RESEARCH PAPER

Mxene-Supported Iron Phthalocyanine Nanocomposites for the High-Sensitivity Electrochemical Detection of Chloramphenicol

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ABSTRACT

The development of efficient electrochemical sensors for food safety monitoring remains a critical challenge. In this study, we report a high-performance electrode modified with a nanocomposite consisting of $Ti_3C_2T_x$ MXene and Iron Phthalocyanine (FeTCSA). The morphology and elemental composition were characterized via Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX), confirming the successful integration of FeTCSA onto the MXene sheets. UV-Vis spectroscopy highlighted the characteristic Q-band and Soret-band transitions of the FeTCSA complex within the composite. The Mx/FeTCSA modified Glassy Carbon Electrode (GCE) exhibited superior electrocatalytic activity toward the reduction of Chloramphenicol (CAP) compared to a bare GCE. Kinetic studies revealed a diffusion-controlled process with enhanced current responses across varying concentrations (10 µM to 50 µM) and scan rates (10 to 250 mV/s). These results demonstrate that the Mx/FeTCSA composite is a promising candidate for the development of robust, low-cost chemical sensors.

Keywords: MXene-Supported Iron Phthalocyanine Nanocomposites, Electrochemical Detection, Chloramphenicol.

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Introduction

Chloramphenicol (CAP) is a broad-spectrum antibiotic widely used in veterinary medicine, but its residues in food products pose significant health risks, including bone marrow suppression and aplastic anemia. Consequently, there is an urgent need for rapid and sensitive detection methods. Electrochemical sensors offer a cost-effective and portable solution.(Wang et al. 2021)

MXenes, a class of two-dimensional (2D) transition metal carbides, have emerged as excellent electrode materials due to their high metallic conductivity, hydrophilicity, and large surface area. Iron Phthalocyanine (FeTCSA) is a well-known macrocyclic complex with remarkable electrocatalytic properties. By combining the conductive network of MXene with the catalytic centers of FeTCSA, we aim to create a synergistic effect that enhances the sensitivity and selectivity of CAP detection.(Wang et al. 2021; Chen et al. 2024)

Materials and Methods

PREPARATION OF MXENE/IRON PHTHALOCYANINE FOR THE DETECTION OF CHLORAMPHENICOL

Preparation of MXene (Ti-C-T_x) dispersion:

- Synthesis of MXene powders: The MXene was formulated by the self-propagating high-temperature route under an N2 environment.
- Powder forms of Ti, Al, and C were (precursors) mixed at a mass ratio of 3:1:2 and grind by ball-milling for 12 h at room temperature. Then, the obtained powder was heated to 1100 °C for 2 h under an N2 atmosphere in a tubular furnace at a ramping rate of 5 °C min^{-1}.

- After cooling down to RT, the black color powder was collected and treated with 40% HF under constant magnetic stirring (1000 rpm) for 24 h.
- Finally, HF-treated material was centrifuged and washed several times with distilled water (dH2O) to neutralize its pH and dried at 70 °C using a hot air oven to get MXene powder
- Subsequently, the obtained Mxene powder was bathed in distilled water for 30 minutes, after that without any further synthesis the Mxene solution was used for the experiment as it is.(Zhang et al. 2026)

Preparation of Mx/FeTCTSA dispersion:

- 1ml of Mxene solution was mixed with 1ml of compound solution (5mg FeTCSA in 10 ml DD H_2O)
- The Mx/FeTCTSA dispersion was bath sonicated for 10 minutes to make them uniformly dispersive and centrifuged at 6000 rpm for 30 minutes.
- The Mx/FeTCTSA dispersion supernatant solution was used for the experiment as it is without any further dilution
- 8µL-10µL of the solution was taken and coated on the GCE glassy surfaces through Drop Casting Method, the GCE was kept in the hot air oven for about 10 minutes to dry the solution
- After 10 minutes the GCE was taken out from the hot air oven and placed outside for a minute or two to make it come to room temperature.
- The dried GCE was then dipped in double distilled water for one minute to remove all the unwanted particles from the glassy surface and dried again to remove all the moisture from its surface.
- The modified GCE was used to perform all the electrochemical studies
- The analyte used for the detection was CHLORAMPHENICOL (CRPL).(Zhang et al. 2026; Chen et al. 2021)

ELECTROCHEMICAL ANALYSIS:

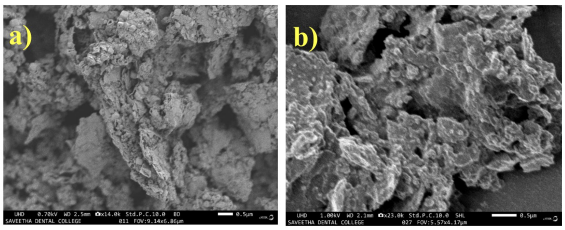
1. **Comparison of Bare GCE and modified GCE in 0.1M PBS:**

- To check the efficiency of the modified electrode it was tested bare under a nitrogen atmosphere. The red CV is the picture of Bare GCE and the blue CV is the picture of modified GCE.
- We can see the difference in the background current as the modified GCE current is more compared to the Bare GCE(Zhang et al. 2026; Chen et al. 2021; Rasheed and Verma 2024)

Results and Discussion

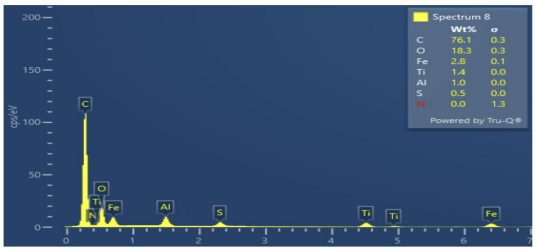
Structural and Morphological Characterization

The surface morphology and microstructure of the synthesized Mx/FeTCSA composite were first investigated using Scanning Electron Microscopy (SEM).(Lai et al. 2023)



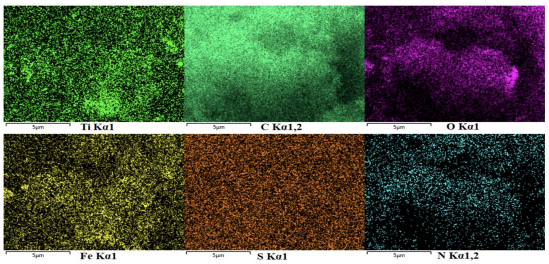
As illustrated in Figure 1, the SEM analysis reveals the typical laminated, flake-like morphology characteristic of Ti_3C_2T_x MXene sheets. Following the self-assembly process, the FeTCSA particles are seen to be uniformly distributed across the MXene layers, which successfully prevents the restacking of the sheets and consequently increases the electroactive surface area.

To confirm the presence and chemical distribution of the constituent elements, Energy Dispersive X-ray Spectroscopy (EDX) was performed on the composite.



The EDX spectrum (Figure 2) clearly confirms the elemental presence of Iron (Fe, 2.8 wt%), Sulfur (S, 0.5 wt%), and Nitrogen (N), which are the signature elements of the FeTCSA complex, verifying its successful incorporation. The high Carbon (76.1 wt%) and Titanium (1.4 wt%) content originate primarily from the MXene backbone.

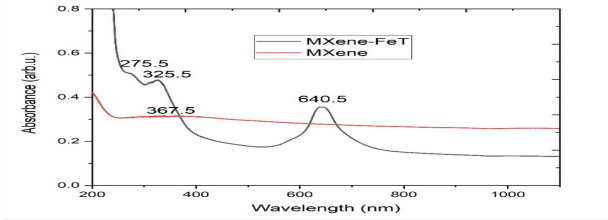
The spatial distribution of these elements was further visualized using elemental mapping.



The elemental mapping results (Figure 3) demonstrate a highly homogeneous distribution of all key elements (Ti,

C, O, Fe, S, N) across the sample, strongly suggesting a successful and uniform hybrid formation at the nanoscale.

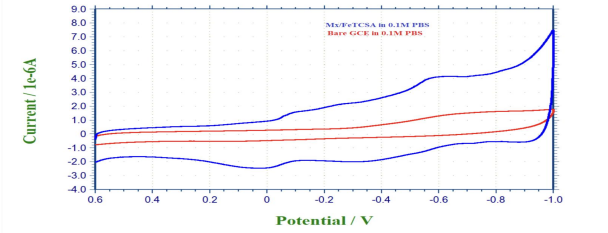
Finally, UV-Vis spectroscopy was employed to provide further evidence of the composite formation and the preserved electronic structure of the phthalocyanine ring.



The UV-Vis spectra (Figure 4) further validate the composite formation. The Mx/FeTCSA spectrum shows a distinct and sharp Q-band at 640.5 nm and characteristic Soret-bands at 275.5 nm and 325.5 nm, which are indicative of the phthalocyanine ring transitions. In contrast, the bare MXene shows a relatively featureless spectrum in the visible range, confirming that the optical signatures observed in the composite arise from the Fe-phthalocyanine.(Zheng et al. 2024)

Electrochemical Performance in PBS

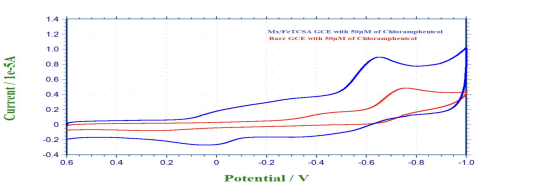
The fundamental electrochemical behavior of the modified electrode was first evaluated in the background electrolyte, 0.1 M Phosphate Buffered Saline (PBS).



As shown in Figure 5, the Mx/FeTCSA modified electrode (blue curve) exhibits a significantly higher background capacitive current compared to the bare Glassy Carbon Electrode (GCE) (red curve). This increase in capacitive current is directly attributed to the larger electroactive surface area provided by the open 2D structure of the non-restacked MXene sheets.(Zheng et al. 2024; Zhang et al. 2023)

Electrocatalytic Detection of Chloramphenicol

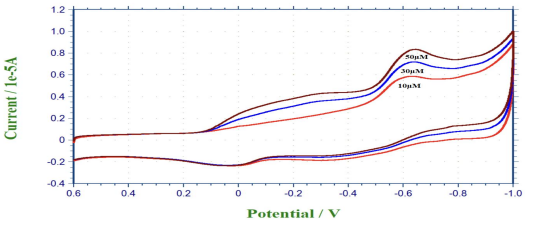
The potential of the Mx/FeTCSA modified electrode for chemical sensing was subsequently tested towards the reduction of Chloramphenicol (CAP).



As illustrated in Figure 6, upon the addition of 50 \mu M Chloramphenicol, the Mx/FeTCSA electrode exhibits a sharp and well-defined reduction peak at approximately -0.65 V. In marked contrast, the bare GCE shows a much lower and broader response. This substantial enhancement highlights the strong electrocatalytic effect of the Fe centers within the phthalocyanine complex, which facilitates the crucial 4e^-/4H^+ reduction of the nitro group in CAP to hydroxylamine, thus significantly improving sensitivity.

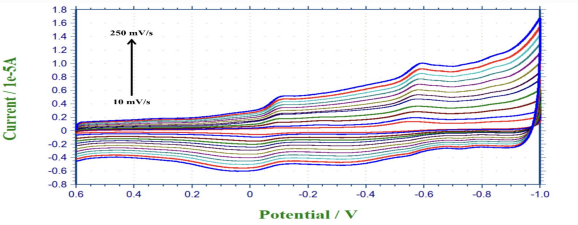
Concentration and Scan Rate Kinetics

The sensitivity of the developed sensor was systematically tested with varying CAP concentrations.



The cathodic peak current (I_{pc}) shows a clear and linear increase as the concentration of Chloramphenicol rises from 10 \mu M to 50 \mu M (Figure 7), demonstrating the feasibility of the sensor for quantitative analytical applications.

Furthermore, the effect of varying the scan rate (10 to 250 mV/s) on the electrochemical response was investigated to understand the reaction kinetics.



As shown in Figure 8, the peak current increases linearly with the square root of the scan rate (\nu^{1/2}). This behavior suggests that the electrochemical reduction of Chloramphenicol at the Mx/FeTCSA modified GCE is a diffusion-controlled process, which is ideal for ensuring reproducible and predictable sensor performance.

Conclusion

In summary, we have successfully fabricated a Mx/FeTCSA nanocomposite for the sensitive electrochemical detection of Chloramphenicol. (Gao et al. 2026)Comprehensive characterization confirms the successful synthesis of the composite, with the FeTCSA complex uniformly distributed across non-restacked MXene layers, preserving the open 2D architecture and large surface area. (Gao et al. 2026; K J et al. 2024)The synergistic combination of MXene's high conductivity and FeTCSA's specific electrocatalytic activity towards nitro group reduction (Gao et al. 2026; K J et al. 2024; Geng et al. 2024)resulted in a sensor with significantly improved current responses and excellent sensitivity across tested concentrations. Kinetic studies revealed a stable, diffusion-controlled reduction process. This study demonstrates a robust and promising platform for the future development of 2D-material-based sensors for applications in food safety and environmental monitoring.(Geng et al. 2023)

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