E-ISSN:2685-8959P-ISSN:2685-8967
Journal of Research and Education Chemistry (JREC)
http://journal.uir.ac.id/index.php/jrec

ENHANCING Zn^{2+} METAL ION DETECTION : LEVERAGING SILVER-MODIFIED PENCIL LEAD ELECTRODE WITH CYCLIC VOLTAMMETRY METHOD

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Abstract

Zn^{2+} metal ions are heavy metals that are essential for the growth and development of organisms but if the amount of zinc (Zn) in excess can be toxic and disrupt the balance of the ecosystem. This research aims to study the electrochemical response of Ag/PLE to detect Zn^{2+} metal ions and determine the optimum conditions of supporting electrolyte and supporting electrolyte pH for the measurement of Zn^{2+} metal ions. In this study, electrochemical method was used to detect Zn^{2+} metal ion. The electrode was modified with a thin layer of silver by electrodeposition with the cyclic voltammetry method. The results showed that Ag/PLE gave a better response than PLE in detecting Zn^{2+} metal ions with acetate buffer solution with pH 4 as supporting electrolyte and optimum pH obtained the curve calibration with a correlation coefficient (R^2) of 0.995, limit of detection (LOD) of 0.44 mM and RSD value (%) of 4.9%.

Keywords : Ion Zn^{2+}, Pencil Lead Electrode, silver thin layer, cyclic voltammetry

Introduction

Zn^{2+} metal ions are heavy metals that are essential for the growth and development of organisms. One of its functions is to play a role in the formation of haemocyanin in the blood system and enzymatic processes (Kalangie et al., 2018). Heavy metal zinc (Zn) can be beneficial to the body if it is still within a reasonable threshold, which is 12-15 mg / day. If the amount of zinc (Zn) is excessive, it can be toxic and disrupt the balance of the ecosystem (Rosyidah & Rachmadiarti, 2023).

Zn^{2+} metal ions are usually sourced from industrial wastes. One of the industrial sectors that has the potential to produce Zn2+ metal ion waste is the rubber processing plant. In the study (Lestari et al., 2013) the liquid waste produced by the rubber processing plant from the inlet was tested and had a Zn level of 0.34 ppm. Zn levels exceed the quality standards that have been set. According to Government Regulation No. 82 of 2001, Zn levels in waters should not exceed 0.05 mg/L. If Zn levels exceed the effluent quality standard threshold, this can have a very detrimental impact on the environment and living things, in accordance with these regulations. Therefore, testing

for Zn2+ metal ions in the environment is required. One method that can be used is the electrochemical method (Lestari et al., 2013).

Heavy metal analysis generally uses spectrophotometric methods with instruments such as Atomic Absorption Spectrophotometry (AAS), Inductively Coupled Plasma Atomic Emission Spectrophotometry (ICP-AES), ICP Optical Emission Spectrophotometry (ICP-OES), and ICP Mass Spectrophotometry (ICP-MS). In addition, another commonly used method is chromatography. These methods are sensitive and accurate, but require high operational costs, large instruments and are not portable, making them difficult to carry into the field (Vasanthi Sridharan & Mandal, 2022). An alternative and more effective method needed to overcome the limitations of these methods can be done using electrochemical methods. The advantages of this method include a simpler analytical process, economical analytical equipment, a high level of sensitivity, and portable instruments that are easy to carry to the field (Sari et al., 2021).

One of the effective electrochemical methods for detecting Zn2+ metal ions is using the cyclic voltammetry method. This cyclic voltammetry provides rapid information about the redox reaction of the analyte compared to other electrochemical methods (Annu et al., 2020). This method provides analysis of various metals and provides both qualitative and quantitative information (Erlita & Setiarso, 2020) and has the advantages of affordable cost, high sensitivity, ease of operation, and the ability to analyze elemental speciation (Maharani et al., 2024).

This study uses Pencil Lead Electrode (PLE) as an electrode made of 80% carbon with the advantages of low cost, good electrical conductivity, more flexible, easy to find, use, and modify (David et al., 2017). In an effort to improve the performance of PLE, modification of the electrode surface is carried out, one of which is by using metals such as silver. Silver (Ag) is resistant to chemicals, has high electrical and thermal conductivity, good resistance to corrosion and has antimicrobial properties that can help inhibit the growth of microorganisms (Valov et al., 2020). These characteristics are the basis for modifying PLE using a thin layer of silver. The process of modifying PLE with a thin layer of silver is done through the electrodeposition method (Asadian et al., 2016). It has been reported in research (Palisoc et al., 2018) regarding the modification of pencil graphite electrodes using silver, bismuth, and nafion in the determination of Pb and Cd

metals. The results showed that the electrode was effective in detecting heavy metals and had high adhesion.

Based on this, in this study, for the first time, PLE was modified with a thin layer of silver to optimize the determination of Zn^{2+} metal ions at optimum conditions using the Cyclic Voltammetry method.

Method

Tools and Materials

The tools used in this study include beakers, micro pipettes, measuring flasks, Petri dishes, magnetic stirrers, spray bottles, 10 mL vials, 100 mL reagent bottles, Teflon tubes, Ag/AgCl reference electrodes, Pt auxiliary electrodes, e-DAQ potentiostat model EA163, Pentel pencil lead electrode (PLE) modified with Thin Silver Layer as a working electrode, and a set of computers. Materials used in this study include $Zn(NO_3)_2$, $AgNO_3$, KNO_3, $HClO_4$,HNO_3, Acetate Buffer, Phosphate Buffer, filter paper and distilled water.

Preparation of Ag/PLE by electrodeposition method

The PLE electrode was modified with a thin layer of silver via electrodeposition in a solution of 5 mM $AgNO_3$ and 0.1 M KNO_3. The potential on the electrode was applied from 1 V to -1 V with a scan rate of 100 mV/s for 1 cycle. This electrode is represented as Pencil Lead Electrode modified silver thin layer (Ag/PLE) (Radha & Sari, 2024).

Electrochemical Characterization of Zn^{2+} Metal Ions Using Non-Modified Electrode PLE and Modified Electrode Ag/PLE by Cyclic Voltammetry Method

Characterization was carried out by cyclic voltammetry using an analyte solution of 10 mM Zn^{2+} metal ion in a supporting electrolyte solution with a scan potential of +0.2 V to -2 V and a scan rate of 100 mV/s set on a potentiostat.

Optimization of Ag/PLE Electrodes as Zn^{2+} Metal Ion Sensors

a. Variation Supporting Electrolyte

Determination of optimum supporting electrolyte conditions was carried out by varying the electrolyte solution in the electrochemical cell, namely 0.1 M $HClO_4$ solution, 0.1 M HNO_3 solution, acetate buffer solution pH 4, and Phosphate buffer solution pH 7 in Zn^{2+} 10 mM test solution. Cyclic Voltammetry

method was applied to the measurement with scan potential set from +0.2 V to -2 V and a scan rate of 100 mV/s.

b. Supporting Electrolyte pH Variations

The pH variations carried out in the optimum electrolyte solution for the supporting electrolyte solutions including: pH variation of Acetate buffer solution: 4,5, dan 6 in the 10 mM Zn^{2+} test solution. The analysis was carried out by Cyclic Voltammetry method using an analyte solution of 10 mM Zn^{2+} metal ions in a supporting electrolyte solution with a scan potential of +0.2 V to -2 V and a scan rate of 100 mV/s set on a potentiostat

Calibration Curve

In the determination of concentration and calibration curve, Zn^{2+} metal ions were detected quantitatively by varying the concentration of Zn^{2+} (1 mM, 2 mM, 4 mM, 6 mM, and 8 mM) under favorable electrolyte conditions and the optimum pH of the electrolyte so as to obtain the detection limit value.

Results And Analysis

Electrochemical Response of Pencil Lead Electrodes modifiedsilver thin layer (Ag/PLE) by Electrodeposition Technique

In this study, modification of PLE with a thin layer of silver by electrodeposition with cyclic voltammetry method was carried out. Silver (Ag) deposited on the surface of PLE was served to increase the sensitivity of the electrode surface (Guimar et al., 2023). PLE modified with a thin layer of silver was carried out by the method of electrodeposition of $AgNO_3$ 5 mM in KNO_3 0.1 M solution in cyclic voltammetry with a scan potential range of+0.2 V to -2.0 V with a scan rate of 100 mV/s. The electrodeposition process involves the redox reaction of Ag as shown in the figure. This electrode is represented as Pencil Lead Electrode modified silver thin layer (Ag/PLE) (Radha & Sari, 2024).

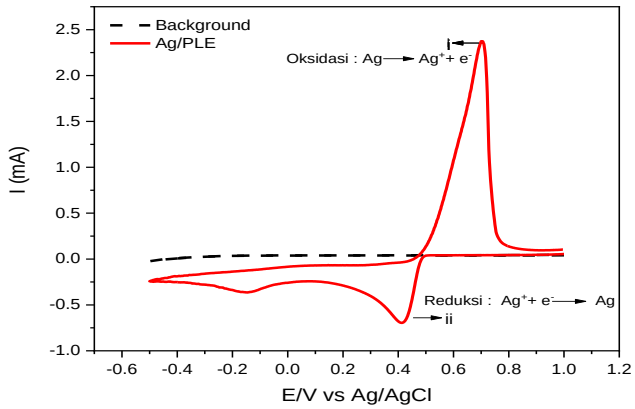


Figure 1. Cyclic voltammogram of modified PLE in 5 mM $AgNO_3$ and 0.1 M KNO3 solution with scan rate of 100 mV/s (Radha & Sari, 2024)

The voltammogram shows the appearance of reduction and oxidation peaks of Ag. The oxidation peak appears at a potential of +0.58 V with a current value of 2.3747 mA and a reduction peak at 0.4140 V with a current value of 0.6951 mA. The redox reaction that occurs at the electrode is as follows.

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

Oxidation: $Ag_{(s)} \rightarrow Ag^{+}_{(aq)} + e^{-}$

Electrochemical characterization of PLE and Ag/PLE towards Zn^{2+} metal ions

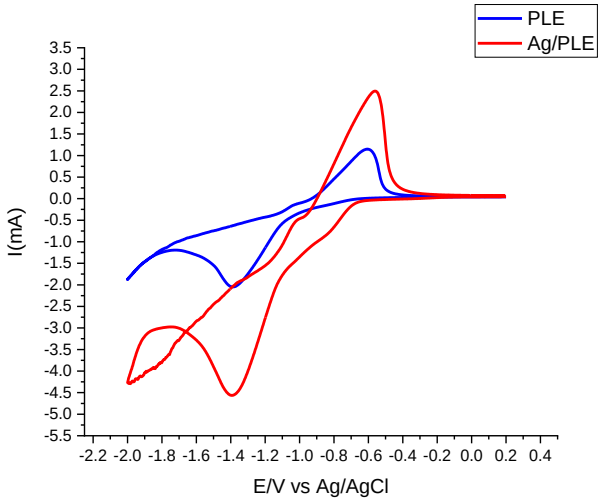


Figure 2. Cyclic voltammograms of PLE and Ag/PLE against 1 mM Zn^{2+} metal ion in 0.1 M pH 4 acetate buffer at a scan rate of 100 mV/s

Characterization was carried out to compare the electrochemical responses of PLE and Ag/PLE electrodes to Zn^{2+} metal ions in the supporting electrolyte solution. The comparison was done to find out which electrode is better in detecting Zn^{2+} metal ions. This test was carried out by adjusting the scan potentialfrom +0.2 V to -2 V with measurement results as shown in Figure 3.

Based on the voltammogram data above, it can be seen that the oxidation peak Zn^{2+} is at a potential of -0.604 V with PLE electrode and -0.558 V with Ag/PLE electrode. The reduction peak is at a potential of -1.460 V with PLE electrode and -1.3490 V with Ag/PLE electrode. The increased detection current in Ag/PLE is due to the electrocatalytic effect of Ag on Zn^{2+} metal ions and the larger electrode surface allows for increased detection current in Ag/PLE (Pura et al., 2023). The reduction and oxidation reactions that occur are as follows.

Reduction : $Zn^{2+}_{(aq)} + 2e^- \rightarrow Zn_{(s)}$

Oxidation : $Zn_{(s)} \rightarrow Zn^{2+}_{(aq)} + 2e^-$

Optimization of the Ag/PLE sensor with determination of Zn^{2+} metal ions

a. Supporting Electrolyte Variations

Supporting electrolyte is a solution that functions as a conducting medium where charge transfer occurs through the movement of electrolyte ions. This solution plays a role in preventing the occurrence of migrant currents due to the difference in ion concentration in the solution and on the electrode surface (Anugrah Putri, 2024). The effect of supporting electrolyte variation can be seen in the figure 4.

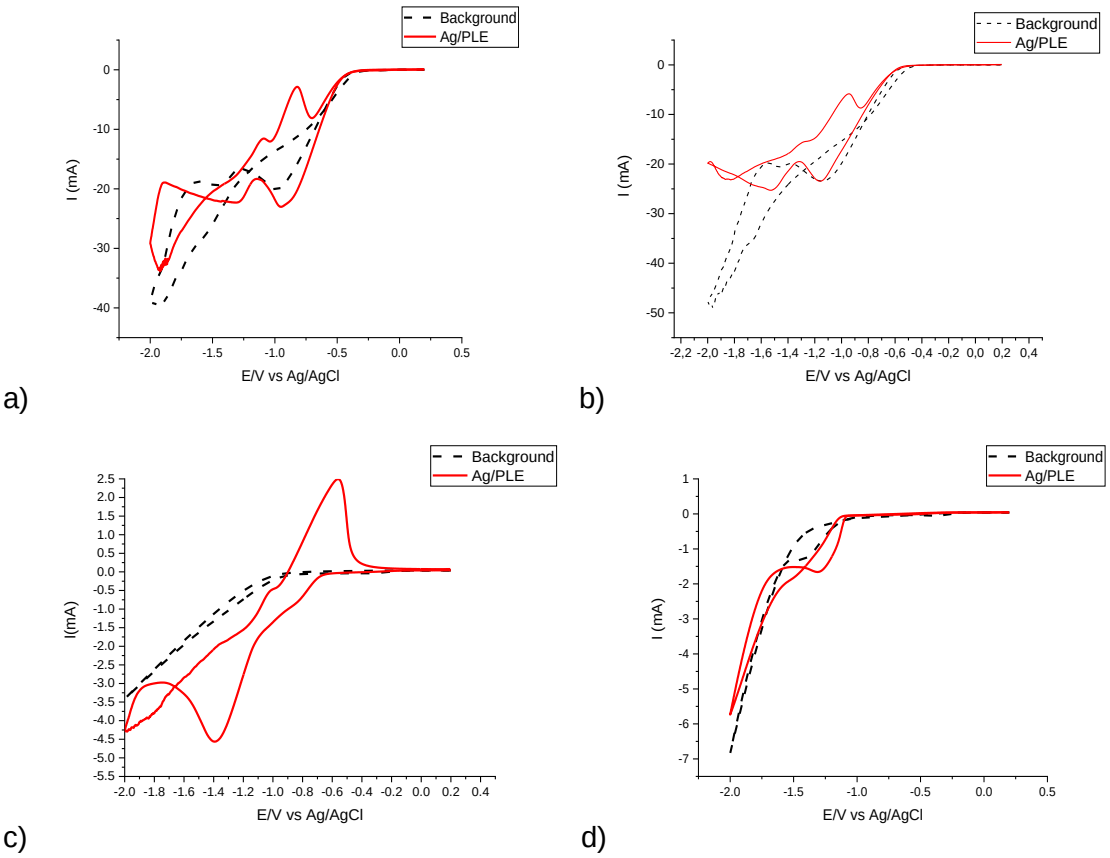


Figure 3. Cyclic voltammograms of Ag/PLE in various supporting electrolytes(a) HNO_3 0,1 M; (b) $HClO_4$ 0,1 M; (c) buffer asetat pH 4; (d) buffer fosfat pH 7 with ascan rate 100 mV/s

Based on the figure, it can be seen that the measurement of Zn^{2+}metal ions in PBS solution (pH 7) the oxidation and reduction peaks of Zn^{2+} metal ions are not visible, which means that the redox reactions of Zn^{2+} metal ions are difficult to occur in these solutions. Redox reactions are difficult to occur due to the possibility of forming insoluble white $Zn_3(PO_4)_2$ precipitates (Vogel, 1979) . The Qsp value of $Zn_3(PO_4)_2$ is greater than the Ksp value of $Zn_3(PO_4)_2$ which causes $Zn_3(PO_4)_2$ to precipitate, making it difficult to oxidize which causes a loss of peak potential and current intensity at the Ag/PLE electrode (Dean, 1999).

In HNO_3 and $HClO_4$ solutions or in acidic conditions show different responses, namely in HNO_3 solution there are peak values of oxidation and reduction at potentials of -0.9380 V and -1.4760 V, while in $HClO_4$ solution there are peak values of oxidation and reduction at potentials of -0.8080 V and -1.6140 V. This is because the redox potential range of Zn^{2+} metal ions is more negative

than the redox potential range of Hydrogen (H^+) so that it interferes with the redox process of Zn^{2+}. This is because the redox potential range of Zn^{2+} metal ions is more negative than the redox potential range of Hydrogen (H^+), thus disrupting the Zn^{2+} redox process. This is because the redox potential range of Zn^{2+} metal ions is more negative than the redox potential range of Hydrogen (H^+), thus interfering with the Zn^{2+} redox process. Therefore, an acidic atmosphere results in a shifted Zn^{2+} redox potential value and a low peak current (Bard, 2001).

Based on this, the best supporting electrolyte in acetate buffer solution (pH 4) with oxidation and reduction peaks obtained are 0.558 V and -1.3490 V. Acetate buffer solution can reduce the influence of ions (such as OH^- and H^+) and can prevent the formation of deposits that can affect the redox reaction of Zn^{2+} (Skoog, 2013). However, acetate buffer has good electrical conductivity to support current during the redox reaction (Scholz, 2003). In addition, Zn^{2+} metal ion is a metal with a negative potential that is very susceptible to corrosion, acetate buffer can reduce the corrosion speed of the metal (Fontana, 1987). Therefore, in this study, acetate buffer (pH 4) was used as the optimal supporting electrolyte.

b. Variation of Supporting Electrolyte pH

In previous tests, it has been found that acetate buffer solution (pH 4) is the most optimum supporting electrolyte solution. The redox process in electrochemical cells is supported by the optimum supporting electrolyte solution (Andriani, 2007). The acetate buffer solution was varied in pH value from 4, 5, and 6 and then tested using Ag/PLE electrodes with the CV method in the potential range of +0,2 hingga -2 V with a scan rate 100 mV/s.

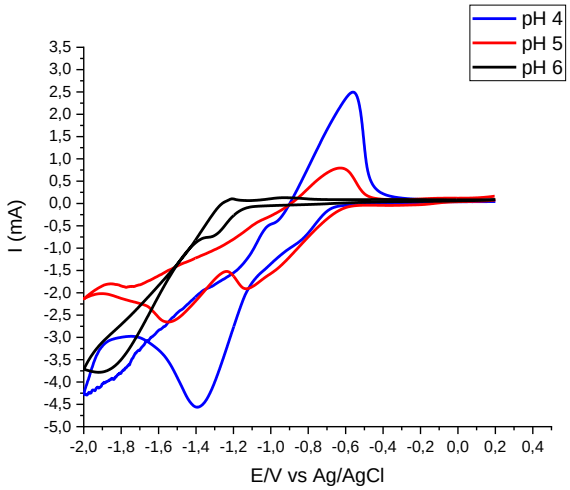


Figure 4. Cyclic voltammograms of Ag/PLE in 10 mM Zn^{2+} test solution and acetate buffer at pH 4 (blue line), pH 5 (red line), and pH 6 (black line) with a scan rate of 100 mV/s

Based on the voltammogram in figure 5 above, it can be seen that acetate buffer with pH 4 is the optimum pH for detecting Zn2 + metal ions which can be seen with the current response at pH 4 of 2.495 mA higher than the currents at pH 5 and 6. This is because at pH 4 the working electrode (Ag / PLE) is in a pH range that allows the Ag to remain stable and reactive without the formation of compounds that can interfere with redox reactions, such as silver hydroxide (AgOH) or silver oxide (Ag_2O) (Smith, 2023).

At pH 5 and 6, there is a decrease in the current value of Zn2+ metal ions caused by the disruption of the performance of the Ag/PLE working electrode. At higher pH, silver (Ag) undergoes a hydrolysis reaction so that Ag reacts with hydroxide ions (OH-) to form AgOH which can affect the continuity of the Zn2+ redox reaction (Chen et al., 2023). Therefore, in this study it was found that acetate buffer pH 4 as the optimum supporting electrolyte pH.

Calibration Curve

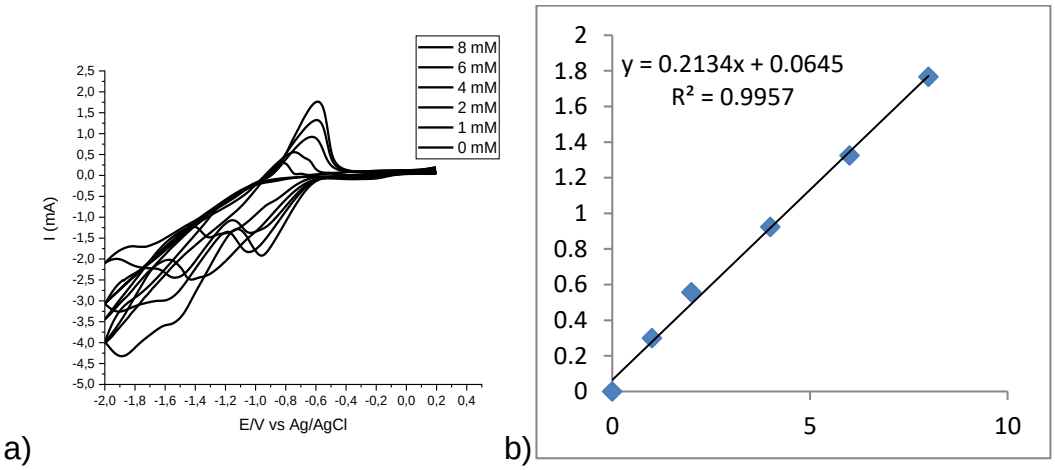
The purpose of carrying out this calibration curve is to test linearity and find out how the ability of an analytical method to obtain test results that match the concentration of the analyte at a certain concentration. After optimizing several parameters that affect the analytical performance of the Ag/PLE modified electrode,

calibration curves were generated using the CV method in the linear range of 1, 2, 4, 6, and 8 mM.

Table 1. Concentration series data of Zn2+ metal ion

Konsentrasi (mM)	E (V)	I (mA)
0	0	0
1	-0,8140	0,29865
2	-0,7440	0,5568
4	-0,6280	0,92303
6	-0,5940	1,32516
8	-0,5880	1,76568

This showed an ideal linear relationship with a detection limit of 0.44 mM and a correlation coefficient value of R2 = 0.995 as shown in Figure 5 below.



a) b)

Figure 5. (a) Cyclic voltammogram of Ag/PLE in acetate buffer pH 4, with varying concentration of Zn^{2+} metal ions, (b) Calibration curve graph of Zn^{2+} metal ions with varying concentration

One of the attractive features of electrocatalysts is their long-term stability. Therefore, the stability of Ag/PLE in the oxidation reaction of Zn^{2+} metal ion was tested by cyclic voltammetry over a potential range from +0.2 V to -2 V in acetate buffer with pH 4 0.1 M and 10 mM Zn^{2+} test solution. The purpose of repeating the cyclic voltammetry (CV) method 5 times is to see the stability and sensitivity of the Ag/PLE electrode to the determination of Zn^{2+} metal ions. It can be seen that the oxidation peak current is relatively stable during the scan (Maharani et al., 2024).

Table 2. CV Recurrence for Determination of Zn^{2+} metal ion

Concentration 10 mM	I (mA)
Repetition 1	2,495
Repetition 2	2,3966
Repetition 3	2,334
Repetition 4	2,2148
Repetition 5	2,2412
Rata-rata	2,33632
%SD	0,114709773
%RSD	4,909848518

In table 3 above, it can be seen for the relative standard deviation (RSD) of 5 repetitions. Read in table 2 for the RSD (%) value obtained of 4.9%. Thus, the results obtained represent acceptable stability and reproducibility.

Conclusion

In this study, PLE electrode modified with a thin layer of silver (Ag/PLE) through electrodeposition was used to detect Zn^{2+} metal ions. The PLE electrode showed better results in detecting Zn^{2+} metal ions due to its larger surface area and electrocatalytic effect compared to PLE. The Ag/PLE electrode was tested based on the effect of supporting electrolyte variation and supporting electrolyte pH using cyclic voltammetry which showed optimum results for Zn^{2+} measurement using acetate buffer solution with pH 4. Measurement of Zn^{2+} ions using Ag/PLE at optimum conditions gave a correlation coefficient (R^2) value of 0.995 and a detection limit of 0.44 mM accompanied by repeatability in CV resulting in an RSD of 4.9% which states that Ag/PLE using the CV method for 5 repetitions has good stability and sensitivity.

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VOL 6 NO 2 BULAN 10 TAHUN 2024
DOI : 10.25299/jrec.2024.vol6(2).18534