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Voltammetric Detection of Zn^{2+} Ions Using a Glassy Carbon Electrode Modified with Napa-Soil-Derived $NiAl_2O_4$

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Abstract
This study reports the development of a glassy carbon electrode (GCE) modified with $NiAl_2O_4$ derived from napa soil for the voltammetric detection of Zn^{2+} ions. The modified electrode was prepared using a drop-casting method and characterized in 4.0 mM $K_3[Fe(CN)_6]$ with 0.1 M KCl as the supporting electrolyte. The results showed an increase in oxidation peak current from 19.93 µA (bare GCE) to 26.80 µA and reduction peak current from −19.03 µA to −24.29 µA after modification, indicating enhanced electron transfer. Zn^{2+} detection was carried out in the concentration range of 1–3 mM, where the reduction peak current increased from −85.49 µA to −170.83 µA with increasing concentration. A linear relationship between concentration and current response was obtained with a regression equation of y = -42.67x – 40.42 and a correlation coefficient (R^2 = 0.9907), indicating good linearity of the sensor. The limit of detection (LOD) was calculated to be 0.21 mM. Among the tested supporting electrolytes, 0.1 M KCl provided the highest and most stable current response. Although the modified electrode shows improved performance, the detection range is still limited to the millimolar level, and further studies are required to evaluate selectivity, reproducibility, and applicability in real samples. These findings demonstrate the potential of napa-soil-derived $NiAl_2O_4$ as a sustainable and cost-effective material for electrochemical sensing applications.

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INTRODUCTION

Heavy metal contamination in aquatic environments has become a serious concern due to its toxic effects on living organisms and human health. Zinc ions (Zn^{2+}), although essential in trace amounts, can be harmful at elevated concentrations; therefore, accurate monitoring is necessary. Electroanalytical methods, particularly voltammetry, are widely employed because of their high sensitivity, low detection limits, and capability to provide detailed information on electron transfer processes at the electrode surface (Lopez-Tellez et al., 2022).

The Glassy Carbon Electrode (GCE) is commonly used as a working electrode in electrochemical analysis due to its high chemical stability, good conductivity, and wide potential window. In addition, glassy carbon materials exhibit excellent structural stability and mechanical strength, making them widely applied in the development of electrochemical sensors (Uskoković, 2021). However, the unmodified GCE still has limitations in sensitivity, thus requiring surface modification to enhance its performance. Previous studies have shown that electrode modification using metal oxides or nanomaterials can increase the active surface area, accelerate electron transfer, and improve the sensitivity for metal ion detection (Zahro & Setiarso, 2023).

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In addition to the selection of active materials, the utilization of local resources has become an important approach in developing more economical and sustainable sensors. Napa soil, which is abundantly found in West Sumatra, is known to be rich in silica and alumina and has potential as a precursor for functional materials. Mawardi and Zainul reported that napa soil contains high mineral content and can be utilized in various environmental applications, including as an adsorbent for heavy metal ions . Therefore, the use of napa soil as a source of alumina in the synthesis of $NiAl_2O_4$ represents an attractive approach for the development of modified electrodes (Mawardi & Zainul, 2015).

Despite the extensive use of modified electrodes for metal ion detection, studies specifically utilizing $NiAl_2O_4$ derived from napa soil as an electrode modifier remain very limited. Napa soil, which is abundant in West Sumatra, contains significant amounts of silica and alumina, making it a promising low-cost precursor for functional materials. However, its application in electrochemical sensors, particularly for Zn^{2+} detection, has not been widely explored. In addition, studies focusing on the optimization of voltammetric parameters to achieve improved sensor performance are still scarce.

Therefore, the novelty of this study lies in the utilization of napa-soil-derived $NiAl_2O_4$ as a modifier for a glassy carbon electrode (GCE) for Zn^{2+} detection. This study aims to (1) develop a $NiAl_2O_4$-modified GCE based on locally sourced napa soil, (2) investigate its electrochemical behavior using cyclic voltammetry, and (3) evaluate the effect of supporting electrolytes on the voltammetric response of Zn^{2+}.

METHOD

Instruments and Materials

All electrochemical measurements were carried out using a potentiostat connected to a conventional three-electrode system consisting of a glassy carbon electrode (GCE, diameter 3 mm) as the working electrode, an Ag/AgCl electrode as the reference electrode, and a platinum (Pt) wire as the counter electrode. An ultrasonic bath was used for electrode cleaning and suspension preparation.

The materials used in this study included $NiAl_2O_4$ derived from napa soil, α-alumina powder (0.05 μm, for electrode polishing), potassium ferricyanide ($K_3[Fe(CN)_6]$), potassium chloride (KCl), potassium nitrate (KNO_3), sodium nitrate ($NaNO_3$), sodium sulfate (Na_2SO_4), sodium chloride (NaCl), sodium acetate (CH_3COONa), hydrochloric acid (HCl), zinc nitrate ($Zn(NO_3)_2$), and deionized water.

Preparation of Bare GCE

Prior to modification, the GCE surface was polished using α-alumina slurry (0.05 μm) on a polishing cloth until a mirror-like surface was obtained. The electrode was then rinsed thoroughly with deionized water, followed by sonication in deionized water for 3 minutes to remove residual particles, and finally dried at room temperature.

Preparation of $NiAl_2O_4$-Modified GCE

A suspension was prepared by dispersing 0.176 g of $NiAl_2O_4$ powder in 10 mL of deionized water, followed by ultrasonication for 15 minutes to obtain a homogeneous dispersion. Then, 5 μL of the suspension was drop-cast onto the surface of the pretreated GCE and allowed to dry at room temperature for 1 hour. The resulting $NiAl_2O_4$-modified GCE was used for subsequent electrochemical measurements.

Electrochemical Characterization

Cyclic voltammetry measurements were performed using 4.0 mM $K_3[Fe(CN)_6]$ containing 0.1 M KCl as the supporting electrolyte. The solution was prepared by dissolving 0.0658 g of $K_3[Fe(CN)_6]$ and 0.373 g of KCl in deionized water in a 50 mL volumetric flask. The voltammograms were recorded in the potential range of 0.1 V to −1.0 V at a scan rate of 100 mV s^{-1}.

Zn^{2+} Detection and Supporting Electrolyte Study

Zn^{2+} detection was carried out using $Zn(NO_3)_2$ solutions with concentrations of 1, 2, and 3 mM. Electrochemical measurements were performed under the same conditions as described above.

To evaluate the effect of supporting electrolytes, different electrolytes (KCl, KNO_3, $NaNO_3$, Na_2SO_4, NaCl, and CH_3COONa) at a concentration of 0.1 M were used. The current responses obtained in each electrolyte were compared to determine the optimal supporting electrolyte for Zn^{2+} detection.

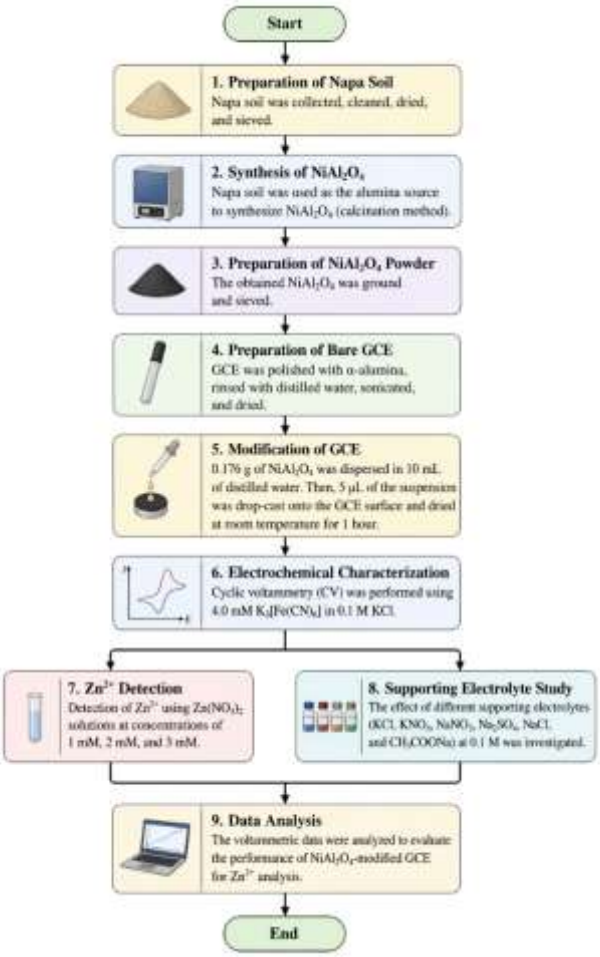

Figure 1. Research flow diagram of $NiAl_2O_4$-modified GCE preparation and Zn^{2+} analysis.

RESULTS AND DISCUSSION

Electrochemical Performance of Modified Electrode

In this study, cyclic voltammetry (CV) was employed to evaluate the electrochemical activity of the $NiAl_2O_4$-modified Glassy Carbon Electrode (GCE). The measurements were carried out using an electrolyte solution containing 4.0 mM $K_3[Fe(CN)_6]$ and 0.1 M KCl. $K_3[Fe(CN)_6]$ was used as a redox probe due to its stable and reversible electrochemical behavior, while KCl served as a supporting electrolyte to enhance solution conductivity and suppress ion migration effects, ensuring that the measured current was predominantly governed by electron transfer processes at the electrode surface (Ahmad & Oh, 2024; Cheah & Chernev, 2021).

The results show a clear difference in current response between the bare GCE and the $NiAl_2O_4$-modified GCE. The bare GCE exhibited an oxidation peak current of 19.93 µA and a reduction peak current of −19.03 µA, indicating relatively slow electron transfer kinetics. After modification with $NiAl_2O_4$, the peak currents increased to 26.80 µA for oxidation and −24.29 µA for reduction. This enhancement indicates that the incorporation of $NiAl_2O_4$ increases the electroactive surface area and facilitates faster electron transfer at the electrode interface (Bard & Faulkner, 2001).

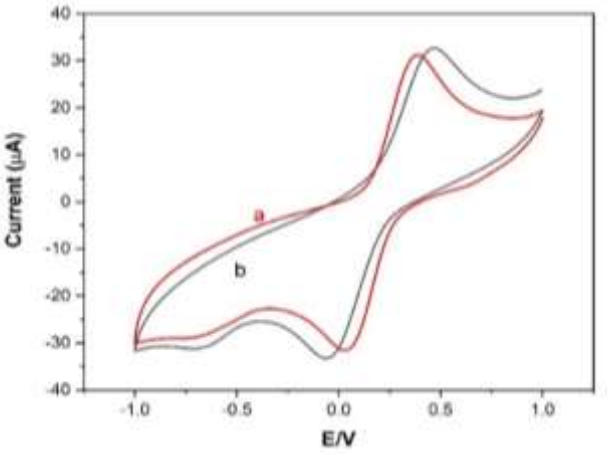

Figure 2. Cyclic voltammograms of (a) bare GCE and (b) $NiAl_2O_4$-modified GCE in 4.0 mM $K_3[Fe(CN)_6]$ containing 0.1 M KCl at a scan rate of 100 mV s^{-1}.

Table 1. Current and Redox Potential Characteristics of a Glassy Carbon Electrode (GCE) Before and After $NiAl_2O_4$ Modification

A. Nonmodification

Oxidation	Reduction
Ipa = 19,93 µA E= 0,47 V	Ipa = -19,03 µA E= -0,049 V

B. Modification Ni Al2O4

Oxidation	Reduction
Ipa = 26,80 µA E= 0,390 V	Ipa = -24,29 µA E=- 0,040 V

In addition to the increase in peak current, the modified electrode exhibited a smaller peak potential separation (ΔEp), indicating improved reversibility of the redox reaction. A lower ΔEp value reflects faster electron transfer kinetics, which can be attributed to the presence of abundant active sites and improved conductivity provided by the $NiAl_2O_4$ layer. This behavior is consistent with electrochemical theory (Ghrissi et al., 2021; Rios et al., 2021).

The enhanced electrochemical performance can be further explained by the spinel structure of $NiAl_2O_4$, which provides a high density of active sites and promotes efficient charge transfer. Spinel-type metal oxides are known to exhibit superior electrochemical properties due to their stable crystal structure and favorable surface characteristics (Morán-Lázaro et al., 2016).

Furthermore, recent studies have emphasized that nanostructured metal oxide materials significantly enhance electrochemical performance by increasing electroactive surface area and improving charge transfer efficiency. These materials exhibit improved conductivity and catalytic activity, resulting in higher current responses and better sensor sensitivity (Si et al., 2022).

These findings are also supported by previous studies on spinel and metal oxide-based electrode materials. (Aouini et al., 2024) reported that $CuMn_2O_4$ spinel-based electrodes exhibited enhanced electrochemical performance due to increased surface activity. (Madagalam et al., 2024) demonstrated that Bi-modified $ZnFe_2O_4$ improved electron transfer and sensitivity. In addition, (Tajik et al., 2022) lso reported that $ZnFe_2O_4$ /RGO nanocomposites significantly enhanced peak currents and electroactive surface area. Therefore, the observed improvement in the $NiAl_2O_4$ -modified GCE in this study is in good agreement with previously reported findings.

Zn^{2+} Detection

The analyte test was conducted to evaluate the ability of the $NiAl_2O_4$-modified GCE to detect Zn^{2+} ions. Measurements were performed using cyclic voltammetry in $Zn(NO_3)_2$ solutions with concentrations of 1 mM, 2 mM, and 3 mM, employing 0.1 M KCl as the supporting electrolyte.

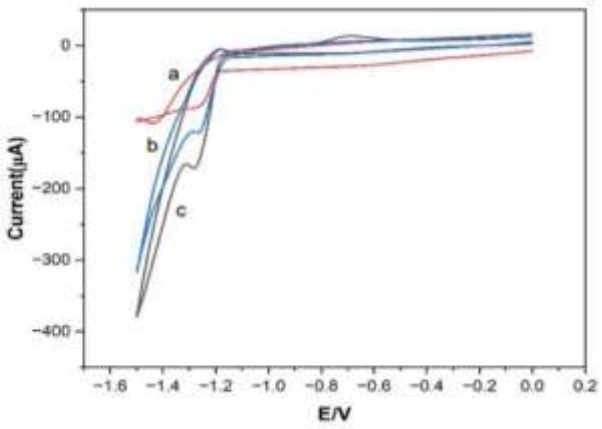

Figure 3. Cyclic voltammograms of $Zn(NO_3)_2$ at concentrations of (a) 1 mM, (b) 2 mM, and (c) 3 mM in 0.1 M KCl.

Figure 3 presents the cyclic voltammograms of Zn^{2+} at different concentrations, while Table 2 summarizes the corresponding reduction peak currents and potentials. The results show that the reduction peak current (Ipc) increases significantly with increasing Zn^{2+} concentration, from −85.49 μA at 1 mM to −170.83 μA at 3 mM. This increase indicates that a greater amount of Zn^{2+} ions is reduced to metallic Zn^0 at the electrode surface as the concentration increases.

Table 2. Peak Current Response and Reduction Potential of Zn^{2+} at Various Concentrations

Variation	Peak Current (μa)	E/V
Zn^{2+} 1mM on 0,1KCl	-85,488	-1,264
Zn^{2+} 2mM on 0,1KCl	-120,98	-1,26
Zn^{2+} 3mM on 0,1KCl	-170,83	-1,288

The electrochemical reaction involved can be expressed as:

$$Zn^{2+} + 2e^- \rightarrow Zn^0$$

The observed increase in peak current with Zn^{2+} concentration indicates that the electrochemical process is predominantly controlled by diffusion of Zn^{2+} ions toward the electrode surface. Under diffusion-controlled conditions, the peak current is proportional to the concentration of electroactive species, which is consistent with previously reported studies (Ringgit et al., 2022; Yamada et al., 2022).

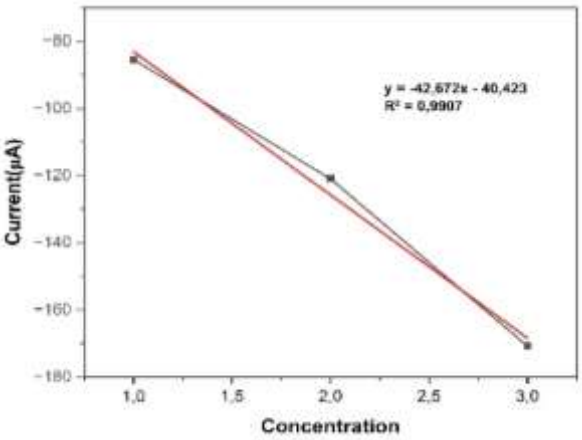

Figure 4. Calibration curve of Zn^{2+} using $NiAl_2O_4$-modified GCE in 0.1 M KCl.

A calibration curve was constructed by plotting the reduction peak current against Zn^{2+} concentration, as shown in Figure 4. The calibration plot exhibits a linear relationship within the studied concentration range. Based on the regression analysis, the linear equation obtained is:

$$\mathbf{y = -42.67x - 40,42}$$

with a correlation coefficient of $\mathbf{R^2 = 0.9907}$, indicating excellent linearity of the electrochemical response. This high R^2 value indicates a strong correlation between Zn^{2+} concentration and current response, confirming the reliability of the modified electrode for quantitative analysis.

The limit of detection (LOD) was calculated using the following equation:

$$LOD=\frac{3\sigma}{m}$$

where σ represents the standard deviation of the blank signal and m is the slope of the calibration curve. The calculated LOD value is **0.21 mM**, indicating that the modified electrode is capable of detecting Zn^{2+} at the millimolar level. However, the obtained LOD is still within the millimolar range, indicating that further optimization is required to achieve detection at lower (trace-level) concentrations.

In addition to the current response, the reduction peak potential shows only a slight variation with increasing Zn^{2+} concentration, indicating that the electrochemical system remains stable and is not significantly affected by reaction kinetics. A similar observation was reported by (Shalaby et al., 2023), where increasing Zn^{2+} concentration influenced the current magnitude without causing a significant shift in peak potential.

The significant enhancement in current response observed in this study demonstrates that the $NiAl_2O_4$ modification plays an important role in improving the electrochemical performance of the GCE. The presence of $NiAl_2O_4$ increases the electroactive surface area and facilitates faster electron transfer, resulting in higher sensitivity. This is consistent with previous studies reporting that nanostructured and metal oxide-modified electrodes provide more active sites and improved electron transfer pathways (Ren et al., 2018; Zolla Azhara, 2024).

Overall, these results indicate that the $NiAl_2O_4$-modified GCE exhibits good sensitivity, stable electrochemical behavior, and a clear concentration-dependent response toward Zn^{2+} ions. Therefore, this modified electrode shows strong potential as an effective electrochemical sensor for Zn^{2+} detection.

Effect of supporting Electrolyte

The variation of supporting electrolytes was investigated to determine the most effective electrolyte for the voltammetric measurement of 3 mM Zn^{2+}. Based on the obtained voltammograms, each supporting electrolyte produced different curve shapes and current responses. This indicates that the type of supporting ions in the solution influences the transport of Zn^{2+} ions toward the electrode surface as well as the stability of the resulting electrochemical signal.

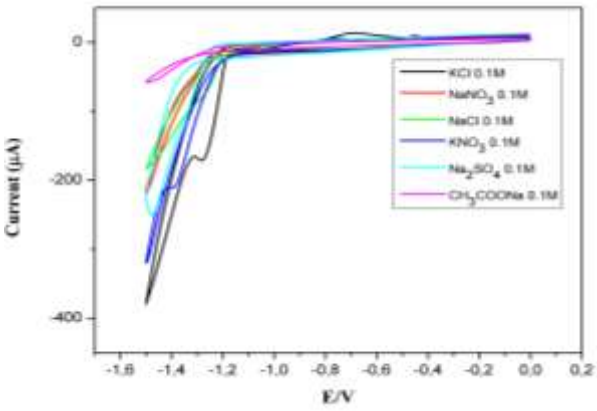

Figure 5. ffect of different supporting electrolytes on the voltammetric response of 3 mM Zn^{2+}

The use of 0.1 M KCl as a supporting electrolyte produced the most distinct and stable reduction peak of Zn compared to other electrolytes. This behavior is attributed to the high conductivity of KCl, which reduces solution resistance (ohmic drop) and enhances the efficiency of Zn^{2+} ion transfer to the electrode surface. In addition, K^+ and Cl^- ions are electrochemically inert within the potential window used, so they do not participate in side reactions that could interfere with the Zn signal. As a result, the voltammetric response shows a sharper peak and improved analytical sensitivity.

In contrast, other electrolytes generated lower peak currents and less stable signals due to differences in ionic strength and ion mobility, which affect Zn^{2+} transport and charge transfer processes at the electrode interface. A supporting electrolyte functions to enhance solution conductivity, suppress migration current, and stabilize the electrical double layer at the electrode surface, thereby improving voltammetric response (Manzoor et al., 2020). This finding is consistent with (Sarah & Sari, 2025)and (Wartati & Sari, 2025), who reported that the type of supporting electrolyte significantly influences peak current intensity and signal stability in metal ion detection.

Furthermore, this trend is supported by open-access studies showing that electrolyte composition strongly affects voltammetric performance through changes in ionic strength, diffusion behavior, and charge transfer resistance (Jia et al., 2020; Rosemarin et al., 2021). These studies confirm that optimized supporting electrolyte conditions are essential to improve sensitivity and analytical precision in electrochemical sensing systems.

Thus, the use of 0.1 M KCl provides optimal electroanalytical conditions for Zn^{2+} detection via cyclic voltammetry. This is due to its high conductivity, low resistance, efficient ion transport, and minimal electrochemical interference, resulting in a stable, sharp, and sensitive voltammetric response. These characteristics strongly support the performance of the $NiAl_2O_4$-modified glassy carbon electrode as an effective electrochemical sensor.

CONCLUSION

This study confirms that the modification of a glassy carbon electrode (GCE) with $NiAl_2O_4$ derived from napa soil enhances its electrochemical performance, as indicated by increased peak currents (19.93 μA to 26.80 μA and −19.03 μA to −24.29 μA) and reduced ΔEp. A linear response toward Zn^{2+} was obtained in the range of 1–3 mM ($y = -42.67x - 40.42$, $R^2 = 0.991$) with a detection limit of 0.21 mM, while 0.1 M KCl provided the most stable signal. However, the sensor performance is still limited to the millimolar range and lacks evaluation of selectivity, reproducibility, and real sample applicability. Therefore, although the material shows promise as a low-cost electrode modifier, further optimization is required for practical applications.

RECOMMENDATION

Future studies should focus on lowering the detection limit to the micromolar level and improving sensitivity through optimization of material composition and electrode fabrication. Comprehensive evaluation of selectivity in the presence of interfering ions, as well as reproducibility and long-term stability tests, is essential. Furthermore, application to real environmental samples and comparison with established methods are strongly recommended to validate the practical applicability of the proposed sensor.

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