



Napa Soil-Derived NiAl_2O_4 /MWCNT-Modified Glassy Carbon Electrode for Cd^{2+} Detection

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Article History

Received: 14-05-2026

Revised: 26-06-2026

Published: 30-06-2026

Keywords: NiAl_2O_4 ; Napa Soil; Multi-Walled Carbon Nanotubes; Glassy Carbon Electrode; Cadmium Ion.

Abstract

Cadmium ions (Cd^{2+}) are toxic environmental pollutants that require sensitive and reliable detection methods. This study aimed to develop a NiAl_2O_4 /MWCNT-modified glassy carbon electrode (GCE) using NiAl_2O_4 synthesized from napa soil as a sustainable local resource for electrochemical Cd^{2+} detection. The electrode was fabricated through a drop-casting method and characterized using cyclic voltammetry. The electrochemical performance of the modified electrode was evaluated for Cd^{2+} detection, including the effects of supporting electrolytes, stability, and analytical performance. The results showed that the incorporation of NiAl_2O_4 and multi-walled carbon nanotubes significantly enhanced the electrochemical response and electron-transfer kinetics compared with the bare GCE. Among the tested electrolytes, 0.1 M KCl provided the highest current response. The sensor exhibited a linear response toward Cd^{2+} concentrations in the range of 1–4 mM with a regression equation of $y = 14.332x + 39.895$ and a correlation coefficient (R^2) of 0.9941. The limit of detection (LOD) and limit of quantification (LOQ) were calculated as 0.2988 mM and 0.9961 mM, respectively. In addition, the electrode demonstrated good stability during repeated measurements. These findings indicate that the napa soil-derived NiAl_2O_4 /MWCNT-modified GCE is a promising, low-cost, and environmentally sustainable platform for Cd^{2+} monitoring.

How to Cite: Ayu, S. D., Mawardi, M., Sanjaya, H., Ningsih, S. K. W., & Suryani, O. (2026). Napa Soil-Derived NiAl_2O_4 /MWCNT-Modified Glassy Carbon Electrode for Cd^{2+} Detection. *Hydrogen: Jurnal Kependidikan Kimia*, 14(3), 472-478. <https://doi.org/10.33394/hjkk.v14i3.21268>



<https://doi.org/10.33394/hjkk.v14i3.21268>

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INTRODUCTION

The increasing intensity of industrialization, mining activities, electronic waste disposal, and chemical usage has significantly contributed to heavy metal pollution worldwide. Among various heavy metals, cadmium ions (Cd^{2+}) are of particular concern because of their high toxicity, persistence, and bioaccumulation potential. Long-term exposure to Cd^{2+} can cause kidney and liver damage, neurological disorders, and carcinogenic effects even at low concentrations (Qu & Zheng, 2024). Therefore, the development of sensitive, rapid, and cost-effective analytical methods for Cd^{2+} detection is essential for environmental monitoring and public health protection.

Compared with conventional techniques such

as Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS), electrochemical sensors offer advantages including high sensitivity, simple operation, rapid response, and lower analytical costs (Gibi et al., 2023). Recent advances in electrochemical sensing have highlighted nanomaterials as effective modifiers for improving sensor performance. MWCNTs offer high electrical conductivity and large surface area, while NiAl_2O_4 provides good electrocatalytic activity and stability. Their combination can enhance electron-transfer kinetics and increase the electroactive surface area, resulting in improved sensor sensitivity (Kanoun et al., 2021).

Alumina (Al_2O_3), an important precursor for NiAl_2O_4 synthesis, is commonly obtained from commercial chemicals or mineral ores. However, naturally occurring aluminosilicate minerals provide an alternative and more sustainable source of alumina. Napa soil, a local mineral resource from West Sumatra, Indonesia, contains abundant aluminosilicate compounds and has been successfully utilized as a source of alumina through acid-leaching processes (Frissherly & Mawardi, 2022). Previous studies also demonstrated that optimization of sulfuric acid concentration can improve alumina recovery and purity from napa soil (Ilmi & Mawardi, 2023). These findings indicate that napa soil has significant potential as a low-cost and environmentally friendly precursor for advanced material synthesis.

Several studies have reported the successful application of nanocomposite-based electrochemical sensors for heavy metal detection. Grabarczyk et al. (2023) demonstrated that carbon nanotubes significantly enhanced Cd^{2+} sensing performance, while Fathi et al. (2023) reported improved electron-transfer kinetics using MWCNT-modified electrodes. Furthermore, Hammami et al. (2024) developed a highly sensitive NiAl_2O_4 -based electrochemical sensor for ascorbic acid detection. Despite these advances, most studies have employed synthetic or commercially available alumina sources. Reports concerning the use of napa soil-derived alumina for NiAl_2O_4 synthesis and its application in NiAl_2O_4 /MWCNT-based electrochemical sensors for Cd^{2+} detection remain limited.

Therefore, a clear research gap exists in the development of environmentally sustainable electrochemical sensors utilizing locally sourced napa soil-derived NiAl_2O_4 . The novelty of this work lies in the synthesis of NiAl_2O_4 from napa soil and its integration with MWCNTs for the modification of a Glassy Carbon Electrode (GCE). This approach combines the electrocatalytic properties of NiAl_2O_4 with the excellent conductivity of MWCNTs while promoting the utilization of local natural resources. The resulting composite is expected to provide enhanced electrochemical performance for Cd^{2+} detection through improved electron-transfer efficiency and increased electroactive surface area.

Based on these considerations, this study aims to synthesize napa soil-derived NiAl_2O_4 , fabricate

a NiAl_2O_4 /MWCNT-modified Glassy Carbon Electrode, and evaluate its electrochemical performance toward Cd^{2+} detection using voltammetric techniques. The findings are expected to contribute to the development of sensitive, economical, and environmentally sustainable electrochemical sensors for heavy metal monitoring.

METHOD

Research Design

A laboratory-based experimental method with a quantitative research design was employed in this study. The work focused on the synthesis of napa clay-derived NiAl_2O_4 , the fabrication of a NiAl_2O_4 /MWCNT-modified Glassy Carbon Electrode (GCE), and the assessment of its electrochemical sensing capability toward Cd^{2+} ion determination.

Instruments and Materials

Electrochemical experiments were performed using a potentiostat with a three-electrode system consisting of a Glassy Carbon Electrode (GCE, 3 mm diameter), an Ag/AgCl reference electrode, and a platinum wire counter electrode. Ultrasonication was applied during electrode cleaning and modifier preparation.

The materials used were napa clay-derived NiAl_2O_4 , MWCNTs, alumina powder (0.05 μm), $\text{K}_3[\text{Fe}(\text{CN})_6]$, KCl, Na_2SO_4 , NaCl, CH_3COONa , HCl, $\text{Cd}(\text{NO}_3)_2$, and deionized water. All reagents were used without further purification.

Electrode Preparation

Preparation of Bare GCE

The GCE was polished with 0.05 μm alumina powder, rinsed with deionized water, ultrasonically cleaned for 3 min, and dried at room temperature.

Preparation of NiAl_2O_4 -GCE

A NiAl_2O_4 suspension was prepared by sonication for 15 min. Then, 5 μL of the suspension was drop-cast onto the GCE surface and dried for 1 h at room temperature.

Preparation of NiAl_2O_4 -MWCNT-GCE

A homogeneous NiAl_2O_4 /MWCNT suspension was prepared by sonication for 15 min. Subsequently, 5 μL of the suspension was drop-cast onto the GCE surface and dried for 1 h before electrochemical analysis.

Electrochemical Characterization

Electrochemical characterization of the electrodes was performed using Cyclic

Voltammetry (CV) in a solution containing 4.0 mM K₃[Fe(CN)₆] and 0.1 M KCl. Measurements were carried out over a potential range of -1.0 to 1.0 V at a scan rate of 100 mV s⁻¹ to evaluate the electron-transfer characteristics of the modified electrodes. This scan rate was selected because it produced clear and stable oxidation and reduction peaks with minimal signal distortion and capacitive current.

Cd²⁺ Detection and Supporting Electrolyte Study

The detection of Cd²⁺ was carried out using a Cd(NO₃)₂ solution under the optimized conditions obtained from electrode characterization. The performance of several supporting electrolytes (0.1 M KCl, Na₂SO₄, NaCl, and CH₃COONa) was compared to determine the most suitable electrolyte for Cd²⁺ detection.

Data Analysis

All measurements were performed in five replicates and reported as mean ± standard deviation. Linear regression analysis was used to construct the calibration curve and determine the coefficient of determination (R²). The LOD and LOQ were calculated using the standard deviation of the response and the slope of the calibration curve.

Stability

Prepare an analyte solution with the lowest concentration used in the previous detection limit test (1 mM), optimum support (0.1 M KCl), and optimum pH. Repeat the peak current (I_p) measurement five times to ensure consistent data.

RESULTS AND DISCUSSION

Electrochemical Performance of Modified Electrode

The cyclic voltammetry results demonstrated that electrode modification significantly improved the electrochemical behavior of the [Fe(CN)₆]^{3-/4-} redox system. The bare GCE exhibited the lowest oxidation and reduction peak currents, indicating relatively slow electron-transfer kinetics. Modification with NiAl₂O₄ increased the peak currents due to the presence of additional electroactive sites, while the NiAl₂O₄/MWCNT/GCE electrode produced the highest current response. This enhancement confirms that the incorporation of MWCNT improved electrical conductivity and facilitated electron transfer between the redox probe and the

electrode surface (Rangaswamy et al., 2024).

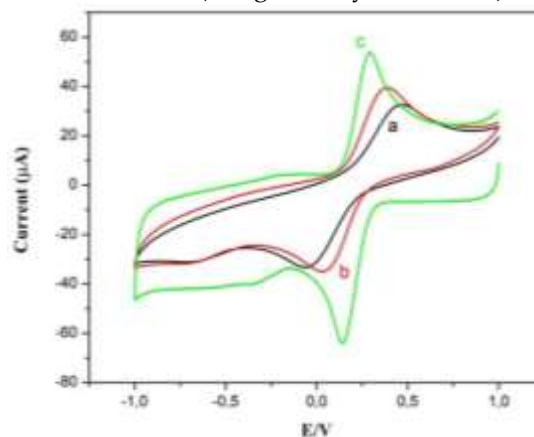


Figure 1 Cyclic voltammograms of (a) Unmodified GCE electrode (b) Modified GCE electrode NiAl₂O₄ in K₃[Fe(CN)₆] in 4.0 mM containing 0.1 M KCl. Scan rate 100 mVs⁻¹, (c) GCE + NiAl₂O₄/MWCNT in K₃[Fe(CN)₆] in 4.0 mM containing 0.1 M KCl

Table 1 Current and Redox Potential Characteristics of a Glassy Carbon Electrode (GCE) Before and After Modification

Non modified	
Oxidation	Reduction
I _{pa} = +31 µA	I _{pc} = -31 µA
E = 0,37 V	E = 0,06 V
Modified with NiAl ₂ O ₄	
Oxidation	Reduction
I _{pa} = +39 µA	I _{pc} = -36 µA
E = 0,34 V	E = 0,08 V
Modified with NiAl ₂ O ₄ /MWCNT	
Oxidation	Reduction
I _{pa} = +54 µA	I _{pc} = -64 µA
E = 0,25 V	E = 0,13 V

The improved electrochemical performance can be attributed to the synergistic effect between NiAl₂O₄ and MWCNT. NiAl₂O₄ provides active sites for redox reactions (Kunde et al., 2020), while MWCNT enhances charge transfer through its high electrical conductivity (Perenlei et al., 2011). Furthermore, the lower peak-to-peak separation (ΔE_p) indicates improved reversibility and faster electron-transfer kinetics, demonstrating the potential of the NiAl₂O₄/MWCNT composite for sensitive Cd²⁺ detection.

The bare GCE exhibited oxidation and reduction peak currents of +31 µA and -31 µA, respectively, which increased to +39 µA and -36 µA after modification with NiAl₂O₄. The NiAl₂O₄/MWCNT/GCE electrode displayed the highest response, with peak currents of +54 µA and -64 µA, indicating enhanced electron-transfer capability and electrocatalytic activity. This

improvement is attributed to the presence of NiAl_2O_4 , which provides abundant active sites for redox reactions and enhances electrocatalytic performance (Hammami et al., 2024). In addition, MWCNTs facilitate electron transport through their high electrical conductivity and large specific surface area, thereby improving charge-transfer efficiency at the electrode surface (Perenlei et al., 2011).

Furthermore, the peak-to-peak separation (ΔE_p) decreased from 0.31 V for the bare GCE to 0.26 V for the NiAl_2O_4 /GCE and 0.12 V for the NiAl_2O_4 /MWCNT/GCE. The lower ΔE_p indicates faster electron-transfer kinetics, improved electrochemical reversibility, and reduced charge-transfer resistance at the electrode–electrolyte interface (Streeter et al., 2008). These results demonstrate the potential of the NiAl_2O_4 /MWCNT composite as an effective sensing platform for Cd^{2+} detection.

Testing the Modified Electrode on Cd^{2+} Analyte

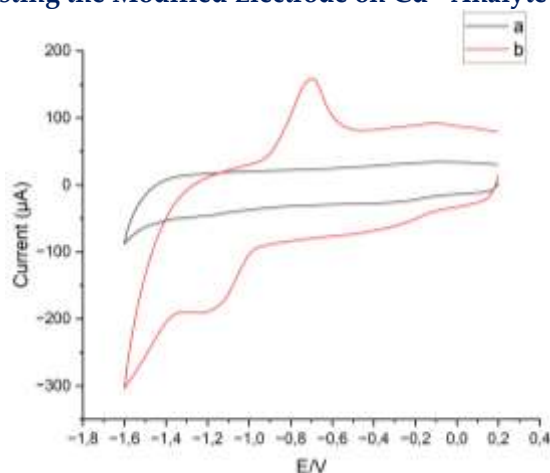


Figure 2 (a) Background KCl 0.1M (b) Analyte Ion Cd^{2+} 2mM with Background KCl 0.1M

Table 2 Electrochemical Characteristics of Bare GCE- NiAl_2O_4 -MWCNT Cd^{2+} in KCl 0,1M

Oxidation	Reduction
$I_{pa} = 155 \mu\text{A}$	$I_{pc} = -195 \mu\text{A}$
$E = -0,75 \text{ V}$	$E = -1,20 \text{ V}$

The cyclic voltammograms revealed a clear difference between the response obtained in 0.1 M KCl solution and that recorded in a 2 mM Cd^{2+} solution containing 0.1 M KCl. The KCl solution produced a relatively low current response because it functions only as a supporting electrolyte and does not participate in redox reactions within the applied potential window. In contrast, the presence of Cd^{2+} resulted in a substantial increase in current, indicating

electron-transfer processes associated with the oxidation and reduction of Cd species at the electrode surface. Similar observations have been reported by Gao et al. (2023), who attributed the enhanced current response to cadmium deposition and stripping processes.

The higher current response obtained in the Cd^{2+} solution confirms the electroactive nature of Cd^{2+} and demonstrates the capability of the developed electrode to detect cadmium ions. The increase in current can be attributed to the participation of Cd^{2+} in electrochemical redox reactions, leading to more efficient charge transfer at the electrode–electrolyte interface (Abd et al., 2023). These results highlight the potential of the NiAl_2O_4 /MWCNT-modified electrode as a sensitive platform for Cd^{2+} detection (Costa et al., 2023).

The presence of Cd^{2+} produced a significant increase in current with well-defined oxidation and reduction peaks. The oxidation peak appeared at approximately -0.75 V ($I_{pa} = +155 \mu\text{A}$), while the reduction peak was observed at around -1.20 V ($I_{pc} = -195 \mu\text{A}$), confirming the electrochemical activity of Cd species at the electrode surface. The oxidation peak corresponds to Cd deposition/stripping processes, whereas the reduction peak is associated with the reduction of Cd^{2+} during the reverse scan. The high peak currents indicate efficient electron transfer and strong interaction between Cd^{2+} ions and the NiAl_2O_4 /MWCNT-modified electrode.

Variety of Supporting Electrolytes

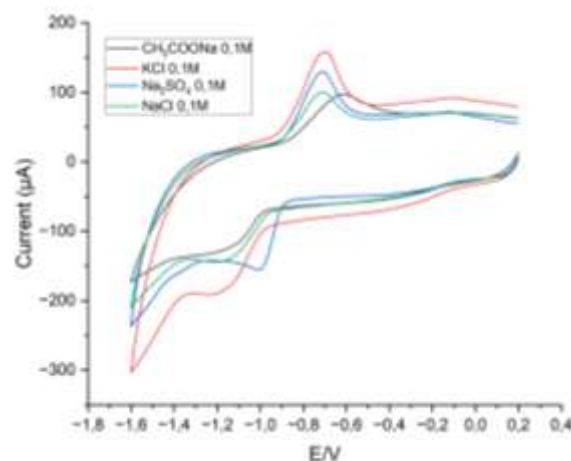


Figure 3 Supporting Variations

The cyclic voltammetry results showed that the supporting electrolyte significantly influenced the electrochemical performance of the

NiAl_2O_4 /MWCNT-modified electrode. Among the investigated electrolytes, 0.1 M KCl produced the highest oxidation and reduction peak currents, whereas 0.1 M CH_3COONa generated the lowest response. This result indicates that KCl provides a more favorable environment for ion transport and electron-transfer processes at the electrode/electrolyte interface.

These findings are consistent with previous studies showing that supporting electrolytes are not entirely inert and that cations such as K^+ and Na^+ can affect interfacial properties and electron-transfer kinetics (Ding et al., 2021). Furthermore Schalenbach et al. (2022) previous studies have shown that supporting electrolytes are vital for maintaining high ionic conductivity and facilitating charge transport, which are essential for efficient electrochemical reactions. Consequently, the differences observed among KCl, NaCl, Na_2SO_4 , and CH_3COONa can be attributed to variations in ion mobility and charge-transfer efficiency. Similar observations were also reported by Brudzisz & Brz, (2021) who demonstrated that electrolyte composition can influence both voltammetric responses and electrocatalytic activity.

Overall, 0.1 M KCl was identified as the most suitable supporting electrolyte for Cd^{2+} detection, providing the highest current response and the most efficient charge-transfer process. Further studies involving electrolyte concentration, solution pH, and interfering ions are required to optimize sensor performance.

Stability Test

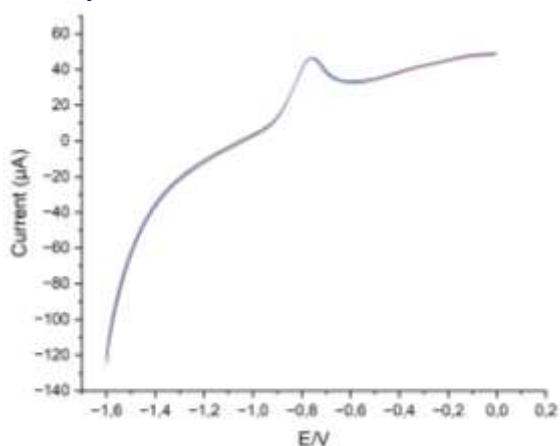


Figure 4 Stability Cd^{2+} 1 mM and Supporting KCl 1 M

The stability of the modified electrode was evaluated in a solution containing 1 mM Cd^{2+} and 0.1 M KCl through five consecutive cyclic voltammetry measurements. As shown in Figure

X, the voltammograms exhibited excellent overlap with no significant shift in peak potential or change in peak shape, indicating a stable electrochemical response during repeated measurements.

The oxidation peak at approximately -0.8 V showed nearly identical current values in all runs, suggesting that the electrode surface remained electrochemically active without significant fouling or deactivation. The consistent current response also indicates strong adhesion of the NiAl_2O_4 /MWCNT modifier layer to the electrode surface, which promotes efficient and reproducible electron transfer. Similar observations have been reported by Tadesse et al. (2013), who associated stable current responses during repeated measurements with good electrode repeatability and operational stability.

Overall, the results demonstrate that the developed electrode possesses good repeatability and stability for Cd^{2+} detection. The negligible variation in peak current and peak potential after five consecutive measurements confirms its potential for reliable electrochemical monitoring applications.

Calibration Curve

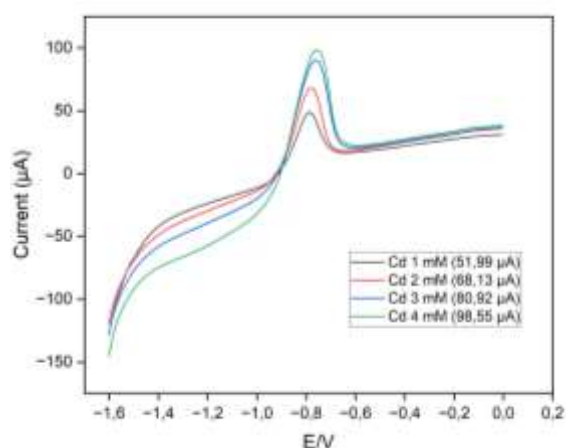


Figure 5 Cyclic voltammograms of Cd^{2+} at concentrations of (a) 1 mM, (b) 2 mM, (c) 3 mM, and 4 mM in 0.1 M KCl.

The calibration curve of Cd^{2+} (Figure 6) in the concentration range of 1–4 mM shows a linear increase in peak current with increasing analyte concentration, namely $51.99 \mu\text{A}$ (1 mM), $68.13 \mu\text{A}$ (2 mM), $80.92 \mu\text{A}$ (3 mM), and $98.55 \mu\text{A}$ (4 mM). This phenomenon indicates that the electrochemical response is dominated by a diffusion-controlled process, where an increase in concentration leads to a higher number of Cd^{2+} ions reaching the electrode surface, resulting in an

increased current response. The linear relationship between concentration and peak current is a typical characteristic of voltammetric sensors for heavy metal ion analysis (Machhindra & Yen, 2022).

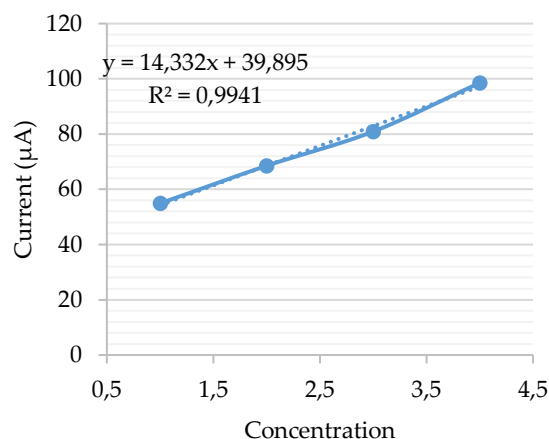


Figure 6 Calibration Curve of Cd²⁺

The obtained linear regression equation $y = 14.332x + 39.895$ with a coefficient of determination (R^2) of 0.9941 demonstrates that the modified electrode exhibits good and stable analytical performance within the investigated concentration range. The R^2 value close to 1 indicates that most of the variation in the current signal can be explained by changes in Cd²⁺ concentration, confirming the system's suitability for quantitative analysis. High linearity is an important indicator in evaluating the performance of voltammetric electrochemical sensors (Mohammed et al., 2024).

The sensitivity represented by the slope value (14.332) reflects the ability of the electrode to respond to changes in Cd²⁺ concentration. In addition, the standard deviation value of 1.4276 was used to calculate important analytical parameters, namely the Limit of Detection (LOD) and Limit of Quantification (LOQ), which are 0.2988 mM and 0.9961 mM, respectively. These parameters represent the minimum detectable and quantifiable concentrations of the sensor system. This calculation method is consistent with standard analytical evaluation procedures for stripping voltammetry-based electrochemical sensors (Reguera, 2007).

The obtained LOD value is still within the typical range for modified electrode-based sensors without advanced nanomaterial enhancement. However, further improvement can be achieved through electrode material

optimization, such as the use of conductive nanocomposites or increasing the effective surface area. Several studies have reported that modification of electrodes with nanostructured carbon materials can significantly enhance sensitivity and reduce LOD to the µM or even nM level (Hossein et al., 2024).

CONCLUSION

A NiAl₂O₄/MWCNT-modified glassy carbon electrode was successfully fabricated using NiAl₂O₄ synthesized from napa soil and applied for electrochemical detection of Cd²⁺ ions. The incorporation of NiAl₂O₄ and MWCNTs enhanced the electrochemical performance of the electrode, resulting in improved sensitivity, stability, and analytical capability toward Cd²⁺ detection. These findings demonstrate the potential of napa soil-derived NiAl₂O₄/MWCNT composites as a low-cost, environmentally sustainable, and promising sensing material for heavy metal monitoring applications.

RECOMMENDATION

Further research should focus on testing the sensor in real environmental samples and evaluating its selectivity against potentially interfering metal ions such as Pb²⁺ and Zn²⁺. Comprehensive analytical validation, including precision, accuracy, and recovery studies, is also recommended to strengthen its potential for practical environmental monitoring.

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