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WO_3/Bi_2WO_6 photoanode enhancement for photoelectrocatalytic water oxidation; scan rate effect optimization in the cyclic voltammetry deposition method

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ABSTRACT

The photoelectrocatalytic approach is a very efficient technology for eliminating microorganisms and organic contaminants. The development of photoanode is widely recognized as a crucial approach to enhancing the efficiency of photoelectrocatalytic cells. The key goal of this methodology is to enhance the efficacy of photoelectrocatalytic oxidation by optimizing composited photoanode fabrication. This research development focuses mainly on fabricating composite WO_3/Bi_2WO_6 semiconductor thin films with high water oxidation efficiency and favorable photoelectrocatalytic *E. coli* degradation applications. Cyclic voltammetry was utilized to create WO_3/Bi_2WO_6 thin coatings on conducting glass while optimizing the photoelectrocatalytic activity via the scan rate parameter. The characteristics of the developed electrode, including charge transfer resistance, optical properties, morphology, crystal structure, chemical composition, and oxidation numbers, were investigated to improve photoelectrocatalytic activity. It was observed that the scanning rate significantly influenced the characteristics of the WO_3/Bi_2WO_6 electrode and the photoelectrocatalytic activity on water oxidation. It was discovered that the WO_3/Bi_2WO_6 electrode prepared with a scan rate of 25 mV/s exhibited the greatest photoelectrocatalytic water oxidation as well as distinguishing characteristics from other conditions. The decision to utilize decreased scanning rates has been determined to optimize the reaction kinetics and improve the film-forming properties of WO_3/Bi_2WO_6. Significantly, the developed electrode can also be used to eliminate 87.5% of *E.coli* in 15 minutes via a photoelectrocatalytic catalytic mechanism. The photoanode composed of WO_3/Bi_2WO_6 has promising capabilities in removing microorganisms and organic pollutants, making it a viable candidate for future advancements in wastewater management applications.

Keywords: Photoelectrocatalytic, Cyclic voltammetry techniques, WO_3/Bi_2WO_6, *E.coli* degradation

INTRODUCTION

The issue of microbial contamination and toxic organic compounds is an additional aspect that impacts the quality of life and the broader ecosystem. Therefore, it is essential to create or offer efficient strategies to handle these issues [1]. One of the most effective advanced oxidation technologies usually developed for removing such pollutants is photoelectrocatalytic (PEC) technology [2-6]. The strategy or guiding principle for the development of such techniques is the selection of suitable semiconductors and the creation of a substrate-based semiconductor film fabrication process. WO_3 and Bi_2WO_6 are two attractive semiconductors extensively used as photoanodes for organic elimination [7-10]. Because of its adequate energy value, it can absorb visible light and has a high positive valence potential appropriate for oxidation processes in aqueous solutions [11]. Preparing the semiconductor film on the substrate is regarded as a significant factor influencing the performance and durability of PEC cells in practical applications. The sol-gel method is a relatively simple and cost-effective technology for making WO_3 and Bi_2WO_6 films since it allows for perfect control over film thickness and composition and can be easily scaled up for large-scale production [12, 13]. However, to achieve the desired crystalline structure, the sol-gel approach may require high-temperature annealing, and film quality might be affected by processing conditions. The hydrothermal approach enables the formation of WO_3 and Bi_2WO_6 films at lower temperatures, increasing crystallinity and photocatalytic activity [8, 14]. However, a longer processing time and careful control of reaction parameters may be required to produce the necessary film qualities. Electrodeposition is a viable option for preparing WO_3 and Bi_2WO_6 films because it is a basic, cost-effective

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technique that does not require expensive equipment or high temperatures. This investigation aims to develop an electrochemical technique for preparing composite WO_3 and Bi_2WO_6 films on conducting glass substrates using cyclic voltammetry (CV) [15, 16]. Cyclic voltammetry is a versatile and powerful technology for fabricating and characterizing semiconductor films, providing precise control, important insights, and scalability for a wide range of optoelectronics, photoelectrochemistry, and beyond applications. We developed the CV method for WO_3 and Bi_2WO_6 film formation by deciding the optimal PEC water oxidation properties, including precursor concentration, applied potential range, and scanning rate. We discovered that the scanning rate considerably impacted the WO_3/Bi_2WO_6 electrode characteristics and the PEC properties for water oxidation. Importantly, our system can also eliminate *E. coli* and is suitable for further development in treating effluent contaminated with microorganisms and toxic organic compounds.

MATERIALS AND METHODS

Chemical and materials

Throughout the experiment, deionized water was utilized to produce all solutions and compounds of the analytical grade. Diethylenetriamine pentaacetic acid; DTPA (Sigma Aldrich), Ammonium hydroxide; NH_4OH (J.T.Baker), Bismuth(III)oxide; Bi_2O_3 (Sigma Aldrich), Ammonium metatungstate hydrate; $(NH_4)_6H_2W_{12}O_40{\cdot}H_2O$ (Sigma Aldrich), Nitric acid; HNO_3 (Univar), Hydrogen peroxide; H_2O_2 (Chem-supply), Ethanol; C_2H_5OH (Rcl Labscan), Sodium chorine; NaCl (Kemaus), Methylene Blue; $C_{16}H_{18}ClN_3S$ (KEMAUS) Sodium hydroxide; NaOH (Univar) were used as chemical precursor materials.

WO_3/Bi_2WO_6 precursor solution preparation

To prepare the WO_3/Bi_2WO_6 precursor solution, 0.83 g of diethylenetriamine pentaacetic acid (DTPA) was dissolved in 2.5 ml of 30% ammonia solution (NH_4OH), and 50 ml of distilled water was then added. The solution was stirred regularly at 80 °C with a mixture of 0.4 g of bismuth(III)oxide (Bi_2O_3) and 0.15 g of ammonium metatungstate hydrate ($(NH_4)_6H_2W_{12}O_{40}.H_2O$). Hydrogen peroxide (77 µl) and nitric acid (1,190 µl) were added to the solution while it was stirred at 80 °C for 15 minutes.

Preparation of WO_3/Bi_2WO_6 electrode

The fluorine-doped tin oxide (FTO) electrode substrate was cleansed using sonication for 10 minutes with detergent, 3 M sodium hydroxide (NaOH), ethanol, and distilled water, respectively. The cleaned FTO was dried and connected with copper wire to serve as a working electrode for the subsequent WO_3/Bi_2WO_6 electrode preparation. Figure 1 displays a three-electrode system configuration for fabricating WO_3/Bi_2WO_6 films using cyclic voltammetry. We use an FTO working electrode and an Ag/AgCl reference electrode to apply a potential ranging from -0.6 V to 0.8 V. For monitoring the current of the CV electrodeposition procedure, and Pt was utilized as a counter electrode. This study emphasized the effect of scan rate in the range of 10-125 mV/s for 30 cycles while keeping the solution temperature at 80 °C. The developed WO_3/Bi_2WO_6 electrode was then sintered for 1 hour at 450 °C. Copper wire was utilized to connect the prepared electrode and epoxy glue was employed to control the working area of the photoanode. The water oxidation photocurrent was used to determine the optimum conditions and performance of the WO_3/Bi_2WO_6 photoanode.

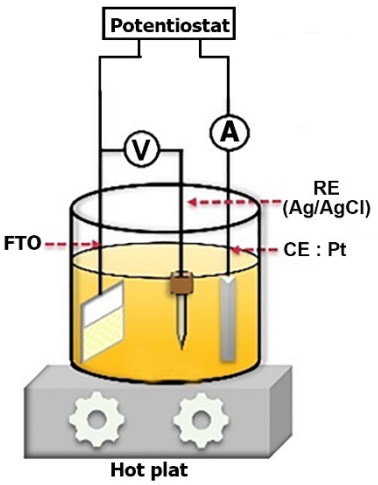

Figure 1 experimental set up for WO_3/Bi_2WO_6 photoanode fabrication using an CV electrodeposition technique.

Characterization and photoelectrocatalytic activity study

The PEC activity for water oxidation was determined using a voltammetry analyzer (Princeton Applied Research, Inc., VersaSTAT 3) in a 0.5 M NaCl electrolyte solution at an applied potential of 1.0 V vs. Ag/AgCl under visible light illumination. Electrochemical impedance spectroscopy (EIS) frequencies ranging from 100 kHz to 0.1 Hz were used to investigate the charge transfer resistance and capacitance at the interfacial electrode/electrolyte. The UV/Vis spectrophotometer (Shimadzu, UV-1601) was used to study the thin WO_3/Bi_2WO_6 film's optical characteristics. Scanning Electron Microscope (SEM, JEOL, JSM 6510) was employed to observe the morphology of the surfaces of thin films. X-ray Diffraction Analysis (XRD, Rigaku, RINT 200TH) was used to investigate the crystal structure. The Energy Dispersive X-ray System (EDX, OXFORD) and X-ray photoelectron spectroscopy (XPS, JEOL, JPS-9010TR) were utilized to confirm the chemical composition and oxidation state of the element at the electrode surface. The effectiveness of PEC *E. coli* degradation was investigated using a two-electrode

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system composed of a WO_3/Bi_2WO_6 photoanode and a stainless-steel cathode electrode.

RESULTS AND DISCUSSIONS

WO_3/Bi_2WO_6 photoanode characterization

Figure 2 illustrates the cyclic voltammogram produced by the reduction reaction of WO_3/Bi_2WO_6 adhesion at the FTO substrate during the scan of a negative potential between 0.8 and -0.6 V. The reduction peak current in figure 2 increases with slower scan rates from 125 to 25 mV/s at approximately -0.2 V potential. When the scan rate was decreased to 10 mv/s, the reduction potential position was altered to -0.3 V. The findings indicate that the response mechanism exhibits variations while adjusting for extremely low scan rates. While oxidation currents would be produced in the region of 0.1 to 0.6 V when the scan rate was positively controlled between -0.6 and 0.8 V, shifting as the scan rate slowed down. This phenomenon may arise due to the release of certain precursor or interfering molecules, resulting in increased purity of the electrodes. This is a beneficial aspect of the cyclic voltammetry film preparation procedure.

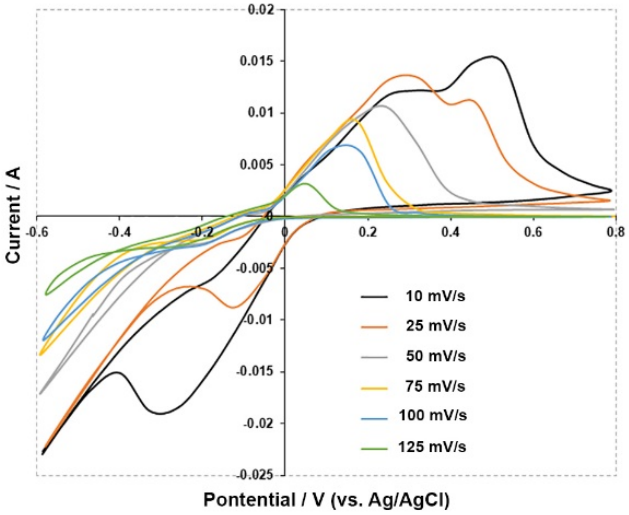

Figure 2 Effects of various scan rates on cyclic voltammograms during WO_3/Bi_2WO_6 deposition using the CV technique.

Figure 3 demonstrates the effect of the scan rate in the CV technique used to prepare WO_3/Bi_2WO_6 electrodes. We discovered that decreasing the scan rate from 125 to 25 mV/s increased the water oxidation photocurrent values of WO_3/Bi_2WO_6 photoanode. This finding elucidates that a decrease in scanning rate allows for increased migration time of the precursor in the solution towards the electrode surface, facilitating a more organized arrangement of the WO_3/Bi_2WO_6 film. Consequently, the resultant film exhibits enhanced efficiency. We discovered that the PEC WO_3/Bi_2WO_6 electrodes were the most effective for oxidizing water at a scan rate of 25 mV/s. When the scan rate was reduced to 10 mV/s, it was seen that the electrode efficiency decreased dramatically. This decrease can be attributed to the scanning rate being too slow, leading to incomplete adhesion of the semiconductor layer.

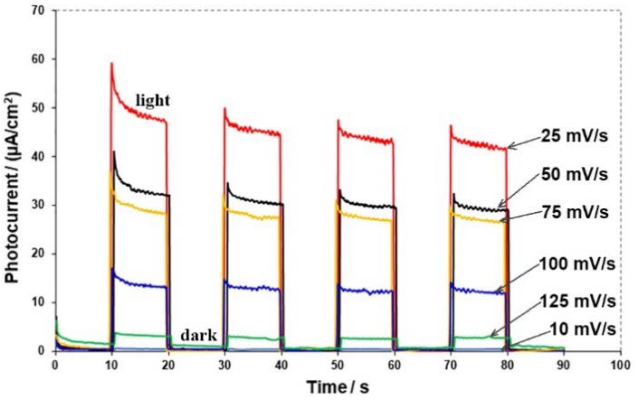

Figure 3 The photocurrent response from water oxidation of WO_3/Bi_2WO_6 photoanode prepared with different scan rates ranging from 10 to 125 mV/s.

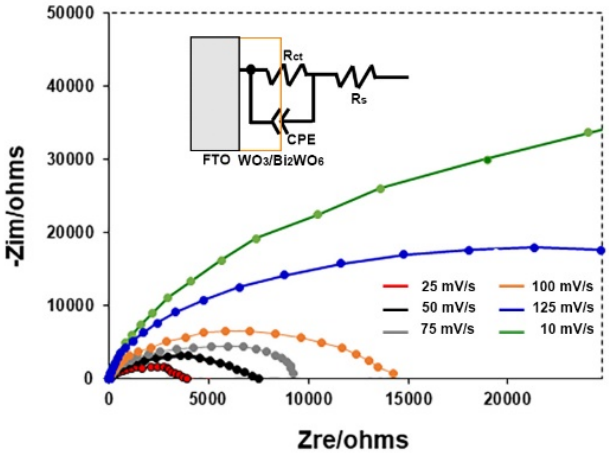

Figure 4 The Nyquist plot of WO_3/Bi_2WO_6 electrode for water oxidation under visible light.

Figure 4 displays the electrochemical impedance spectroscopy (EIS) data employed to examine charge transfer resistance characteristics inside the interfacial region of the WO_3/Bi_2WO_6 photoanode. The Inset of figure 4 depicts a circuit consisting of charge transfer resistance (Rct), solution resistivity (Rs), and electrical capacitance (CPE) that most closely reflects the reaction at the electrode-solution interface. The Nyquist plot in the provided figure depicts the characteristics of the WO_3/Bi_2WO_6 electrodes produced using a scan rate of 25 mV/s. The semicircle with the shortest radius on the plot indicates the lowest charge transfer resistance, which correlates with the maximum photocurrent observed in the previous findings. This relationship suggests that the high PEC properties of electrodes under optimal conditions result from their low charge transfer resistance, which is controlled by a slow

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scanning rate when forming the film semiconductor with the desired properties.

Figure 5 displays X-ray diffraction patterns that illustrate the crystalline structure of the chemical composition of the FTO substrate and WO_3/Bi_2WO_6 electrode prepared at varied scan rates.

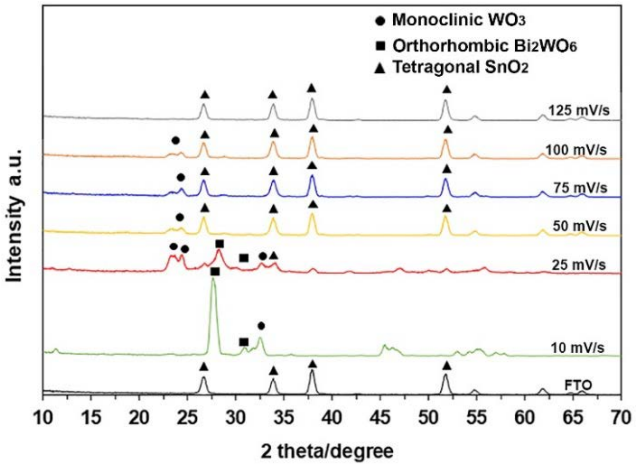

Figure 5 X-ray diffraction pattern of a bare FTO substrate compared to a WO_3/Bi_2WO_6 electrode produced with different scan rates conditions ranging from 10 to 125 mV/s.

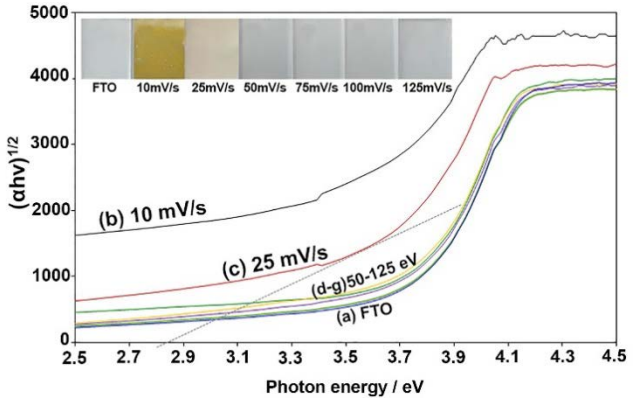

Figure 6 Correlation of absorbance coefficient and bandgap energy, inset of electrode photograph of (a) bare FTO substrate and FTO/WO_3/Bi_2WO_6 electrodes prepared with the different scan rates of (b) 10, (c) 25, (d) 50, (e) 75, (f) 100 and (g) 125 mV/s.

We discovered that the FTO substrate exhibited diffraction peaks at 26.7°, 34.1°, 38.02°, and 52.0°, which corresponded to tetragonal SnO_2 [17]. It was found that the prepared electrode at scan rates of 10 and 25 mV/s presented the main XRD peak at 2θ of 28.02°, 32.6°, which indicates the orthorhombic Bi_2WO_6 crystal structure on the FTO surface [18]. However, for the prepared electrode at a higher scan rate than 25 mV/s, a small amount of Bi_2WO_6 film is present.

It was observed that the produced electrode exhibits a small presence of Bi_2WO_6 semiconductor when applied to scan rates exceeding 25 mV/s. The X-ray diffraction (XRD) analysis reveals that the composite WO_3/Bi_2WO_6 exhibits optimal formation. At the optimal scan rate of 25 mV/s, it can produce both a monoclinic WO_3 structure at 2θ of 23.6° and 24.4° and 32.5° [15] an orthorhombic Bi_2WO_6 structure, which results in excellent PEC properties.

Figure 6 exhibits the optical properties of the WO_3/Bi_2WO_6 electrode, including band energy and a photograph, as a result of the scan rate preparation procedure compared to the FTO substrate.

The WO_3/Bi_2WO_6 thin film prepared at a scan rate of 10 mV/s has the greatest thickness, the highest absorption properties, and the darkest yellow color, whereas increasing the scanning rate results in electrodes with softer colors and thinner films. The preceding results demonstrate that a slow scanning rate increases the bonding period of the semiconductor film to the electrode surface, resulting in a thick film, and the thickness decreases as the scan rate increases. The Tauc equation, shown in Equation 1, helped us confirm that the band gap energy (Eg) was 2.85 eV at the best scan rate of 25 mV/s for the electrode preparation conditions.

$$(\alpha h\upsilon)^{1/2} = \beta(h\upsilon - E_g) \quad (1)$$

where β is a constant, α is the molar extinction coefficient, and Eg is the band gap energy. This Eg value was consistent with the optical absorption properties of the WO_3/Bi_2WO_6 semiconductor [19].

Figure 7 displays SEM images illustrating the morphology of composited WO_3/Bi_2WO_6 thin films on FTO substrates prepared at various scan rates with significantly different particle sizes and distributions. The particles of WO_3/Bi_2WO_6, which were prepared at a scan rate of 10 mV/s, exhibited a much larger particle size than those prepared under other conditions. This can be attributed to the extended accumulating period during the deposition process of the WO_3/Bi_2WO_6 thin films on the electrode substrate under the higher scan rate condition. The particles exhibited comparable size and dispersion. The samples prepared at a scan rate of 25 mV/s displayed increased porosity and greater surface roughness. The utilization of a scan rate of 25 mV/s in the preparation of the electrode has been found to significantly improve the morphologies of its surfaces. This improvement is characterized by an increased surface area and roughness, which enhances the effectiveness of solution contact and electron transport at the electrode surface. The obtained outcome provides robust evidence in favor of the PEC characteristics. Based on the investigation into the impact of scanning rate on the formation of WO_3/Bi_2WO_6 films using the CV technique, it can be inferred that a decreased scanning rate holds promise for augmenting the degree of precision in the deposition procedure. Consequently, this leads to the creation of films that demonstrate enhanced uniformity and distinct characteristics. Conversely, an increased

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scanning rate may result in decreased control over the film's creation and a decline in quality. In summary, the scanning rate employed in cyclic voltammetry influences the kinetics of redox reactions, mass transport, film formation processes, and potential distribution within the electrochemical system. Slower scanning rates have been chosen to optimize reaction kinetics and increase the film-forming characteristics.

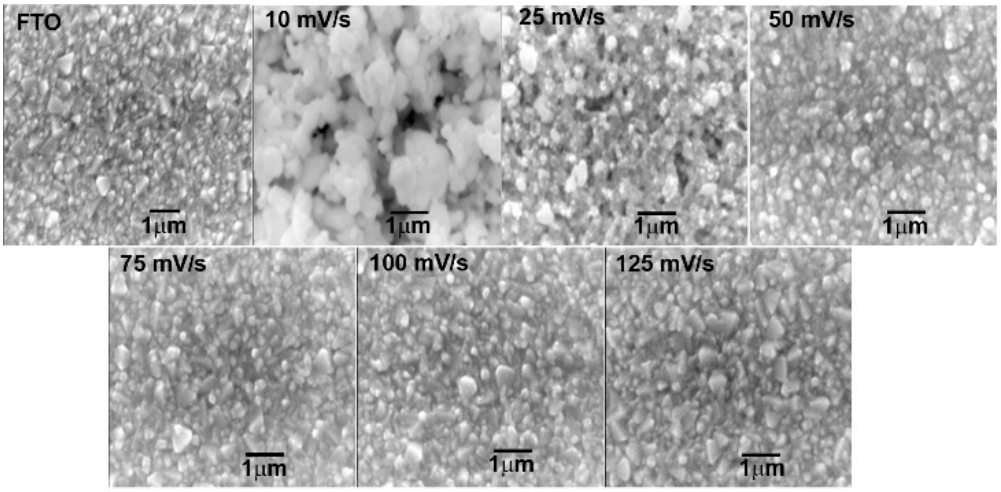

Figure 7 SEM images of a bare FTO substrate and an FTO/WO_3/Bi_2WO_6 electrode prepared with scan rates ranging between 10 and 125 mV/s.

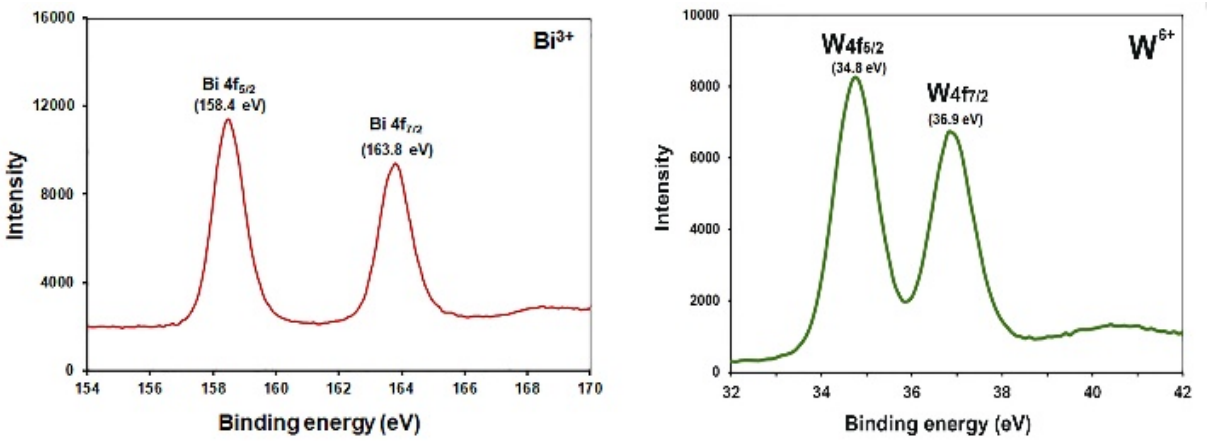

Figure 8 XPS spectra of Bi and W at FTO/WO_3/Bi_2WO_6 photoanode.

The composition of W, Bi, and O on the FTO/WO_3/Bi_2WO_6 electrodes was investigated by EDX analysis. Our findings unequivocally establish the presence of WO_3 and Bi_2WO_6 compounds on the FTO substrate. Furthermore, we investigate the chemical composition and oxidation state of all elements present in the composite WO_3/Bi_2WO_6 electrode utilizing the X-ray Photoelectron Spectroscopy (XPS) technique. The XPS spectra of all elements present in the WO_3/Bi_2WO_6 film were obtained by calibrating the binding energy using C1s (284.6 eV) as a reference. The experimental results indicate that the O1s orbital exhibits a binding energy of 530.8 eV. The XPS spectra of $Bi4f_{5/2}$ and $Bi4f_{7/2}$ orbitals exhibit binding energies of 163.8 eV and 158.4 eV, respectively, as shown in Figure 8. The energy levels of $W_{4f5/2}$ and $W_{4f7/2}$ were measured to be 34.8 eV and 36.9 eV, respectively. The findings validate the oxidation states of Bi^{3+} and W^{6+} seen at the WO_3/Bi_2WO_6 composite electrode [20]. The XPS and EDX findings offer significant insights that may be utilized to validate the chemical composition and chemical state of WO_3/Bi_2WO_6 present on the FTO substrate. The present study employed the optimum WO_3/Bi_2WO_6 photoanode to investigate the effectiveness of *E. coli* degradation under visible light irradiation and a bias potential of 1.5 V. Consequently, the PEC cell that was presented showed the capability to degrade 87.50 % of *E. coli* over a duration of 15 minutes. This finding confirms that the WO_3/Bi_2WO_6 electrodes that were produced are really effective in facilitating PEC oxidation, thereby enabling the elimination of *E. coli* in aqueous solutions. Moreover, these electrodes hold promise for potential application in removing microorganisms from wastewater.

CONCLUSIONS

We have successfully prepared the WO_3/Bi_2WO_6 photoanode using the CV method and applied it to the degradation of *E. coli* using the proposed PEC

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cell. The scan rate in the CV method substantially impacts the characteristics and PEC activity of the composite WO_3/Bi_2WO_6 thin film fabrication. The optimal scanning rate condition is crucial for enhancing light absorption, surface morphology, and electron transport between the electrode and the electrolyte during the fabrication of WO_3/Bi_2WO_6 thin film electrodes. The optimal WO_3/Bi_2WO_6 photoanode fabrication condition for the highest PEC water oxidation properties was a scanning rate of 25 mV/s. We can conclude that the cyclic voltammetry scanning rate impacts the kinetics of redox reactions, mass transport, film formation processes, and potential distribution within the electrochemical system. The decision to take advantage of slower scanning rates has been made to optimize the kinetics of the reaction and enhance the film-forming characteristics of WO_3/Bi_2WO_6. The WO_3/Bi_2WO_6 composite material exhibits a notable capability for effectively eliminating *E.coli* in the photoelectrochemical (PEC) process. This finding suggests that the material holds promise for potential applications in eliminating other microorganisms present in wastewater.

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