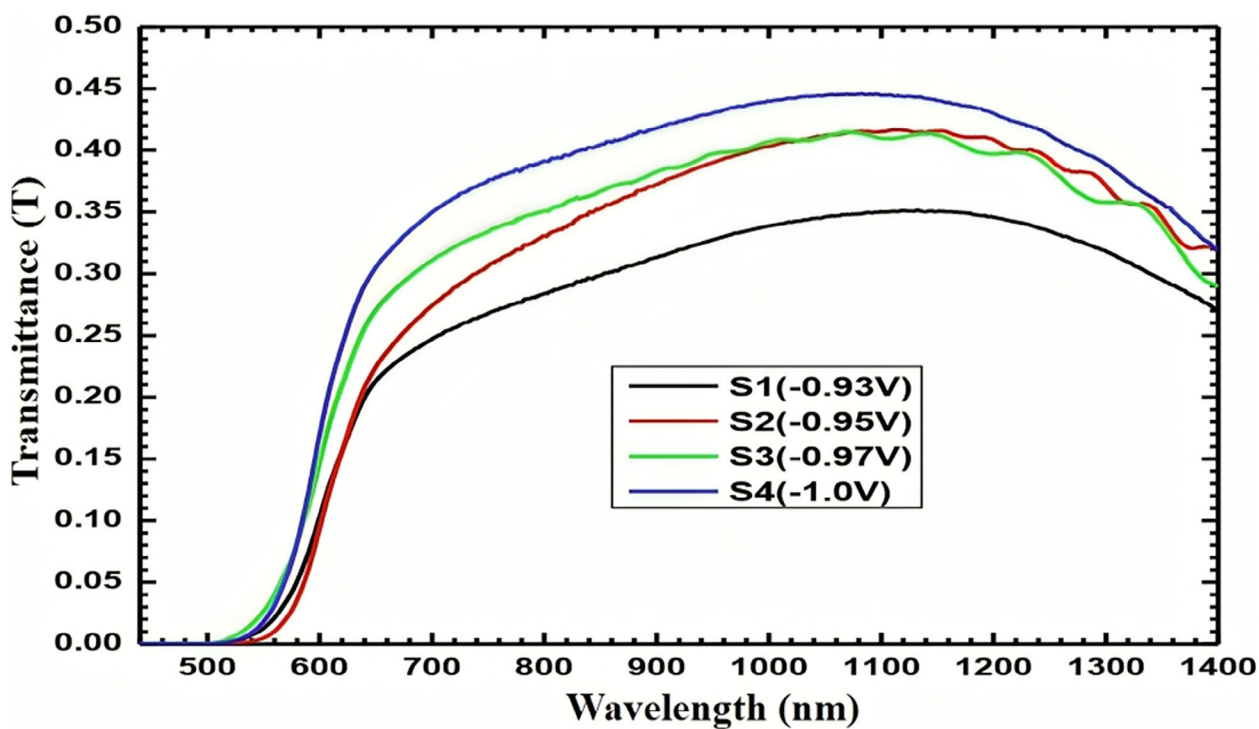


FIGURE 3 Transmittance versus wavelength graph for the different applied voltage.

absorbance with sufficient transmittance is essential. The transparent conductive layer on top of the active layer should allow most of the incident light to reach the semiconductor layer, ensuring efficient absorption and energy conversion.

4.3 | Absorption coefficient (α) measurements

The absorption coefficient α were calculated from the following equation:

$$\alpha = \frac{1}{t} \ln \left(\frac{1}{T} \right)$$

where the film thickness is determined using the weighing method, and ' T ' is the transmittance of the cuprous oxide thin films.

The values of the optical absorption coefficient, (α) in dependence of photon energy, ($h\nu$) for the cuprous oxide thin films at different deposition time were shown in the following figure. The above figures represent the dependency of absorption coefficient (α) on photon energy ($h\nu$) for different applied voltage of cuprous oxide thin films. It was realized that the absorption coefficient (α) increases with increasing photon energy ($h\nu$) for all samples of cuprous oxide thin films. It was also observed that the absorption coefficient initially decreases with increasing applied voltage of cuprous oxide thin films. The maximum absorption coefficient of cuprous oxide thin films were 18.0×10^3 , 21.0×10^3 , 14.0×10^3 and 9.4×10^3 cm^{-1} for different applied voltages (i.e. -0.93 , -0.95 , -0.97 and -1.0 V, respectively) and the minimum absorption coefficient of cuprous oxide nanoparticles were 2.37×10^3 , 1.73×10^3 , 1.44×10^3 and 1.23×10^3 cm^{-1} for different applied voltages (i.e. -0.93 , -0.95 , -0.97 and -1.0 V, respectively). The maximum absorption coefficients were observed at different values for various applied voltages (i.e. -0.93 , -0.95 , -0.97 and -1.0 V). The variation in maximum absorption coefficients with different applied voltages implies that the deposition conditions, specifically the voltage, play a crucial role in determining the optical properties of the cuprous oxide thin films. The maximum absorption coefficient indicates the film's ability to absorb light efficiently, which is essential for its performance in solar cell applications.

4.4 | Bandgap measurements

In both crystalline and amorphous materials, the optical transition involved can be determined based on the dependency of α on photon energy ($h\nu$) via the Tauc relation:

$$\alpha h\nu = C(h\nu - E_g)^n$$

where $h\nu$ is the incident photon energy, h is Planck's constant, ν the frequency of the incident solar radiation, C is an energy independent constant and is a measure of the steepness of band

TABLE 1 Energy band gap (E_g) variation of Cu_2O thin films with applied voltage.

Sample thin films	Applied voltage (V)	Band gap (eV)
Cu_2O	-0.93	2.06
Cu_2O	-0.95	2.00
Cu_2O	-0.97	2.12
Cu_2O	-1.0	2.10

tail density of states (Urbach region), E_g is the optical band gap energy and n is an index depending on the type of optical transition involved in the photon absorption process. The index n equals 0.5, 2, 1.5 and 3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions, respectively [46]. Figure 4 depicts that the absorption coefficient (α) (cm^{-1}) versus energy (E) plots of cuprous oxide thin films for different applied voltage (i.e. -0.93 , -0.95 , -0.97 and -1.0 V).

Thus, the indirect band-gap energy, E_i can be estimated by plotting $(\alpha h\nu)^{1/2}$ versus $h\nu$ curves and extrapolating the linear portion of the curve to $(\alpha h\nu)^{1/2} = 0$. The direct band gap energy, E_d was computed by plotting $(\alpha h\nu)^2$ with respect to the photon energy and extrapolating the linear portion of the curve to $(\alpha h\nu)^2 = 0$ along the photon energy axis.

Extrapolating the linear portion of the curve to $(\alpha h\nu)^2 = 0$ in the photon energy axis gives the values of band gap energy. In Figure 5, $(\alpha h\nu)^2$ as a function of $h\nu$ was plotted to obtain the E_g of the cuprous oxide thin films. The $(\alpha h\nu)^2$ versus $h\nu$ plots provide a detailed understanding of the bandgap energy variations in cuprous oxide thin films with different applied voltages. These variations are critical for tailoring the material's optical properties for specific applications, such as in photovoltaic devices, where efficient absorption of solar radiation is essential for energy conversion. The E_g was determined from the intercept of linear part of the curves extrapolated to a zero in the energy axis. The values of the E_g for cuprous oxide thin films were tabulated in Table 1. It was also observed from the Table 1, that five different types of cuprous oxide thin films have band gap around 2.06–2.18 eV for different applied voltage. This indicates that the band gap of cuprous oxide thin films depends on the variation of applied voltage. This variation suggests that the electronic structure and energy band arrangement within the cuprous oxide thin films are influenced by changes in applied voltage during the deposition process. When a photon influences the surface of a PV device, three possible results may occur. The photon may be absorbed by the material, reflected back into the atmosphere, or transmitted through the cell. If the photon is reflected or transmitted, a loss mechanism has occurred and the photon will not generate power. If the photon is successfully absorbed into the material, it may excite an electron from the valence band of the material to the conduction band. For photovoltaic operation, electron-hole pair generation is required by the excitation of an electron from the valence band to the conduction band [47]. The energy of the photon relative to that of the band gap will determine the fate of the

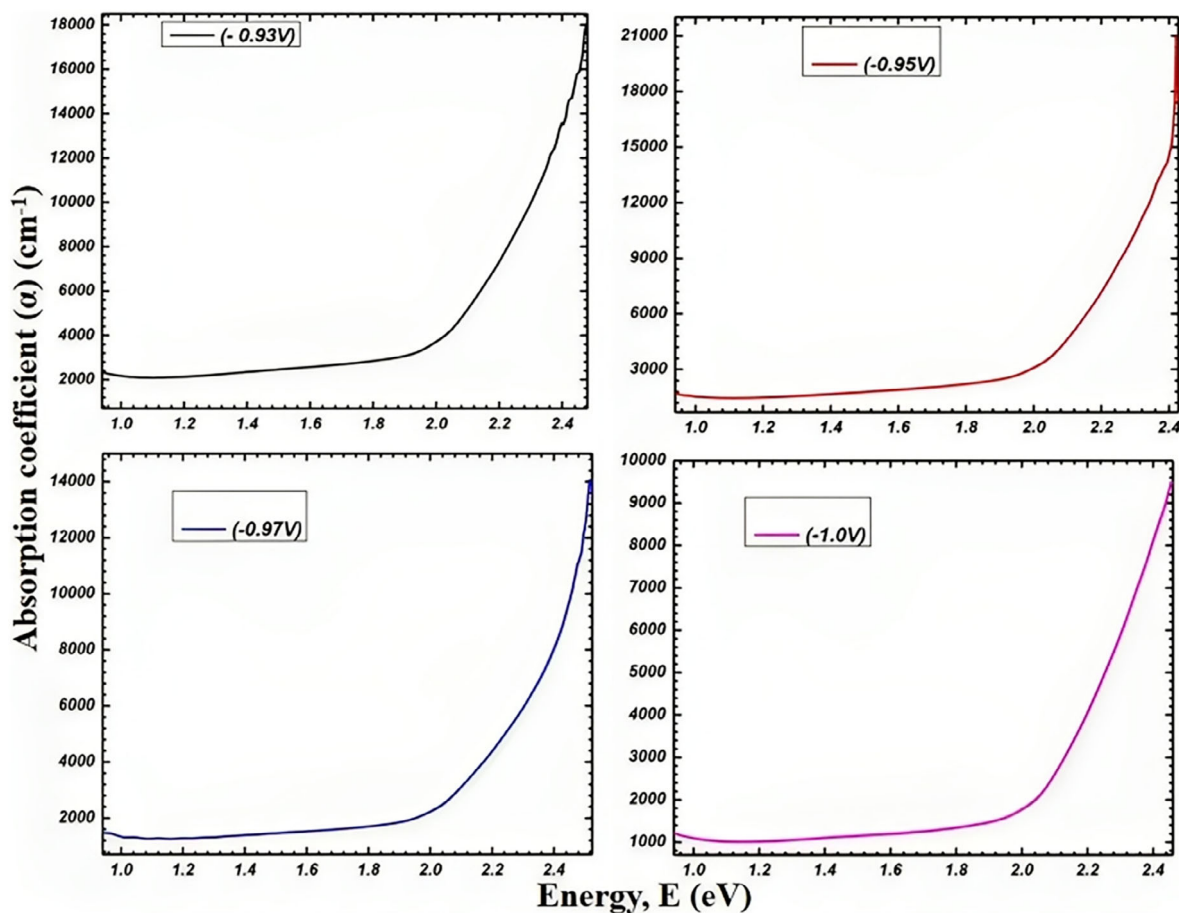


FIGURE 4 Absorption coefficient (α) (cm^{-1}) versus energy (E) plots of cuprous oxide thin films for different applied voltage (i.e. -0.93 , -0.95 , -0.97 and -1.0 V).

photon as it interacts with the material. If the energy of the photon is less than that of the band gap, the photon will not excite an electron into the conduction band. If the energy of the photon is precisely the same as that of the material's band gap, the photon will excite an electron into the conduction band. If the photon has energy larger than that of the band gap, the photon will excite an electron and the excess energy is wasted as thermal energy.

4.5 | Structural characterization of cuprous oxide thin films

4.5.1 | X-ray diffraction analysis

The XRD patterns of Cu_2O reveal prominent peaks at specific angles, indicating the crystallographic orientation of the Cu_2O thin films. At an applied voltage of -0.95 V, the most pronounced peak is observed at 42.6 degrees (200) [48, 49]. Similarly, at -1.0 V, a significant peak is evident at 42.7° (200), and at -1.5 V, the most prominent peak occurs at 36.6 degrees (111). These observations suggest that the crystal growth orientation of Cu_2O deposition is notably influenced by the applied voltage values. Different peaks correspond to different crystallographic

TABLE 2 Variation of crystallite size with applied voltages for different samples of Cu_2O thin films.

Applied voltage (V) in volts	Observed angle, (2θ) in degree	(hkl) plane	FWHM in degree	Crystallite size (nm)
-0.95 V	42.60	(200)	0.36	23.69
-1.0 V	42.72	(200)	0.30	28.54
-1.5 V	36.63	(111)	0.39	21.72

planes, providing insights into the preferential growth directions of the crystals. The crystallite size of the prepared Cu_2O thin films is determined using the Scherrer formula, which takes into account the XRD peaks' full width at half maximum (FWHM) [50].

The obtained crystallite size ranges from 21.72 nm to 23.69 nm under different applied voltage conditions (0.95 , -1.0 and -1.5 V) shown in Table 2. The Scherrer formula uses the wavelength of the primary $\text{CuK}\alpha$ radiation, angle of incidence (θ), and the experimentally determined FWHM to calculate the average crystallite size. Table 2 and Figure 6 depict how the crystallite size varies with applied voltage. At -0.95 V, the crystallite size is 23.69 nm, at -1.0 V, it increases to 28.54 nm, and at -1.5 V, and it decreases to 21.72 nm. The notable increase

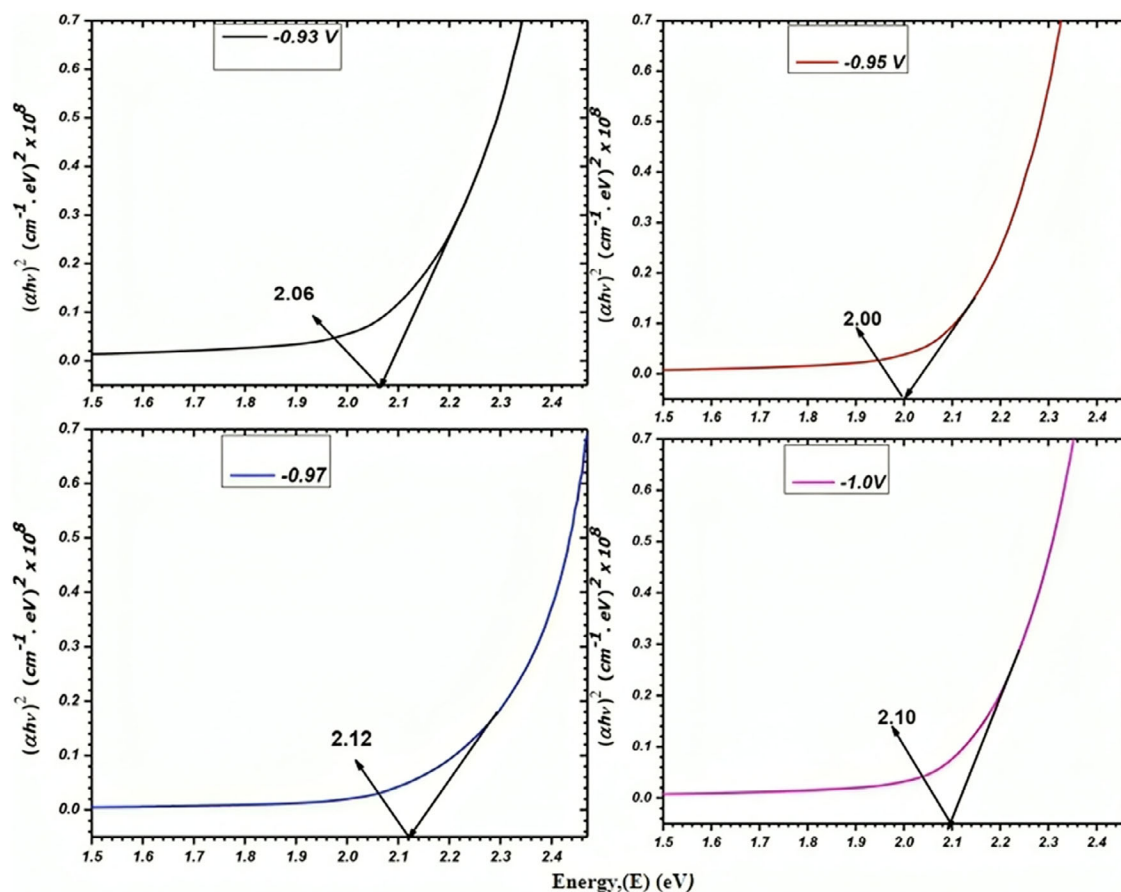


FIGURE 5 $(\alpha h\nu)^2$ versus $h\nu$ plots for Cu_2O (-0.93 V), Cu_2O (-0.95 V), Cu_2O (0.97 V) and Cu_2O (-1.0 V) thin films.

in crystallite size at -1.0 V suggests enhanced crystal growth under this voltage condition. The sudden decrease in crystallite size at -1.5 V implies a change in the growth mechanism or other factors influencing the crystal formation at this specific voltage. The analysis indicates that the crystallite size of cuprous oxide thin films is influenced by applied voltage during the electrochemical deposition process. An increase in applied voltage generally leads to larger crystallite sizes, but there is a sudden decrease at -1.5 V. These findings have implications for tailoring the properties of thin films by controlling the electrochemical deposition conditions, providing valuable insights for applications in material science and device fabrication [51, 52].

4.6 | Morphological characterization of Cu_2O thin films

4.6.1 | Scanning electron microscopy (SEM) of Cu_2O thin films

Figure 7 shows Scanning electron microscopy (SEM) pictures of cuprous oxide thin films deposited onto FTO glass substrates at various applied potentials (-0.95 , -1.0 and -1.5 V). SEM examinations were carried out to evaluate the surface morphology, grain size and uniformity of Cu_2O thin films. These images

TABLE 3 Variation of grain size with applied potentials.

Sample thin films	Value of pH	Applied potential, V (V)	Grain size (nm)
Cu_2O	9.5	-0.95	463
Cu_2O	9.5	-1.0	628
Cu_2O	9.5	-1.5	1230

allowed for an examination of the grain structure of the films at different applied potentials, revealing a trend of increasing grain size with higher applied potentials.

Table 3 indicates a clear trend of increasing grain size with higher applied potentials. The grain size values for Cu_2O thin films are 463, 628 and 1230 nm for applied potentials of -0.95 , -1.0 and -1.5 V, respectively. The increase in grain size suggests that higher applied potentials promote the growth of larger crystalline grains during the electrodeposition process. This could be attributed to enhanced nucleation and growth kinetics at more cathodic potentials, leading to the formation of larger crystalline structures. Figure 7a,b shows irregularly shaped grains with smoother contours and no sharp angles for samples deposited at -0.95 and -1.0 V. In contrast, Figure 7c exhibits clearly defined cubic particles. The emergence of well-defined cubic particles at -1.5 V suggests that the electrodeposition

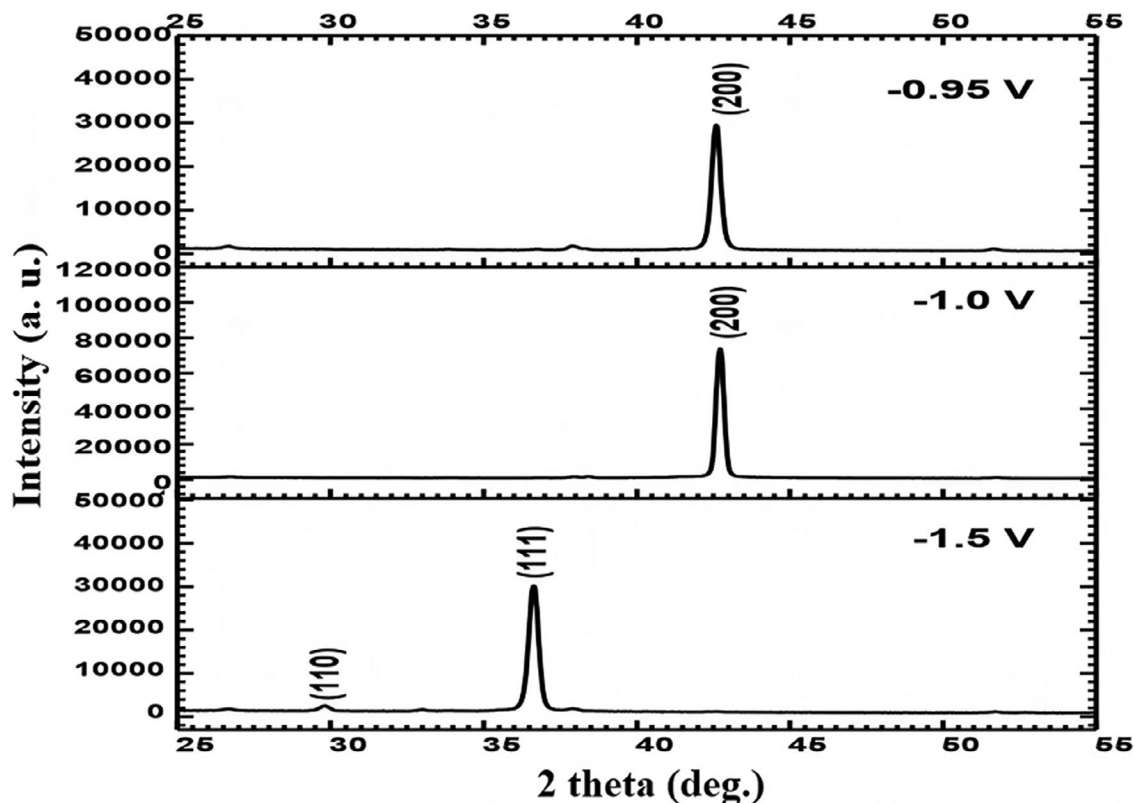


FIGURE 6 XRD spectra of the prepared Cu_2O films produced in different ECD processing conditions.

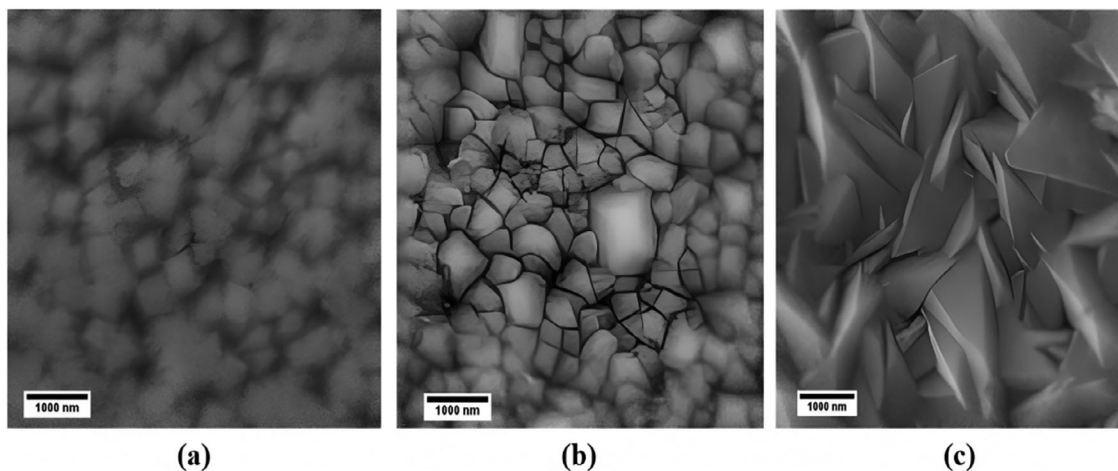


FIGURE 7 SEM images of Cu_2O thin films deposited by ECD processing at (a) -0.95 V, (b) -1.0 V and (c) -1.5 V.

process at more cathodic potentials promotes the formation of distinct crystal shapes, potentially related to a more controlled growth environment. The presence of clearly defined cubic particles at -1.5 V is noted and compared with similar observations in previous studies [53, 54]. The trend towards larger grain sizes and the development of distinct cubic particles with increasing applied potentials underscores the importance of precise control over the deposition parameters for tailoring the structural properties of these films in practical applications.

5 | CONCLUSIONS

In the pursuit of advancing solar cell technology, researchers have been exploring cuprous oxide (Cu_2O) thin films. Notably, Cu_2O films at -0.93 V exhibited low transmittance, consistent with prior research. XRD patterns unveiled changes in the crystalline structure of the films in response to varying voltages. The most intriguing observation was the intensity ratio of cuprous oxide, which showed a convergence at -0.95 and -1.0 V,

followed by a sudden divergence at -1.5 V, resembling a crescendo in a musical composition. SEM images provided insight into the morphology of the Cu_2O thin films, revealing varying grain sizes. In contrast, films at -0.95 and -1.0 V displayed irregularly shaped grains, resembling an artist's brushwork in an abstract landscape. This research uncovers the intricate relationship between applied voltage and the optical and structural properties of Cu_2O thin films. It suggests the exciting possibility of fine-tuning these films by manipulating voltage variations, offering promising prospects for advancements in solar cell technology, where light and energy harmoniously interact.

AUTHOR CONTRIBUTIONS

Rabiul Awal: Conceptualization; formal analysis; investigation; methodology; writing—original draft. **Nilufer Yesmin Tanisa:** Methodology; writing—review and editing. **Md. Arifur Rahman:** Writing—review and editing. **Shamim Ahmed:** Writing—review and editing.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflicts of interest.

DATA AVAILABILITY STATEMENT

Not Applicable.

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