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RESEARCH PAPER

Development of a Cr(VI) metal ion detection sensor using a modified pencil lead electrode (PLE) with a silver thin layer using the voltammetry method

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Abstract: Chromium(VI) metal ions have harmful effects on the environment and organisms. Chromium(VI) particles can cause chromosomal abnormalities, cross-linking, disruption of the cell cycle, and DNA damage within cells. Therefore, due to its high toxicity, it is crucial to monitor the concentration of chromium(VI) metal ions in the environment. This study aims to detect Cr(VI) metal ions in a simple, rapid, and effective manner. Electrochemical methods are employed to investigate the performance of PLE and Ag/PLE electrodes and to determine the optimal conditions for electrodeposition cycles and supporting electrolyte concentrations in detecting Cr(VI) metal ions. The electrochemical method is based on the specific reaction of the analyte, which generates an electrical signal. The PLE surface is modified with a silver thin layer through electrodeposition using cyclic voltammetry. The silver thin layer used for electrode modification offers several advantages, including improved electron transport and enhanced electrocatalytic response. The results indicate that the performance of Ag/PLE is superior to that of PLE in detecting Cr(VI) metal ions under optimal supporting electrolyte conditions, specifically 0.1 M HNO_3 with one electrodeposition cycle. Calibration curve analysis for Cr(VI) ions yielded the regression equation y = 0.6688x + 0.0085, with a correlation coefficient (R^2) of 0.9958 and a limit of detection (LOD) of 0.18 mM.

Keywords: ion logam Cr(VI), Pencil Lead electrode, Silver Thin layer, Voltametri

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INTRODUCTION

Chromium is a d-block transition metal that can in various oxidation states. The oxidation states of chromium range from -2 to +6, however, Cr(III) and Cr(VI) are the thermodynamically stable forms found in nature [1]. The toxicity of Cr(VI) is significantly higher than that of Cr(III). While Cr(III) in small amounts, plays an important role in metabolic processes, Cr(VI) exhibits toxic, mutagenic, and carcinogenic effects within cells [2]. At physiological pH, Cr(VI) is present as tetrahedral chromate anions, similar in structure to phosphate and sulfate, which allows them to easily cross cell membranes. In contrast, Cr(III) cannot readily penetrate cell membranes due to its octahedral complex structure. This structural difference explains why Cr(III) is less prevalent in the environment compared to Cr(VI), as Cr(VI) is mainly released through industrial activities, including electroplating, leather tanning, wood polishing and preservation, and as a pigment in the production of synthetic gems and jewelry [3]. Cr(VI) particles can cause chromosomal abnormalities, cross-linking, disruption of the cell cycle, and DNA damage within cells [4]. Therefore, monitoring Cr(VI) concentrations in water bodies is crucial for public health.

Various analytical techniques are used to detect Cr(VI), such as atomic absorption spectroscopy, UV-Vis spectroscopy, and ICP-MS. These methods have high sensitivity, but they require sophisticated, expensive, and time-consuming techniques in the analysis process [2]. Therefore, a fast and effective method is needed to detect heavy metal ions, such as electrochemical methods [5]. Electrochemical methods are analytical methods that involve the reaction of analytes to produce an electrical signal as output. This method has several advantages, including simplicity, sensitivity, and low cost [2]. One electrochemical method for detecting heavy metal ions is voltammetry [5], such as Cyclic Voltammetry (CV) and Linear Sweep Voltammetry (LSV) [6]. The CV method can provide rapid information about the redox reaction of the analyte [7]. Meanwhile, LSV is a method that can achieve high sensitivity with a small background current [8].

The pencil lead electrode (PLE) is a carbon-based electrode used in this study. PLE consists of graphite (75–80%), organic binder (13%), and spindle oil (8%) [7]. The graphite surface of the PLE is the active site that plays a crucial role in electrochemical reactivity. Type B pencils have a higher graphite content compared to type H pencils. The more graphite particles present, the more conductive the PLE surface becomes [9]. PLE has several advantages, including high electrical conductivity, simplicity, ease of availability, low cost [10], and a wide potential range [11]. However, PLE requires modification to enhance its sensitivity and selectivity [2].

Previous studies have reported the use of PLE with various modifications for determining Cr(VI) ions via electrochemical techniques. For instance, Au/PLE electrodes were investigated using cyclic voltammetry (CV) [2], while PAni/DPC/PLE composites were also studied with CV [12]. Notably, silver has emerged as a viable material for electrode modification [13]. The silver thin layer used in such modifications offers multiple advantages, including improved electron transport, enhanced electrocatalytic response [14], and increased resistance to surface contamination [15].

In this study, Cr(VI) metal ions were detected using PLE modified with a silver thin layer by voltammetry using HNO_3 as the supporting electrolyte [16]. The use of HNO_3 as the supporting electrolyte was chosen because the Ag/PLE electrode exhibited better response compared to other supporting electrolyte solutions such as HCl, $HClO_4$, and H_2SO_4 in detecting Cr(VI) ions [16]. PLE is modified using the electrodeposition technique to enhance the catalytic surface of PLE [17]. Electrodeposition is a technique for depositing layers of modifying substances, such as metals, on the electrode surface when a potential is applied [6]. The advantages of this technique include its speed and the high purity of the particles [18]. This study offers a sensitive, fast, inexpensive, and simple analytical method.

MATERIALS AND METHODS

Materials

The materials used in this study included $K_2Cr_2O_7$ (Merck), $AgNO_3$ (Merck), HNO_3 (Merck), distilled water, and filter paper. The equipment used in this study includes a micropipette, chemical glassware, Teflon tubes, petri dishes, vials, and an e-DAQ EA163 potentiostat. The electrodes used consist of a working electrode, namely a Pencil Lead Electrode (PLE) coated with a thin layer of silver (Ag/PLE), a reference electrode Ag/AgCl, and an auxiliary electrode Pt.

Preparation of Ag/PLE using the electrodeposition method

The Pencil Lead Electrode (PLE) was modified with a silver thin layer using the electrodeposition method with a 5 mM $AgNO_3$ solution in a 0.1 M KNO_3 electrolyte solution, scanning the potential from +1.0 V to 0 V at a scan rate of 100 mV/s for one cycle. This electrode is known as a pencil lead electrode modified with a thin silver layer (Ag/PLE) [14].

Characterization of unmodified electrodes (PLE) and modified electrodes (Ag/PLE) for the detection of Cr(VI) metal ions using the Linear Sweep Voltammetry (LSV) technique

Electrochemical measurements of PLE and Ag/PLE toward 5 mM Cr(VI) ions were conducted in a supporting electrolyte of 0.1 M HNO_3. The measurements were performed using the LSV method with a scan potential from +1.6 V to -0.8 V at a scan rate of 100 mV/s.

Variation in Supporting Electrolyte Concentration

Variations in the concentration of HNO_3 supporting electrolyte in a 5 mM Cr(VI) ion solution were studied using the LSV method. Measurements were performed with a scan potential from +1.5 V to 0.5 V. The HNO_3 concentration variations included concentrations of 0.1, 0.01, and 0.001 M.

Calibration curve

The calibration curve was performed using a supporting electrolyte of 0.1 M HNO_3 with Cr(VI) ion concentrations of (0; 0.5; 1; 1.5; 2; 2.5) mM. Measurements were conducted using the LSV method with one electrodeposition cycle, a scan potential from +1 V to 0 V, and a scan rate of 100 mV/s.

Selectivity

The selectivity of Ag/PLE against other interfering ions such as Cu(II), Fe(III), Zn(II), Cd(II), and Pb(II) ions was carried out at the same concentration as for the detection of Cr(VI) ions, namely 5 mM. This measurement was performed in a supporting electrolyte of 0.1 M HNO_3 using the LSV method with a scan potential from +1.5 V to -0.5 V and a scan rate of 100 mV/s.

RESULTS AND DISCUSSION

Modification of PLE with a silver thin layer (Ag/PLE)

The silver thin layer used for PLE surface modification was prepared using a 5 mM $AgNO_3$ solution in 0.1 M KNO_3 via cyclic voltammetry with a scan potential from +1.0 V to 0 V and a scan rate of 100 mV/s for one cycle [14]. The electrodeposition voltammogram is shown in Figure 1

Based on the voltammogram in Figure 1, it can be seen that there are (i) reduction peaks and (ii) oxidation peaks from Ag/PLE. The occurrence of reduction and oxidation processes in the voltammogram indicates the success of the electrodeposition process [14]. The oxidation potential value obtained is 0.58 V with a current of 1.785 mA, while the reduction potential is 0.29 V with a current of -0.859 mA. PLE electrodeposition with a silver thin layer has been performed previously, showing that the oxidation potential is at +0.58 V and the reduction peak at +0.27 V [14]. The reactions involved in the electrodeposition process can be explained as follows:

i. Reduction : $Ag^{+}_{(aq)} + e^{-} \rightarrow Ag_{(s)}$
ii. oxidation : $Ag_{(s)} \rightarrow Ag^{+}_{(aq)} + e^{-}$

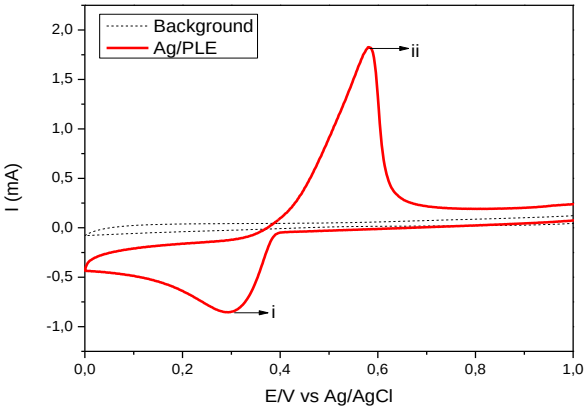

Figure 1. Cyclic voltamogram of electrodeposition of 5 mM $AgNO_3$ in 0.1 M KNO_3 on the surface of PLE with a scan rate of 100 mV/s

In this electrodeposition method, when a potential is applied, a metal-based modifier layer can be deposited on the electrode surface [6]. The advantages of modification using this electrodeposition method include low cost, ease of process [19] and high particle purity [18]. Meanwhile, surface modification of PLE with a thin silver layer can enhance the sensitivity of the electrode surface [20], improve electron transport, and enhance electrocatalytic response [14].

Characterization of unmodified electrodes (PLE) and modified electrodes (Ag/PLE) for the detection of Cr(VI) metal ions using the Linear Sweep Voltammetry (LSV) technique.

The characterization of Ag/PLE and PLE electrodes for Cr(VI) ions was performed using the LSV method. The LSV method can achieve higher sensitivity with minimal background current [8]. This study was conducted in a supporting electrolyte of 0.1 M HNO_3 against 5 mM Cr(VI) ions with a potential scan from +1.5 V to -0.5 V and a scan rate of 100 mV/s. The characterization results can be seen in Figure 2.

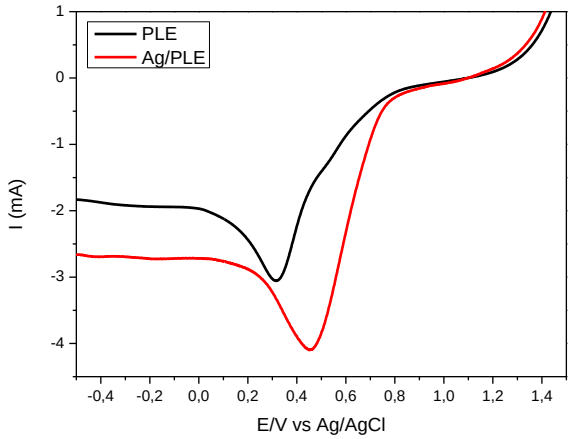

Figure 2. voltamogram LSV PLE and Ag/PLE of 5 mM Cr(VI) metal ions in 0.1 M HNO_3 at a scan rate of 100 mV/s.

Based on the voltamogram in Figure 2, it can be seen that Ag/PLE provides better electrode performance than PLE in detecting Cr(VI) ions. The increase in Ag/PLE detection current is due to the larger electrode surface area [21] and the electrocatalytic effect of Ag [14]. The shift in the reduction peak potential is caused by the electrocatalytic effect of the thin silver layer [2]. The reaction occurring on the electrode surface is

Reduction: $HCrO_4^- + 7H^+ + 3e \longrightarrow Cr^{3+} + 4H_2O$

oxidation: $Cr^{3+} + 4H_2O \longrightarrow HCrO_4^- + 7H^+ + 3e$

Effect of electrodeposition cycle

Variations in the Ag electrodeposition cycle on the PLE surface were investigated using a 5 mM Cr(VI) test solution in 0.1 M HNO_3 solution by cyclic voltammetry. Electrodeposition variations were performed with variations of 1 cycle, 5 cycles, and 10 cycles. The variations in the electrodeposition cycle can be seen in Figure 3.

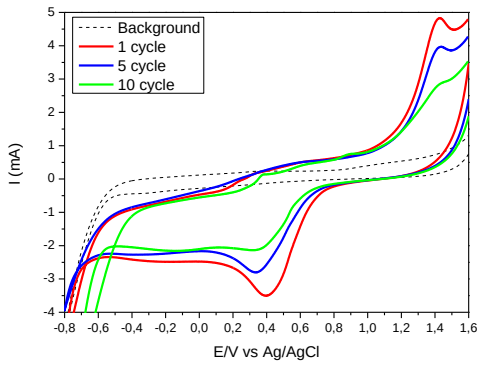

Figure 3. Voltammogram CV of Ag electrodeposition cycle variations in 0.1 M HNO_3

Based on the voltammogram in Figure 3 above, it can be seen that 1 cycle provides a higher current value compared to 5 cycles and 10 cycles. The 1-cycle variation results in a thin Ag layer on the PLE surface, while 5 and 10 cycles cause the formation of a thick Ag layer on the PLE surface [22]. The effectiveness of the electrode is inversely proportional to the thickness of the modification on the PLE surface [23]. As the Ag layer on the PLE surface becomes thicker, it reduces the active surface area of the electrode [24]. Therefore, the measured Cr(VI) current value decreases as the number of electrodeposition cycles increases.

Effect of supporting electrolyte concentration

In this test, variations in supporting electrolyte concentration were performed using the LSV method on 5 mM Cr(VI) ions. The use of HNO3 as the supporting electrolyte was chosen because the Ag/PLE electrode provides a better response compared to other supporting electrolyte solutions such as HCl, $HClO_4$, and H_2SO_4 in detecting Cr(VI) metal ions [16]. The variations in HNO_3 supporting electrolyte concentration consisted of 0.1 M, 0.01 M, and 0.001 M. Measurements were performed by scanning the potential from +1.5 V to 0.5 V at a scan rate of 100 mV/s. The voltammogram results from the variations in supporting electrolyte concentration can be seen in the figure 4.

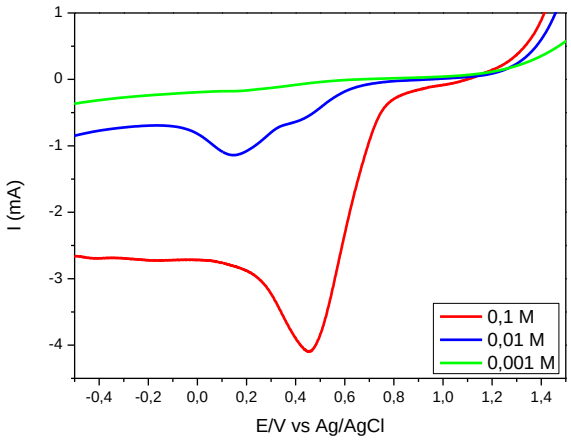

Figure 4. Voltammogram LSV Ag/PLE against variations in HNO_3 concentration with a scan rate of 100 mV/s.

Based on the voltammogram in Figure 4, it can be seen that HNO_3 with a concentration of 0.1 M shows a higher current compared to concentrations of 0.01 M and 0.001 M. Therefore, 0.1 M HNO_3 is the optimal

concentration for detecting Cr(VI) ions. This is because as the concentration of the supporting electrolyte increases, more hydrogen ions are involved in the reaction, thereby increasing the conductivity of the solution [25]. The shift in the cathodic peak potential may occur due to interference from the Oxygen Reduction Reaction (ORR). ORR causes a decrease in protons (H^+), which can reduce sensor response and result in a shift in the reduction potential [26].

Calibration curve

Calibration curves can be used to determine the linearity of a particular analytical method. In this study, calibration curve testing was performed at concentrations of 0; 0.5; 1; 1.5; 2; and 2.5 mM using LSV in a 0.1 M HNO_3 supporting electrolyte solution, with one electrodeposition cycle and a potential scan from +1.0 V to 0 V at a scan rate of 100 mV/s. The results of the calibration curve voltammogram can be seen in Figure 5.

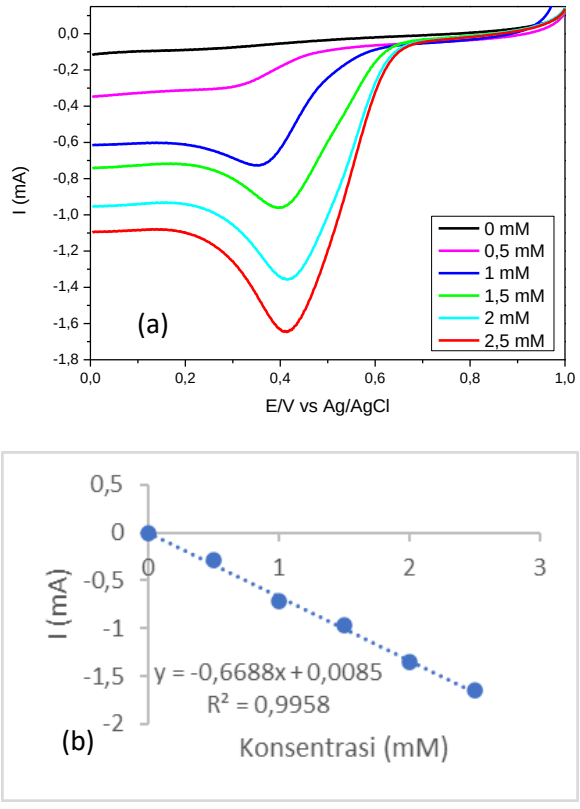

Figure 5. voltamogram LSV Ag/PLE with a scan rate of 100 mV/s (b) Cr(VI) calibration curve with concentrations ranging from 0 to 2.5 mM.

Based on the voltammogram in Figure 5 above, it can be seen that there is a linear relationship between the concentration of Cr(VI) ions and the current produced. This is because the concentration of the target compound is directly proportional to the strength of the current produced [27]. Therefore, the higher the Cr(VI) concentration, the greater the current generated. Based on the analysis data, the regression equation is y = 0.6688x + 0.0085, with a correlation coefficient (R^2) of 0.9958 and a limit of detection (LOD) of 0.18 mM.

Selectivity

Selectivity testing is very important in practical applications to ensure the sensor's ability to accurately identify and measure analytes [28]. The selectivity of Ag/PLE for the detection of Cr(VI) ions was investigated through testing with coexisting interfering ions such as Pb(II), Zn(II), Cd(II), Fe(III), and Cu(II). The selectivity of Ag/PLE toward other interfering ions can be seen in Figure 6.

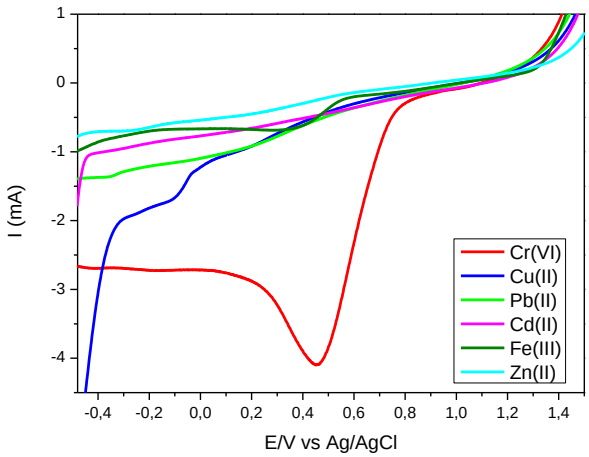

Figure 6. Voltammogram of Ag/PLE selectivity against interfering ions

Based on the analysis of the voltammogram in Figure 6, it can be seen that Ag/PLE does not show a significant response to Pb(II), Zn(II), Cd(II), Fe(III), and Cu(II) ions. The lack of response of Ag/PLE to these interfering ions identifies that the Ag/PLE sensor is selective for the detection of Cr(VI) ions compared to other ions [28].

CONCLUSION

In this study, PLE modified with a thin layer of silver by electrodeposition was investigated for the detection of Cr(VI) metal ions. Ag/PLE showed better performance than PLE for Cr(VI) detection. This is because silver can increase the sensitivity of the electrode and enhance the electrocatalytic

response. The Ag/PLE electrode was tested to determine the effects of electrodeposition cycles and supporting electrolyte concentration. Measurement of Cr(VI) ions under optimal conditions yielded a regression equation of y = 0.6688x + 0.0085 with a correlation coefficient (R^2) of 0.9958 and a limit of detection (LOD) of 0.18 mM.

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