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Cobalt Decoration on Cu_2O Photocatalyst via Electrodeposition for Enhanced Methylene Blue Degradation

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Abstract

Cuprous oxide (Cu_2O) is a promising material for photocatalysis due to its narrow bandgap, cost-effectiveness, and non-toxicity. However, its practical application is hindered by the rapid recombination of electron-hole pairs and low stability. This study addresses these limitations by decorating Cu_2O film with a cobalt (Co) cocatalyst. The Cu_2O/Co film was successfully synthesized on indium tin oxide (ITO) substrate through electrodeposition. Characterisation results confirmed the presence of small, agglomerated Co deposits that scattered on Cu_2O grain boundaries. Electrochemical impedance spectroscopy showed that Cu_2O/Co exhibited a charge transfer resistance of 21.94 Ω, which is lower compared to Cu_2O at 24.16 Ω. This indicates that the addition of Co enhances interfacial charge transfer kinetics. Photodegradation test of Cu_2O with Co then revealed a higher degradation efficiency of 76.51% than that of Cu_2O film at 67.55%, highlighting the potential of Co as a cocatalyst for Cu_2O.

Keywords: cobalt cocatalyst, Cu_2O, electrodeposition, methylene blue, photodegradation

1. Introduction

Methylene blue (MB) is a highly toxic cationic dye widely used in the textile dyeing industry, including batik production [1]. During the MB dyeing process, only about 5% of the dye is used, while the remainder is discarded. The high toxicity of this waste, despite the small amount used, makes it a significant environmental hazard. This dye can cause permanent damage to human eyes and aquatic animals, cancer, jaundice, cell mutation, vomiting, and cyanosis [2]. Various methods have been developed for MB wastewater treatment, including coagulation [3], nanofiltration [4,5], phytoremediation [6,7], adsorption [8], and electrocoating [9,10]. In recent decades, photodegradation has been the most widely developed method because it can efficiently degrade MB waste. The material photocatalysts used in this process can be synthesized through various techniques, and among these methods, electrodeposition is highly promising. This method offers several advantages such as low synthesis temperature, low production costs, and high product purity [11]. Additionally, electrodeposition allows control over the stoichiometry, thickness, and microstructure of the resulting film by adjusting the deposition parameters.

Cuprous oxide (Cu_2O) is a nanostructured oxide classified as a “p-type” semiconductor material due to the presence of copper electron vacancies in its crystal structure, resulting in a low bandgap value of approximately 1.9-2.17 eV [12]. This characteristic makes Cu_2O a highly photoactive material in the visible light

region, suitable for both photoelectrochemical and photovoltaic applications [13]. This material has great potential for use as a catalyst in the photodegradation of dyes such as MB, as it is a low-cost, readily available, and non-toxic catalyst [14]. However, a drawback of Cu_2O is that its electron-hole pairs are prone to recombination, resulting in low quantum efficiency [15]. Pure Cu_2O nanoparticles often exhibit limited photocatalytic efficiency. A significant factor contributing to this low activity is self-oxidation. The resulting species forms a passivating layer on the nanoparticle surface, which blocks incident light leading to a rapid decrease in performance [16]. Therefore, alternatives are needed to enhance the efficiency of utilizing the electron-hole pairs generated by light.

The degradation of MB dye, a persistent pollutant in the environment, remains a significant global challenges. While photocatalytic degradation using Cu_2O nanoparticles has emerged as a promising solution, its practical application is often hindered by low stability and rapid recombination of charge carriers. To address these limitations, the addition of metal cocatalysts such as Au, Pt, Zn, Co, and Ni has been studied to improve the stability of Cu_2O nanoparticles [17]. In this study, a cobalt (Co) cocatalyst was used because of its comparable ability to precious metal-based catalysts, cost-effectiveness, and ability to improve the stability of Cu_2O [18]. The dispersed Co on the Cu_2O particlescan act as an electron trap, creating a local electric field that supports charge separation and reduces the likelihood of recombination [11]. Additionally, the formation of a metal-semiconductor interface, a Schottky junction, between Co and Cu_2O accelerates electron transfer to the reaction sites on the surface, enhancing the efficiency of redox reactions [19]. The presence of Co can also enhance the structural stability of Cu_2O by inhibiting the transformation of Cu^+ into Cu^{2+}, which is detrimental. In this study, the decoration of Cu_2O with Co was performed using the electrodeposition method to assess its efficiency in degrading MB dye.

2. Materials and Method

2.1 Fabrication of Cu_2O and Cu_2O/Co thin film

The materials used for this research were copper sulfate pentahydrate ($CuSO_4.5H_2O$, Merck), lactic acid ($C_3H_6O_3$, Merck), sodium sulfate (Na_2SO_4, Merck), sodium hydroxide (NaOH, Merck), cobalt(II) sulfate heptahydrate ($CoSO_4.7H_2O$, Merck), methylene blue (MB, $C_{16}H_{18}ClN_3S$, Merck), and indium tin oxide (ITO) as the substrate. Supporting materials such as distilled water and ethanol were also used.

2.2 Fabrication of Cu_2O and Cu_2O/Co thin film

The electrodeposition method used in this study was controlled using CS310 workstation with a three-electrode system, where Ag/AgCl (KCl 3M) was used as the reference electrode, platinum as the counter electrode, and indium tin oxide (ITO) as the working electrode. Cu_2O was deposited on an ITO substrate that had been repeatedly rinsed with ethanol and distilled water. The deposition process was carried out at a constant potential of -0.3 V for 1 h at 60°C. A solution of 0.05 M $CuSO_4.5H_2O$ was used as the electrolyte. The pH of the solution was then adjusted to pH 10.00 using 10 M NaOH solution. Surface modification of Cu_2O with cobalt as a cocatalyst was performed using a constant potential of -1.25 V. Cobalt was deposited by immersing the Cu_2O film into a 0.1 M $CoSO_4 \cdot 7H_2O$ solution at room temperature for 15 seconds.

2.3 Characterization

The synthesized samples were characterized to determine their structural, morphological, elemental, and optical properties. The crystalline phases were identified by X-Ray diffraction (XRD). Morphological analysis was examined using scanning electron microscope (SEM), while elemental composition analysis was verfied using energy dispersive X-Ray fluorescence (EDXRF). The optical properties of the films were determined using Tauc-plot curves from reflectance spectra measured by sphere reflectance integrated-UV-Vis (UV-Vis ISR).

2.4 Electrochemical measurements

Photoelectrochemical measurements were conducted using electrochemical impedance spectroscopy (EIS), and linear sweep voltammetry (LSV) techniques. All measurements were performed using a standar three-electrode system and controlled using a Corrtest CS310 workstation under simulated solar irradiation (AM 1.5 G). EIS measurements were performed in the frequency range between 100 kHz and 0.1 Hz. The photocurrent was analyzed using LSV at a bias voltage between -1 V and 1.5 V against Ag/AgCl (3 M KCl) at room temperature, with scan rate of 50 mV/s.

2.5 Photodegradation study

The photocatalytic activity of the samples was evaluated by the degradation of MB solution under visible light irradiation. Data were collected at 5-min intervals for the first 60 min, then at 10-min intervals up to 120 min. The concentrations were monitored using UV-VIS spectrophotometer. The degradation efficiency was calculated using Equation 1.

$$\frac{C_0 - C}{C_0} \times 100\% \quad (1)$$

Where C_0 represents the initial concentration and C denotes the concentration measured at specified irradiation times.

3. Results and Discussion

3.1 Structure, phase, and morphology

The XRD patterns of the samples are shown in Fig 1. The phase of Cu_2O was confirmed present in both Cu_2O and Cu_2O/Co according to the COD reference data No. 96-100-0064. The main diffraction peaks were detected at 2θ of 42.51°, 62.87°, and 73.62°, which are associated with the (200), (220), and (311) planes, respectively. The other observed diffraction peaks correspond of the ITO substrate, similar to the XRD reported by Ayeshamariam and Sanjeeviraja [20]. After modification with Co, the diffraction peak on the (220) plane shifted to 62.94°, while the other peaks remained relatively stable. The selective shift observed only on this plane suggests that Co interacts preferentially with certain crystal facets, posibbly due to variations in surface energy and atomic arrangement. Despite the peak shift, no new diffraction peaks were observed, confirming that the fundamental cubic structure of Cu_2O remains unchanged. This shift of the (220) diffraction peak toward a higher angle indicates the presence of local lattice strain due to the deposition of Co cocatalyst on the Cu_2O surface, without altering the overall crystal structure of Cu_2O [20]. The undetectable peak of the Co metal phase diffraction is likely due to the low spectrum intensity of Co relative to Cu_2O and its very fine particle distribution, making it difficult to distinguish from the Cu_2O pattern [21]. Overall, the XRD results indicate that the addition of Co does not cause a change in the main phase of Cu_2O, but it can influence the surface structure and local microstrain, which has the potential to enhance the electrochemical and photocatalytic characteristics of the material, as reported in a similar system by [22].

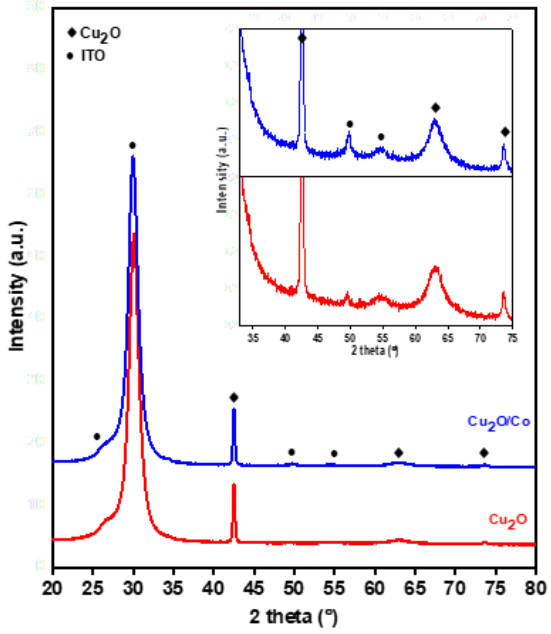

Figure 1. XRD diffractograms of Cu_2O and Cu_2O /Co.

SEM micrographs in Fig 2 show the morphology of Cu_2O and Cu_2O/Co synthesized via electrodeposition. The surface morphology of Cu_2O shows grains with clearly defined grain boundaries and relatively uniform sizes, with an average grain size of 2.28 µm. The addition of the Co cocatalyst to Cu_2O shows a different morphology, where small particles scattered on the grain surface, forming agglomerates. In transition metal cocatalysts such as Co, the particles are more stable when they adhere in groups rather than being evenly distributed across the entire surface, as shown by the SEM images [23,24]. The distribution of Co particles is not completely homogeneous but is localized in certain areas, particularly around grain boundaries.

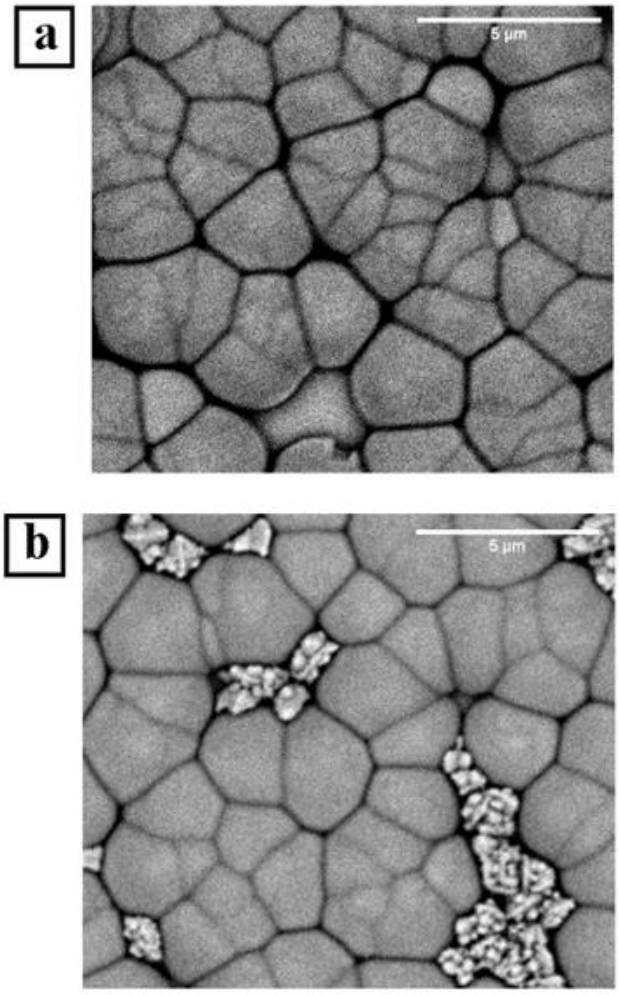

Figure 2. SEM micrographs of the (a) Cu_2O and (b) Cu_2O/Co.

The characterization of chemical element content was performed using XRF. Based on Table 1, it was observed that sample Cu_2O shows no impurities, while sample Cu_2O/Co detected the presence of Co at 2.586% with Cu still being the dominant element at 97.414%. The presence of Co proves that the Co deposition process was successful.

Although the detected Co content is relatively small, this value is sufficient to indicate that Co is successfully bound to the Cu_2O surface without forming a significantly separate cobalt oxide phase. These results are consistent with the study [14] which reported that the addition of small amounts of Co to Cu_2O can enhance photocatalytic activity without disrupting the Cu_2O phase structure. Thus, the XRF results confirm that the presence of Co is not a contaminant but a cocatalyst incorporated into Cu_2O to enhance photocatalytic efficiency.

Table 1. Chemical elements of Cu_2O and Cu_2O/Co.

Sample	Composition	
	Cu (%)	Co (%)
Cu_2O	99.987	0.0
Cu_2O /Co	97.414	2.586

3.2 Electrochemical impedance analysis

Electrochemical impedance spectroscopy (EIS) measurements and Nyquist plots obtained for Cu_2O and Cu_2O/Co are presented in Fig 3. The impedance spectra were fitted using an equivalent circuit model to obtain the bulk resistance (R_s) and charge transfer resistance (R_{ct}) values. The Nyquist plots show a characteristic semicircular shape, where the diameter of the semicircle represents the R_{ct} value at the interface between the photocatalyst and the electrolyte [20]. As shown in the Fig 3, Cu_2O/Co has a smaller semicircular diameter compared to pure Cu_2O. Quantitatively, the Rct value for Cu_2O/Co is 21.94 Ω, lower than that of Cu_2O at 24.16 Ω. This decrease in charge transfer resistance indicates that the addition of Co as a cocatalyst enhances the interfacial charge transfer kinetics [22], resulting in more effective charge separation on Cu_2O as the primary photocatalyst [23]. This improvement is primarily attributed to the role of Co as a cocatalyst in facilitating interfacial charge transfer, as evidenced by the lower R_{ct} value. However, the increase in the R_s value indicates that the overall bulk conductivity of Cu_2O/Co does not experience a significant increase, suggesting that Co plays a more dominant role at the interface level rather than in enhancing the intrinsic conductivity of the bulk material.

The estimated electrochemically active surface area (ECSA) was calculated using the Equation 2, where C_{dl} represents the electrochemical double-layer capacitance and C_s is the specific capacitance of the material per unit area with the value of 0.001758 F/cm² [20].

$$ECSA = \frac{C_{dl}}{C_s} \quad (2)$$

The ECSA value for Cu_2O/Co reaches approximately 1.51 cm², significantly larger than that of Cu_2O at 0.15 cm². These results indicate an increase in the number of active surface sites and improved electron transfer efficiency at the interface, consistent with the lower R_{ct} value, and confirm that Co enhances the surface kinetics of this photocatalytic system. Although the average particle size of Cu_2O/Co is slightly larger than that of Cu_2O, the ECSA of Cu_2O/Co increases significantly. This indicates that the incorporation of Co enhances the effective electrochemically active surface area through increased surface roughness or porosity, surface decoration by Co deposits, and modifications in surface properties that raise the specific capacitance [33].

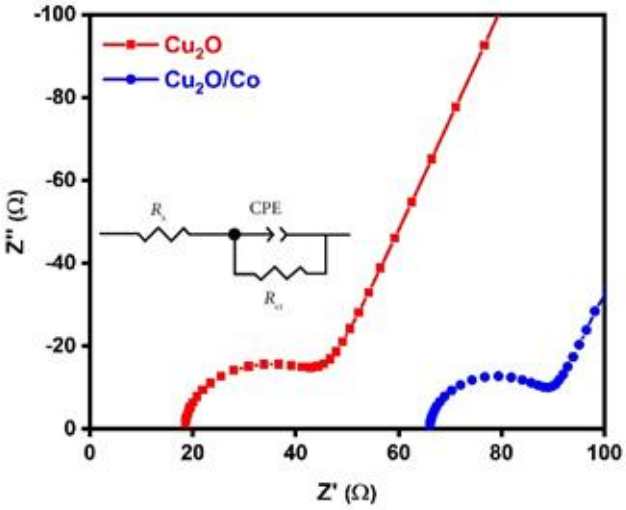

Figure 3. Nyquist plots measured under irradiation for Cu_2O and Cu_2O/Co.

3.3 Photoelctrochemical analysis

The photoelectrochemical (PEC) performance of the synthesized samples was evaluated under conditions with and without simulated sunlight irradiation. In Fig 4, Cu_2O exhibits a very small current response in the dark, indicating that the generated electric current is influenced by light irradiation. Under illuminated conditions, the absorbed photon energy triggers electron excitation from the valence band to the conduction band, thereby generating electron-hole pairs that contribute to the generated photocurrent [24]. Based on the test results, the photocurrent density of Cu_2O reached 4.55 mA/cm² at a potential of 0.15 V vs Ag/AgCl.

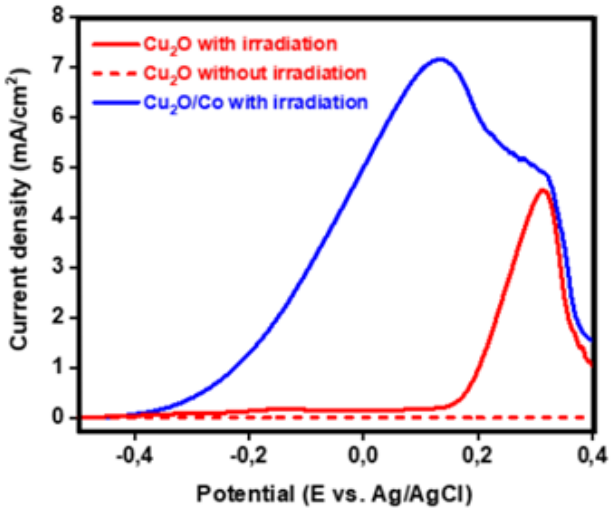

Figure 4. Photocurrent responses without and with irradiation for Cu_2O and Cu_2O/Co.

After the deposition of Co cocatalyst, this value increased to 7.15 mA/cm² at a potential of 0.13 V. Additionally, the onset potential of Cu_2O/Co shifted negatively to −0.4 V, compared to 0.15 V for Cu_2O, indicating that the photoelectrocatalytic reaction can occur at a lower potential bias. These results indicate that the addition of Co as a cocatalyst effectively facilitates more efficient charge separation and suppresses electron-hole pair recombination on Cu_2O [20]. The presence of Co also promotes the transfer of light-excited electrons, thereby showing potential to enhancing photocatalytic performance. This is supported by EIS measurement results showing a decrease in R_{ct} values after Co deposition.

3.4 Photocatalytic degradation of Methylene Blue

Figure 6 presents the degradation efficiency of the MB degradation, showing that Cu_2O was able to degrade MB by 67.55%, while Cu_2O/Co increased the degradation by 1.13 times compared to Cu_2O, reaching 76.51%. This increase can be attributed to the synergistic effect between Cu_2O and Co, which facilitates more efficient separation and transfer of charge carriers (electrons and holes) [25].

In the Cu_2O/Co thin film, the presence of Co provides additional active sites that promote the migration of photoexcited electrons, thereby reducing the recombination rate. Better charge separation extends the lifetime of electron-hole pairs and enhances redox reactions with MB pollutants [26]. Additionally, Co can act as a cocatalyst, enhancing the system's reduction capability by forming highly reactive hydroxyl radicals. These ·OH radicals will react with MB, producing compunds like carbon dioxide, water, and inorganic ions, as shown in the following reaction:

$$Cu_2O + h\nu \rightarrow e^- + h^+ \quad (3)$$

$$O_2 + e^- \rightarrow O_2^{\bullet -} \quad (4)$$

$$2\ O_2^{\bullet -} + 2\ H+ \rightarrow H_2O_2 + O_2 \quad (5)$$

$$Cu^+ + H_2O_2 \rightarrow Cu^{2+} + {\cdot}OH + OH^- \quad (6)$$

$$Co^{3+} + e^- \rightarrow Co^{2+} \quad (7)$$

$$Co^{2+} + H_2O_2 \rightarrow Co^{3+} + {\cdot}OH + OH^- \quad (8)$$

$$MB + {\cdot}OH \rightarrow CO_2 + H_2O + \text{inorganic ions} \quad (9)$$

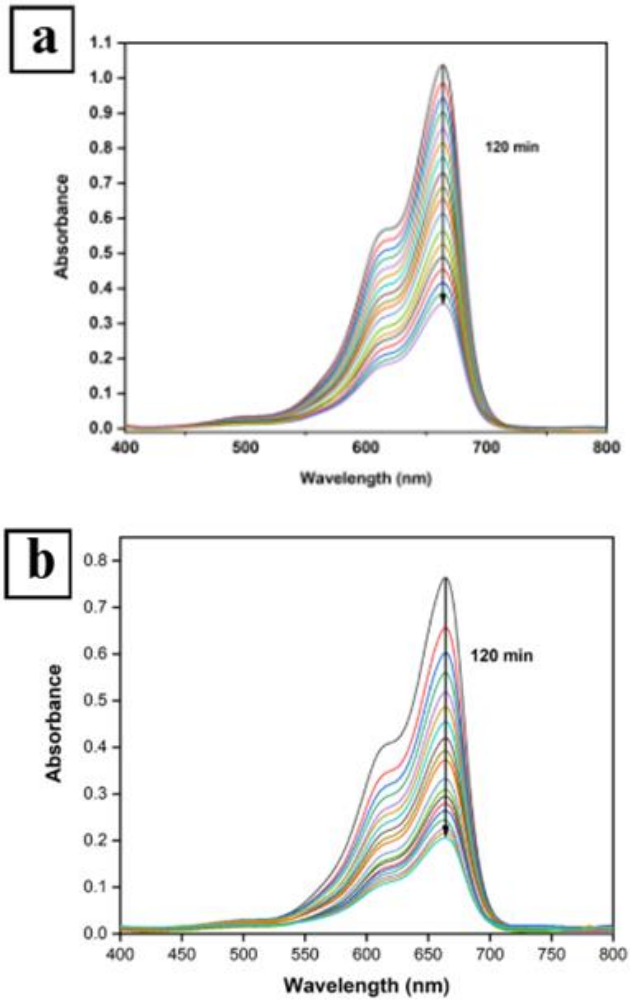

Figure 5. UV-VIS absorbance spectra of (a) Cu_2O and (b) Cu_2O/Co.

When a photocatalyst like Cu_2O is exposed to light, electron-hole pairs are formed. To initiate the degradation reaction, electrons must be transferred to the surface and interact with dissolved oxygen, while holes react with water or hydroxide ions to form hydroxyl radicals (·OH) [10]. However, this process can be disrupted by electron-hole recombination within the material before they can react at the surface. A low R_{ct} indicates high charge mobility, meaning more electrons or holes reach the surface and participate in the photocatalytic reaction. The

lower the R_{ct} value, the more efficient the charge transfer to the interface, thereby enhancing hydroxyl formation, which functions to degrade methylene blue.

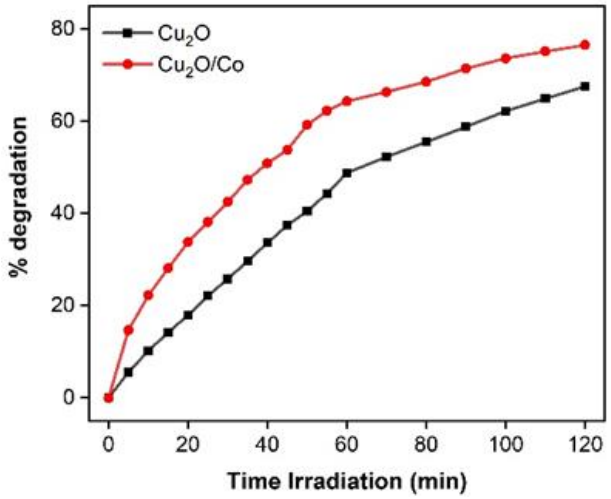

Figure 6. Photodegradation of MB using Cu_2O and Cu_2O/Co.

3.5 Optical analysis

The UV-Vis transmission results were used to estimate the optical bandgap (E_g) of the Cu_2O and Cu_2O/Co thin films. The E_g values were determined by plotting the $(F(R)hv)^2$ against photon energy and extrapolating the linear portion of the plot to its intercept with energy axis, as shown in Fig 7. Using this method, E_g was found to be around 2.00 eV for both samples. The invariance of the band gap may result from the likelihood that Co deposits are present only on the surface as a cocatalyst without substantially substituting Cu ions within the Cu_2O crystal lattice [31]. Consequently, the valance band and structures of Cu_2O are preserved. Therefore, although the addition of Co enhances photocatalytic activity by improving charge separation and electron transfer, its effect is not reflected in a modification of the band gap of Cu_2O. Although a change in band gap can influence photocatalytic activity by affecting light absorption, it is not the sole determining factor. Photocatalytic performance is more strongly governed by charge separation efficiency, generation of reaction of reactive oxygen species (ROS), and electron transfer kinetics. Therefore, even if the band gap of Cu_2O remains nearly unchanged after the deposition of Co, the photocatalytic activity can still be significantly enhanced due to improved charge separation and interfacial reaction.

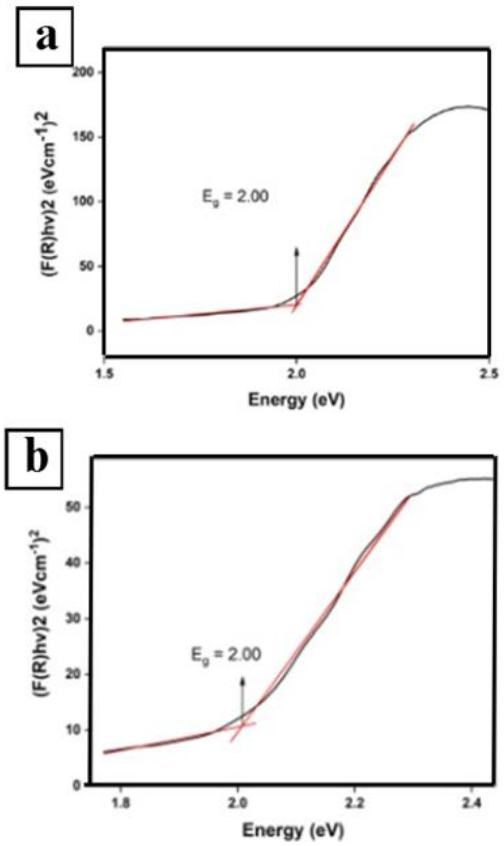

Figure 7. Bandgap values of (a) Cu_2O and (b) Cu_2O/Co.

4. Conclusion

Cu_2O photocatalysts with the addition of Co cocatalyst were successfully synthesized via electrodeposition on ITO substrates for methylene blue (MB) degradation. Degradation experiments showed that the presence of Co deposits in Cu_2O increased the degradation efficiency from 67.55% to 76.51% under visible light irradiation. Impedance data showed that the Cu_2O/Co film had a lower charge transfer resistance (R_{ct}) compared to pure Cu_2O, namely 21.94 Ω and 24.16 Ω, respectively. This reduction indicates an increase in charge separation efficiency and mobility on the material surface. Thus, Cu_2O/Co has high potential as an environmentally friendly photocatalyst for organic wastewater treatment applications.

Author contributions

Bella Pricilya: Investigation, Formal analysis, Writing-original draft, Visualization. **Nindys Aprillia**: Investigation, Writing-Review and Editing. **Shyla Noureen Zahra**: Visualization, Formal Analysis. **Firgie Wulandari**: Conceptualization, Methodology, Software, Formal Analysis. **Faisal Abnisa**: Supervision, Project Administration.

Conflicts of interest

The authors declare no conflicts of interest.

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