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ORIGINAL PAPER

CuO/TiO_2 nanocomposite photo-anode for dye-sensitized solar cells

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Abstract

Titanium dioxide (TiO_2) nanoparticles were synthesized via a hydrothermal method using titanium bis(ammonium lactato) dihydroxide as a precursor and confirmed by X-ray diffraction (XRD) analysis to exhibit the crystalline anatase phase. Ultraviolet–visible (UV–Vis) spectroscopy revealed that TiO_2 primarily absorbs in the ultraviolet region. High-resolution transmission electron microscopy (HRTEM) characterized the particle morphology and size distribution. A CuO/TiO_2 nanocomposite was then prepared, with XRD confirming the coexistence of monoclinic CuO and anatase TiO_2 phases. UV–Vis spectra demonstrated enhanced light absorption in the visible region due to CuO incorporation. Photo-anodes based on these materials were tested in dye-sensitized solar cells (DSSCs) fabricated via the doctor-blade technique. *I–V* measurements showed that DSSCs with CuO/TiO_2 photo-anodes achieved a 2.1% efficiency, surpassing pure TiO_2-based DSSCs (1.7%). This improvement is attributed to better light absorption and electron transfer. These findings highlight the potential of CuO/TiO_2 nanocomposites to enhance DSSC performance, contributing to the advancement of cost-effective solar energy technologies.

Introduction

As the world moves toward a more sustainable future, adopting renewable energy systems has become crucial to reducing environmental impacts. Renewable energy sources, such as biomass, geothermal, hydropower, wind power, and solar offer clean alternatives to fossil fuels, lowering carbon emissions and reducing reliance on finite resources. These systems enhance energy efficiency and promote long-term sustainability [1]. Solar energy technologies, such as photovoltaic (PV) cells, harness sunlight to generate electricity through the photovoltaic effect, where sunlight excites electrons in semiconductor materials, producing an electric current [2].

The photovoltaic solar cell has three generations. The first generation, which is manufactured on silicon wafers [3], is known as single crystal and polycrystal silicon solar cells; the main advantage of this type of solar cell is a high power conversion efficiency of around 24%. The most significant disadvantage is the high manufacturing cost and complexity. Thin-film solar cells are the second generation of solar cells [4] that were developed to reduce the high cost of fabrication and the need for source materials. The disadvantage is that it is toxic, and the efficiency is still low owing to the complexity of fabrication. The third-generation solar cell is a new promising technology made from nanomaterials. This type of solar cell, which includes polymer-based solar cells, nanocrystal-based solar cells, concentrated solar cells, and dye-sensitized solar cells, aims to broaden the active material's light absorption spectrum while lowering fabrication costs [5].

Dye-sensitized solar cells (DSSCs) are gaining attention for their high efficiency and low cost [6]. This research focuses on DSSCs owing to their low-temperature fabrication, the use of nanostructures (e.g., metal oxides) with large surface areas, and their wide light absorption spectrum, making them ideal for photoelectrode materials [7].

The DSSCs consist of four parts: the semiconductor material, dye, electrolyte, and counterelectrode [8]. TiO_2 is

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generally used in DSSCs. DSSCs work by using a photosensitive dye to absorb sunlight and generate excited electrons, which are then transferred to a semiconductor electrode, creating an electric current. The cell relies on an electrolyte to complete the circuit and maintain electron flow [9]. The disadvantages of DSSCs are the use of synthetic dye and the use of platinum as a counterelectrode [10]. To address these issues, natural dyes are being used to replace synthetic dyes, and platinum counterelectrodes are being replaced with carbon/graphite to reduce production costs, despite the fact that the resulting efficiency remains low. The short lifetime for the electron is the main drawback of semiconductors due to the wider bandgap [11], so electron–hole pairs that are generated can easily undergo a recombination process. To reduce such charge recombination, efforts have been made to make it easier for electrons to reach the counterelectrode [12] by reducing the bandgap of the TiO_2 semiconductor. Some efforts, such as growing metal or adding dopant to TiO_2, are required. Metal-oxide semiconductors (MOS) are promising materials for solar cell applications [13], acting as transparent conducting front electrodes and as hole or electron transport layers, as well as extending the TiO_2 range from UV to visible light and increasing the TiO_2 surface area. MOS have gained popularity in recent years owing to their low cost and low-cost production methods [14].

Copper oxide (CuO) is a *p*-type semiconductor with a 1.2-eV indirect bandgap and a monoclinic crystal structure. It possesses excellent electrical, optical, and thermal properties, making it suitable for a wide range of applications, including thermal conductors, lithium-ion batteries, catalysis, gas sensors, antimicrobial agents, field-emission emitters, and photovoltaics [15].

Archana Ashok et al. [16] synthesized a CuO/TiO_2 nanocomposite via the sol–gel method to modify the optical reactivity of TiO_2 from the UV to visible spectrum, highlighting CuO/TiO_2 as a promising candidate for photoanodes in DSSC. Rokhmat et al. [17] reported that growing copper particles using a fix current electroplating method improves the performance of a TiO_2/CuO solar cell. Ali A. Ahmed et al. [18] fabricated a CuO/TiO_2 nanocomposite via an electrochemical technique and reported that the DSSC performance was improved. Although several studies have focused on the synthesis of TiO_2/CuO (Cu_2O) solar cells with various techniques, the efficiencies remain low. In this study, a novel method for synthesizing CuO/TiO_2 nanocomposites using a simple approach is presented, demonstrating significant improvement in dye-sensitized solar cell performance.

Experimental

Materials

Titanium bis(ammonium lactato) dihydroxide (TiBALDH) solution, 50 wt% in H_2O ($C_6H_{18}N_2O_8Ti$; MW: 294.08) was purchased from Sigma Aldrich, deionized (DI) water from Thermo Scientific, a nanopure D3750 hollow fiber filter device from Barnstead, and ammonia solution (~ 30% ammonium hydroxide solution; NH_3, M: 17.09 g/mol) from R&M Chemicals. Copper chloride dihydrate ($CuCl_2 \cdot 2H_2O$; MW: 170.48) was obtained from Sigma Aldrich. Sodium hydroxide (NaOH, MW: 40 g/mol) was obtained from HmbG Chemicals and absolute ethanol from Sigma Aldrich.

Methods

To synthesis TiO_2 via a hydrothermal method, 0.1 mL TiBALDH solution was added to 40 mL deionized (DI) water under stirring condition. Ammonia was added to modify the pH to 10.5, and the resulting solution was place into Teflon-lined hydrothermal reactors (homemade) and heated at 170 °C for 24 h. The resulting products were washed several times with DI water to remove any unreacted material before being dried in an oven at 60 °C for 24 h [19].

CuO nanoparticles were prepared by a precipitation method; 1.7 g $CuCl_2 \cdot 2H_2O$ was dissolved in 100 mL deionized (DI) water under vigorous stirring. NaOH solution (0.1 M) was added to modify the pH to 14, and the black precipitate was washed by using absolute ethanol and (DI) water for many times until the pH reached 7 before being dried in an oven at 60 °C for 24 h [20].

To prepare CuO/TiO_2 nanocomposites, 20 ml $CuCl_2 \cdot 2H_2O$ (0.0125 M) was placed in a magnetic stirrer, then 0.5 mmol TiBALDH with 20 ml absolute ethanol was added and stirred vigorously for 1 h at room temperature. Next, 0.25 M NaOH has added dropwise into the reaction solution until the pH of the mixture reached 10. After that, it was placed in a Teflon-lined hydrothermal reactor and heated to 170 °C for 24 h. The black CuO/TiO_2 composite precipitate was extracted by centrifugation and rinsed several times with distilled water and absolute ethanol before drying at 60 °C for 24 h [21].

Preparation of DSSCs

First, 0.3 g of TiO_2 and CuO/TiO_2 powder were added to the mortar, followed by one drop of acetic acid and one drop of ammonia, achieving viscosity high enough to make a thick paste film without requiring high temperature to achieve practical interconnection. Then, this was ground for 15 min to avoid aggregation as reported.

Fluorine-doped tin oxide (FTO) glass substrate, which was used as a current collector, was first cleaned in an ultrasonic bath for 15 min by ethanol and acetone for 10 min each. The three edges of the FTO glass were covered with adhesive tape as a frame to provide noncoated areas for electrical contact. The TiO_2 and CuO/TiO_2 mixture pastes were coated onto the FTO glass using the doctor-blade technique. After application, the films were air-dried for approximately 10 min to reduce surface irregularities. The films were then heated to 150 °C in a hot plate for 20 min to facilitate interconnection of the paste and FTO substrate.

After the films had cooled to 60 °C, the electrodes were immersed in beetroot dye solution for 3 h. After dye adsorption, the film was cleaned with pure ethanol to remove the excess dye and dried in air, yielding a dye-covered electrode. The carbon counterelectrode was prepared by using a candle. A drop of iodine electrolyte solution was injected into the cell [22].

Results and discussion

The structure of the samples was characterized as follows: X-ray diffraction analysis (MAXima-X, XRD-7000 Shimadzu, Japan) was carried out with Cu K_α radiation at a wavelength of 1.5418 Å), morphologies were obtained by high-resolution transmission electron microscopy (HRTEM; JEOL JEM 2100F, Japan), and absorbance spectra were taken by ultraviolet–visible (UV–Vis) spectroscopy (Shimadzu UV-3600, Japan).

XRD analysis

Figure 1 shows the XRD patterns of TiO_2, CuO, and CuO/TiO_2 nanocomposite, exhibiting a strong diffraction pattern at scattering angles in the range $20° \leq 2\theta \leq 80°$. Diffraction scattering angles (2θ) of 25.19°, 37.79°, 48.07°, 54.03°, 55.15°, 62.91°, 68.88°, 70.43°, and 75.13° were observed. These results indicate the formation of TiO_2 in tetragonal anatase phase. Several groups have reported the XRD patterns of TiO_2 synthesized at pH 10 and 11, respectively, where the only phase observed was crystalline anatase; this result is in good agreement with Kinsinger et al. [19]. These results indicate that the hydrothermal method is suitable for preparing the TiO_2 anatase phase directly.

The lattice parameters of the sample were calculated by using the Maud program, yielding lattice constants *a* and *c* of 3.81 Å and 9.02 Å, respectively. These results indicate the formation of the anatase phase of TiO_2, as reported in several studies such as that by K. Madhusudan et al. [23].

The XRD patterns of the CuO nanoparticles are shown in Fig. 1b. XRD peaks are located at $2\theta = 32.54°$, 35.53°, 38.74°, 46.26°, 48.76°, 53.51°, 58.17°, 61.52°, 66.29°, 68.09°, 72.47°, and 75.20°. The diffraction patterns

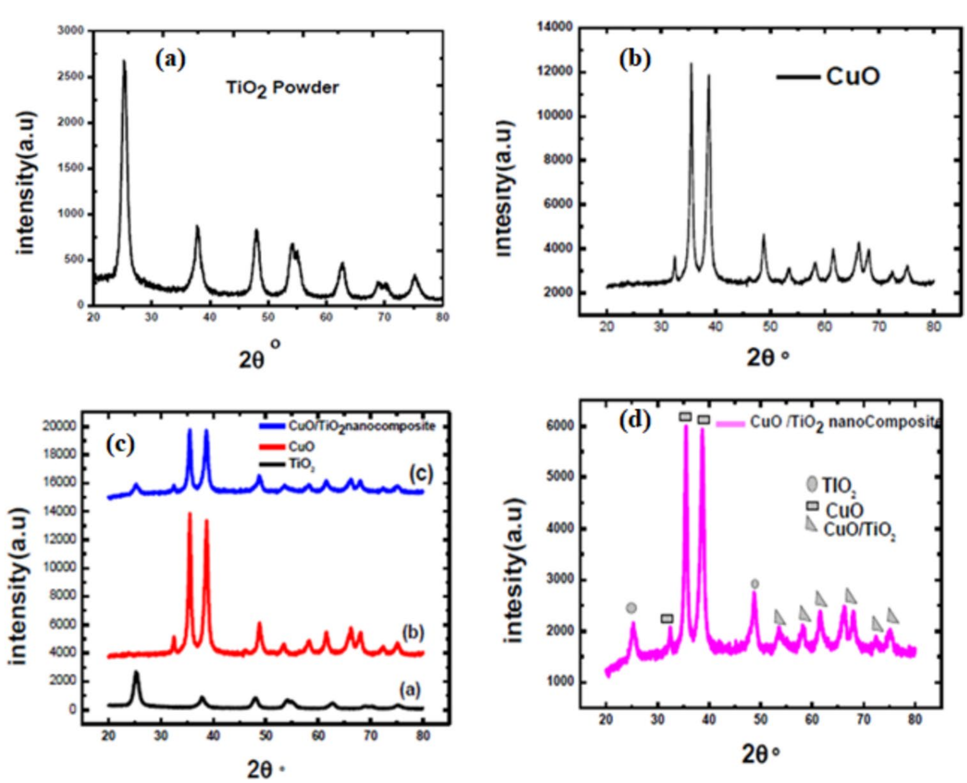


Fig. 1 XRD patterns of **a** TiO_2 prepared by hydrothermal method, **b** CuO, and **c**, **d** CuO/TiO_2 nanocomposite

correspond to (110), (002), (200), and $(\bar{2}02)$ plane orientations and confirm the formation of CuO in monoclinic phase (space group *C*2/*c*) with lattice constants $a = 4.96$ Å, $b = 3.37$ Å, and $c = 4.99$ Å, in good agreement with literature values [21].

Figure 1b indicates the high purity of the CuO synthesized at room temperature (at 29 °C) according to the sharp and intense diffraction pattern, the narrow full-width at half-maximum (FWHM), and the absence of diffraction patterns related to impurities.

Figure 1d exhibits the XRD patterns of TiO_2, CuO, and CuO/TiO_2 nanocomposite, as shown in Fig. 1d. All the diffraction patterns indicate the presence of anatase phase of TiO_2 and monoclinic phases of CuO phase, and imply that a CuO/TiO_2 composite was formed, as also reported by Chao Chen [24]. In Fig. 1c, the patterns corresponding to TiO_2 phase are marked by a circle, while those of monoclinic CuO are marked by a square, and the common pattern content of CuO/TiO_2 composite is marked by a triangle.

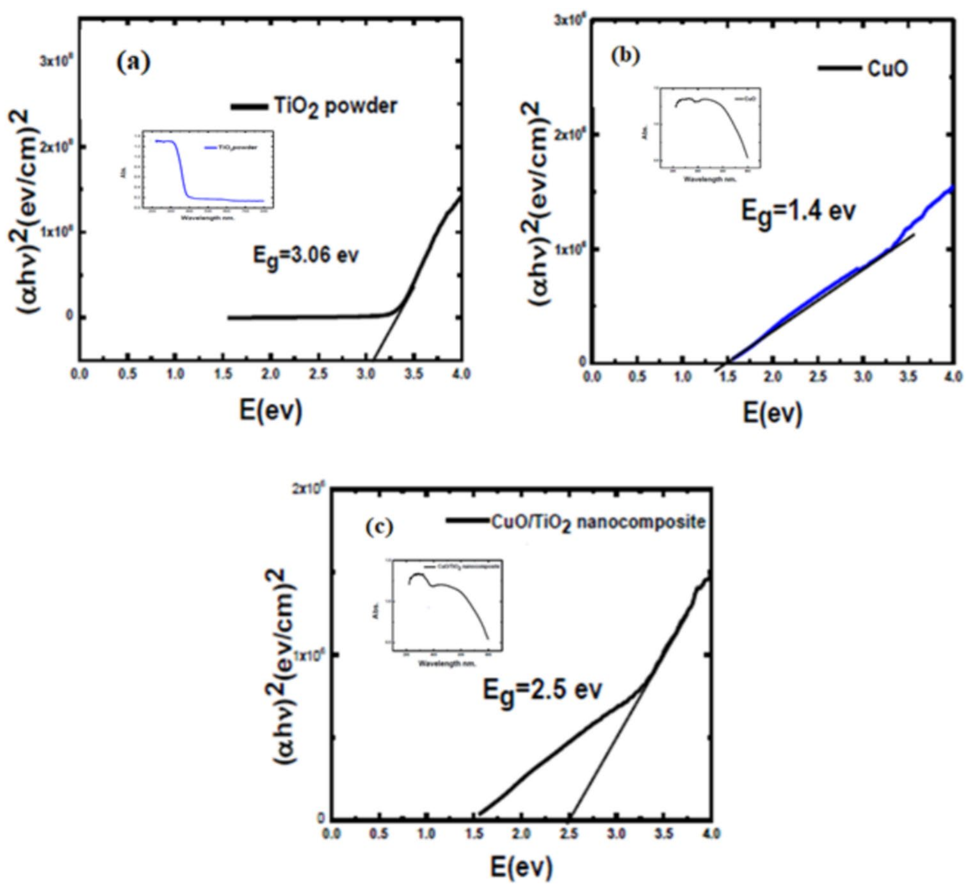
UV–visible analysis

The UV–vis spectra of TiO_2 synthesized by the hydrothermal method are shown in Fig. 2a, showing that TiO_2 absorbs light in the range of the UV region. The bandgap calculated from the relation between $(\alpha h\upsilon)^2$ and E (eV) was 3.06 eV. These results correspond to TiO_2 anatase phase, as reported in many studies. Sergio Valencia et al. suggested that the value of the optical bandgap of TiO_2 is around 3–4 eV. For amorphous materials, the bandgap values are above 3.4 eV, as reported in Ref. [25], so this result of 3.06 eV from Fig. 2a indicates the crystallinity of TiO_2, in agreement with the XRD results.

The optical absorption spectrum was used to determine the optical properties of the CuO structure. Figure 2b show the absorption and bandgap of CuO. Figure 2b shows broad absorption by CuO in the range of the visible region, while a bandgap value of 1.4 eV was obtained from Fig. 2b. These results mean that we prepared CuO structure in good agreement with literature [13]. Asha Radhakrishnan et al. studied the structural and optical absorption and reported a bandgap value for CuO in the range of 1.2–1.8 eV [26]. These results indicate the formation of crystalline CuO.

Figure 2c shows the absorption spectrum and bandgap of the CuO/TiO_2 nanocomposite at room temperature. It is clear there are two absorption peaks in the spectrum: the absorption peak below 400 nm corresponds to TiO_2, while the other peak from 400 to 800 nm indicates absorption by CuO nanoparticles. Figure 2c illustrates a bandgap value of 2.5 eV for the nanocomposite material, indicating that

Fig. 2 Absorption and bandgap of **a** TiO_2, **b** CuO and, **c** CuO/TiO_2 nanocomposite



the bandgap of TiO_2 was reduced by introducing the CuO nanoparticles.

This result is approximately similar to the value reported. Yao et al. reported a bandgap for CuO/TiO_2 of 2.61 eV [27].

HRTEM analysis

Figure 3a–e shows HRTEM images at different magnifications of 20 nm, 10 nm, and 5 nm. Figure 3a clearly shows the CuO and TiO_2 as a composite, while the HRTEM images in Fig. 3b, c clearly show the presence of rough and tiny nanoparticles with average particle size of ~9.54 nm. Additionally, more detailed analysis of the lattice fringes in Fig. 3d, e reveals clear lattice fringe planes corresponding to the lattice spacing of CuO and TiO_2, where the fringe spacing is 0.25 nm for the (002) plane of CuO and 0.24 nm for the (103) plane of anatase TiO_2. These results indicate the formation of CuO/TiO_2 nanocomposite, similar to the results obtained by Xin Tian [28]. These results, in addition to the XRD results, confirm the successful synthesis of CuO/TiO_2 nanocomposite.

I–V characteristics

To evaluate the influence of the nanocomposite on the efficiency of DSSCs, different photoanodes based on TiO_2 and CuO/TiO_2 were fabricated. To evaluate the photosensitivity of these materials, we measured the performance of CuO/TiO_2 thin-film solar cells and compared them with pure TiO_2 dye-sensitized solar cells. The performance of a dye-sensitized solar cell can be evaluated by using the maximum power output (P_{max}), short circuit current (I_{SC}, mA), open-circuit voltage (V_{OC}, V), fill factor, and overall efficiency (η, %) at a constant light level exposure [22]. P_{max} is the maximum efficiency of the DSSC for the conversion of sunlight into electricity. P_{max}, FF, and η are defined as follows [27]:

$$P_{max} = I_{max} \times V_{max}, \tag{1}$$

$$\mathrm{FF} = P_{max} I_{sc} \times V_{oc}, \tag{2}$$

$$\eta = P_{max}/P_{in} \text{ or } I_{sc} \times V_{oc} \times \mathrm{FF}/P_{in}. \tag{3}$$

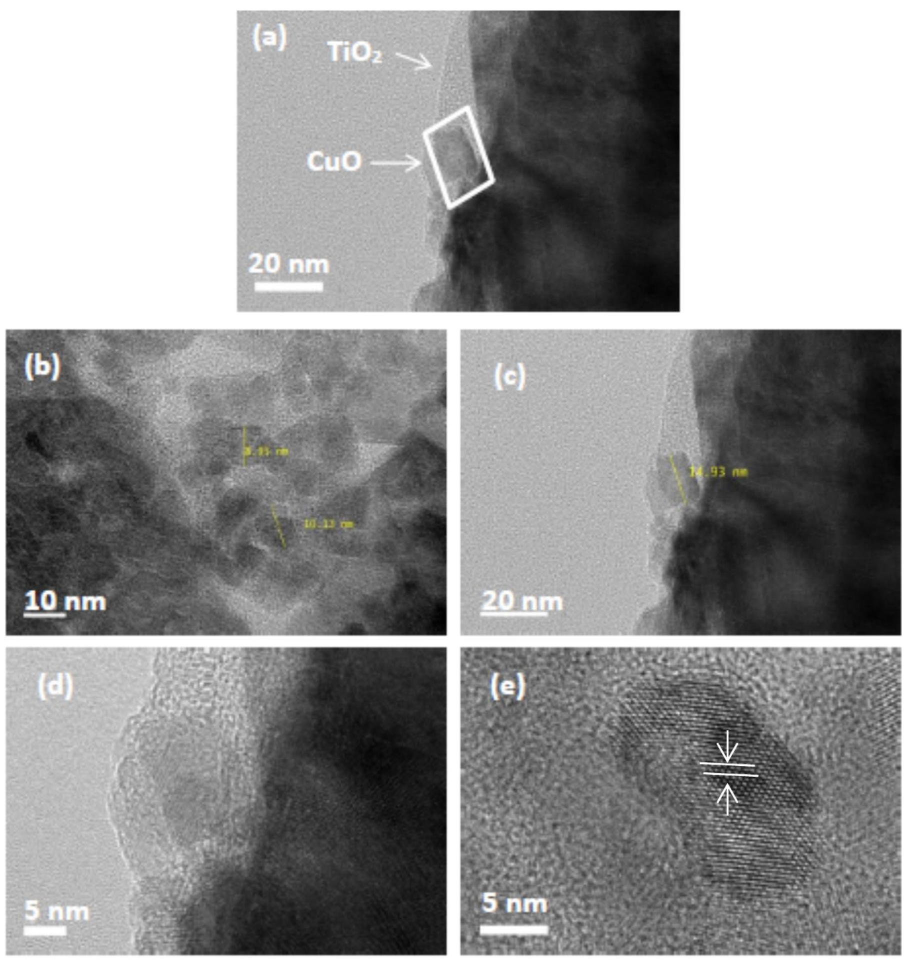


Fig. 3 **a–e** HRTEM images of the CuO/TiO_2 nanocomposite

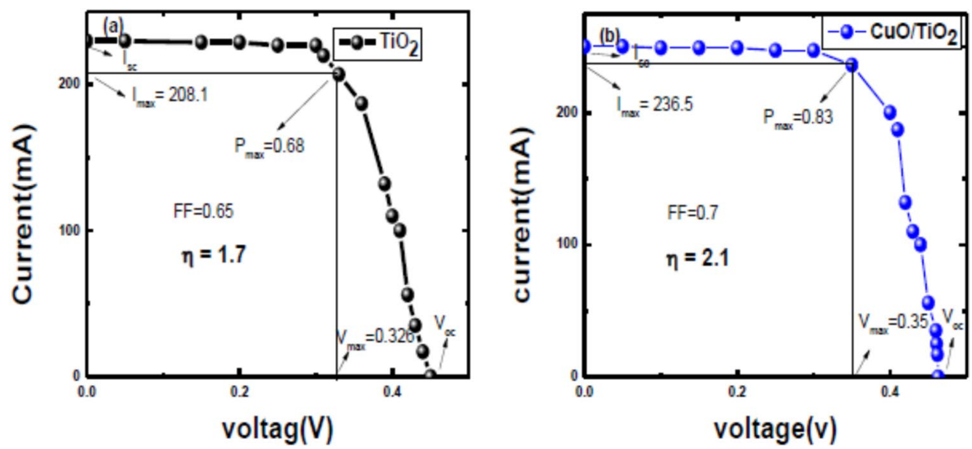


Fig. 4 **a**, **b** *I*–*V* characteristics for the pure TiO_2 and CuO/TiO_2 nanocomposite

The intensity of the incident light (P_{in}) was 0.384 mW/cm^2. The *I*–*V* characteristics for the pure TiO_2 and CuO/TiO_2 nanocomposite are shown in Fig. 4a and b, respectively. The figure displays the relation between the voltage and the current.

CuO/TiO_2 composites significantly improve the electron transfer, increasing the short-circuit current and maximum power. This leads to higher efficiency in dye-sensitized solar cells compared with pure TiO_2, with minimal change in the open-circuit voltage. The maximum power (P_{max}) for CuO/TiO_2 was 0.83, yielding an efficiency of 2.1%, while pure TiO_2 achieved a P_{max} of 0.68 and an efficiency of 1.7%. These results are in good agreement with Refs. [16, 18].

Conclusions

This study introduces a CuO/TiO_2 nanocomposite as an effective photo-anode for dye-sensitized solar cells (DSSCs). TiO_2 nanoparticles were synthesized via a hydrothermal method, resulting in the anatase phase with a high crystallinity, as confirmed by XRD. UV–Vis analysis showed a bandgap of 3.06 eV, and HRTEM revealed an average particle size of 11.84 nm. The CuO/TiO_2 composite was prepared next, with XRD and HRTEM confirming the successful formation of CuO monoclinic and TiO_2 anatase phases. The composite showed two absorption peaks, one in the UV and one in the visible region, indicating enhanced light absorption, with a reduced bandgap of 2.5 eV.

When applied as a photo-anode in DSSCs, the CuO/TiO_2 nanocomposite demonstrated improved performance compared with pure TiO_2. This improved efficiency is attributed to better light harvesting and enhanced electron transport. The CuO/TiO_2 nanocomposite is thus a promising material for improving the performance of DSSCs.

Author contributions H.A. Elbushra conducted the chemical synthesis, characterization, data analysis, plotting, discussion, and manuscript writing. J.Y.C. Liew and Z. A. Talib supervised in Malaysia, overseeing the synthesis and characterization processes. M. Ahmed collaborated in Malaysia, assisting with experiments, characterization, and results revision. H.Wardi and N. Eassa supervised the research in Sudan, providing guidance and consultation through the study. All authors reviewed and approved the final manuscript.

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Data availability All the presented data were obtained by the authors and are accessible if needed. Me Hdeel (H. A. Elbushra) and Prof. Wardi (H. Wardi) do not have official institutional email addresses owing to the current war situation in Sudan.

Declarations

Conflict of interest On behalf of all authors, the corresponding author states that there is no conflict of interest.

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