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$NiAl_2O_4$ -Modified Glassy Carbon Electrode From Napa Soil For Pb^{2+} Ions Using Voltammetry

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Abstract
Lead (Pb^{2+}) is a toxic heavy metal that poses serious risks to environmental and human health, creating a need for sensitive and reliable detection methods. This study aimed to develop a glassy carbon electrode (GCE) modified with $NiAl_2O_4$ derived from Napa soil for the electrochemical detection of Pb^{2+} ions using voltammetric techniques. The electrochemical performance of the modified electrode was evaluated by cyclic voltammetry in 4 mM $K_3[Fe(CN)_6]$ containing 0,1 M KCl. The results showed that the $NiAl_2O_4$-modified GCE exhibited enhanced electrochemical activity compared with the bare GCE, as indicated by an increase in anodic peak current from 19.93 µA to 26.60 µA (33.5%) and cathodic peak current from 19.03 µA to 24.29 µA (27.6%), reflecting improved electron-transfer capability and a larger electroactive surface area. The modified electrode successfully detected Pb^{2+} ions, producing well-defined oxidation and reduction peaks at −0.32 V and −0.52 V, respectively. Among the supporting electrolytes investigated, 0,1 M KCl provided the highest current response, and increasing the KCl concentration from 0.05 M to 0,10 M further enhanced the electrochemical signal. In addition, the electrode demonstrated good repeatability during repeated measurements. These findings indicate that the $NiAl_2O_4$-modified GCE based on Napa soil is a promising, low-cost, and sensitive electrochemical sensor for Pb^{2+} detection with improved electrochemical performance.

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INTRODUCTION

Lead (Pb^{2+}) is one of the most hazardous heavy metal pollutants due to its toxicity, persistence, and bioaccumulation in living organisms(Shalaby et al., 2023). Long-term exposure to Pb^{2+} can cause neurological disorders, kidney damage, reproductive dysfunction, and developmental impairment in humans(Xiao et al., 2020). Consequently, the development of sensitive and reliable methods for Pb^{2+} detection is essential for environmental monitoring and public health protection.

Electrochemical techniques have emerged as attractive approaches for heavy metal detection because of their simplicity, low cost, rapid response, and high sensitivity toward heavy metal ions(Bard et al., 2001). Voltametric techniques such as cyclic voltammetry (CV) and linear sweep voltammetry (LSV) are widely employed for the determination of Pb^{2+} because they provide accurate electrochemical information and enable trace-level analysis(Shalaby et al., 2023).

Glassy carbon electrodes (GCE) are frequently used in electrochemical sensing owing to their wide potential window, low background current, excellent chemical stability, and mechanical strength(Bard et al., 2001). However, bare GCEs often exhibit limited sensitivity and relatively slow electron-transfer kinetics, resulting in lower analytical performance for heavy metal detection (Xiao et al., 2020). Therefore, electrode surface modification is necessary to improve the electro-

chemical response and sensing capability.

Metal oxide materials have been extensively investigated as electrode modifiers because they can enhance conductivity, increase electroactive surface area, and provide catalytic active sites for electron-transfer reactions. Nickel aluminate ($NiAl_2O_4$), a spinel-type oxide, exhibits excellent thermal stability, chemical resistance, and electrocatalytic properties, making it a promising material for electrochemical sensing applications (Arunkumar & Nesaraj, 2020; Hammami et al., 2024; Miazga et al., 2016).

The presence of nickel species within the spinel structure can facilitate charge transfer, while alumina contributes to structural stability and surface porosity (Rangaswamy et al., 2024). In recent years, natural clay materials have attracted attention as low-cost precursors for functional electrode materials. Napa soil contains aluminosilicate components that can be converted into porous metal oxide structures with high surface area and adsorption capacity (Ilmi & Mawardi, 2023). The utilization of Napa soil as a source material for $NiAl_2O_4$ synthesis not only reduces production costs but also promotes the use of environmentally friendly and sustainable resources.

Despite the growing interest in metal-oxide-modified electrodes for heavy metal sensing, studies on the use of naturally derived $NiAl_2O_4$ materials remain scarce. In particular, the electrochemical performance of $NiAl_2O_4$ synthesized from Napa soil as a glassy carbon electrode modifier for Pb^{2+} detection has not been systematically investigated. Information regarding its ability to enhance electron-transfer processes, improve electroactive surface area, and optimize sensing performance is still limited. Therefore, this study introduces Napa-soil-derived $NiAl_2O_4$ as a sustainable and low-cost electrode modifier and systematically evaluates its application for Pb^{2+} detection, providing new insights into the utilization of locally available natural resources for electrochemical sensing.

Although numerous modified electrodes have been reported for Pb^{2+} detection(Zhang et al., 2020), studies concerning the application of Napa-soil-derived $NiAl_2O_4$ as a modifier for glassy carbon electrodes are still limited. Therefore, the development of a $NiAl_2O_4$-modified GCE based on Napa soil represents a novel approach for improving the electrochemical detection of Pb^{2+} ions.

This study aims to evaluate the electrochemical characteristics of a $NiAl_2O_4$-modified glassy carbon electrode derived from Napa soil and investigate its performance for Pb^{2+} detection using voltametric techniques. The effects of electrode modification, supporting electrolyte type, and supporting electrolyte concentration were systematically studied to determine the optimum conditions for Pb^{2+} analysis.

METHOD

Instruments and Materials

A potentiostat connected to a standard three-electrode system comprising 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 was used for all electrochemical measurements. An ultrasonic bath was used for electrode cleaning and suspension preparation.

The materials used in this study included NiAl2O4derived 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 (NaNO3), sodium sulfate (Na_2SO_4), sodium chloride (NaCl), sodium acetate (CH3COONa), hydrochloric acid (HCl), lead (II) nitrate ($Pb(NO_3)_2$), and deionized water.

Preparation of Non-Modified GLASSY Carbon Electrode (GCE).

The GCE surface was polished using α-alumina slurry (0,05 μm) on a polishing cloth until a mirror-like surface was achieved before modification. The electrode was then thoroughly cleaned with deionised water, sonicated in deionised water for three minutes to get rid of any remaining particles, and then allowed to dry at room temperature.

Preparation of Glassy Carbon Electrode (GCE) modified $NiAl_2O_4$.

The GCE was polished with α-alumina powder until the electrode surface was shiny. After sonication for 3 minutes, 0,176 g of NiAl2O4 was dispersed in 1 ml of water. Then, 5 μL was drop-cast onto the GCE surface. The

Napa earth-based $NiAl_2O_4$-modified GCE was then ready for use.

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}. The scan rate was chosen to provide a balance between signal sensitivity and peak resolution while minimizing capacitive current effects.

Repeatability

The repeatability of the $NiAl_2O_4$-modified glassy carbon electrode (GCE) was evaluated through five consecutive measurements of 1 mM Pb^{2+} solution under the optimum experimental conditions. The measurements were carried out using the same electrode in 0,1 M KCl as the supporting electrolyte and a scan rate of 100 mV s^{-1}. The oxidation peak current obtained from each measurement was recorded and compared to assess the consistency of the electrochemical response. The repeatability of the sensor was determined based on the relative change in peak current during repeated measurements. A small variation in current response indicates good repeatability and reliability of the modified electrode for Pb^{2+} detection.

Pb^{2+} Detection and Electrolyte Optimization

The Pb^{2+} detection study was conducted using a $Pb(NO_3)_2$ solution with a concentration of 1 mM. Electrochemical measurements were carried out under the same conditions as described above. To evaluate the effect of supporting electrolytes, different electrolytes (KCl, NaCl, CH_3COONa, $NaNO_3$, and KNO_3) with a concentration of 0,1 M were used. The current responses obtained for each electrolyte were compared to determine the optimal supporting electrolyte for Pb^{2+} detection. After identifying the most suitable supporting electrolyte, its concentration was varied to find the optimum starting from 0,05M ; 0,06M ; 0,07M ; 0,08M ; 0,09M ; 0,1M.

The concentrations of the redox probe and supporting electrolyte, as well as the scan rate, were selected based on commonly reported electrochemical characterization conditions and preliminary experiments that provided stable voltammetric responses and good peak resolution.

RESULTS AND DISCUSSION

Electrochemical Response of Modified Electrodes

Pb^{2+} ions detection using a modified Glassy Carbon Electrode with a modified nickel and alumina base based on Napa soil

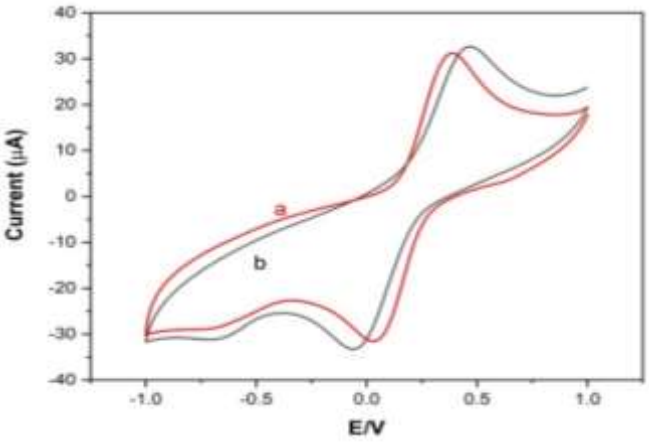

Figure 1. Cyclic Voltammograms of (a) Unmodified GCE electrode (b) Modified GCE electrode $NiAl_2O_4$ in $K_3[Fe(CN)_6]$ in 4,0 mM containing 0,1 M KCl. Scan rate 100 mVs^{-1}.

Table 1. Peak Current of Unmodified GCE electrode

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

Table 2. Peak Current of Unmodified GCE electrode (Modification $NiAl_2O_4$)

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

The electrochemical characteristics of the bare glassy carbon electrode (GCE) and $NiAl_2O_4$-modified GCE prepared from Napa clay were evaluated using cyclic voltammetry (CV) in a solution containing 4 mM $K_3[Fe(CN)_6]$ and 0,1 M KCl as the supporting electrolyte. The $Fe(CN)_6^{3-}/Fe(CN)_6^{4-}$ redox couple is commonly employed as an electrochemical probe to assess electron transfer kinetics and the electroactive surface area of modified electrodes(Bard et al., 2001).

As shown in Figure 1, both electrodes exhibited well-defined oxidation and reduction peaks corresponding to the reversible redox reaction of the ferri/ferrocyanide couple. However, significant differences were observed

in the peak current response after modification with $NiAl_2O_4$. The bare GCE exhibited an anodic peak current (Ipa) of 19.93 μA at 0.27 V and a cathodic peak current (Ipc) of −19.03 μA at −0.049 V. In contrast, the $NiAl_2O_4$-modified GCE displayed higher peak currents, with Ipa and Ipc reaching 26.60 μA and −24.29 μA, respectively. The anodic and cathodic peak currents increased by approximately 33.5% and 27.6%, respectively, after modification. This enhancement indicates that the $NiAl_2O_4$ layer effectively improved the electroactive surface area and facilitated electron transfer between the redox probe and the electrode surface. According to the Randles–Ševčík equation, the peak current is directly proportional to the electroactive surface area under diffusion-controlled conditions; therefore, the increase in peak current suggests successful enlargement of the accessible electrochemical surface(Bard et al., 2001).

The improved electrochemical performance can be attributed to the unique properties of the $NiAl_2O_4$ spinel structure. $NiAl_2O_4$ possesses good chemical stability and electrocatalytic activity, while the incorporation of nickel species provides additional active sites that promote charge transfer processes. Furthermore, the porous structure derived from Napa clay contributes to a larger specific surface area, allowing more electroactive species to interact with the electrode surface during the redox process. Consequently, the modified electrode exhibits enhanced conductivity and faster electron transfer kinetics compared to the unmodified GCE(Muhammad et al., 2020).

Furthermore, the improved electrochemical response can be attributed to the spinel structure of $NiAl_2O_4$. Nickel aluminate possesses good chemical stability and electrocatalytic properties, while nickel species within the spinel framework provide additional active sites for electron-transfer reactions. When deposited onto the GCE surface, $NiAl_2O_4$ nanoparticles create a rougher and more porous surface morphology, increasing the contact area between the electrolyte and electrode surface. As a result, electron transport becomes more efficient, leading to enhanced redox currents. Similar improvements in current response and sensing performance have been reported for $NiAl_2O_4$-modified GCEs used in electrochemical sensing applications (Rangaswamy et al., 2024). In addition, the voltammogram of the modified electrode exhibited a more pronounced and stable redox profile than the bare GCE, indicating improved reversibility of the $Fe(CN)_6^{3-}/Fe(CN)_6^{4-}$ redox system. This behavior suggests that the presence of $NiAl_2O_4$ reduces the resistance to charge transfer and promotes faster electron exchange between the redox probe and the electrode surface. Enhanced electron-transfer kinetics are highly desirable in electrochemical sensors because they contribute to higher sensitivity, better signal reproducibility, and lower detection limits.

Overall, the CV results confirm the successful modification of the GCE surface with $NiAl_2O_4$ derived from Napa clay. The significant increase in oxidation and reduction peak currents demonstrates that the modification improves the electroactive surface area, conductivity, and electron-transfer capability of the electrode. These characteristics are expected to facilitate the accumulation and electrochemical detection of Pb^{2+} ions, thereby improving the analytical performance of the developed sensor

Pb^{2+} Ion Detection with Modified GCE Electrode

Pb^{2+} ions detection using a modified Glassy Carbon Electrode with a modified nickel and alumina base based on Napa soil. The purpose of this modification is to increase the sensitivity of the electrode. After testing the modified electrode, Pb^{2+} ions can be detected, which can be obtained

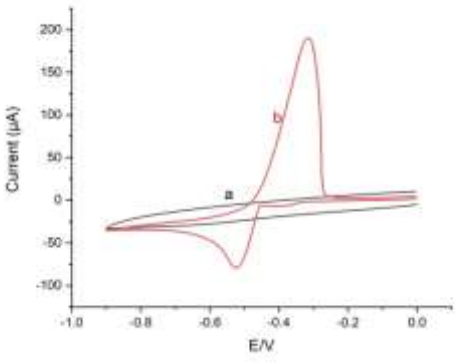

Figure 2. Cyclic Voltammograms of (a) Background KCl 0,1M (b) Analyte Pb^{2+} 1mM in 0,1M KCl

Table 3. Peak Current of 0,1M KCl background solution containing 1 mM Pb^{2+}

Oxidation	Reduction
Ipa = 186,23 μA	Ipc = -57,56 μA
E= -0,32 V	E= -0,52 V

Based on the cyclic voltammogram in figure 2, a significant difference is seen between the 0,1M KCl background solution containing 1 mM Pb^{2+}. The KCl background curva shows a relatively stable current without any oxidation or reduction peaks, indicating that KCl only functions as a supporting electrolyte and does not provide a redox response in the measurement potential range(Alshawi et al., 2022). After the addition of 1 mM Pb^{2+} ions, a pair of oxidation and reduction peaks appeared, indicating the occurrence of the Pb^{2+}/Pb^{0} electron transfer process on the electrode surface. The oxidation peak was obtained at a potential of −0.32 V with a current of 186.23 μA, while the reduction peak appeared at a potential of −0.52 V with a current of −57.56 μA. The appearance of these peaks indicates that Pb^{2+} ions were successfully detected electrochemically through a redox reaction on the electrode surface(Shalaby et al., 2023).

The reduction and oxidation reactions of Pb^{2+} that occur can be written as follows:

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

In the negative potential sweep process, Pb^{2+} is reduced to Pb^{0}, which is deposited on the electrode surface. Meanwhile, in the reverse potential sweep, Pb^{0} is oxidized to Pb^{2+}, which is returned to the solution(Riyanto, 2014). The higher oxidation current value compared to the reduction current indicates that the electrode has good electrocatalytic activity towards Pb^{2+} ions. In addition, the large difference in oxidation and reduction peak potentials (ΔEp) indicates that the system is quasi-reversible, which indicates the existence of limitations in electron transfer kinetics on the electrode surface(Sun et al., 2018).

In this context, the electrochemical response obtained is strongly influenced by the use of a glassy carbon electrode (GCE) modified with Ni–Al_2O_3 material derived from Napa soil (natural clay). This modification increases the active surface area of the electrode and provides additional active sites from Ni species and the porous Al_2O_3 structure obtained from the activation of Napa soil(Ilmi & Mawardi, 2023), thereby enhancing the adsorption and accumulation of Pb^{2+} ions on the electrode surface. The presence of Ni particles also acts as catalytic centers that accelerate electron transfer in the Pb^{2+}/Pb^{0} redox system, resulting in an increased peak current intensity compared to the unmodified electrode.

The increase in peak current intensity in the Pb^{2+} solution compared to the KCl blank indicates that the diffusion process of Pb^{2+} ions toward the electrode surface proceeds effectively. According to Shalaby et al., (2023), an increase in the active surface area and conductivity of electrode materials can enhance the sensitivity of sensors toward heavy metal ions such as Pb^{2+}. In this case, the combination of GCE/Ni–Al_2O_3 based on Napa soil significantly contributes to the improvement of the electrochemical response. Thus, the obtained voltammogram demonstrates that the Ni–Al_2O_3-modified GCE based on Napa soil is capable of effectively detecting Pb^{2+} ions through the formation of distinct oxidation and reduction peak pairs. In addition, 0,1 M KCl solution has been proven to be an effective supporting electrolyte, as it does not interfere with the electrochemical response of Pb^{2+}.

Repeatability

The repeatability of the $NiAl_2O_4$-modified glassy carbon electrode was evaluated to determine its ability to maintain a consistent electrochemical response during repeated measurements of Pb^{2+} ions. Repeatability is an important parameter in electrochemical sensor development because it reflects the reliability and durability of the electrode performance over time. The repeatability test was conducted under optimum experimental conditions using repeated measurements of Pb^{2+} solution, and the obtained voltammograms are presented in Figure 3.

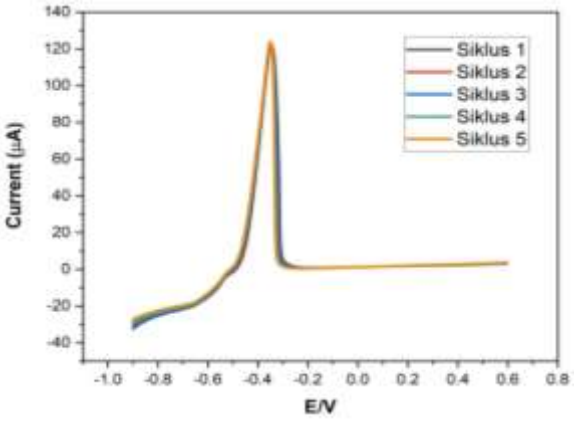

Figure 3. Voltammogram of the LSV method of the $Pb(NO_3)_2$ analyte stability test in 0,1M KCl supporting solution (a) cycle 1 (b) cycle 2 (c) cycle 3 (d) cycle 4 (e) cycle 5

Sensor repeatability is a critical parameter in evaluating electrochemical sensor performance because it indicates the electrode's ability to produce a consistent response during repeated measurements. In this study, the repeatability of a modified $NiAl_2O_4$ electrode was tested using the Linear Sweep Voltammetry (LSV) method against a Pb^{2+} solution in 0,1 M KCl medium for five consecutive measurement cycles.

The voltammograms in Figure 3 show that the measurement curves from the first to the fifth cycle nearly overlap. The peak current generated in each cycle is in the relatively similar range, approximately 120–125 μA, with very little peak potential shift. These results indicate that the electrode has good reproducibility and repeatability during repeated measurements.

The absence of a significant decrease in peak current indicates that the electrode surface has not experienced significant fouling or passivation due to adsorption of reaction products or accumulation of Pb^{2+} ions on the electrode surface. This condition indicates that the $NiAl_2O_4$ material has good chemical and electrochemical repeatability, allowing it to maintain its electrocatalytic activity throughout the measurement process (Rangaswamy et al., 2024).

Furthermore, the similarity in the voltammogram shape during each cycle indicates that the electron transfer process between the Pb^{2+} ions and the electrode surface occurs consistently. This repeatability is related to the presence of the spinel structure of $NiAl_2O_4$, which has high thermal and chemical stability, making it less susceptible to degradation during repeated electrochemical processes .The relatively constant peak current also indicates that active sites on the electrode surface remain available to interact with Pb^{2+} ions throughout the five measurements. This indicates that the modified material is able to maintain the active surface area and electron transfer efficiency required for heavy metal ion detection (Muhammad et al., 2020).

Overall, the repeatability test results show that the $NiAl_2O_4$ modified GCE electrode has excellent stability in detecting Pb^{2+} ions in 0,1 M KCl media. The overlapping of the voltammogram curves from the first cycle to the fifth cycle and the absence of a significant decrease in peak current prove that the sensor has good repeatability and has the potential to be used for repeated Pb^{2+} analysis applications with a high level of reliability.

Supporting Electrolyte

To investigate the effect of supporting electrolyte type on the electrochemical response of Pb^{2+} ions, cyclic voltammetry measurements were performed. The supporting electrolyte plays a crucial role in electrochemical systems as it enhances solution conductivity, stabilizes the electric field within the electrochemical cell, and minimizes ion migration effects, thereby ensuring that the observed current is primarily governed by diffusion-controlled processes. In this study, several supporting electrolytes were used, namely 0,1 M KCl, 0,1 M NaCl, 0,1 M KNO_3, 0,1 M $NaNO_3$, and 0,1 M CH_3COONa, to evaluate the influence of ionic composition on the Pb^{2+} peak current response.

Differences in the cation and anion composition of each supporting electrolyte are expected to significantly influence electron transfer processes, electrochemical system stability, and the diffusion rate of Pb^{2+} ions toward the electrode surface. Therefore, a comparative analysis of the cyclic voltammograms obtained in different supporting electrolytes is essential to determine the optimal condition that yields the best electrochemical response.

The following figure presents the cyclic voltammograms of Pb^{2+} ions recorded in different supporting electrolytes used in this study.

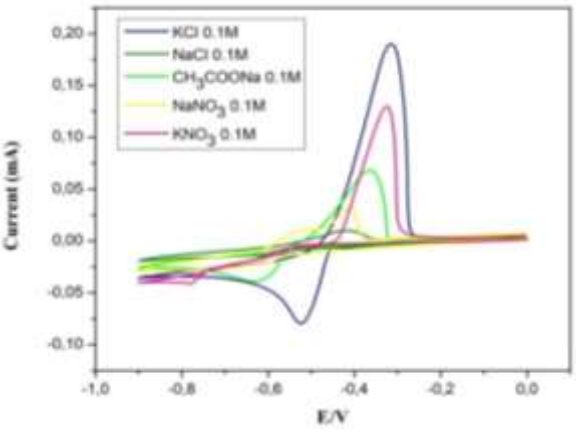

Figure 4. Cyclic voltammograms of supporting variations for the analyte Pb^{2+} (a) Supporting KCl 0,1M (b) Supporting KNO_3 0,1M (c) Supporting CH_3COONa 0,1M (d) Supporting $NaNO_3$ 0,1M (d) Supporting NaCl 0,1M

Based on the cyclic voltammogram in Figure 4, it can be observed that variations in the type of supporting electrolyte significantly influence the electrochemical response of Pb^{2+} ions. The supporting electrolytes used, namely 0,1 M KCl, 0,1 M NaCl, 0,1 M CH_3COONa, 0,1 M $NaNO_3$, and 0,1 M KNO_3, produced different curve shapes and peak current magnitudes. These differences indicate that both the cationic and anionic species in the supporting electrolyte affect electron transfer processes, solution conductivity, and the diffusion of Pb^{2+} ions toward the electrode surface (Shalaby et al., 2023)

In the voltammogram, 0,1 M KCl produced the highest oxidation and reduction peak currents compared to the other supporting electrolytes. The high peak current indicates that KCl enhances solution conductivity and accelerates electron transfer at the electrode surface. K^+ ions have high ionic mobility and are chemically inert, meaning they do not readily interact with either the electrode surface or Pb^{2+} ions. In addition, Cl^- ions help maintain ionic stability during the redox process. Therefore, KCl is commonly used as a supporting electrolyte in heavy metal voltammetric analysis because it provides higher sensitivity and better signal stability (Wang et al., 2023).

The 0,1 M KNO_3 solution also showed a relatively high current response, although still lower than that of KCl. This is attributed to NO_3^- ions having good ionic transport properties but being less effective than Cl^- in maintaining stable electron transfer processes at the electrode surface. Nevertheless, KNO_3 is still widely used as a supporting electrolyte due to its inert nature and its resistance to side reactions within certain potential ranges (Alshawi et al., 2022). For 0,1 M NaCl, a lower peak current was obtained compared to KCl. This difference is influenced by the type of cation used, namely Na^+. The ionic mobility of Na^+ is lower than that of K^+, resulting in less efficient ion migration in the solution. Consequently, the electron transfer rate and the sensitivity of Pb^{2+} detection are also reduced.

The 0,1 M CH_3COONa supporting electrolyte produced relatively smaller currents and less sharp peak shapes. This may occur because acetate ions (CH_3COO^-) can partially interact with Pb^{2+} ions to form weak complexes, reducing the concentration of free Pb^{2+} ions available for redox reactions at the electrode surface. The decrease in free Pb^{2+} ions leads to lower peak currents compared to KCl and KNO_3 (Shalaby et al., 2023). Meanwhile, 0,1 M $NaNO_3$ exhibited the lowest current response among all the supporting electrolytes used. The combination of Na^+ and NO_3^- results in suboptimal conductivity for Pb^{2+} electron transfer processes. This indicates that the selection of a supporting electrolyte is not only influenced by ionic strength but also by the ability of its constituent ions to support electrochemical system stability and facilitate charge transfer (Alshawi et al., 2022).

In general, supporting electrolytes are selected due to several important roles in voltammetric systems, namely increasing solution conductivity, reducing solution resistance (ohmic drop), maintaining electrode potential stability, and accelerating ion transport during redox processes. Supporting electrolytes must also be inert, not react with the analyte or electrode surface, and remain stable within the measurement potential range (Bard et al., 2001).

Thus, the voltammetric results demonstrate that 0,1 M KCl is the most suitable supporting electrolyte for Pb^{2+} detection, as it produces the highest peak current and the clearest redox peak shape. This indicates that KCl provides the most optimal electrochemical environment for electron transfer processes of Pb^{2+} at the electrode surface.

Effect of KCl Concentration on the Electrochemical Response of Pb^{2+}

The effect of KCl concentration as a supporting electrolyte on the electrochemical response of Pb^{2+} was investigated using Linear Sweep Voltammetry (LSV), as shown in Figure 5.

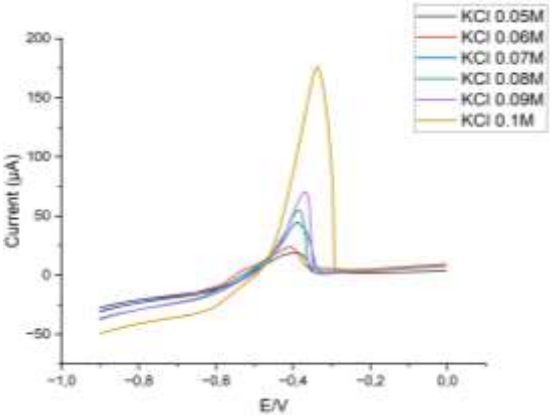

Figure 5. Voltammogram of LSV method of KCl concentration variation for analyte Pb^{2+} (a) KCl concentration 0,1M (b) KCl concentration 0,09M (c) KCl concentration 0,08M (d) KCl concentration 0,07M (e) KCl concentration 0,06M (f) KCl concentration 0,05M

Various KCl concentrations, namely 0,05, 0,06, 0,07, 0,08, 0,09, and 0,10 M, were evaluated to determine the optimum electrolyte concentration for Pb^{2+} detection. The results demonstrate that increasing the KCl concentration significantly enhances the current response and improves the shape of the voltammetric peak.

As shown in Figure 5, the peak current gradually increased as the KCl concentration increased from 0,05 to 0,10 M. The lowest current response was observed at 0,05 M KCl, whereas the highest peak current, approximately 175 μA, was obtained at 0,10 M KCl. The increase in peak current indicates that a higher concentration of supporting electrolyte improves the conductivity of the solution and facilitates the electrochemical reaction of Pb^{2+} at the electrode surface.

The role of the supporting electrolyte is to provide sufficient ionic strength, minimize solution resistance (ohmic drop), and suppress the migration effect of electroactive species, allowing mass transport to be dominated by diffusion. Consequently, electron transfer between Pb^{2+} ions and the electrode surface becomes more efficient, resulting in a stronger analytical signal (Muhammad et al., 2020). Therefore, the increase in current response observed with increasing KCl concentration can be attributed to the enhanced charge-transfer process and improved ionic conductivity of the electrolyte solution.

At lower KCl concentrations (0,05–0,07 M), relatively broad peaks with lower current intensities were observed. This behavior suggests that the ionic conductivity of the solution was insufficient to support efficient electron-transfer kinetics. Under these conditions, the transport of Pb^{2+} ions toward the electrode surface becomes less effective, resulting in lower electrochemical activity and reduced sensitivity of the sensor (Zaib & Athar, 2017).

A significant increase in peak current was observed at KCl concentrations of 0,08 and 0,09 M, indicating improved electrochemical conditions for Pb^{2+} oxidation or reduction. However, the optimum response was achieved at 0,10 M KCl, where the highest and sharpest voltammetric peak was obtained. This result suggests that 0,10 M KCl provides the most favorable environment for charge transport and electron-transfer reactions at the electrode/electrolyte interface.Furthermore, the enhanced response at 0,10 M KCl may also be associated with the improved performance of the $NiAl_2O_4$-modified electrode. Nickel aluminate possesses good electrocatalytic properties and provides additional active sites that facilitate electron-transfer reactions. Combined with adequate electrolyte conductivity, the modified electrode exhibits a higher current response and improved electrochemical sensitivity (Rangaswamy et al., 2024).

Overall, the LSV results demonstrate that KCl concentration plays a crucial role in the electrochemical detection of Pb^{2+}. Among the investigated concentrations, 0,10 M KCl produced the highest peak current and the most defined voltammetric profile, indicating that it is the optimum supporting electrolyte concentration for Pb^{2+} determination using the $NiAl_2O_4$-modified GCE.

CONCLUSION

The $NiAl_2O_4$-modified glassy carbon electrode (GCE) derived from Napa soil exhibited improved electrochemical performance compared with the unmodified electrode. Cyclic voltammetry characterization using the $Fe(CN)_6^{3-}/Fe(CN)_6^{4-}$ redox probe showed that the anodic peak current increased from 19.93 μA to 26.60 μA (33.5%), while the cathodic peak current increased from 19.03 μA to 24.29 μA (27.6%), indicating enhanced electron-transfer capability and electroactive surface area. The modified electrode successfully detected Pb^{2+} ions, producing well-defined oxidation and reduction peaks associated with the Pb^{2+}/Pb^0 redox process.

The supporting electrolyte significantly influenced the electrochemical response, with 0,1 M KCl providing the highest peak current and the most distinct voltammetric profile. Increasing the KCl concentration from 0.05 M to 0,10 M further enhanced the current response, with the optimum performance obtained at 0,10 M KCl. These findings demonstrate that the $NiAl_2O_4$-modified GCE based on Napa soil is a promising low-cost electrochemical sensor for Pb^{2+} detection, offering improved sensitivity and electrochemical performance under optimized conditions.

RECOMMENDATION

Although the $NiAl_2O_4$-modified glassy carbon electrode exhibited good electrochemical

performance and satisfactory repeatability toward Pb^{2+} detection, further studies are recommended to evaluate its long-term storage stability and performance in real environmental samples. In addition, investigations on the selectivity of the electrode in the presence of other metal ions and optimization of operational parameters such as pH and scan rate are necessary to further improve the sensitivity and practical applicability of the developed sensor.

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