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Voltammetric Detection of Cu^{2+} Using a Napa Soil-Derived $NiAl_2O_4$/MWCNT-Modified Glassy Carbon Electrode

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Abstract
Copper ions (Cu^{2+}) are essential trace elements but may become toxic at elevated concentrations, requiring reliable monitoring methods. In this study, a $NiAl_2O_4$/MWCNT-modified glassy carbon electrode (GCE) derived from napa soil was developed for the voltammetric determination of Cu^{2+} ions. The modified electrode was prepared by combining $NiAl_2O_4$ synthesized from napa soil with multi-walled carbon nanotubes (MWCNTs) through a drop-casting technique. Electrochemical characterization demonstrated that the incorporation of $NiAl_2O_4$ and MWCNTs significantly enhanced the electrochemical performance of the electrode by improving electron-transfer efficiency and increasing the electroactive surface area. The modified electrode exhibited a strong voltammetric response toward Cu^{2+} ions, while $HClO_4$ was identified as the most suitable supporting electrolyte among the investigated media. Selectivity studies showed that Cu^{2+} remained detectable in the presence of Pb^{2+}, although partial signal suppression was observed at higher Pb^{2+} concentrations. These findings indicate that the $NiAl_2O_4$/MWCNT-modified GCE is a promising low-cost and sustainable platform for Cu^{2+} determination in aqueous environments.

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INTRODUCTION

Heavy metal contamination in aquatic environments has become a major environmental concern due to its persistence, toxicity, and tendency to accumulate in living organisms. Among various heavy metals, copper ions (Cu^{2+}) are essential micronutrients involved in numerous biological processes; however, excessive exposure can pose serious risks to both ecosystems and human health. Elevated Cu^{2+} concentrations have been associated with liver and kidney damage, gastrointestinal disorders, and neurological impairment. Furthermore, the continuous release of copper-containing wastes from industrial activities, mining operations, and agricultural runoff contributes to the increasing levels of Cu^{2+} in water systems. Therefore, reliable and sensitive methods for monitoring Cu^{2+} are essential to prevent environmental contamination, maintain water quality, and protect public health (Briffa et al., 2020).

Voltammetric techniques have been widely employed for the determination of trace metal ions owing to their high sensitivity, rapid response, low operational cost, and ability to provide valuable information on electrochemical processes occurring at electrode surfaces. Among the various working electrodes used in voltammetric analysis, the glassy carbon electrode (GCE) is widely preferred because of its wide potential window, low background current, excellent chemical stability, and good electrical conductivity. However, the analytical performance of bare GCE may be limited by its relatively low electrocatalytic activity and insufficient electroactive surface area, particularly for trace-level metal ion detection. Therefore,

surface modification using functional nanomaterials has become an effective strategy to improve electron-transfer kinetics, increase the number of active sites, and enhance analytical sensitivity (Abd-Elsabur et al., 2023; Pimpilova, 2024).

In recent years, various nanomaterials have been extensively investigated to improve the performance of electrochemical sensors. Among them, spinel-type metal oxides such as nickel aluminate ($NiAl_2O_4$) have attracted considerable attention due to their excellent structural stability, large surface area, and favorable electrochemical properties that facilitate electron-transfer reactions. $NiAl_2O_4$ has been successfully applied as an electrode material for electrochemical sensing because of its good electrocatalytic activity, chemical stability, and rapid charge-transfer capability. In addition, multi-walled carbon nanotubes (MWCNTs) have attracted significant interest in electrochemical sensing owing to their high electrical conductivity, large specific surface area, and excellent electron-transport properties, making them promising materials for electrode modification and signal enhancement (Rangaswamy et al., 2024).

Napa soil is a naturally occurring material abundantly found in West Sumatra, Indonesia, and is primarily composed of silica (SiO_2) and alumina (Al_2O_3) minerals. Due to its high alumina content, napa soil has attracted considerable interest as a low-cost and locally available raw material for the synthesis of various functional inorganic materials. The utilization of naturally abundant resources such as napa soil not only reduces production costs but also supports the development of sustainable and environmentally friendly materials. In addition, the presence of alumina-rich phases makes napa soil a promising precursor for the preparation of spinel-type metal oxides. Therefore, napa soil offers significant potential as an alternative source for the synthesis of $NiAl_2O_4$, providing an economical and sustainable approach for developing advanced materials for electrochemical applications (Mawardi & Zainul, 2015).

Although $NiAl_2O_4$ and MWCNT-based materials have individually demonstrated promising electrochemical properties for sensor applications, most reported studies rely on commercially available chemical precursors for material synthesis. The utilization of naturally occurring alumina-rich resources as sustainable precursors for $NiAl_2O_4$ production remains largely unexplored. In particular, napa soil has rarely been investigated as an alternative alumina source for the preparation of electroactive spinel materials. Moreover, previous studies have primarily focused on the synthesis and characterization of $NiAl_2O_4$ materials, while their integration with MWCNTs for electrochemical sensing applications has received limited attention. To the best of our knowledge, no study has reported the fabrication of a $NiAl_2O_4$/MWCNT composite synthesized from napa soil-derived materials for voltammetric Cu^{2+} detection. This gap highlights the need to develop sustainable, low-cost, and environmentally friendly electrode materials while maintaining high electrochemical performance for heavy metal monitoring.

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

METHOD

Instruments and Materials

Electrochemical measurements were performed using a potentiostat coupled with a conventional three-electrode electrochemical cell. A glassy carbon electrode (GCE, 3 mm diameter) was employed as the working electrode, an Ag/AgCl electrode served as the reference electrode, and a platinum (Pt) wire was used as the counter electrode. An ultrasonic bath was utilized for cleaning the electrodes and preparing homogeneous suspensions.

The materials used in this study included Cu nanoparticles synthesized from copper nitrate precursor, α-alumina powder (0.05 μm) for polishing the GCE surface, potassium ferricyanide ($K_3[Fe(CN)_6]$), potassium chloride (KCl), potassium nitrate (KNO_3), sodium asetate (CH_3COONa), perchloric acid ($HClO_4$), hydrochloric acid (HCl), copper(II) nitrate ($Cu(NO_3)_2$), and deionized water.

Preparation of Bare GCE

The GCE surface was first polished using

a 0.05 µm α-alumina slurry on a polishing cloth until a mirror-like surface was obtained. It was then rinsed thoroughly with deionized water to remove any polishing residues and sonicated in deionized water for 3 minutes to eliminate remaining particles. Finally, the electrode was dried at room temperature prior to use.

Preparation of $NiAl_2O_4$-MWCNT Modified GCE

A suspension was prepared by dispersing 0.005 g of $NiAl_2O_4$ and 0.095 g of MWCNT in 10 mL of deionized water, followed by ultrasonication for 15 minutes to achieve a homogeneous dispersion. Then, 5 µL of the resulting suspension was drop-cast onto a pretreated glassy carbon electrode (GCE) and left to dry at room temperature for one hour. The modified $NiAl_2O_4$/MWCNT electrode was subsequently used for electrochemical measurements.

Electrochemical Characterization

Cyclic voltammetry (CV) measurements were performed using a 4.0 mM $K_3[Fe(CN)_6]$ solution with 0.1 M KCl as a 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, then diluting to a volume of 50 mL using a volumetric flask. Voltammograms were recorded over a potential range of 0.1 V to 1.0 V with a scan rate of 100 mV s^{-1}. A scan rate of 100 mV s^{-1} was selected because it provided a stable voltammetric response with well-defined oxidation and reduction peaks, ensuring reliable electrochemical measurements.

Cu^{2+} Detection and Supporting Electrolyte Study

Cu^{2+} detection was performed using a 2 mM $Cu(NO_3)_2$ solution under the same electrochemical conditions described in the previous section. The influence of supporting electrolytes on the voltammetric response was evaluated using 0.1 M $HClO_4$, KNO_3, and CH_3COONa solutions. The resulting current responses were compared to identify the most suitable supporting electrolyte for Cu^{2+} determination.

Calibration Curve of Cu^{2+}

A series of Cu^{2+} standard solutions with concentrations of 1.2, 1.4, 1.6, 1.8, and 2.0 mM were prepared and measured using the optimized supporting electrolyte 0.1 M $HClO_4$. Voltammograms were recorded for each concentration under identical experimental conditions, and the corresponding peak currents were obtained. The calibration curve was constructed by plotting the peak current against Cu^{2+} concentration to evaluate the linear relationship between the current response and analyte concentration

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

RESULTS AND DISCUSSION

Electrochemical Performance of Modified Electrode

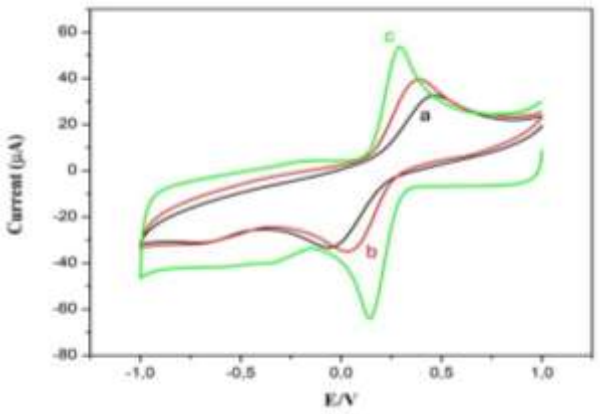

Figure 2. Cyclic voltammograms of (a) bare GCE, (b) $NiAl_2O_4$-modified GCE, and (c) $NiAl_2O_4$/MWCNT-modified GCE in 4.0 mM $K_3[Fe(CN)_6]$ containing 0.1 M KCl at a scan rate of 100 mV s^{-1}.

Table 1. Electrochemical Characteristics of Bare GCE, $NiAl_2O_4$/GCE, and $NiAl_2O_4$/MWCNT/GCE in 4.0 mM $K_3[Fe(CN)_6]$ Containing 0.1 M KCl

a. Bare GCE

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

b. $NiAl_2O_4$-Modified GCE

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

c. $NiAl_2O_4$/MWCNT- Modified GCE

Oxidation	Reduction
Ipa = 54 µA E = 0,25 V	Ipc = -64 µA E = 0,13 V

The electrochemical performance of bare GCE, $NiAl_2O_4$/GCE, and $NiAl_2O_4$/MWCNT/GCE was evaluated using cyclic voltammetry in 4.0 mM $K_3[Fe(CN)_6]$ and 0.1 M KCl. The ferri/ferrocyanide redox system is widely used as an electrochemical probe to evaluate electron-transfer behavior and the effectiveness of electrode surface modification (Stortini et al., 2020). As shown in Figure 2 and Table 1, the anodic and cathodic peak currents increased progressively after electrode modification. The bare GCE exhibited anodic and cathodic peak currents of 19.93 µA and −19.03 µA, respectively, whereas $NiAl_2O_4$/GCE showed increased currents of 26.80 µA and −24.29 µA. The highest current response was obtained for $NiAl_2O_4$/MWCNT/GCE, with anodic and cathodic peak currents of 54 µA and −64 µA, respectively. These results indicate that electrode modification enhanced the electrochemical activity of the electrode surface.

The increase in peak current after $NiAl_2O_4$ modification suggests the presence of additional electroactive sites and improved electron-transfer processes at the electrode/electrolyte interface. $NiAl_2O_4$-based materials have been reported to exhibit enhanced electrochemical activity due to their porous structure, high surface area, and favorable charge-transport properties (Hu et al., 2024). Similar improvements in electrochemical response have also been associated with increased electroactive surface area and enhanced electron-transfer efficiency in nanostructured electrode materials (Beas-Bernuy et al., 2023; Selim et al., 2023)

A more pronounced enhancement was observed after the incorporation of MWCNTs into the $NiAl_2O_4$ matrix. The excellent electrical conductivity and large surface area of MWCNTs facilitate charge transport and increase the accessibility of electroactive species to the electrode surface, resulting in higher current responses (Selim et al., 2023). The substantial increase in both anodic and cathodic peak currents obtained for $NiAl_2O_4$/MWCNT/GCE indicates a synergistic interaction between $NiAl_2O_4$ and MWCNTs, which promotes electron-transfer kinetics and improves the electrochemical activity of the modified electrode.

In addition to the increase in peak current, a shift in anodic peak potential was also observed following electrode modification. The anodic peak potential decreased from 0.47 V for bare GCE to 0.39 V for $NiAl_2O_4$/GCE and further to 0.25 V for $NiAl_2O_4$/MWCNT/GCE. This shift toward lower potential indicates that the oxidation process became more favorable, reflecting faster charge-transfer processes and improved electrocatalytic activity at the electrode surface. The lower oxidation potential obtained for $NiAl_2O_4$/MWCNT/GCE further confirms the beneficial effect of combining $NiAl_2O_4$ with MWCNTs in facilitating electron transfer (Magar et al., 2023).

Overall, the results demonstrate that the combination of $NiAl_2O_4$ and MWCNTs significantly improved the electrochemical performance of the electrode through the synergistic contribution of electroactive sites and conductive pathways. Similar behavior has been reported for metal oxide–MWCNT nanocomposites, where the integration of conductive carbon networks with electroactive metal oxides enhances charge transport and sensing performance toward heavy metal ions (Mohammed et al., 2024). These characteristics are advantageous for electrochemical sensing applications and indicate the potential of $NiAl_2O_4$/MWCNT/GCE as a sensing platform for Cu^{2+} determination.

Electrochemical Response of Cu^{2+}

Cyclic voltammograms of the blank solution (0.1 M $HClO_4$) and 2.0 mM Cu^{2+} were recorded to evaluate the electrochemical behavior of the modified electrode. No significant redox peak was observed in the blank solution, whereas the addition of Cu^{2+} generated a distinct pair of oxidation and reduction peaks. As presented in Table 2, the anodic peak current (Ipa) and cathodic peak current (Ipc) were 260 µA and

−158.14 μA, respectively, with peak potentials of 0.11 V and −0.086 V. The appearance of these peaks indicates the electrochemical response of Cu^{2+} at the $NiAl_2O_4$/MWCNT-modified GCE.

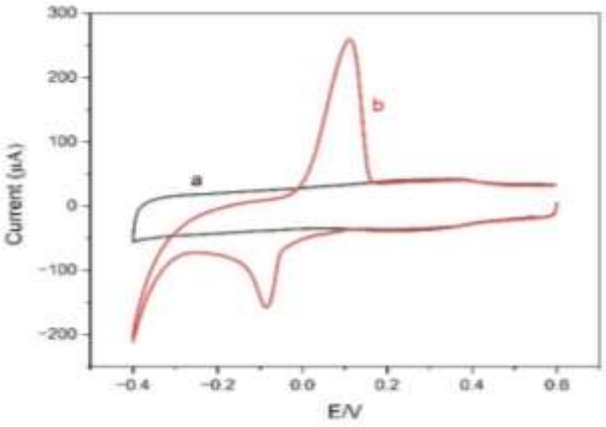

Figure 3. Cyclic voltammograms of (a) 0.1 M $HClO_4$ blank solution and (b) 2.0 mM Cu^{2+} in 0.1 M $HClO_4$ supporting electrolyte.

Table 2. Electrochemical Characteristics of Bare GCE, $NiAl_2O_4$/GCE, and $NiAl_2O_4$/MWCNT/GCE in 4.0 mM $K_3[Fe(CN)_6]$ Containing 0.1 M KCl

Oxidation	Reduction
Ipa = 260 μA	Ipc = -158.140 μA
E = 0,11 V	E = -0,0860 V

The electrochemical behavior of Cu^{2+} at the modified electrode is governed by a reversible redox reaction, which can be described as follows:

$$Cu^{2+} + 2e^- \rightleftharpoons Cu^0$$

During the cathodic scan, Cu^{2+} ions are reduced and deposited onto the electrode surface as metallic copper. In the subsequent anodic scan, the deposited Cu^0 is oxidized back to Cu^{2+}, producing the anodic stripping peak observed in the voltammogram. This redox process confirms that the detected signal originates from the electrochemical transformation of Cu^{2+} at the electrode interface.

The relatively high anodic current obtained indicates efficient electron-transfer processes between Cu^{2+} ions and the modified electrode surface. This behavior can be attributed to the incorporation of MWCNTs, which enhance electrical conductivity and facilitate charge transport during the redox reaction. Previous studies have reported that ZIF-67/MWCNT-modified electrodes exhibit accelerated electron-transfer kinetics and enhanced current response for Cu^{2+} detection due to the conductive network provided by MWCNTs (Li et al., 2024).

The results demonstrate that the $NiAl_2O_4$/MWCNT-modified GCE exhibits good electrochemical performance toward Cu^{2+} detection. The enhanced current response highlights the effectiveness of the modified electrode for Cu^{2+} determination and confirms the important role of carbon nanotube-based materials in improving electrochemical sensor performance (Tamborelli et al., 2025).

Effect of supporting Electrolyte

The supporting electrolyte plays an important role in electrochemical measurements because it affects ionic conductivity and electron-transfer efficiency during the redox process. Therefore, optimization of the supporting electrolyte is necessary to obtain the maximum voltammetric response for Cu^{2+} detection

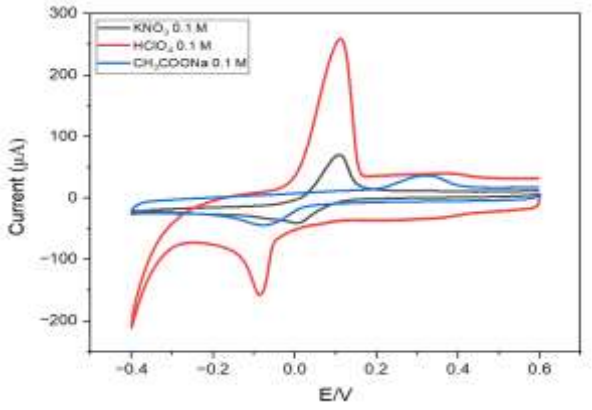

Figure 4. Effect of different supporting electrolytes on the voltammetric response of 2 mM Cu^{2+}

The cyclic voltammetric responses of 2.0 mM Cu^{2+} in different supporting electrolytes, namely $HClO_4$, KNO_3, and CH_3COONa. The results indicate that the supporting electrolyte significantly affects the electrochemical behavior of Cu^{2+} at the $NiAl_2O_4$/MWCNT-modified GCE, as evidenced by differences in peak current and voltammogram profile. The electrolyte composition influences ionic conductivity, mass transport, and the efficiency of charge-transfer processes occurring during the oxidation and reduction of Cu^{2+}. Similar electrolyte-dependent behavior has been reported for Cu^{2+} sensors based on nanostructured materials, where changes in the supporting electrolyte directly affected the magnitude of the electrochemical signal and analytical sensitivity (C. Zhang et al., 2024).

Among the investigated electrolytes, $HClO_4$ produced the highest current response, indicating that acidic conditions provide more favorable environments for the electrochemical oxidation and reduction of Cu^{2+}. The enhanced response can be attributed to the higher ionic conductivity of the acidic medium, which facilitates ion migration

and accelerates charge-transfer processes at the electrode surface. Consequently, Cu^{2+} ions can be more efficiently involved in the electrochemical reaction, resulting in increased peak currents. Similar findings have been reported for heavy-metal ion sensors, where acidic supporting electrolytes improved electron-transfer kinetics and enhanced analytical sensitivity (Dinu et al., 2024).

In contrast, the current response obtained in KNO_3 was lower than that observed in $HClO_4$. Although nitrate ions are generally considered electrochemically inert and are frequently employed as supporting electrolytes, the neutral medium provides less favorable conditions for charge transport than strongly acidic solutions. As a result, the oxidation and reduction processes of Cu^{2+} become less efficient, leading to a decrease in the voltammetric signal. The influence of electrolyte composition on charge-transfer behavior has also been observed in metal–organic framework-based electrochemical sensors for heavy-metal ion determination (Tran et al., 2023).

The lowest current response was observed in CH_3COONa solution, indicating that acetate-containing media were less favorable for Cu^{2+} determination under the experimental conditions employed. This behavior may be attributed to lower ionic mobility and less efficient charge-transfer processes compared with strongly acidic electrolytes. Consequently, the transport of Cu^{2+} ions toward the electrode surface becomes less effective, resulting in a lower voltammetric signal. Similar trends have been reported in Cu^{2+} electrochemical detection studies, where the selection of supporting electrolyte significantly influenced the current response and analytical performance of the sensor (Zulfa & Kumala Sari, 2024).

Calibration Curve and Detection Limit

Figure 6 shows the calibration curve obtained from the relationship between the anodic peak current and Cu^{2+} concentration in the range of 1.2–2.0 mM. The anodic peak current increased linearly with increasing Cu^{2+} concentration, indicating that the electrochemical response of the $NiAl_2O_4$/MWCNT-modified GCE was dependent on the analyte concentration. This result demonstrates the capability of the modified electrode to provide a consistent electrochemical response toward Cu^{2+} ions.

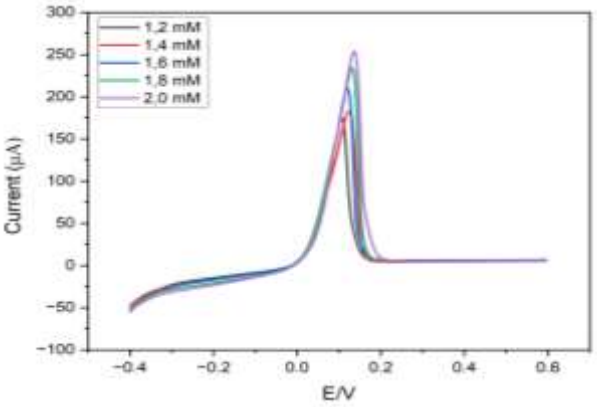

Figure 5. Electrochemical responses of the $NiAl_2O_4$/MWCNT-modified glassy carbon electrode toward Cu^{2+} at different concentrations (1.2–2.0 mM) in 0.1 M $HClO_4$.

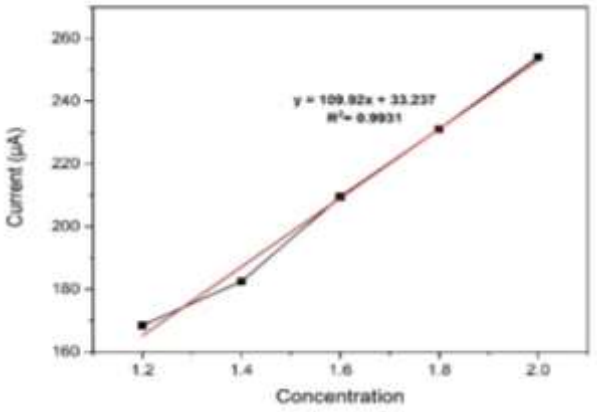

Figure 6. Calibration curve of Cu^{2+} showing the relationship between current and concentration.

Linear regression analysis of the calibration data yielded the following equation:

$$\mathbf{y = 109.92x + 33.237}$$

The regression model produced a correlation coefficient of $\mathbf{R^2 = 0.9931}$, indicating excellent linearity within the investigated concentration range. The high coefficient of determination suggests that the anodic peak current was strongly correlated with Cu^{2+} concentration, demonstrating the reliability of the developed sensor for quantitative analysis. The obtained linear relationship confirms that the $NiAl_2O_4$/MWCNT-modified GCE can generate a stable and reproducible analytical response toward Cu^{2+} ions.

The sensitivity of the developed $NiAl_2O_4$/MWCNT-modified GCE was determined from the slope of the calibration curve and was calculated to be 109.92 μA mM^{-1}. This sensitivity value indicates that the electrode can generate a significant change in current response for a small variation in Cu^{2+} concentration, demonstrating its capability for quantitative

detection. The high sensitivity can be attributed to the synergistic effect between $NiAl_2O_4$ and MWCNTs, where $NiAl_2O_4$ provides abundant electroactive sites while MWCNTs enhance electrical conductivity and facilitate rapid electron transfer. In addition, the large electroactive surface area provided by the nanocomposite promotes greater interaction between Cu^{2+} ions and the electrode surface, resulting in improved signal generation. Enhanced electrocatalytic activity, increased electroactive surface area, and improved charge-transfer efficiency have been recognized as key factors contributing to the high sensitivity of electrochemical sensors based on $NiAl_2O_4$ nanostructures and MWCNT-containing nanocomposites. These characteristics confirm that the $NiAl_2O_4$/MWCNT-modified GCE possesses favorable analytical performance for Cu^{2+} determination (Hammami et al., 2024; Zhou, 2024).

The limit of detection (LOD) was calculated to evaluate the analytical capability of the developed sensor for the determination of Cu^{2+} using the standard deviation of the analytical response (σ = 3.2109) and the slope of the calibration curve according to the equation:

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

The calculated LOD value was **0.0787 mM**, indicating that the developed electrode is capable of detecting Cu^{2+} ions at relatively low concentration levels. A low detection limit is an important analytical parameter because it reflects the ability of the sensor to distinguish analyte signals from background noise and reliably detect the presence of target ions. The obtained LOD demonstrates satisfactory analytical performance of the $NiAl_2O_4$/MWCNT-modified GCE for Cu^{2+} detection within the investigated concentration range. The achieved detection limit suggests that the $NiAl_2O_4$/MWCNT nanocomposite effectively improves the electrochemical response toward Cu^{2+} ions, thereby enhancing signal discrimination at low concentrations. This behavior can be attributed to the synergistic interaction between the conductive MWCNT network and the electroactive $NiAl_2O_4$ particles, which facilitates charge transfer and promotes efficient analyte oxidation. Consequently, the developed sensor exhibits sufficient sensitivity for Cu^{2+} determination and shows potential for application in environmental monitoring and water quality assessment. The importance of achieving low detection limits for reliable trace-level heavy metal analysis in aqueous systems has been highlighted in previous studies on electrochemical Cu^{2+} sensors (Du et al., 2024).

Selectivity toward Cu^{2+} Detection

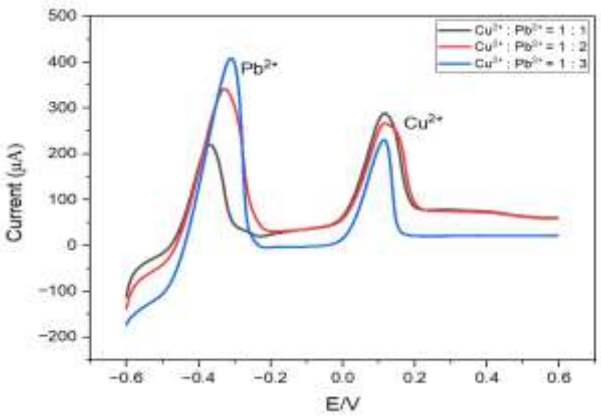

Figure 7. Effect of Pb^{2+} on the voltammetric response of Cu^{2+}.

The selectivity of the $NiAl_2O_4$/MWCNT-modified GCE toward Cu^{2+} was evaluated in the presence of Pb^{2+} at Cu^{2+} concentration ratios of 1:1, 1:2, and 1:3, as shown in Figure 7. Two distinct oxidation peaks corresponding to Pb^{2+} and Cu^{2+} were observed, indicating that both metal ions could be electrochemically distinguished on the modified electrode surface. The clear separation of the voltammetric peaks demonstrates the ability of the developed sensor to selectively detect Cu^{2+} in the presence of Pb^{2+} under the investigated conditions. This finding is consistent with previous studies employing modified carbon paste electrodes for the electrochemical determination of Pb^{2+} and Cu^{2+}, where well-resolved voltammetric signals enabled effective differentiation between the two metal ions during analysis (Laghlimi et al., 2020).

As the concentration of Pb^{2+} increased from a Cu^{2+}:Pb^{2+} ratio of 1:1 to 1:3, the peak current of Pb^{2+} increased significantly, while the Cu^{2+} peak current gradually decreased. This behavior suggests the occurrence of competitive interactions between Cu^{2+} and Pb^{2+} during the electrochemical process, resulting in partial suppression of the Cu^{2+} response at higher Pb^{2+} concentrations. The coexistence of electroactive metal ions can influence the voltammetric response of the target analyte due to competition occurring at the electrode surface during the redox process, which consequently affects the

measured current signal (Y. Zhang et al., 2020).

The observed decrease in the Cu^{2+} peak current with increasing Pb^{2+} concentration suggests the occurrence of competitive electrochemical interactions between the two metal ions during the detection process. As the amount of Pb^{2+} in the solution increases, competition for electroactive sites and electron-transfer pathways at the electrode interface may become more significant, leading to a partial suppression of the Cu^{2+} response. Nevertheless, the Cu^{2+} peak remained clearly distinguishable at all investigated concentration ratios, indicating that the $NiAl_2O_4$/MWCNT-modified GCE retained sufficient selectivity toward Cu^{2+} even in the presence of increasing concentrations of Pb^{2+}. This behavior demonstrates that the developed electrode possesses an acceptable tolerance toward Pb^{2+} interference under the experimental conditions employed in this study.

Although a gradual decrease in the Cu^{2+} peak current was observed with increasing Pb^{2+} concentration, the Cu^{2+} response remained detectable at all investigated concentration ratios, indicating that the modified electrode retained its analytical capability for Cu^{2+} determination in the presence of Pb^{2+}. The persistence of the Cu^{2+} signal suggests that the interference caused by Pb^{2+} did not completely suppress the electrochemical response of the target ion under the studied conditions. The retained electrochemical response may be associated with the presence of MWCNTs, which provide conductive pathways and a large electroactive surface area that can facilitate electron-transfer processes during electrochemical measurements. The incorporation of MWCNTs into modified electrodes has been reported to enhance conductivity and charge-transfer efficiency, thereby improving the electrochemical detection of heavy metal ions (Mourya et al., 2021).

CONCLUSION

A $NiAl_2O_4$/MWCNT-modified glassy carbon electrode derived from napa soil was successfully developed for Cu^{2+} determination. The combination of $NiAl_2O_4$ and MWCNTs improved the electrochemical properties of the electrode and enhanced its voltammetric response toward Cu^{2+} ions. The modified electrode also demonstrated acceptable selectivity in the presence of Pb^{2+}, highlighting its potential for future environmental monitoring applications.

RECOMMENDATION

Further research is required to evaluate the analytical performance of the developed $NiAl_2O_4$/MWCNT-modified GCE, particularly in terms of detection limit, sensitivity, reproducibility, and long-term stability. The selectivity of the sensor should also be examined against a wider range of potentially interfering metal ions. In addition, validation using real environmental water samples and comparison with conventional analytical techniques are necessary to assess the practical applicability of the proposed sensing platform.

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