

Full Paper

Potentiality Slag Nickel Waste as A Candidate Working Electrode for Electrochemical Detection of Pb²⁺ Ion

Kusdin Kusdin,¹ Faizal Mustapa,^{1,*} and Irwan Irwan^{2,*}

¹*Department of Marine Sciences, Institut Teknologi dan Bisnis Muhammadiyah Kolaka, Kolaka 93511, Southeast Sulawesi, Indonesia*

²*Department of Pharmacy, Faculty of Sciences and Technology, Institut Teknologi dan Kesehatan Avicenna, Kendari 93117, Southeast Sulawesi, Indonesia*

*Corresponding Author, Tel.: +6282345154139

E-Mails: ichalgowamekong28@gmail.com (F. Mustapa); nazrilirwan16@gmail.com (I. Irwan)

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Abstract- The escalating threat of heavy metal contamination in water sources necessitates the development of efficient and cost-effective detection methods. This study explores the possibility of nickel slag waste (NSW) as a sustainable electrode material for the electrochemical sensor of Pb²⁺ ions through cyclic voltammetry (CV) and square wave voltammetry (SWV). The composition and morphology of the SNW were characterized using X-ray Fluorescence (XRF) and Scanning Electron Microscopy Energy Dispersive X-ray Spectroscopy (SEM-EDX). The analysis revealed a complex composition dominated by silica (SiO₂), magnesium oxide (MgO), and iron (Fe) with significant amounts of other elements such as nickel (Ni), aluminum (Al₂O₃), and titanium dioxide (TiO₂). The presence of these elements contributes to the electrode's high conductivity, structural integrity, and catalytic activity, which are crucial for efficient electrochemical sensing. Optimal detection was achieved at pH 5 being the optimal condition for Pb²⁺ detection due to favorable speciation and surface chemistry. The Gr/SNW composite electrode exhibited high sensitivity, with a limit of detection (LOD) as low as 0.005 ppm, and demonstrated strong linearity in Pb²⁺ concentration measurements. Additionally, the electrode showed excellent repeatability in performance, with RSD value, which was found to be 0.09%. These findings suggest that nickel slag waste is a promising candidate for developing low-cost, efficient electrochemical sensors for heavy metal detection.

Keywords- Nickel slag waste; Graphene; Electrochemical detection; Pb²⁺ ions; Cyclic voltammetry

1. INTRODUCTION

Growing amounts of heavy metal contamination in water sources are becoming a serious threat to the environment and public health. Heavy metals such as cadmium (Cd), lead (Pb), and mercury (Hg) are known for their toxicity and persistence in the environment, posing severe risks to aquatic life and human health [1,2]. Numerous studies have demonstrated that heavy metals can persist in the environment, accumulating in soil and water, leading to bioaccumulation in plants and animals [3,4]. The potential for this bioaccumulation to make its way into the human food chain presents serious health hazards. For example, exposure to lead, even at low levels, can result in neurodevelopmental abnormalities in children, which can lead to decreased cognitive performance and behavioral problems [5]. World Health Organization (WHO) has established a maximum allowable concentration of lead in drinking water at 10 $\mu\text{g/L}$ to mitigate these health risks [6,7]. The United States' Environmental Protection Agency (EPA) establishes a 15 $\mu\text{g/L}$ action threshold for lead in drinking water, highlighting the necessity of rigorous monitoring and cleanup procedures to avoid lead contamination [8].

Traditional methods for detecting lead (Pb) have been widely utilized such as atomic absorption spectroscopy (AAS) [9], inductively coupled plasma mass spectrometry (ICP-MS) [10], and X-ray fluorescence (XRF) [11]. These techniques are highly sensitive and capable of detecting trace amounts of lead with high accuracy. However, these methods have several limitations. To operate them, they often require expensive, complex equipment, extensive sample preparation, and trained staff [12,13]. Moreover, they are usually laboratory-based methods, making them unsuitable for real-time or on-site lead contamination monitoring. Electrochemical detection such as cyclic voltammetry (CV) offers a promising alternative due to its sensitivity, rapid response, and portability. It was widely employed for the detection and analysis of various chemical species, particularly in the field of heavy metal detection [14–16]. This technique utilizes a three-electrode system comprising a working electrode, a counter electrode (Pt wire) and a reference electrode (Ag/AgCl). The working electrode is of paramount importance as it directly interacts with the analyte and determines the sensitivity, selectivity, and overall performance of the electrochemical sensor [17–19].

One innovative approach in this field is the utilization of waste materials as cost-effective and sustainable electrode materials. A byproduct of the nickel refining process, nickel slag has attracted interest recently because of its possible uses in a number of disciplines, such as material science and environmental remediation. This interest stems from its unique chemical composition and physical properties, which make it a suitable candidate for such applications. Several studies have reported that the Nickel slag consists of oxides and silicates, with varying proportions of nickel, iron, and other metal components [20]. The chemical composition of nickel slag often includes significant amounts of nickel oxide (NiO), iron oxide (FeO/Fe₂O₃), and other metal oxides such as Titanium dioxide (TiO₂) and copper oxide (CuO) [21]. These oxides contribute to the slag's conductivity and catalytic properties, which are essential for

efficient electrochemical sensing. Nickel, in particular, is known for its catalytic activity towards various redox reactions involved in heavy metal detection processes [22,23].

In this work, we have explored the potential of nickel slag waste as a candidate electrode for the electrochemical sensor of heavy metals like Pb ions. Nickel slag, a byproduct of nickel production, presents a sustainable and cost-effective alternative in electrochemical applications due to its unique composition and properties. By harnessing this waste material, we aim to contribute to environmental sustainability and technological advancement in heavy metal detection methodologies. We hope this article will provide insight into the primary opportunities for using nickel slag waste as an electrode candidate for electrochemical sensors.

2. MATERIALS AND METHOD

2.1. Preparation and Activation of Nickel Slag Waste

This investigation employed nickel slag that was acquired from PT. Antam Pomalaa in Kolaka Regency. The slag samples, initially in lump form, were washed with water to remove contaminants. Subsequently, the slag was ground using a disc mill. After obtaining the slag in powder form, it was sieved using a 250 mesh. The next step involved activation, where 25 grams of the slag powder was dispersed into 100 mL of 1.5 M H₂SO₄ solution while being stirred with a magnetic stirrer for 6 hours. The mixture was then filtered and washed with hot water until the pH was neutral. Finally, the nickel slag powder was calcined at 700°C for 1 hour.

2.2. Electrode Material Characterization

The mineral composition of SNW was analyzed using X-ray Fluorescence (XRF). The surface morphology and elemental composition of SNW were examined using Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM-EDS). Additionally, all electrochemical experiments were conducted using a three-electrode system, including an Ag/AgCl reference electrode, a Pt wire auxiliary electrode, and a Gr/SNW working electrode. Electrochemical analysis was performed with a DY2100B Potentiostat connected to a monitor.

2.3. Preparation of Gr/SNW Working Electrode

The working electrode was prepared by weighing 0.7 g of Gr/SNW and placing it into a watch glass containing 0.3 g of paraffin oil. A thermometer was positioned above the surface of the paraffin oil to monitor the oil temperature. Once the paraffin oil reaches a temperature of 80°C, the mixture is mechanically stirred until the electrode composite is fully homogeneous. The homogeneous composite is then inserted into a tubular electrode body (with an aperture diameter of approximately 3 mm) and connected to a copper wire.

2.4. Performance Testing of Gr/SNW Electrode for Pb²⁺ Ion Detection

The performance of the Gr/SNW electrode was evaluated in a Pb²⁺ solution with a concentration of 1 ppm. Measurements were conducted with a scan rate of 100 mVs⁻¹ throughout a potential range (-0.8 V to 0.8 V) in a 0.01 M NaNO₃ electrolyte solution. The limit of detection (LOD) was determined by testing the optimal Gr/SNW electrode across five minimum concentrations namely 0.1 ppm, 0.3 ppm, 0.5 ppm, 0.7 ppm and 1.0 ppm. Subsequently, the final average LOD value was calculated as the standard deviation from five measurements. After identifying the optimal measurement conditions, analytical parameters were tested, such as the effects of modifier mass, pH, scan rate, linear measurement range, limit of detection, and repeatability.

3. RESULTS AND DISCUSSION

3.1. Characterization of SNW composition, and morphology

The X-ray fluorescence (XRF) analysis of slag nickel waste (Figure 1), showed a complex and rich elemental composition that highlights its potential for use as electrode material in electrochemical applications, particularly for detecting heavy metals. The analysis demonstrated that the slag contains many transition metals, with iron (Fe), Magnesium oxide (MgO), and silica dioxide (SiO₂) being the most prevalent with percentages of 8.06%, 20.97%, and 34.85% respectively. These metals are known for their excellent electrical conductivity and catalytic properties, making them highly suitable for electrochemical processes [24,25]. The presence of these elements in significant quantities suggests that slag nickel waste could serve as an efficient and effective electrode material, capable of facilitating electron transfer in electrochemical reactions [26].

Moreover, the XRF results identified the presence of other elements such as Ni, Al₂O₃, TiO₂, CaO, Cr₂O₃, MnO, and Zn. Although present in smaller quantities, this element plays a vital role in enhancing the electrochemical performance of electrode material. Chromium and manganese, for example, can provide additional active sites for the adsorption and reduction of heavy metal ions, thereby improving the sensitivity of the electrode in detecting various contaminants. On the other hand, TiO₂ is well-known for its catalytic qualities, which may improve the electrode's electrochemical performance even further. The combination of these elements in slag nickel waste not only reinforces its structural properties but also indicates its potential durability and longevity as an electrode material, which is crucial for long-term applications in environmental monitoring and heavy metal detection.

NSW composites' morphology was analyzed using SEM-EDX at magnifications of 1000x and 3000x. SEM provides detailed images that reveal the surface characteristics and microstructural features of the composite materials. Figure 2a can be seen at 1000x

magnification, the SEM analysis highlights the overall distribution and arrangement of nickel slag particles within the matrix [26,27].

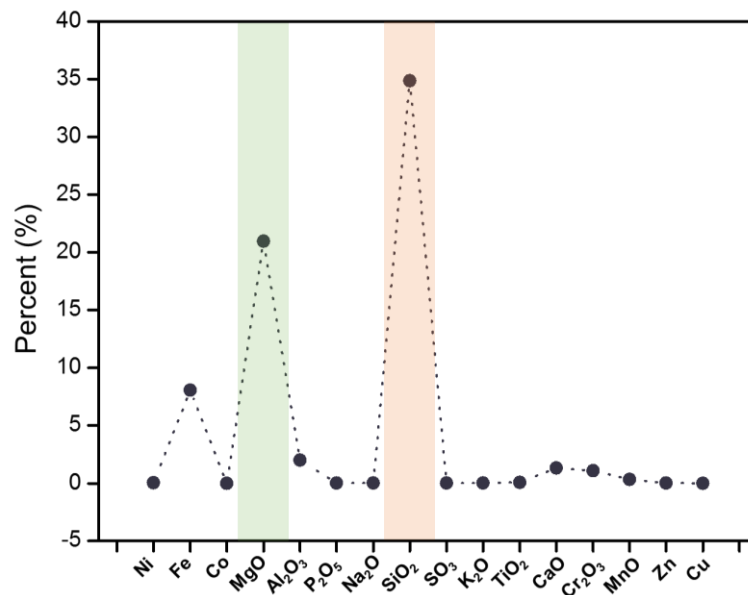


Figure 1. The composition of SNW analyzed using XRF

This level of magnification observation of larger structural features, such as the interface between the nickel slag and the binder material contributes to the composite's mechanical properties. Moreover, the presence of cracks or voids at this magnification can also be assessed, providing insights into the integrity and durability of the composite. Conversely, when the magnification is increased to 3000x (Figure 2b), the SEM images reveal finer details, such as the surface roughness of the slag particles and the morphology of the binder phase. This higher resolution reveals of specific microstructural phenomena, such as the formation of agglomerates or the dispersion of slag particles within the matrix.

Furthermore, the chemical composition of SNW was analyzed by EDX, displayed in Figure 2c. EDX analysis also confirms the presence of important elements in determining the potential of the composite as a candidate working electrode in electrochemical sensor applications. Some key elements detected such as Silica (Si), magnesium (Mg), iron (Fe), Nickel (Ni), and aluminum (Al) with percentages of 16.26%, 13.71%, 9.93%, 0.93%, and 1.49% respectively (Table 1). One important component of SNW is the presence of elements like Si, Mg, and Fe. Where silica provides structural stability, magnesium enhances conductivity, and iron offers redox activity. Additionally, the existence of Nickel (Ni), although present in a relatively low amount is a key element in SNW due to its excellent catalytic properties. Nickel's ability to facilitate electron transfer reactions makes it an ideal candidate for improving the electrochemical performance of the electrode. Nickel can also enhance the electrode's ability

to detect target compounds by providing more active sites for interaction, thus increasing the electrode's overall efficiency.

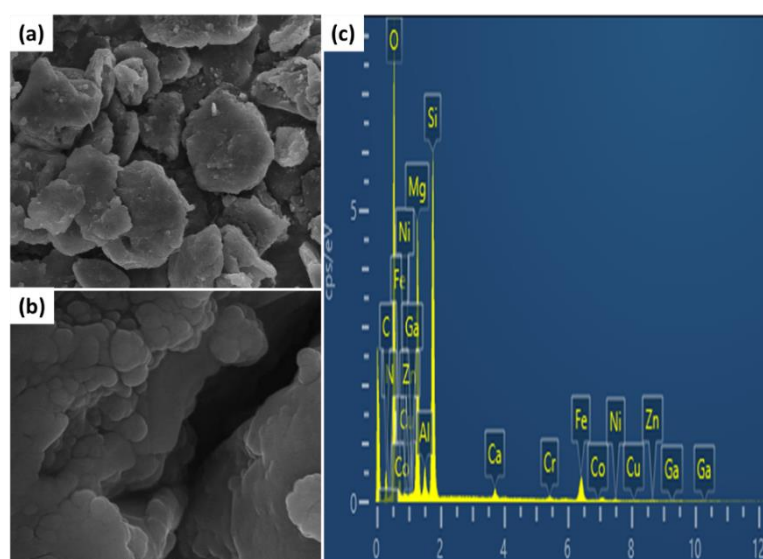


Figure 2. SEM-EDX morphology analysis of SNW composite, (a) 1000x, (b) 3000x magnification, and (c) SNW composite EDX spectrum

Table 1. The elements of SNW composite analyzed by EDX spectra

No	Element	Wt (%)
1	C	9.65
2	N	0.00
3	O	45.76
4	Mg	13.71
5	Al	1.49
6	Si	16.26
7	Ca	0.81
8	Cr	0.76
9	Mn	0.42
10	Fe	9.93
11	Co	0.03
12	Ni	0.93
13	Cu	0.00
14	Zn	0.11
15	Ga	0.14

3.2. Electrochemical examination of Gr/SNW electrode against Pb ion detection

In this study, the working electrode was composed of graphene (Gr), paraffin oil, and slag nickel waste (SNW) with varying amounts of modifier (0.01 g, 0.02 g, and 0.03 g). The composite was then inserted into an electrode body made of glass tube with a diameter (0.4 m), and a 4.0 cm copper wire was inserted to facilitate electron transfer. The electrochemical tests

were conducted using a portable potentiostat with CV and SWV to measure the peak currents of redox, represented as I_{pc} and I_{pa} for cathodic and anodic charges, and E_{pc} and E_{pa} for cathodic anodic potentials.

3.2.1. Variation mass of SNW as a modifier

In this experiment, we evaluated the influence of varying SNW masses on the electrochemical detection of lead (Pb) ions using cyclic voltammetry, to identify the optimal slag mass for sensitive and accurate detection (Figure 3). The masses examined were (0.01 b/b, 0.02 b/b, and 0.03 b/b), and the potential window of -0.8 to 0.8 V with a scan rate was $100 \text{ mV}\cdot\text{s}^{-1}$ [28]. It's visible that the mass of SNW significantly affects the electrochemical performance of the electrode. At an SNW mass of 0.01 g, the current response was relatively low, suggesting insufficient active sites available for Pb ion adsorption and subsequent electrochemical reaction. This lower activity could be attributed to the limited surface area provided by the smaller mass, leading to reduced interaction between the electrode surface and the Pb ions.

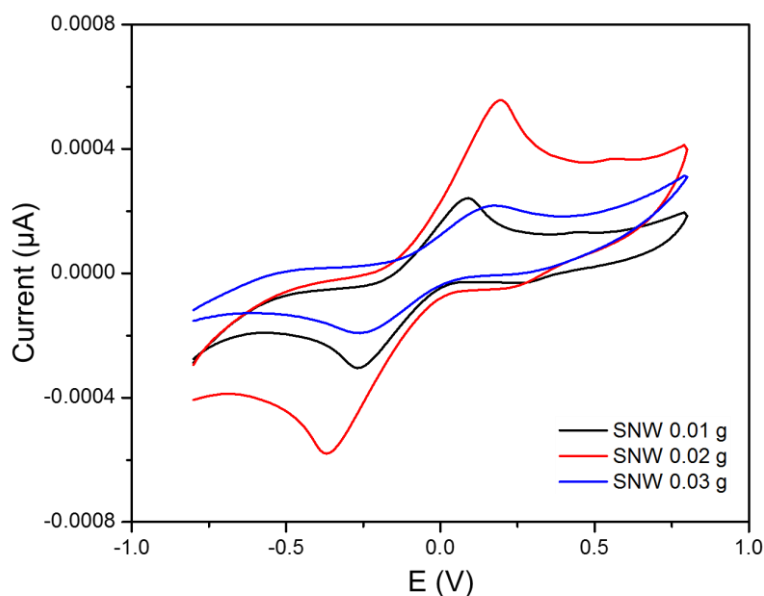


Figure 3. CV of Gr/SNW composite with the mass variation of SNW

As the SNW mass increased to 0.02 g, there was a notable enhancement in the current response. This increase is indicative of a greater number of active sites and an improved surface area-to-volume ratio, which facilitated better electron transfer kinetics. The 0.02 g slag mass provided an optimal balance between surface area and electrode conductivity, resulting in more efficient Pb ion detection. This mass exhibited the highest sensitivity and the most distinct peak currents, which are critical parameters for accurate electrochemical detection. However, further increasing the SNW mass to 0.03 g did not lead to a proportional increase in the current response. Instead, there was a slight decrease in peak current intensity. This phenomenon could

be explained by the high SNW mass causing particle agglomeration or aggregation, which can impede electron transmission and decrease effective surface area. Furthermore, a thicker SNW layer may have raised the electrode's resistance, decreasing the electrochemical process's overall efficiency.

3.2.2. Variation of scan rate

The influence of scan rate on electrochemical measurements, particularly in cyclic voltammetry (CV), is a crucial aspect of understanding the behavior of various electroactive species. Figure 4 illustrates the effect of varying scan rates from 0.02 to 0.2 V/s using a Gr/SNW in an analyte solution of 1 ppm Pb^{2+} ion containing 0.1M NaNO_3 . It is clear that at lower scan rates (0.02 V/s), the system has more time to achieve equilibrium, allowing for a more complete and irreversible redox process. This results in a lower peak current because the electrode has ample time to reach its equilibrium potential, leading to a more stable and less intense signal. Conversely, as the scan rate increases (0.2 V/s), the system is less able to achieve equilibrium, leading to a more irreversible redox process. This results in a higher peak current because the electrode is not given sufficient time to reach its equilibrium potential, causing a more rapid and intense signal [29]. The higher peak current at faster scan rates is indicative of a more dynamic and kinetically controlled process. This suggests that the detection of Pb^{2+} ions at higher scan rates is more sensitive to the kinetic parameters of the electrode reaction, potentially offering better detection limits for Pb^{2+} ions.

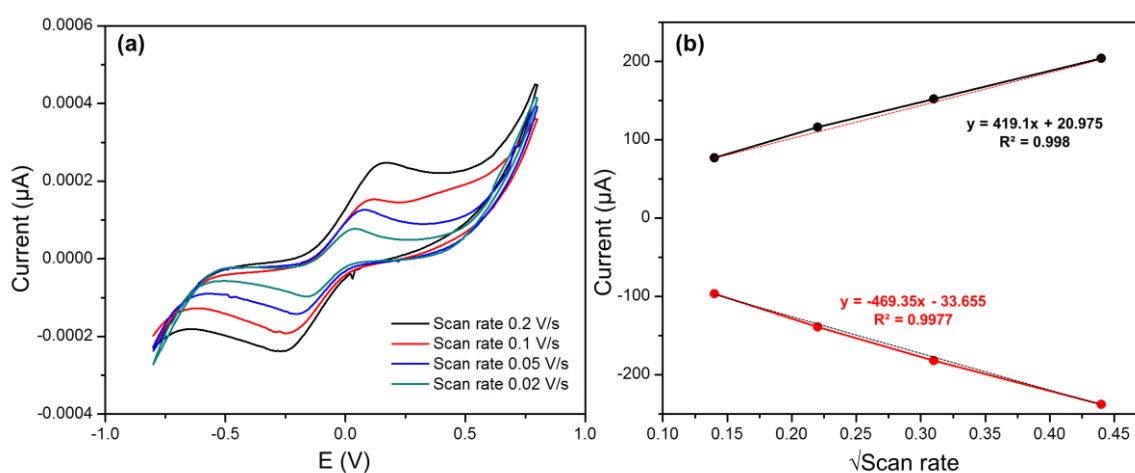


Figure 4. (a) CV of Pb ion redox over Gr/SNW with different scan rates, (b) curve between current (I_{pa} = black; I_{pc} = red) peaks versus $\sqrt{\text{scan rate}}$

3.2.3. Effect of pH

In this study, the effect of pH plays a crucial role in the electrochemical detection of Pb^{2+} ions using cyclic voltammetry (CV) with a Gr/SNW electrode. The pH affects the speciation of lead ions in the solution and the surface chemistry of the electrode, ultimately influencing

the electrochemical response. The pH range of 3 to 7 was examined, using a 100 mVs^{-1} scan rate and a potential window (-0.8 to 0.8 V). As demonstrated in Figure 5, hydrogen ion (H^+) concentrations are significantly high at lower pH values (pH 3 and 4), which causes H^+ and Pb^{2+} to compete for active sites on the electrode surface. This competition results in decreased adsorption of Pb^{2+} ions, thereby reducing the observed peak current [30]. Additionally, at these lower pH levels, Pb^{2+} ions might exist in a more hydrated or complexed form, which is less electroactive, further contributing to the reduced peak current.

However, a discernible rise in peak current was noted as the pH rose to 5, suggesting that pH 5 is the optimal condition for the electrochemical sensing of Pb^{2+} ions using the Gr/SNW electrode. At this pH, the balance between the deprotonation of the electrode surface and the speciation of Pb^{2+} is favorable for efficient electron transfer [31]. The Gr/SNW electrode likely provides an optimal environment for the adsorption of Pb^{2+} ions, which enhances the sensitivity of the detection. This increased sensitivity is reflected in the higher peak current observed at pH 5. In contrast, the peak current begins to decrease as the pH gets closer to neutral (pH 6 and 7). This explanation for this case is attributed to the formation of lead hydroxide species ($\text{Pb}(\text{OH})^+$ and $\text{Pb}(\text{OH})_2$), which are less electroactive and less likely to be reduced at the electrode surface. On the electrode surface, these hydroxide species may precipitate, causing passivation and a subsequent reduction in the electrochemical response [32].

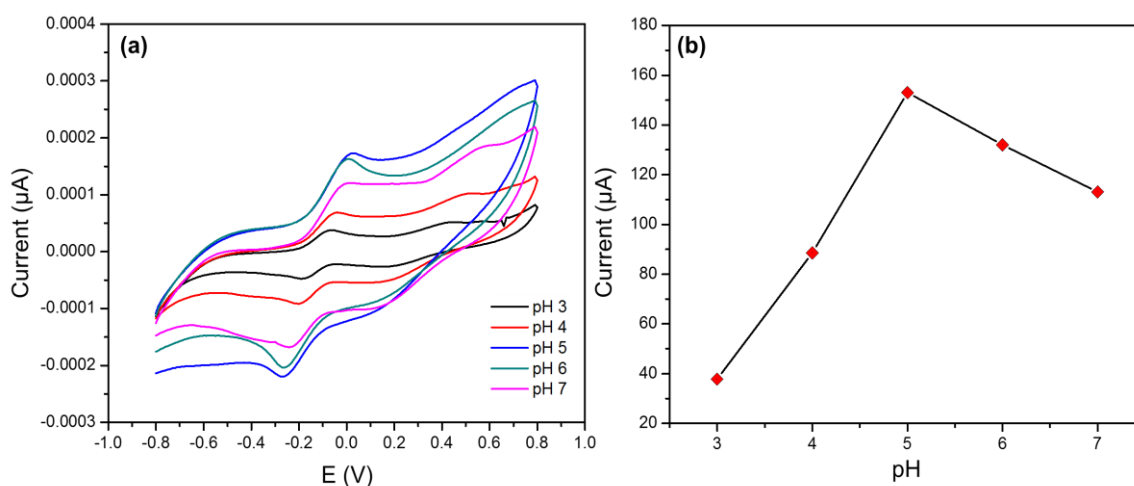


Figure 5. (a) CV of Pb^{2+} ion containing the citric buffer with pH variation, (b) oxidation current against pH curve

3.2.4. Linearity and limit of detection tests

Square wave voltammetry (SWV) was used to measure the linearity and limit of detection (LOD) for Pb^{2+} ion detection utilizing the Gr/SNW electrode. This is essential for figuring out how sensitive and useful the electrode is for trace metal analysis [33]. The SWV is a pulse voltammetric technique that offers several advantages, including high sensitivity and rapid analysis. In this study, the concentration range examined namely 0.1 ppm - 1 ppm at pH 5. The

results in Figure 6, indicated a strong linear correlation between the peak current and Pb^{2+} ion concentration. Moreover, the linearity was evaluated by plotting the peak current against the concentration of Pb^{2+} (Figure 6b). It can be seen, that a strong linear relationship is indicated by a high correlation coefficient (R^2) near 1, indicating that the Gr/SNW electrode responds proportionately to variations in Pb^{2+} concentration within the tested range. This linearity is essential for quantifying unknown samples, as it ensures that the electrode can reliably measure concentrations within this range without significant deviations. Furthermore, the LOD for Pb^{2+} was determined could reach as low as 0.005 ppm, which is critical given the toxic nature of lead and its health implications. The electrochemical behavior of Pb^{2+} at the Gr/SNW electrode was characterized by distinct peak currents at specific voltages, which facilitated the quantification of lead ions in the tested concentration range. Table 2 displays the comparison of the proposed electrode materials in this work with the other previously reported Pb^{2+} ion electrochemical detection.

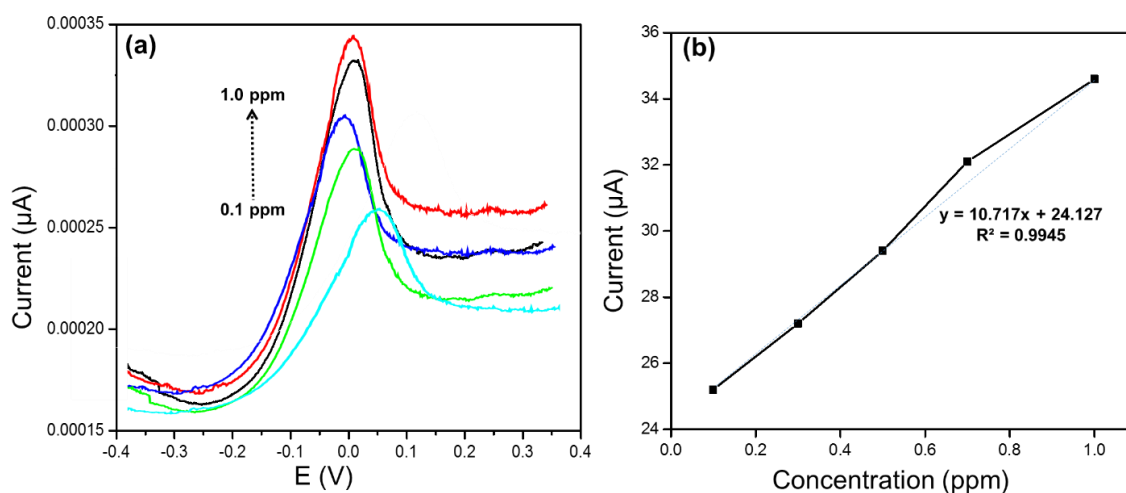


Figure 6. (a) SWV voltammogram of Pb^{2+} ion at various concentrations; (b) Pb^{2+} ion concentration plotted against peak oxidation current

Table 2. Comparing several working electrode materials for Pb^{2+} ion detection with current research

Electrode material	Electrochemical techniques	Analyte	Sensitivity	Reference
AErGO	DPASV	Pb^{2+}	0.0924 mg L ⁻¹	[34]
TiO ₂ -Ionophores BEK6	CV	Pb^{2+}	0.1756 ppm	[19]
ERGO/GCE	SWASV	Pb^{2+}	0.2 ng/mL	[35]
GCE/CPE	DPASV	Pb^{2+}	0.0292 μmol/L	[36]
r-CeO ₂ /EG	DPV	Pb^{2+}	0.21 μg L ⁻¹	[37]
AgNP@GNs	DPV	Pb^{2+}	246.4 μA/μg L ⁻¹	[38]
Gr/SNW	SWV	Pb^{2+}	0.005 ppm	This work

3.2.5. Repeatability test

The repeatability test of Pb^{2+} ion detection was conducted for 20 consecutive measurements. Repeatability, is a crucial parameter in electrochemical analysis, reflecting the consistency and reliability of the electrode's performance over multiple experiments [39]. Figure 7, demonstrated a relatively stable peak current across the 20 measurements, indicating good repeatability of the Gr/SNW electrode. The standard deviation of the peak current was minimal, suggesting that the electrode surface was consistent in its electrochemical behavior throughout the test. The RSD value, which was found to be 0.09%, indicates an exceptionally high degree of repeatability and precision in the cyclic voltammetry measurements. The low % RSD value suggests that the Gr/SNW electrode provides highly consistent responses when detecting Pb^{2+} ions across multiple measurements. This level of repeatability is crucial for ensuring the accuracy and reliability of analytical results, particularly in applications requiring precise quantification of trace elements.

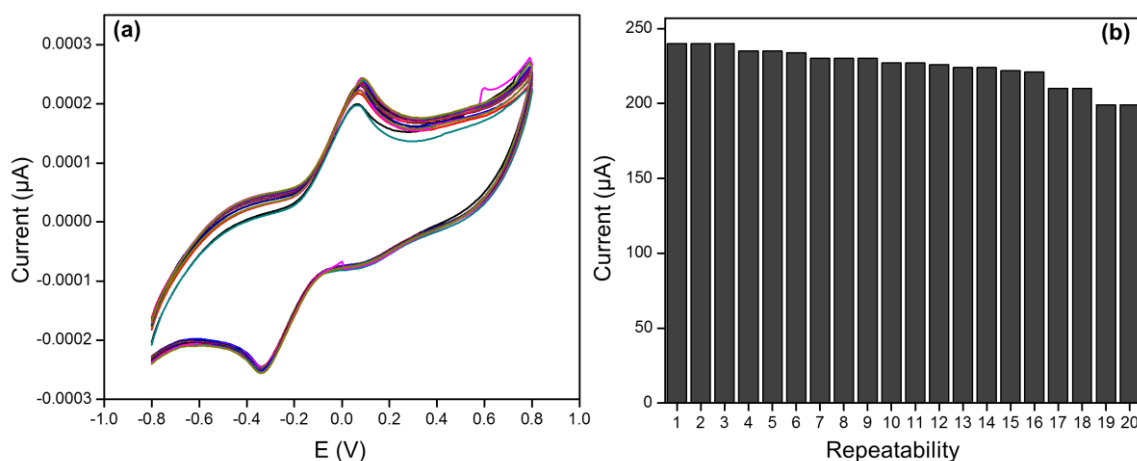


Figure 7. Repeatability test of Gr/SNW electrode (a) cyclic voltammogram of repetition, (b) histogram peak current over 20 repetitions

4. CONCLUSION

The study explores the potential use of nickel slag waste as a cost-effective and sustainable electrode material for the electrochemical detection of lead ions (Pb^{2+}) in water. The material electrodes were characterized by various spectroscopy such as XRF and SEM-EDS and analytical methods such as CV and SWV. The Gr/SNW electrode exhibited a wide linear response range for the determination of Pb^{2+} ions in the solution. The electrode can achieve a LOD of 0.005 ppm and has good repeatability. The findings highlight the potential of using nickel slag waste materials as a candidate working electrode in electrochemical applications, contributing to environmental sustainability and technological advancement in heavy metal detection.

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Declarations of interest

The authors declare no conflict of interest in this reported work.

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