



BiFeO₃/carbon black-modified screen-printed electrode for sensitive electrochemical determination of furazolidone

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ABSTRACT

Antibiotic resistance is a major concern to the global health, highlighting the need for new techniques for the targeted identification and analysis of specific antibiotic drugs. This study addresses the electrochemical detection of the pharmaceutical agent furazolidone (FZD). A BiFeO₃/carbon black-modified screen-printed carbon electrode (BiFeO₃/CB@SPCE) was evaluated for the electrochemical determination of furazolidone (FZD). Although BiFeO₃/carbon composites have previously been explored for electrochemical sensing, the present study focuses on their application in a disposable SPCE format for FZD-specific cathodic detection. The electrochemical response of FZD was evaluated across a concentration range of 0.01–130 μM and pH range of 3.0–11.0 in phosphate-buffered saline (PBS). Interference studies were conducted using common ions and bio-molecules. The fabricated biosensor exhibited high stability, reproducibility, and a low detection limit (LOD) of 0.045 μM. A wide linear response range (0.01–130 μM) was achieved owing to the synergistic effects of the nanocomposite, outperforming comparable BiFeO₃/CB-based electrodes reported at 200 μM. The highest cathodic current (−42.59 μA at −0.38 V) was observed at pH 7. The sensor showed excellent selectivity in the presence of Na⁺, K⁺, Ca²⁺, Mg²⁺, Fe³⁺, Cu²⁺, ascorbic acid, citric acid, glucose, and fructose. These results demonstrate good repeatability, anti-interference ability, and these results demonstrate the analytical applicability of the BiFeO₃/CB@SPCE for sensitive FZD determination in aqueous, biological, and pharmaceutical samples.

1. Introduction

Furazolidone (FZD) is a nitrofurantoin-based antibacterial agent that has

been used to treat veterinary and human pathogens for several years. Highly disruptive *Helicobacter pylori* infections are customarily treated with FZD [1]. However, the presence of FZD in ecosystems is known to

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damage ecology and disrupt monoamine oxidase [2]. Nearby water bodies are vulnerable to excess FZD and its eventual runoff into wastewater, and their continued accumulation increases the risk of microbial antibiotic resistance. FZD contamination may cause fever and related health issues in individuals with weakened immune systems. As such, the European Union imposed a limit on the use of nitrofurantoin and its derivatives ($0.5 \mu\text{g/kg}$) in November 2022 (EU 2023/411).

It is important to detect FZD levels to realize its impact on ecology, and studies using gas chromatography [3], high-performance liquid chromatography, and liquid chromatography–mass spectrometry [4] have been conducted. In addition, the use of modern methods, such as calorimetric, photothermal detection [5], surface-enhanced Raman scattering–based detection [6], fluorescence detection [7], and electrochemical detection [8,9] is gaining increasing attention. To overcome the limitations of conventional analytical methods, electrochemical sensors have emerged as promising alternatives because of their rapid response, high sensitivity, relatively low cost, portability, and suitability for decentralized and potentially continuous monitoring [10–13]. Furthermore, electrochemical sensing can be performed using different electrode platforms, including glassy carbon electrodes, carbon paste electrodes, and screen-printed carbon electrodes.

Typically, for biosensor fabrication, the desired properties of the electrode material include high electrical conductivity, large electroactive surface area, and stability with good surface functionalization possibilities. Bismuth ferrite (BiFeO_3) is a unique perovskite-structured room-temperature multiferroic material with excellent magnetic and electronic properties and is an ideal candidate for electrochemical sensor electrodes owing to its cost-effectiveness, highly manipulable nature, and reproducible properties [14,15], [11,12]. However, its intrinsically poor electrical conductivity limits the rate of electron transfer between the analyte (FZD) and the interface of the electrode. In contrast, carbon black (CB), which is a derivative of charred hydrocarbon, has several beneficial electrochemical characteristics that can be exploited for the fabrication of biosensors. CB can be used in its native state [16], [13] or as composites with metals [17], [14] or semiconducting oxides [18], [15]. Compared with other carbon-based materials, CB has a high surface area to volume ratio, tunable electrical properties, relatively low cost to synthesize, along with good thermal and mechanical stability, making it an excellent candidate for use in nanocomposite matrices for electrochemical sensor fabrication.

BiFeO_3 /carbon-based composites have previously been investigated as electrode materials for electrochemical sensing, including closely related systems reported in Refs. [19, 20]. Therefore, the present work does not claim the introduction of a fundamentally new BiFeO_3 /carbon material class. Instead, this study investigates the analytical applicability of a BiFeO_3 /CB composite on a disposable screen-printed carbon electrode for the cathodic determination of FZD. The specific contributions include optimization of the potential window and solution pH for FZD reduction, evaluation of electron-transfer and mass-transport behavior, DPV-based quantification over 0.01 – $130 \mu\text{M}$, and validation in tap water, river water, urine, and pharmaceutical tablet samples.

2. Material and methods

2.1. Synthesis of BiFeO_3

The co-precipitation technique was adapted for the synthesis of BiFeO_3 . Initially, bismuth nitrate (Bi_2NO_3) (0.1 M) and iron (III) nitrate nanohydrate (0.1 M) were dissolved in 20 mL of deionized water in separate beakers. The solutions were individually stirred for 15 min . Finally, both solutions were transferred to a new beaker to form the precursor solution. The solution mixture was stirred for 1 h , then 50 mL of sodium hydroxide was added to maintain a pH of 12 . Additionally, cetyltrimethylammonium bromide (0.05 M) surfactant was added to remove aggregation. This solution was kept under stirring for 1 h , then transferred to a Teflon autoclave followed by heating at 180°C for 12 h

in a hot air oven. The autoclave was kept at room temperature to cool for 24 h , the product was separated and rinsed alternatively with water and ethanol, dried for 12 h , and then calcined at 450°C in a box furnace to obtain the final product.

2.2. Ultrasonic-assisted preparation of BiFeO_3 -CB nanocomposite

To prepare the BiFeO_3 /CB nanocomposite, 2 mg of BiFeO_3 and 1 mg of CB were mixed, corresponding to a fixed BiFeO_3 : CB mass ratio of $2:1$. The mixture was dispersed in $700 \mu\text{L}$ of deionized water and $300 \mu\text{L}$ of ethanol and ultrasonicated for 1 h to ensure uniform distribution of BiFeO_3 and CB. The preparation procedure is illustrated in Fig. S1.

3. Results and discussion

3.1. Microstructural, structural, and chemical composition analysis

Field emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDX), and elemental mapping techniques were employed to analyze the morphological traits and the elemental distribution of the synthesized materials. SEM image of BiFeO_3 (Fig. 1A (a and b)) clearly reveals aggregates composed of crystalline particulates rather than isolated nanocrystals, indicating the inherent limitations of the BiFeO_3 aggregation effects. For CB (Fig. 1A (c and d)), the surface features are spherical with an average size of 80 nm . However, for BiFeO_3 /CB (Fig. 1A (e and f)), the nanocomposite matrix forms a homogeneous network of interconnected carbon layers encapsulated over the BiFeO_3 nanocrystals, which enhance the structural uniformity and electrical conductivity. Both the overlay image and EDAX elemental spectrum are presented in Fig. 1B (a and b), which evidences the elements present in BiFeO_3 /CB. Furthermore, the elementally mapped micrographs portray the uniform distribution of Bi, C, O, and Fe (Fig. 1B (c–f)), respectively, showing the composite structure is formed.

The structure and morphology of the BiFeO_3 /CB nanocomposite were examined using transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM), and selected-area electron diffraction (SAED). The TEM images in Fig. 2(a–d) clearly show the BiFeO_3 /CB composite, where the BiFeO_3 nanorods and CB are uniformly distributed across the textured surfaces at different magnifications. The SAED pattern (Fig. 2(e)) displays diffraction rings indexed to the (101) and (012) planes of BiFeO_3 , which concur with the X-ray diffraction (XRD) data. The HRTEM image (Fig. 2(f)) shows well-defined lattice fringes with no evident structural defects. The observed interplanar spacings of approximately 0.40 and 0.28 nm correspond to the (101) and (012) planes, respectively, further supporting the XRD results (Fig. 3(a)). These observations confirm the crystalline quality and structural stability of the BiFeO_3 nanorods within the composite.

The crystal structures of BiFeO_3 , commercial CB, and the BiFeO_3 /CB composite were determined using XRD analysis. This technique accurately identified the crystalline phases of the materials. The XRD pattern of CB (Fig. 3(a)) shows two distinct peaks at 25.9° and 43.6° , corresponding to the (002) and (110) crystallographic planes of pristine carbon. These findings align with those in the existing literature, confirming the structural integrity and purity of CB. For BiFeO_3 , prominent diffraction peaks were observed at 2θ values of 22.49° , 31.81° , 32.14° , 39.51° , 45.81° , 51.38° , 57.01° , and 67.09° (JCPDS No. 00–020-0169), corresponding to the (101), (012), (110), (021), (202), (113), (122), and (220) planes. These peaks confirm the unique atomic arrangement and crystalline structure of BiFeO_3 , indicating its high purity. Additionally, the diffraction pattern of the BiFeO_3 /CB composite matched the characteristic peaks of BiFeO_3 , demonstrating the successful formation of the nanocomposite [21–23], [16–18].

Fig. 3(b) shows the Raman spectra of CB, BiFeO_3 , and the BiFeO_3 /CB nanocomposite. BiFeO_3 displays characteristic modes in the regions of 70 – 300 and 416 – 700 cm^{-1} . The peak at 174 cm^{-1}

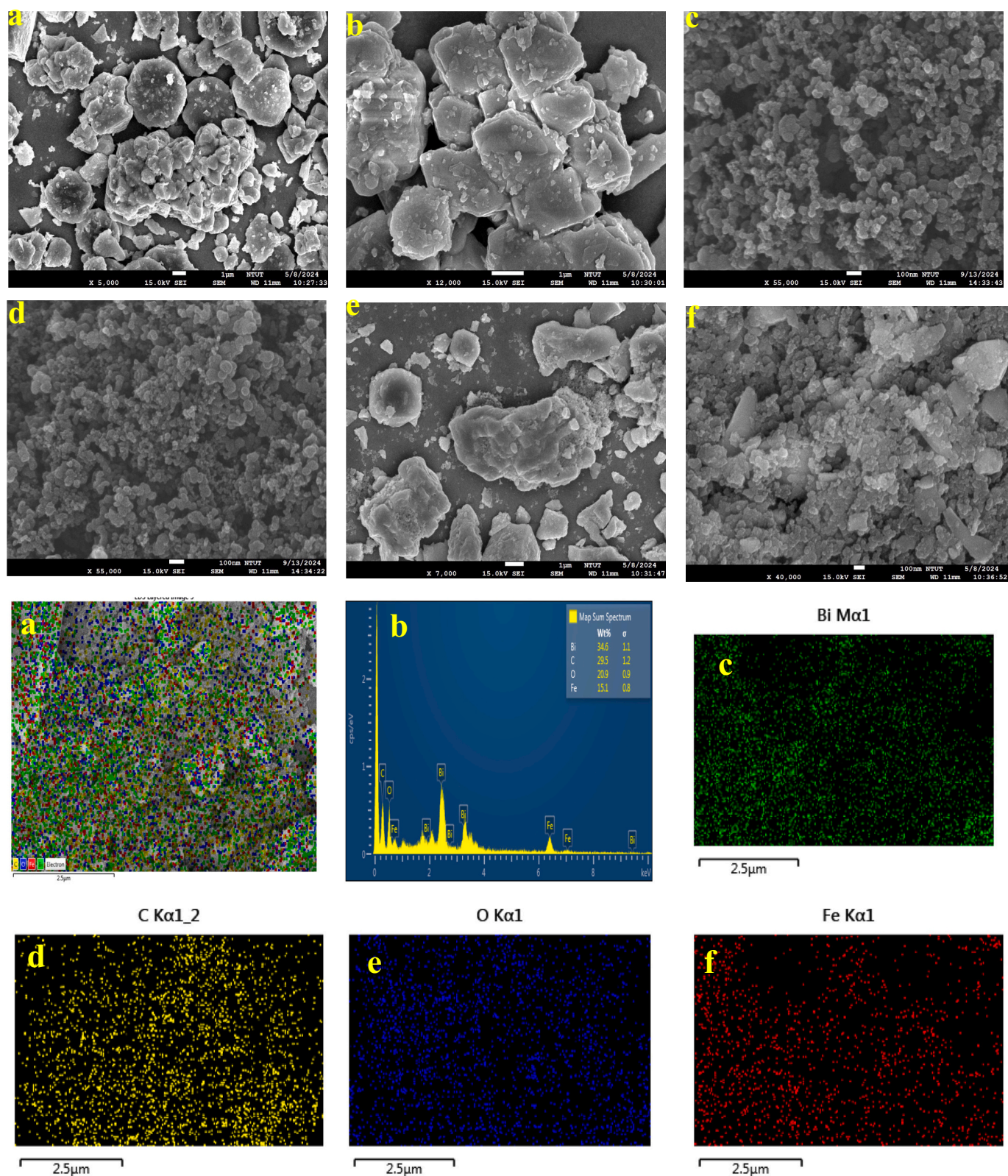


Fig. 1. A, (a) and (b): Field-emission scanning electron microscopy (FE-SEM) images of BiFeO₃; (c) and (d): carbon black (CB); (e) and (f): BiFeO₃@CB nanocomposite.

B, (a) Overlay image of the elements present in the BiFeO₃@CB nanocomposite; (b) energy-dispersive X-ray analysis (EDAX) spectrum; EDAX elemental mapping images of (c) Bi, (d) C, (e) O, and (f) Fe.

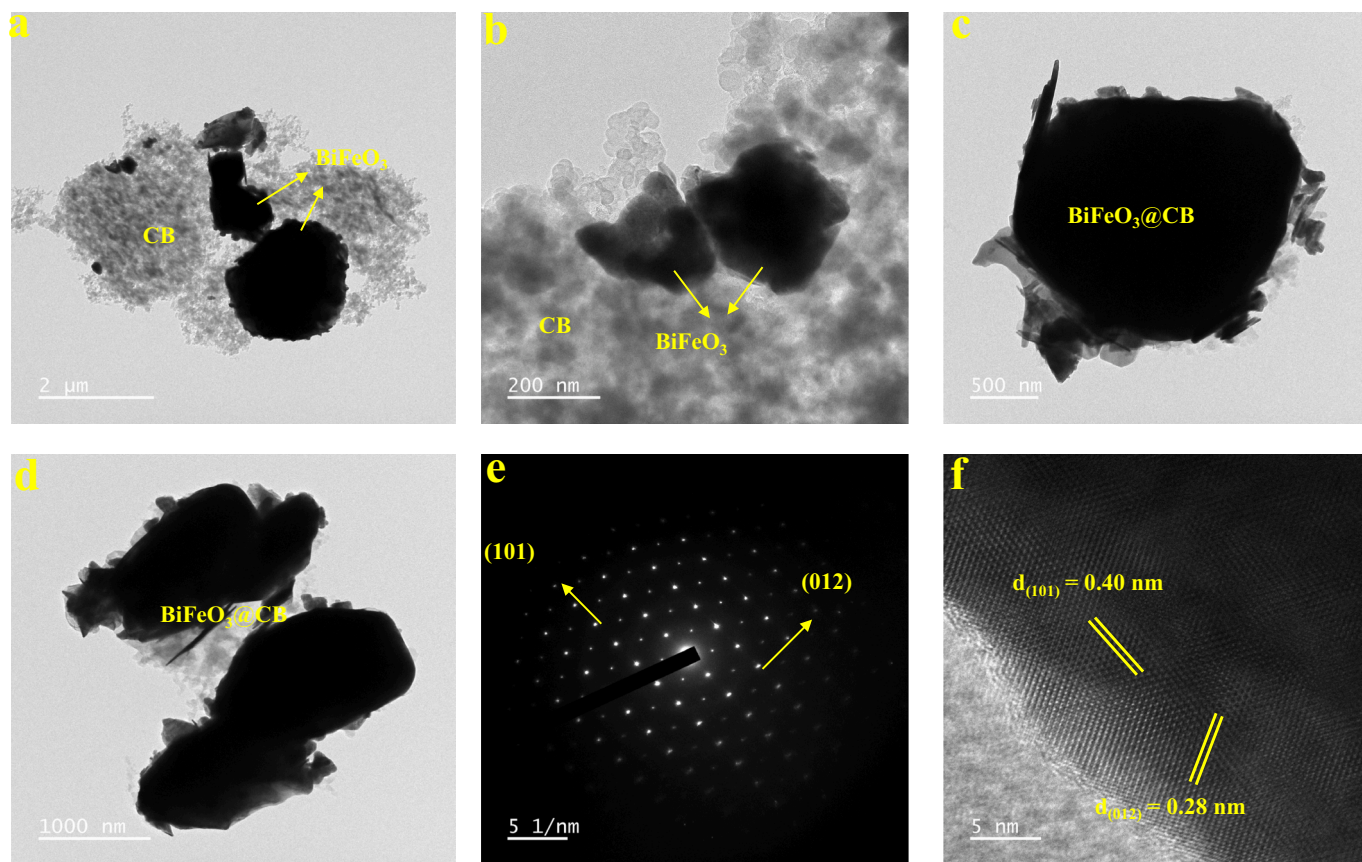


Fig. 2. Transmission electron microscopy (TEM) images of $\text{BiFeO}_3@\text{CB}$ at different scales: (a) 2 μm , (b) 200 nm, (c) 500 nm, and (d) 1000 nm. The corresponding selected area electron diffraction (SAED) patterns are shown in (e), whereas high-resolution TEM (HR-TEM) images are displayed in (f).

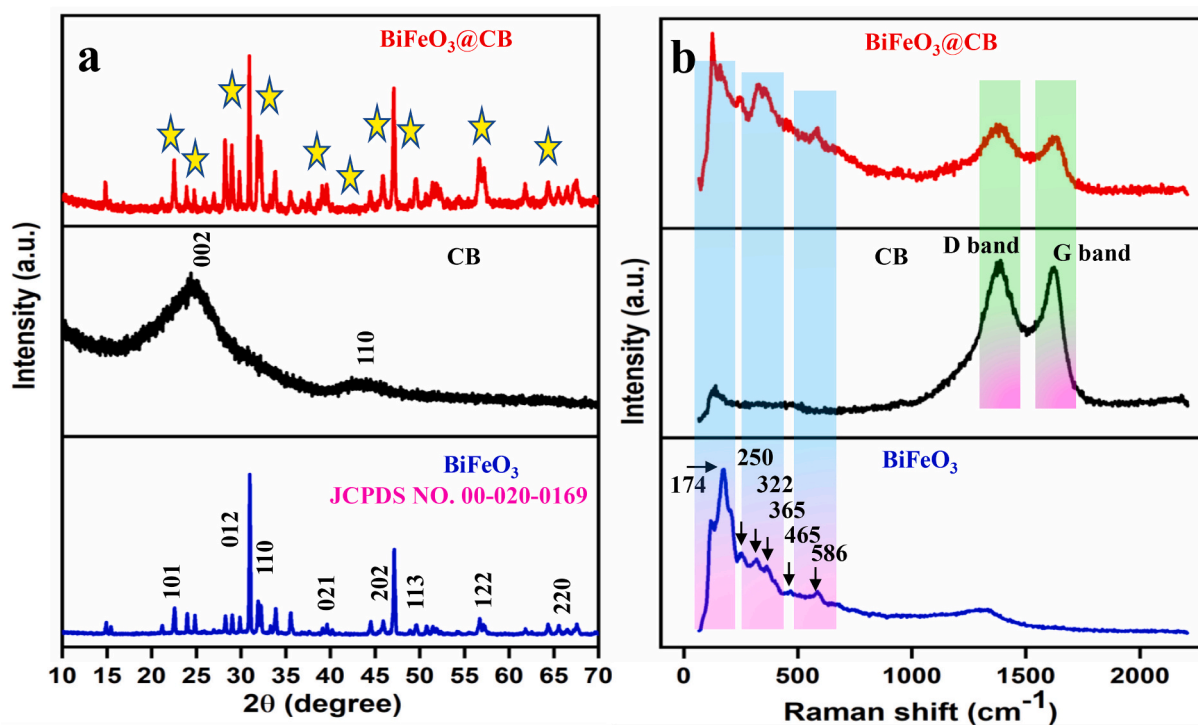


Fig. 3. (a) X-ray diffraction (XRD) patterns and (b) Raman spectra of BiFeO_3 , CB, and the $\text{BiFeO}_3@\text{CB}$ nanocomposite.

corresponds to the A_1 (Bi—O) mode, whereas the bands at 250, 322, 365, 465, and 586 cm^{-1} are assigned to E modes, where the lower-energy

peaks involve Bi—O and Bi—O—Fe vibrations and the higher-energy peaks are mainly associated with Fe—O bending and stretching within

FeO_6 octahedra. CB exhibits two distinct bands: the D band at 1379 cm^{-1} , indicating structural disorder, and the G band at 1623 cm^{-1} , attributed to sp^2 -hybridized graphitic domains. The modifications observed in the nanocomposite suggest that the particle size, lattice strain, and interfacial interactions enhance its potential for electrochemical applications [24–27], [19–22].

The XPS analysis (Fig. 4(a)) confirmed the topological electronic structure and elemental composition of the BiFeO_3/CB nanocomposite, revealing the presence of Bi, Fe, C, and O. In Fig. 4(b), the $\text{Bi } 4f_{5/2}$ peak at 162.93 eV is assigned to Bi^{3+} , indicating oxidized bismuth species.

Fig. 4(c) shows Fe peaks at 715.65 eV ($\text{Fe } 2p_{3/2}$), 729.96 eV ($\text{Fe } 2p_{1/2}$), along with a satellite feature at 718.77 eV , which confirms the presence of Fe^{3+} in iron oxides. The $\text{O } 1s$ spectrum (Fig. 4(d)) displays a peak at 534.9 eV , which is attributed to surface-adsorbed oxygen or hydroxyl groups. As shown in Fig. 4(e), the $\text{C } 1s$ spectrum of CB exhibits a single peak at 288.7 eV , corresponding to COOH or C=O functionalities. These results collectively verify the successful synthesis of BiFeO_3 -based materials containing multiple oxygen species [28–31], [23–26].

3.2. Analysis of electrochemical performance based on electrochemical impedance spectroscopy (EIS) and CV

Fig. 5 presents the comprehensive electrochemical analysis of the bare and modified SPCEs using EIS and CV to investigate their electrocatalytic behaviors. The results of these investigations provide valuable information on the electron transfer kinetics, redox behavior, diffusion processes, and electrochemically active surface areas (ECSAs) of the electrode interfaces. The Nyquist plots in Fig. 5(a), obtained from EIS measurements using 0.1 M KCl and $5\text{ mM } [\text{Fe}(\text{CN})_6]^{3-/4-}$ with a 5 mV amplitude over a frequency range of $0.01\text{--}100\text{ kHz}$, reveal key insights into the interfacial resistance and ion diffusion. The bare SPCE exhibited a high charge transfer resistance (R_{ct}) of $2758\ \Omega$, reflecting limited

electron mobility. In contrast, modification of the electrode surface led to marked improvements: R_{ct} values decreased to $1940\ \Omega$ for $\text{BiFeO}_3/\text{SPCE}$, $906\ \Omega$ for CB/SPCE , and only $382\ \Omega$ for $\text{BiFeO}_3/\text{CB}/\text{SPCE}$, as determined through Randles equivalent circuit fitting.

This substantial reduction, particularly in the composite electrode, indicated enhanced interfacial charge transport facilitated by the synergistic interaction between BiFeO_3 and CB, resulting in better conductivity and electrochemical reactivity. Eq. (1) was used to determine the charge-transfer rate from the obtained R_{ct} values [32], [27].

$$R_{ct} = RT / (n^2 F^2 A K_s C), \quad (1)$$

where F is the Faraday constant, R is the gas constant, n is the number of transferred electrons, A is the electrode area, T is the absolute temperature, and C is the concentration of the redox probe. Fig. 5(b) presents the CV profiles for all electrodes recorded in $5\text{ mM } [\text{Fe}(\text{CN})_6]^{3-/4-}$ with 0.1 M KCl at 50 mV/s , a standard redox probe. The modified electrodes showed significantly greater redox peak currents and reduced peak-to-peak separations than those of the unmodified SPCE, implying faster electron exchange and improved reaction reversibility. Among them, $\text{BiFeO}_3/\text{CB}/\text{SPCE}$ demonstrated the most pronounced redox response with anodic and cathodic peak currents of 97.40 and $-97.05\ \mu\text{A}$, respectively, outperforming $\text{BiFeO}_3/\text{SPCE}$ ($I_{pa} = 68.29\ \mu\text{A}$, $I_{pc} = -71.66\ \mu\text{A}$) and CB/SPCE ($I_{pa} = 74.24\ \mu\text{A}$, $I_{pc} = -88.40\ \mu\text{A}$). This confirms that the composite electrode benefits from the cooperative enhancement effect between the two components. Further insights into the redox mechanism were obtained from scan-rate-dependent CV studies (Fig. 5(c)) conducted on $\text{BiFeO}_3/\text{CB}/\text{SPCE}$ at scan rates ranging from 20 to 300 mV/s . A direct proportionality was observed between the peak current and square root of the scan rate (Fig. 5(d)), with the linear equations $I_{pa} = 0.5115x + 6.1292$ ($R^2 = 0.9976$) and $I_{pc} = -0.4437x - 6.5956$ ($R^2 = 0.9958$) confirming a diffusion-controlled electrochemical

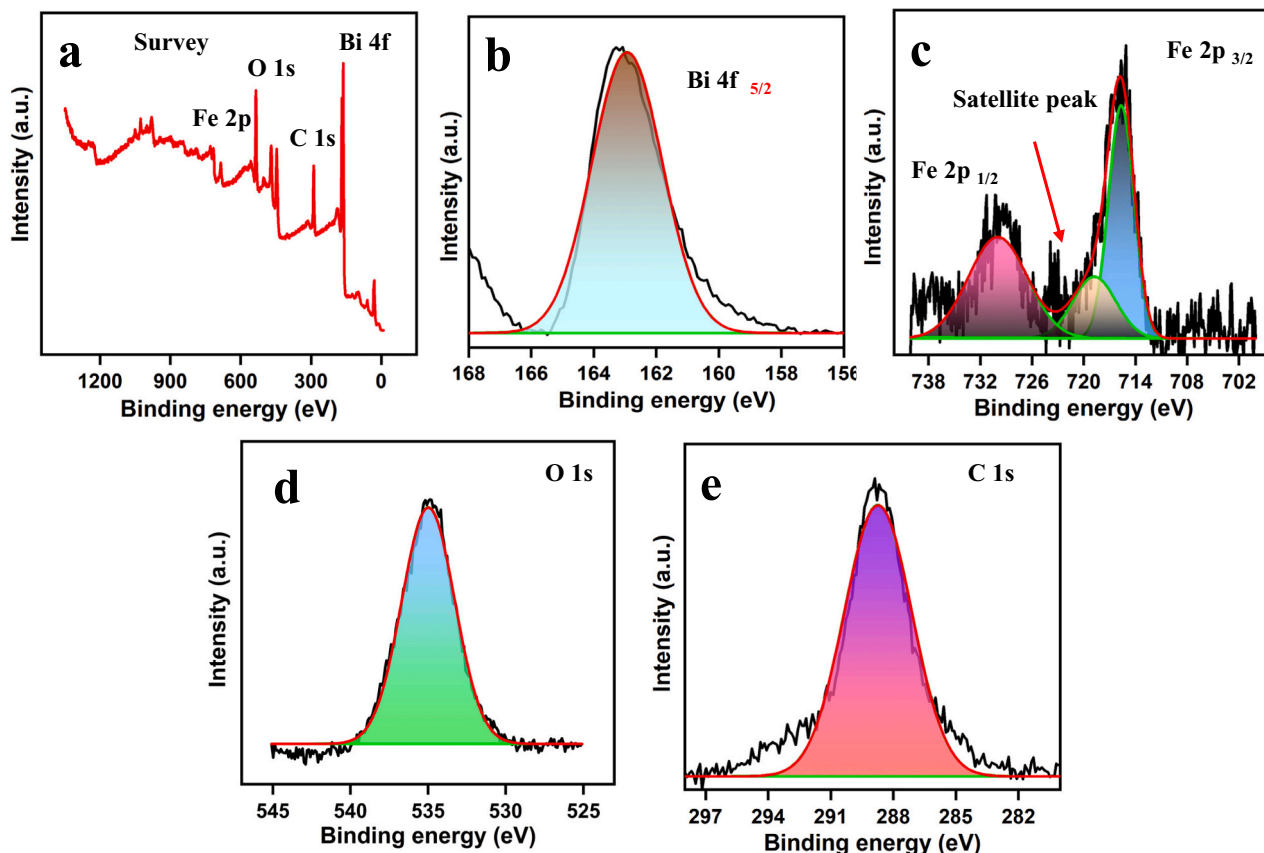


Fig. 4. (a) X-ray photoelectron spectroscopy (XPS) survey spectrum of BiFeO_3/CB ; high-resolution XPS spectra of (b) $\text{Bi } 4f$, (c) $\text{Fe } 2p$, (d) $\text{O } 1s$, and (e) $\text{C } 1s$.

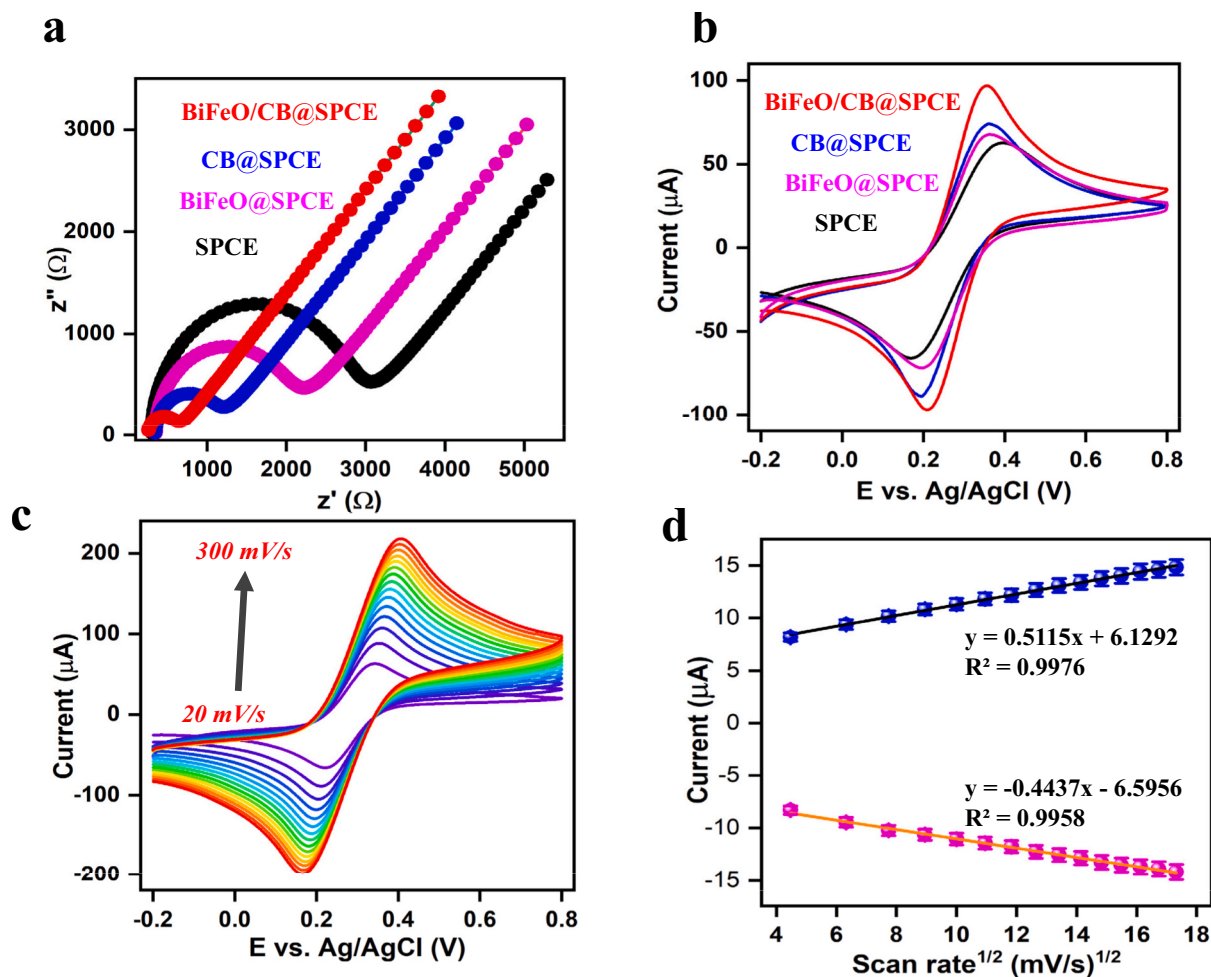


Fig. 5. Electrochemical characterizations: (a) Nyquist plots obtained from electrochemical impedance spectroscopy (EIS) for bare screen-printed carbon electrode (SPCE), BiFeO₃@SPCE, CB@SPCE, and BiFeO₃/CB@SPCE, measured in 0.1 M KCl containing 5 mM [Fe(CN)₆]^{3-/4-} using a 5 mV amplitude over a frequency range of 0.01–100 kHz; (b) cyclic voltammetry (CV) profiles of the same electrodes in 5 mM [Fe(CN)₆]^{3-/4-} with 0.1 M KCl at a scan rate of 50 mV/s; (c) CV curves of BiFeO₃/CB@SPCE recorded at scan rates from 20 to 300 mV/s; and (d) corresponding plot of peak current versus the square root of scan rate, indicating a diffusion-controlled process.

mechanism consistent with the Randles–Sevcik relationship. The ECSA was calculated using the Randles–Sevcik equation (Eq. (2)) [33], [28].

$$I_p = (2.69 \times 10^5) n^{3/2} D^{1/2} V^{1/2} AC, \quad (2)$$

where n is the number of transferred electrons, I_p is the peak current, A the electrode area, D is the diffusion coefficient, C is the concentration, and V is the scan rate. Additionally, the ECSA, calculated using the Randles–Sevcik equation, was found to be 0.158 cm² for bare SPCE, 0.174 cm² for BiFeO₃@SPCE, 0.190 cm² for CB@SPCE, and 0.249 cm² for the BiFeO₃/CB@SPCE composite. The significantly higher ECSA of the composite electrode highlights the greater availability of active sites for redox reactions, supporting its enhanced analytical performance for FZD detection. Altogether, these electrochemical findings highlight the superior characteristics of the BiFeO₃/CB@SPCE sensor, including its reduced charge-transfer resistance, elevated redox current response, diffusion-governed kinetics, and high surface area, collectively enabling efficient and sensitive electrochemical sensing. The improved response arises mainly from the complementary functions of the two components: BiFeO₃ provides electroactive surface sites, whereas CB improves electrical connectivity and increases the accessible electrochemical area. These results support the use of the composite for FZD sensing; however, the analytical contribution of the present study is associated primarily with its implementation on a disposable SPCE and its FZD-specific electrochemical evaluation rather than with the introduction of a new

BiFeO₃/carbon material architecture.

Potential optimization for FZD detection was conducted on BiFeO₃/CB@SPCE using CV at 50 mV/s in 0.05 M phosphate-buffered saline (PBS) with 200 μ M FZD. Initially, different starting potentials (+0.15 to −0.05 V) were tested with a fixed final potential of −0.7 V. The highest reduction current (−45.84 μ A) was observed from +0.05 to −0.7 V, making +0.05 V the optimal starting potential. Then, by varying the final potential (−0.6 to −0.8 V) with +0.05 V fixed, the best response (−45.84 μ A) again occurred at −0.7 V. The optimized potential range was therefore set from +0.05 to −0.7 V. To improve electrochemical sensitivity, systematic optimization of the potential window for FZD detection was conducted on a BiFeO₃/CB-modified SPCE and investigated using CV. The effective detection of FZD depends on the careful selection of potential limits that promote the optimal redox activity at the electrode interface.

As shown in Fig. S3(a), the initial potential was varied from +0.15 to −0.05 V while maintaining the final potential at −0.70 V. Among the evaluated conditions, initiating the potential sweep at +0.05 V produced the highest reduction current of −45.84 μ A. In the second step (Fig. S3(b)), the final potential was varied from −0.60 to −0.80 V while the initial potential was fixed at +0.05 V. The highest current response was obtained at −0.70 V. Therefore, the optimized potential window for FZD detection was established as +0.05 to −0.70 V. This two-step optimization established the ideal potential window as +0.05 to −0.7

V, which consistently generated a maximum reduction current of $-45.84 \mu\text{A}$. This optimized window ensures high electrochemical responsiveness, thereby enabling the sensitive and reliable quantification of FZD using the $\text{BiFeO}_3/\text{CB@SPCE}$ system.

Using the optimized potential window, different electrodes were tested with $200 \mu\text{M}$ FZD at a scan rate of 50 mV/s in 0.05 M PBS (Fig. 6(a)). The bare SPCE showed a cathodic peak current of $-12.59 \mu\text{A}$ at -0.43 V . In contrast, $\text{BiFeO}_3/\text{SPCE}$ showed an increased cathodic current of $-15.71 \mu\text{A}$ at -0.42 V . Furthermore, CB@SPCE showed a higher current of $-30.49 \mu\text{A}$ at -0.36 V due to the synergistic effect and increased surface area. The nanocomposite further enhanced the current to $-42.59 \mu\text{A}$ at -0.38 V , indicating strong synergy between BiFeO_3 and CB. Thus, $\text{BiFeO}_3/\text{CB@SPCE}$ was selected for further study. A bar chart comparing the cathodic current responses of bare SPCE, $\text{BiFeO}_3/\text{SPCE}$, CB@SPCE , and $\text{BiFeO}_3/\text{CB@SPCE}$ is presented in Fig. 6(b).

Fig. 6 provides a comparative illustration of the electrochemical performances of various modified electrodes for FZD detection and the influence of pH on the redox behavior using CV profiles. As shown in Fig. 6(a) the bare SPCE exhibited a cathodic peak current of $-12.59 \mu\text{A}$ at -0.43 V , serving as a reference for evaluating electrode modifications. Upon modification with BiFeO_3 , the cathodic current showed a slight increase to $-15.71 \mu\text{A}$ with SPCE, with a marginal anodic shift to -0.42 V , indicating limited catalytic activity enhancement. A more significant improvement was observed with CB modification, which resulted in a significantly higher current of $-30.49 \mu\text{A}$ at -0.36 V , attributed to the high conductivity and large surface area of CB that facilitate efficient electron transfer and increase the number of electroactive sites. The most pronounced enhancement was achieved with the BiFeO_3/CB composite-modified SPCE, which generated a cathodic peak current of $-42.59 \mu\text{A}$ at -0.38 V . This superior performance reflects the synergistic effect between BiFeO_3 , which potentially contributes to the catalytic and adsorptive properties, and CB, which provides enhanced

electron transport pathways and an increased surface area. The corresponding bar chart in Fig. 6(b) further supports this trend, clearly showing the stepwise improvement in the current response across the different electrodes and confirming the effectiveness of the $\text{BiFeO}_3/\text{CB@SPCE}$ design. The effect of pH on the electrochemical response of FZD was investigated over the pH range of 3.0 – 11.0 . As shown in Fig. 6(c), both the cathodic peak current and peak potential varied with pH. The maximum cathodic current was obtained at pH 7.0 , indicating favorable conditions for the electrochemical reduction of FZD. The cathodic peak potential shifted linearly toward more negative values with increasing pH, following the regression equation $E_{\text{pc}} (\text{V}) = -0.0325\text{pH} - 0.1421$, with $R^2 = 0.9905$, as shown in Fig. 6(e). The corresponding slope was -32.5 mV pH^{-1} , confirming the involvement of protons in the FZD reduction process. According to the Nernst relationship, $dE_{\text{pc}}/d\text{pH} = -0.0591(m/n) \text{ V pH}^{-1}$, where m and n represent the numbers of protons and electrons involved, respectively. The calculated m/n ratio was approximately 0.55 , suggesting an approximate proton-to-electron ratio of $1:2$ in the potential-determining step. However, because the pH-dependent slope provides only the proton-to-electron ratio, the absolute reduction stoichiometry cannot be assigned without an independent determination of the number of transferred electrons [32], [27]. The proposed FZD reduction mechanism is illustrated in Fig. S2.

Fig. 7 presents the results of a detailed assessment of the electrochemical performance of the $\text{BiFeO}_3/\text{CB@SPCE}$ sensor obtained through an examination of its response to varying FZD concentration and an evaluation of its reaction kinetics through a scan rate analysis. In Fig. 7(a), the CV curves illustrate that the cathodic peak current increased progressively as FZD concentration rose from 20 to $200 \mu\text{M}$. This trend confirms the sensitivity of the sensor to FZD and indicates a clear concentration-dependent electrochemical response. The corresponding calibration curve in Fig. 7(b) shows a strong linear relationship between

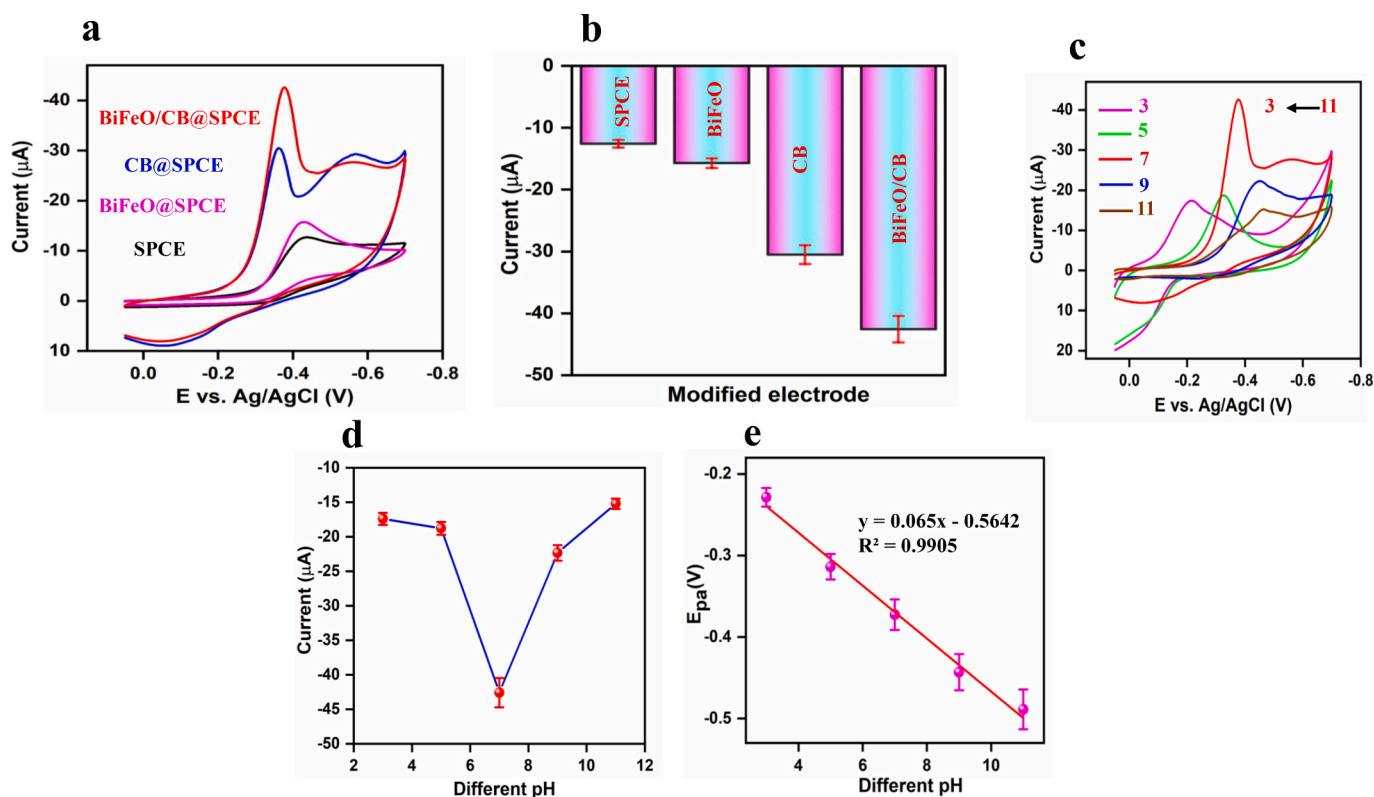


Fig. 6. (a) Comparative cyclic voltammetry (CV) performance of modified electrodes (SPCE, $\text{BiFeO}_3/\text{SPCE}$, CB@SPCE , and $\text{BiFeO}_3/\text{CB@SPCE}$) tested with $200 \mu\text{M}$ furazolidone (FZD) in 0.05 M phosphate-buffered saline (PBS; pH 7.0); (b) bar chart illustrating the current responses of SPCE, $\text{BiFeO}_3/\text{SPCE}$, CB@SPCE , and $\text{BiFeO}_3/\text{CB@SPCE}$; (c) CV analysis across pH values ranging from 3.0 to 11.0 at a scan rate of 50 mV/s ; (d) and (e) linear plots of pH versus current (μA) and pH versus potential (V), respectively.

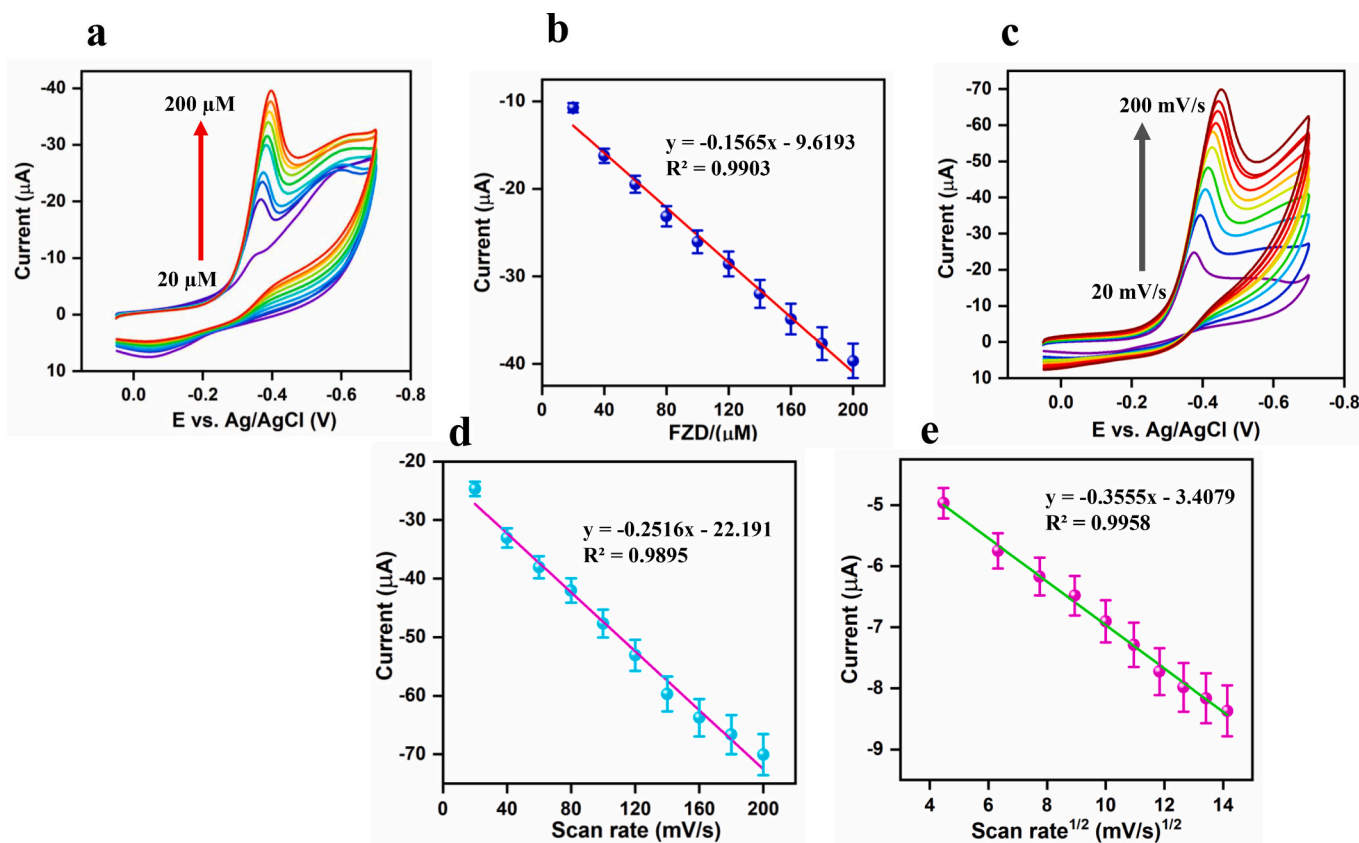


Fig. 7. (a) Cyclic voltammetry (CV) responses of FZD (20–200 μM) at BiFeO₃/CB@SPCE; (b) linear plot of FZD concentration versus reduction current; (c) CV responses recorded at different scan rates in the presence of 200 μM FZD; (d) linear plot of current versus scan rate; and (e) linear plot of current versus the square root of scan rate.

the FZD concentration and peak reduction current, demonstrating the suitability of the sensor for accurate quantitative determination within the tested concentration range. This linearity highlights the reliability and effectiveness of the BiFeO₃/CB@SPCE sensor in detecting FZD. To investigate the reaction mechanism, scan rate-dependent CV measurements were performed. As shown in Fig. 7(c), the reduction current increases with the scan rate, and this is a typical indication of a kinetically favorable redox process. Notably, only slight shifts in the peak potential were observed with increasing scan rates, suggesting rapid electron transfer at the electrode surface. The nature of the electrochemical process was further analyzed by plotting the peak current against both the scan rate (Fig. 7(d)) and square root of the scan rate (Fig. 7(e)). The linear fit of the current versus scan rate yielded a correlation coefficient (R^2) of 0.9895, whereas the current versus square root of the scan rate showed slightly stronger linearity, with $R^2 = 0.9958$. Based on electrochemical principles, a linear relationship between the peak current and scan rate is typically associated with a surface-confined reaction mechanism, whereas a linear dependence on the square root of the scan rate is indicative of diffusion-controlled behavior. Therefore, the higher correlation with the square root of the scan rate suggests that the reduction in FZD at the BiFeO₃/CB@SPCE is primarily controlled by the diffusion of the analyte from the bulk solution to the electrode surface.

Reliability and consistency are essential attributes for any electrochemical sensor intended for quantitative analysis and real-world applications. These aspects were assessed for the BiFeO₃/CB@SPCE sensor, and the results are presented in Fig. S4. Repeatability was evaluated by recording five successive CV measurements using a single BiFeO₃/CB@SPCE in 100 μM FZD ($n = 5$), as shown in Fig. S4(a). The cathodic peak-current magnitudes were 29.95, 29.78, 29.65, 29.31, and 29.04 μA, giving a mean current of 29.55 ± 0.37 μA and an RSD of 1.24%,

which confirms that the sensor delivers a stable performance under identical experimental conditions. Reproducibility was evaluated using five independently fabricated BiFeO₃/CB@SPCEs under the same experimental conditions ($n = 5$), as shown in Fig. S4(b). The cathodic peak-current magnitudes were 28.97, 28.97, 28.90, 28.83, and 28.76 μA, corresponding to a mean current of 28.89 ± 0.09 μA and an RSD of 0.32%. These low RSD values demonstrate the good repeatability of the electrochemical measurements and the reproducibility of the electrode-fabrication procedure, demonstrating that the fabrication method produces reliable and uniform electrodes with minimal batch-to-batch variability, which is an important criterion for scalable production and practical deployment. Cycling stability was evaluated using a single BiFeO₃/CB@SPCE ($n = 1$) in 50 μM FZD prepared in 0.05 M PBS (pH 7.0). One hundred consecutive CV cycles were recorded over the potential range of +0.05 to −0.70 V at a scan rate of 50 mV/s. After 100 cycles, the electrode retained 95% of its initial cathodic peak-current response, as shown in Fig. S4(c). This result indicates stable performance during repeated cycling for the tested electrode. Because only one electrode was evaluated, mean $\pm$ standard deviation and error bars were not calculated, indicating a strong resistance to electrochemical degradation or fouling and confirming its long-term operational durability.

3.3. DPV measurements

To evaluate the performance of the BiFeO₃ sensor in detecting FZD, DPV was conducted in 0.05 M PBS (pH 7.0) across different concentrations of FZD ranging from 0.01 to 130 μM. As the FZD concentration increased, a proportional rise in the cathodic current was observed, reaching a peak of −3.43 μA at 130 μM (Fig. 8(a)), indicating an improved reduction efficiency. The sensor gave a linear response over the concentration range of 0.01–130 μM (Fig. 8(b)), suggesting a

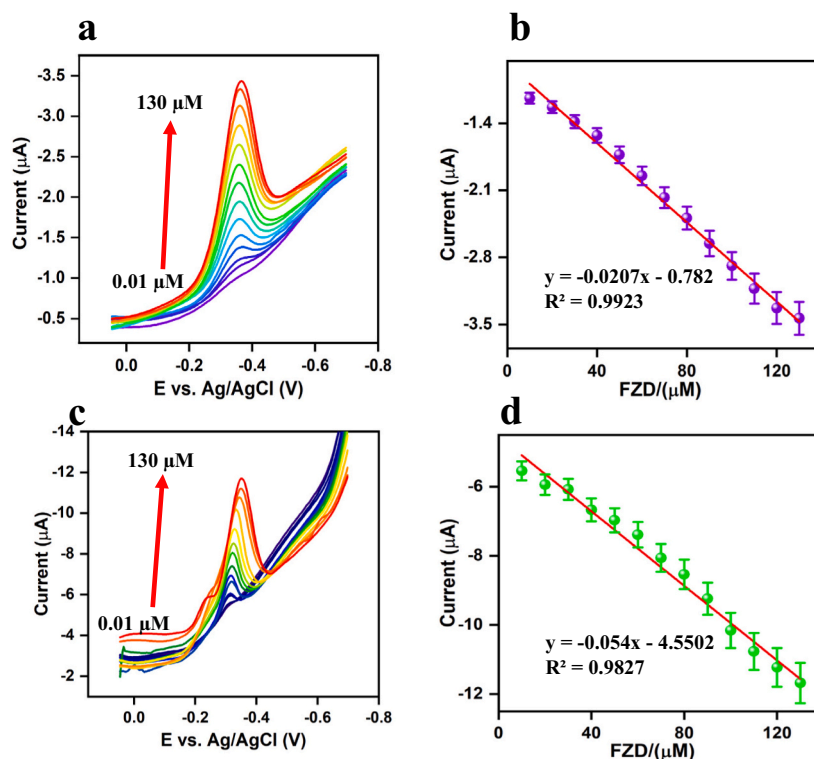


Fig. 8. (a) Differential pulse voltammetry (DPV) responses of FZD at BiFeO₃@SPCE; (b) linear plot showing FZD concentration versus cathodic current; (c) DPV responses of FZD at CB@SPCE; and (d) corresponding linear plot of FZD concentration versus cathodic current.

diffusion-controlled process. The calibration curve yielded a linear regression equation of $y = -0.0207x - 0.782$ with a correlation coefficient (R^2) of 0.9923. The slope (-0.0207) was used to calculate the LOD for the BiFeO₃-based FZD sensor [34].

The standard deviation (σ) and calibration slope (S) were used to calculate the detection limit ($LOD = 3\sigma/S$), yielding $0.045 \mu\text{M}$. The sensitivity was $1.0 \mu\text{A}/\mu\text{M}/\text{cm}^2$, obtained from the slope normalized to the electrode's active area. Table 1 compares the analytical performance of the BiFeO₃/CB@SPCE with previously reported methods for FZD determination. The performance of the CB sensor for FZD detection was assessed using DPV in 0.05 M PBS (pH 7.0) over a concentration range of 0.01–130 μM . An increase in FZD concentration led to a rise in the

cathodic current, reaching $-11.68 \mu\text{A}$ at 130 μM (Fig. 8(c)), indicating enhanced reduction activity. A linear response was observed within the tested range (Fig. 8(d)), confirming a diffusion-controlled process. The calibration plot showed a linear regression equation of $y = -0.054x - 4.5502$ with an R^2 value of 0.9827. A slope (0.054) was used to determine the LOD of the CB-based FZD sensor.

As shown in Table 1, the BiFeO₃/CB@SPCE provided an LOD of $0.045 \mu\text{M}$, which was lower than those reported for NiFe₂O₄/rGO ($0.050 \mu\text{M}$), S,N-GQDs/MMWCNTs ($2.1 \mu\text{M}$), Gr/Au/GCE ($0.064 \mu\text{M}$), MWCNTs/GCE ($0.0832 \mu\text{M}$), and TiO₂/rGO/CPE (0.43 and $0.55 \mu\text{M}$). Although the Gr/Au/GCE and MWCNTs/GCE platforms exhibited wider upper concentration limits, the present sensor combined a low detection limit, a linear range of 0.01 – $130 \mu\text{M}$, and a disposable screen-printed electrode configuration.

The present system should also be considered in relation to previously reported BiFeO₃/carbon-based electrochemical platforms. Those studies established the usefulness of combining BiFeO₃ with conductive carbon phases for electrochemical sensing. Accordingly, the material-level concept used here is related to the earlier reports. The specific advancement of the present work is its evaluation for the cathodic determination of the nitrofurantoin drug FZD using a disposable SPCE, together with FZD-specific potential-window and pH optimization, diffusion-behavior analysis, interference evaluation, and application to four real-sample matrices.

BiFeO₃/CB@SPCE sensor's performance toward FZD detection was investigated using DPV in 0.05 M PBS (pH 7.0) across concentrations ranging from 0.01 to $130 \mu\text{M}$, and the results are shown in Fig. 9. As the FZD concentration increased, the cathodic current rose steadily, reaching a maximum of $-13.24 \mu\text{A}$ at $130 \mu\text{M}$ (Fig. 9(a)), demonstrating efficient reduction capability.

The corresponding calibration plot (Fig. 9(b)) revealed a linear relationship with the regression equation $y = -0.0696x - 3.9072$ and a correlation coefficient (R^2) of 0.9943, indicating a diffusion-controlled process. Based on the slope (-0.0696), the LOD of the sensor was calculated. To compare the individual contributions of the components,

Table 1

Comparison of different electrochemical sensors for FZD detection.

Electrodes	LOD (μM)	Linear range (μM)	Methods	References
^a NiFe ₂ O ₄ /rGO	0.05	0.1–10.0; 10.0–150.0	ⁱ DPV	[35]
^b S, N-GQDs/ MMWCNTs	2.1	8.0–250	ⁱⁱ FES	[36]
^c Gr/Au/GCE	0.064	1–674	ⁱⁱⁱ AMP	[37]
^d MWCNTs/GCE	0.0832	0.49–590	ⁱ DPV	[38]
^e TiO ₂ /rGO/CPE	0.55 and 0.43	1–150	ⁱ DPV	[39]
^f BiFeO ₃ /CB@SPCE	0.045	0.01–130	ⁱ DPV	This work

^a Nickel–ferrite oxide nanoparticles decorated with reduced graphene oxide.

^b Sulfur- and nitrogen-doped graphene quantum dots/multi-walled carbon nanotube.

^c Graphene/gold/glassy carbon electrode.

^d Multi-walled carbon nanotube/glassy carbon electrode.

^e Titanium dioxide/reduced graphene oxide/carbon paste electrode.

^f Bismuth ferrite/Carbon black@Screen-printed carbon electrode.

ⁱ Differential pulse voltammetry.

ⁱⁱ Fluorescence.

ⁱⁱⁱ Amperometric.

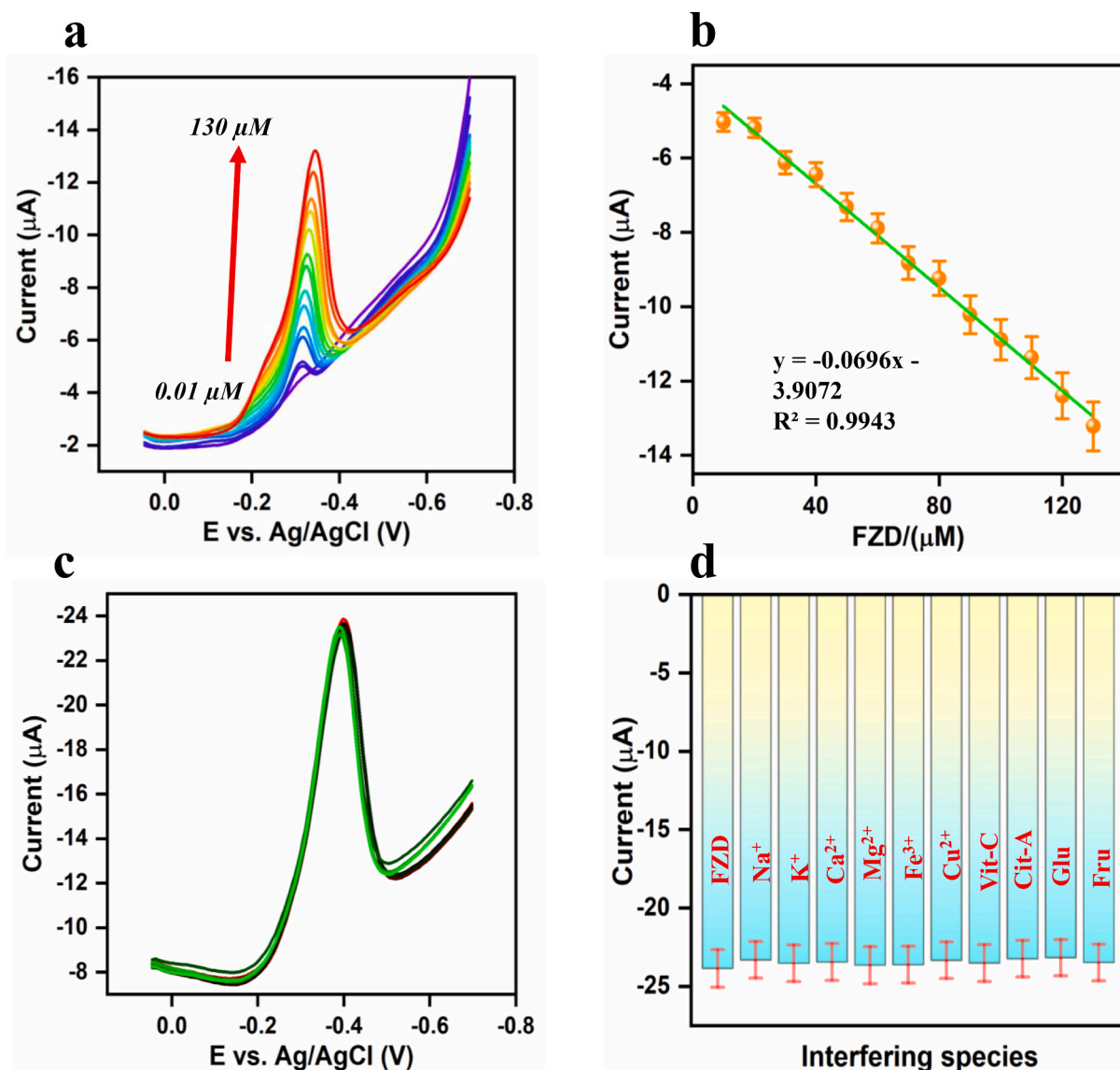


Fig. 9. (a) DPV responses of FZD at BiFeO₃/CB@SPCE; (b) linear plot showing FZD concentration versus cathodic current; (c) DPV analysis assessing the effect of potential interfering species (Na⁺, K⁺, Ca²⁺, Mg²⁺, Fe³⁺, Cu²⁺, ascorbic acid (Vit-C), citric acid (Cit-A), glucose (Glu), and fructose (Fru)) in the presence of 200 μM FZD; and (d) interference bar diagram.

the DPV responses of the BiFeO₃@SPCE, CB@SPCE, and BiFeO₃/CB@SPCE composites were analyzed. BiFeO₃ alone produced a relatively weak cathodic current, reflecting its limited electrocatalytic activity. The CB exhibited a stronger response owing to its superior electron-transfer properties. Notably, the composite electrode displayed the highest current response of −13.24 μA at 130 μM FZD, confirming the synergistic effect between BiFeO₃ and CB. This enhancement is attributed to the improved conductivity, larger electroactive surface area, and additional active sites available for FZD reduction. The selectivity of the BiFeO₃/CB@SPCE sensor was evaluated by recording the DPV response of 200 μM FZD in the presence of equimolar concentrations of Na⁺, K⁺, Ca²⁺, Mg²⁺, Fe³⁺, Cu²⁺, ascorbic acid, citric acid, glucose, and fructose. The relative current change was calculated using $\Delta I (\%) = [(I_{FZD+interferent} - I_{FZD}) / I_{FZD}] \times 100$, where I_{FZD} and $I_{FZD} + \text{interferent}$ represent the cathodic peak currents obtained for FZD alone and FZD in the presence of each interferent, respectively. The current changes observed for Na⁺, K⁺, Ca²⁺, Mg²⁺, Fe³⁺, Cu²⁺, ascorbic acid, citric acid, glucose, and fructose were −2.31%, −1.38%, −1.72%, −0.88%, −1.01%, −2.18%, −1.47%, −2.60%, −2.89%, and −1.59%, respectively. The maximum absolute current change was

2.89% in the presence of glucose, indicating that the tested species caused only limited interference with the FZD response under the investigated conditions. Each condition was measured once ($n = 1$).

3.4. Real sample analysis

Diluted river water, tap water, urine, and pharmaceutical tablets were used as real samples for the evaluation of detection capability of the BiFeO₃/CB@SPCE sensor. For water and urine samples, 5 mL of the collected sample was mixed with 5 mL of deionized DMSO. As FZD is not naturally present in these samples, 10 mg of FZD spiking was done with each sample. Commercial FZD tablets were sourced from a local pharmacy in Taipei, Taiwan, and 10 mg of the finely ground FZD tablets were dissolved in 10 mL DMSO for analysis. All the prepared samples were ultrasonicated for 30 min to ensure homogeneity. To accurately quantify FZD in complex matrices, DPV was performed using the standard addition method under optimized conditions for each sample. The results demonstrated that the sensor provides good recovery values, confirming its reliability and accuracy, as shown in Table S1.

Fig. 10 shows the DPV-based analysis of FZD in different real-world

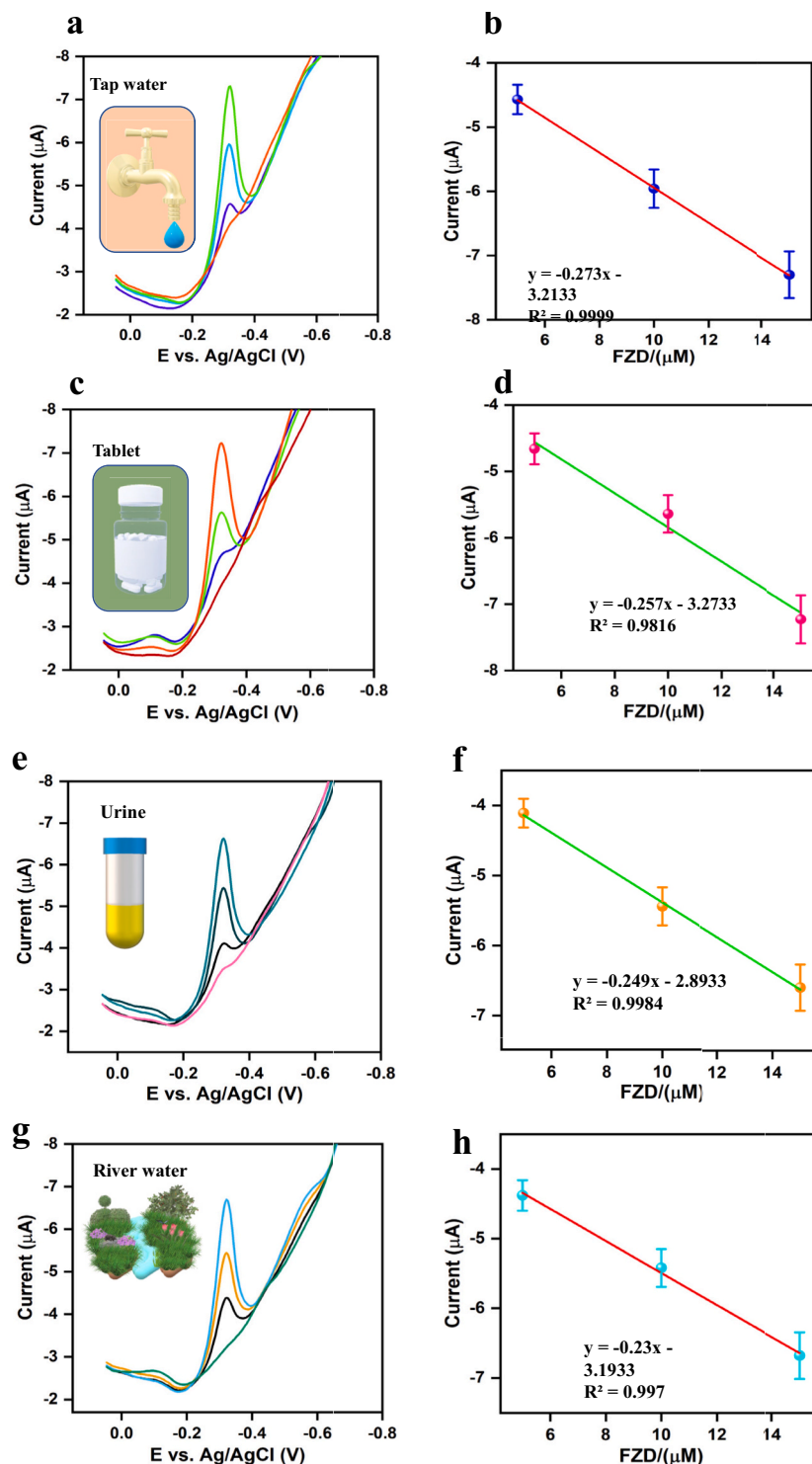


Fig. 10. A) Differential pulse voltammetry (DPV) responses recorded for FZD detection in standard solutions and real samples: Tap water and pharmaceutical tablet. B) Urine and River water over a concentration range of 5–15 μ M. All measurements were performed in 0.05 M phosphate-buffered saline (PBS) at pH 7.0.

samples. The sensor was tested using tap water, dissolved pharmaceutical tablets, urine, and river water samples. For all these samples, measurements were conducted within the concentration range of 5–15 μ M using 0.05 M PBS (pH 7.0).

4. Conclusion

A BiFeO₃/CB-modified SPCE was evaluated for the electrochemical determination of FZD. The composite electrode exhibited lower charge-

transfer resistance and a larger electrochemically active surface area than the individual BiFeO₃- and CB-modified electrodes. Under the optimized conditions, the sensor provided a linear response from 0.01 to 130 μ M with an LOD of 0.045 μ M and satisfactory performance in tap water, river water, urine, and pharmaceutical tablet samples. Although BiFeO₃/carbon composites have previously been reported for electrochemical sensing, the contribution of the present study lies in the application of this platform to FZD-specific cathodic detection using a disposable SPCE, together with systematic optimization and real-sample

validation. This study summarizes the systematic analysis of modified coprecipitation-hydrothermal synthesis of BiFeO₃-CB nanocomposites as electrode materials for the ultrasensitive electrochemical detection of FZD. The nanocomposite performed well, showing high linearity, good reproducibility, and significant stability overall during the investigations. Tap water, river water, human urine, and pharmaceutical tablet samples were used to evaluate the practical applicability of the BiFeO₃/CB@SPCE sensor. The sensor performed well overall in the laboratory and in real sample analyses, in relation to the synergistic effect between the excellent surface area and electrochemical properties of BiFeO₃. The stability, low LOD of 0.045 μ M, excellent sensitivity, low interference from other compounds, and wide linear range of 0.01–130 μ M makes the BiFeO₃-CB nanocomposite a potent candidate for use in the detection of environmental contaminants such as FZD.

Declaration of generative AI and AI-assisted technologies in the manuscript preparation process

There are no generative AI and AI assisted Technologies in the manuscript preparation.

CRediT authorship contribution statement

Pandiyan Parkavi: Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Methodology, Formal analysis, Data curation, Conceptualization. **Saravana Pandi Sivaji:** Writing – review & editing, Writing – original draft, Visualization, Validation, Software, Investigation, Formal analysis, Data curation. **Shen-Ming Chen:** Writing – review & editing, Writing – original draft, Supervision, Project administration, Investigation, Funding acquisition. **Balaji Ramachandran:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis. **Karthik Kiran Sarigamala:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis. **Narendhar Chandrasekar:** Writing – review & editing, Writing – original draft, Validation, Project administration, Methodology, Formal analysis. **Mohammed F. Albeshr:** Writing – review & editing, Funding acquisition. **Vyoma Singh:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis. **Muthusamy Govarthan:** Writing – review & editing, Writing – original draft, Methodology. **Subha Veeramani:** Writing – review & editing, Writing – original draft, Formal analysis. **Chung-Yu Guan:** Methodology, Formal analysis. **Moon Soo Kim:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis. **Michael Taeyoung Hwang:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.microc.2026.119640>.

Data availability

Data will be made available on request.

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