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Anodic Stripping Voltammetry for simultaneous the Determination of Lead and Cadmium with Graphene Oxide Nanoparticles

Lazim Abdullah Salih [1], Nashwan H. Ali* [2]

[1] *Department of Chemistry, Collage of Education, University of Samarra, Iraq*

[2] *Department of Applied Chemistry, Collage of applied Science, University of Samarra, Iraq*

Abstract:- A highly sensitive electrochemical sensing system was developed for the quantitative determination of lead (Pb^{2+}) and cadmium (Cd^{2+}) ions using a glassy carbon electrode modified with graphene oxide nanoparticles (GONPs/GCE). The Graphene Oxide Nanoparticles GONPs were synthesized by the well-known Hummers' method, then dispersed in 0.5wt% Nafion solution in 1:1DI water/ethanol mixture and drop-cast onto the surface of GCE. Square-wave anodic stripping voltammetry (SWASV) was employed to take advantage of the improved electrochemical behavior of the GONPs/GCE. Best operating conditions include pre-concentration at -1.2 V for 50\s in pH=7 phosphate buffer/ KNO_3 electrolyte supporting medium.

Well-separated oxidation peaks are observed at about -0.5 Vfor lead Ions and -0.7 V for cadmium ions. The sensor showed very good linearity over a range between $0\,.5-50\ \mu g\ L^{-1}$ for both metals with low detection limits found as only 0.09 $\mu g\ L^{-1}$ for Pb $^{+2}$ 0.23 $\mu g\ L^{-1}$ in Cd^{+2}. Precision, reported as relative standard deviation (RSD) of 1.84% for Pb^{+2} and 1.90 % for Cd^{+2} was also obtained, highly reproducible results. The developed GONPs/GCE-based method was effectively applied to the analysis of tap and industrial water samples, yielding satisfactory recovery values.

Keywords: *Anodic stripping voltammetry, graphene oxide Nps, Heavy metals, Lead, Cadmium.*

1. Introduction

Heavy metals are components that are both beneficial and harmful, but they can also be over abundated. Lead and cadmium are the most harmful metals to humans. Exposure to lead and cadmium is associated with a wide range of adverse health effects, including carcinogenic risks, hepatic damage, neurological disorders, cardiovascular complications, and other systemic impairments [1-3]. These metals are discharged into the environment through various industrial activities, including electroplating processes, metallurgical operations, and paper manufacturing [4]. These components can negatively affect crops by polluting the soil and water. Cadmium and lead ions are soluble in water and could be taken up by plant tissue. Cadmium has been observed in both wheat and rice, additionally, lead has been observed in both spinach and cabbage [5]. The maximum permissible concentrations of cadmium and lead in drinking water have been established at 3 $\mu g\ L^{-1}$ and 10 $\mu g\ L^{-1}$, respectively [6].

Several analytical methods have been developed to identify heavy metals. These include electrothermal atomic absorption spectrometry (ET-AAS), flame atomic absorption spectrometry (FAAS), ICP-MS, and ICP-OES. These spectrometry's are said to be "inert" or

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"passive" in nature [7]. All of these approaches require high-maintenance instrumentation in a laboratory that is well equipped with trained operators that have long procedures for preparing samples; however, they produce very accurate results [8,9]. Recently, a new method to determine heavy metal ions has been proposed as an electrochemical approach. Portable device! Easy procedure! Quick response analysis is only marginally more expensive than other existing methods [10]. Anodic stripping voltammetry is one of the most popular heavy metal detection methods because its pre-concentrates the analyte followed by a buildup, then stripping the electrodes. All of this is done within a single step. In the preconcentration phase, metal ions are deposited onto the electrode through reduction, followed by rapid oxidation, allowing the anodic peak current to reflect their concentration [11-14]. Anodic stripping voltammetry (ASV) measurements were conducted using a conventional three-electrode system, consisting of a reference electrode (RE), a counter electrode (CE), and a working electrode (WE). The choice and surface modification of the working electrode play a crucial role in voltametric techniques, as they directly influence the analytical performance. Selecting an appropriate electrode material can significantly enhance method sensitivity, selectivity, reproducibility, and stability [4,15–17]. Over the past decade, graphene oxide nanoparticle-modified electrodes have been successfully developed as alternatives to conventional glassy carbon electrodes. Compared to unmodified glassy carbon, these electrodes exhibit enhanced electrochemical properties, a wide potential window, and excellent chemical stability. Moreover, the abundant functional groups on graphene oxide facilitate easy surface functionalization, allowing for effective attachment of various heavy metal ions. The accumulation capability of graphene oxide nanoparticles (GONPs) enhances the sensitivity of detecting heavy metals. Accordingly, GONPs-modified electrodes have been developed for the voltametric determination of Pb^{2+} and Cd^{2+} ions [18-20].

Graphene oxide nanoparticles combined with glassy carbon electrodes (GCE) offer a powerful and versatile platform for the electrochemical determination of lead and cadmium, two toxic heavy metals. This combination enhances the sensitivity and selectivity of the electrodes, enabling the detection of trace levels of these metals in environmental samples. The high surface area and excellent conductivity of graphene oxide facilitate rapid electron transfer, improving the response time and overall performance of the sensors. Additionally, the ability to functionalize the electrodes with specific ligands or modifiers allows for tailored interactions with lead and cadmium, further increasing detection accuracy. As a result, modified GCEs with graphene oxide are emerging as effective tools for monitoring heavy metal contamination in water and soil, contributing to environmental safety and public health.

2.MATERIALS AND METHODS

2.1 Chemicals and Reagents.

Graphite flakes, $NaNO_3$, and $KMnO_4$ were employed to create graphene oxide. These

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chemicals have a concentration of 98.6%, H_2O_2, and hydrochloric acid were added to the graphene flakes. Ethanol, potassium ferricyanide, potassium chloride, sulfuric acid, potassium dihydrogen phosphate, and disodium phosphate recognized as effective buffering agents were utilized in the electrochemical expertmints. To investigate potential interferences, metal salts including mercury (II) chloride, magnesium (II) acetate, zinc (II) acetate, nickel (II) nitrate, cobalt (II) acetate, copper (II) acetate, and iron (II) nitrate were employed. Reagents were sourced from Merck and Sigma-Aldrich, and Milli-Q water (resistivity 18.2 MΩ·cm) was employed in all experimental procedures.

2.2 Instrumentation

Voltammetric readings were recorded with the Corrtest Competence (China). In-situ cultivation of GO was accomplished on a 3.0 mm carbon dioxide-supplied electrode that functioned as the working electrode. The reference electrode had a composition of Ag/AgCl/KCl (3.0 $mol\ L^{-1}$), and the counter or helper electrode was a platinum wire. All electrochemical experiments were conducted in A 15 mL glass cell that was covered with a Teflon cell cover.

The pH was measured with a Hanna HI2002-02 pH meter.

2.3 Preparation of stock solution for Pb^{2+}

Weigh approximately 0.016 mg of $Pb(NO_3)_2$ using an analytical balance and transfer into a 100 mL volumetric flask. Then, added deionized water to dissolve the Pb $(NO_3)_2$.

Preparation of stock solution for Cd^{+2}

Weigh approximately 0.021 mg of Cd $(NO_3)_2$•H_2O and transfer into a 100 mL volumetric flask. Then, added deionized water to dissolve the Cd $(NO_3)_2$•H_2O

Phosphate buffer solution (PBS)

Add 1.76 g sodium phosphate monobasic ($NaH_2PO_4·H_2O$) and 2.38 g sodium phosphate dibasic ($Na_2HPO_4·7H_2O$) to about 800 mL distilled water. Allow it to dissolve with continuous stirring. Transfer this solution into a 1 L volumetric flask, also transferring the beaker washings, make up the volume to 1 L with distilled water. Check pH, adjust, if necessary, by adding small quantities of either salt so as to get final desired $pH = 7.4$, mix well and store buffer solution in labeled container for future use.

2.5 Synthesis of GO NPs using Hummers' method [21]:

An ice flask was set up containing 2g of graphite in 1500 ml at 0-5 C° to which 50 ml concentrated sulfuric acid was added followed by1.3 g sodium nitrate. Ten grams solid potassium permanganate were then gradually added over two hours while the temperature was kept below 20 C° with the aid of a mechanical stirrer. The reaction mixture was initially

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heated to 35°C and maintained at this temperature for 30 minutes. Subsequently, deionized water was added, and the temperature was increased to 90°C with continuous stirring for an additional 30 minutes. Afterwards, 30% hydrogen peroxide was added dropwise until the solution turned bright yellow and gas evolution ceased. The resulting product was then filtered and thoroughly washed several times with a 5% hydrochloric acid solution to remove residual metal ions, followed by rinsing with deionized water to eliminate any remaining acids.

2.6 Nafion mixture solution

Nafion solution was prepared by diluting the commercial 5 wt % Nafion stock solution (purchased from China) to a final concentration of 5 wt %. The diluent was a mixture of DI water and ethanol ETOH in a 1:1 volume ratio. Specifically, 100 microliter of the 5% stock was mixed with 900 microliter of the DI water/ethanol mixture.

The resulting solution was thoroughly homogenized using an ultrasonic bath for 15 min. This diluted Nafion solution was subsequently used as a binder to prepare the final GO/Nafion composite ink for drop-casting onto the glassy carbon electrode GCE, thereby enhancing the mechanical stability and adhesion of the sensing film.

2.7 Preparation of modified glassy carbon electrode

Just before every single experiment, alumina powder of 0.3 and 0.5 µm was used to polish the Glassy Carbon Electrode, followed by rinsing with deionized water, then ultrasonic cleaning in distilled water for three minutes. The modifying compound was prepared by homogenously dispersing synthesized GONPs in a mixture containing 3 µM Nafion; the suspension is subjected to ultrasonication right before use so as to ensure good dispersion. Deposition through drop-casting applied five microliters (µL) GO suspension onto the surface of pre-cleaned GCE after which it allowed drying at room temperature for two hours.

2.8 Conventional three-electrode system

To assemble a conventional three-electrode system for anodic stripping voltammetry (ASV) using a graphene oxide-modified glassy carbon electrode (GCE), the electrochemical cell was first prepared, and the modified GCE was inserted as the working electrode. A reference electrode (Ag/AgCl) and a counter electrode (platinum wire) were then placed in the cell, ensuring proper spacing between all electrodes. Connect the electrodes to a potentiostat, then filled the cell with a supporting electrolyte solution 0.1 mol/L KNO_3. Performed cyclic voltammetry to condition the modified electrode before conducting ASV measurements, which involve a pre-concentration step at a – 1.2 potential followed by a potential sweep to detect the oxidation of accumulated metal ions.

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Area of Study and Sample collections

Tap water and industrial water samples were collected from Samarra city. Immediately upon collection, each water sample was filtered through a micro-filter with a pore size of 0.45 µm. The filtrates and the filters containing residues were treated separately. The filtrates were acidified with 1 ml of concentrated HNO_3 per liter, ensuring that the pH value of the acidified filtrates lies between 2.0 and 3.0.

Analytical procedure

The electrode surface was prepared prior to every measurement by mechanical polishing with 0.3 µm alumina on a microcloth polishing pad, and then rinsed by ethanol and deionized water thoroughly. After that, the electrode was activated in 0.5 M H_2SO_4 by cyclic voltametric sweeping from $-0.1\ V$ to $1.2\ V$ at a scan rate of 50 mV s^{-1}.

The ASV method, which is also known as anodic stripping voltammetry, involves two main steps: the accumulation step and the stripping step. the first step is to make the working electrode (GO/GCE) negative, this is typically done by holding it at a voltage of -1.2 V against a reference electrode that is Ag/AgCl. At this point, the ions of Pb^{+2} and Cd^{2+} in the sample solution are removed and deposited (electroplated) onto the surface of the electrode, this creates a thin layer of Pb and Cd metals. The decrease in half-reactions is as follows:

Pb^{+2} + 2e- → Pb (s)

Cd^{+2} + 2e- → Cd (s)

The amount of Pb and Cd deposited on the electrode is proportional to their respective concentrations in the sample solution and the duration of the accumulation step.

After the accumulation step, the potential of the working electrode is scanned in the positive direction (anodic scan).

As the potential becomes more positive, the deposited Pb and Cd metals are oxidized (stripped) from the electrode surface, releasing Pb^{+2} and Cd^{+2} ions back into the solution. The oxidation half-reactions are:

Pb (s) → Pb^{+2} + 2e-

Cd (s) → Cd^{+2} + 2e-

The oxidation of Pb and Cd results in the generation of characteristic anodic stripping peaks, which are measured as the current response. The peak potentials for Pb and Cd are typically around -0.5 V and -0.7 V vs. Ag/AgCl, respectively, allowing for their separation and quantification.

The use of the GO/GCE further enhances the performance of this ASV method by providing

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increased surface area, improved electron transfer kinetics, and enhanced catalytic activity, leading to improved sensitivity, selectivity, and lower detection limits for the simultaneous determination of Pb^{+2} and Cd^{+2}.

RESULTS AND DISCUSSION

The square-wave voltammogram displays the electrochemical response of a 50 µM solution of **Pb** and **Cd** in 0.1 mol/L KNO3. The voltammogram was run by square-wave anodic stripping voltammetry (SWASV).

A potential of $\mathbf{-1.2\ V}$ was applied for 120 seconds to deposit Pb and Cd onto the modified glassy carbon electrode during the preconcentration step. Then, a scan was run which gave clear oxidation peaks for $\mathbf{Pb^{+2}}$ and $\mathbf{Cd^{+2}}$ at about $\mathbf{-0.5\ V}$ and $\mathbf{-0.7\ V}$, respectively, versus Ag/AgCl reference electrode. Sharp well-defined peaks mean successful preconcentration and sensitive detection of Pb and Cd by this electroanalytical technique.

The square-wave voltammogram shows the electrochemical response for a 50 µM solution of Pb and Cd in 0.1 mol/L KNO_3. The voltammogram was obtained using square-wave anodic stripping voltammetry (SWASV)

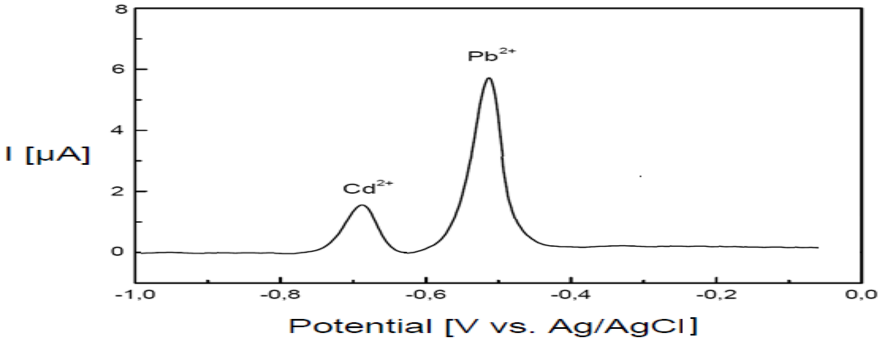

Figure.1."Anodic Stripping Voltammetry (ASV) stripping Peaks of Cd^{+2} and Pb^{+2}"

Optimization for determination of Pb^{+2} and Cd^{+2} at the Modified electrode

In order to establish the most suitable experimental conditions for the anodic stripping voltammetry determination Pb^{+2} and Cd^{+2} at Modified electrode. The detailed studies involve pH effect, Deposition times, Electrolyte Composition and the Scan Rate.

Effect of pH

The peak currents of Pb^{2+} and Cd^{2+} were evaluated in various supporting electrolytes, including HCl, HNO_3, and different phosphate buffers. To examine the effect of pH across the entire range, all electrolytes were prepared at the same concentration (1×10^{-2}mol/L^{-1}).

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The highest anodic stripping peak currents were observed in phosphate buffers of varying pH values (Fig. 2), with pH 7 identified as the optimum condition for sensitive detection.

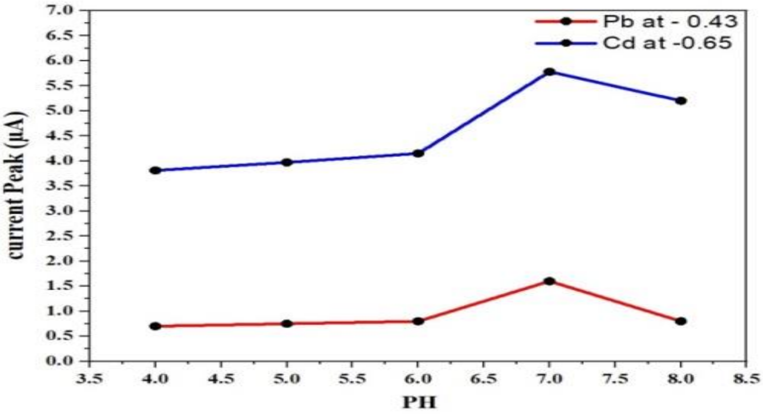

Figure 2. . Investigation of the effect of pH on the peak current intensity of Pb^{2+} and Cd^{2+} ions using ASV, indicating the optimal operating conditions for maximum electrochemical response.

Effect of accumulation time

The dependence of different anodic stripping currents on the amount of time spent in the same place was studied. The proposed method's sensitivity was enhanced by the longer time of accumulation because of the higher concentration of lead and cadmium on the altered electrode. However, this tendency would not be maintained all of the time due to the limited number of active sites for pb and Cd, so the time required to accumulate 50 S was chosen as the best.

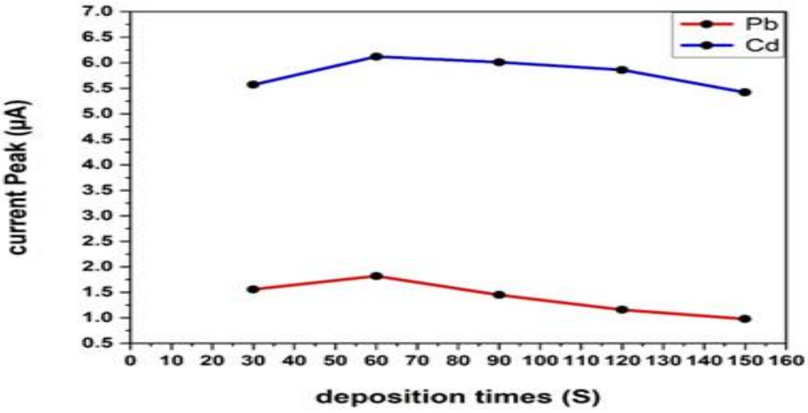

Figure. 3. Effect of Deposition Time on the peak current of Pb^{+2} and Cd^{+2} ions

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Effect of electrolyte composition

the study of electrolyte composition in ASV for detecting Pb^{+2} and Cd^{+2} at modified electrodes is crucial for enhancing analytical performance, and developing reliable methods for monitoring heavy metals, so, it was take from $0.05 - 0.5$ M for KNO_3, the 0.1 M electrolyte concentration appears to be a suitable choice for the determination of Cdand Pb using the modified electrode in ASV, as it provides a good balance between sensitivity, resolution, and practical considerations.

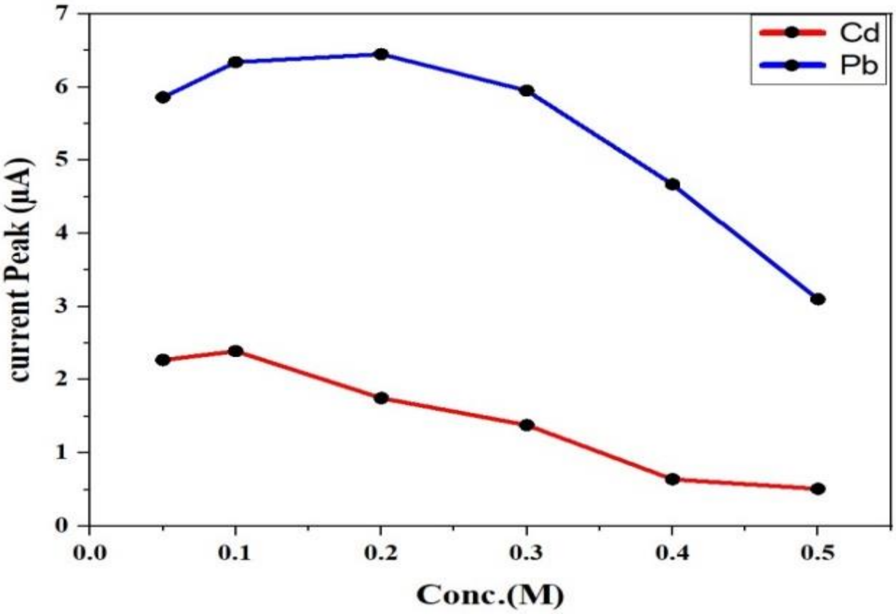

Figure 4. Curves of Pb^{+2} and Cd^{+2} Relationship Between ion concentration (M) and Peak current (µA).

Effect of Scan Rate

Based on the analysis of scan rate effects, 50 mV/s was selected as the optimal scan rate for the simultaneous determination of Cd and Pb. This rate provides an effective compromise, offering well-defined peaks and sufficient sensitivity for both metals. While the peak current for Cd increases steadily with scan rate, the response for Pb is optimized around 50 mV/s, as higher rates lead to peak deterioration and a loss of resolution, thereby ensuring reliable analytical performance.

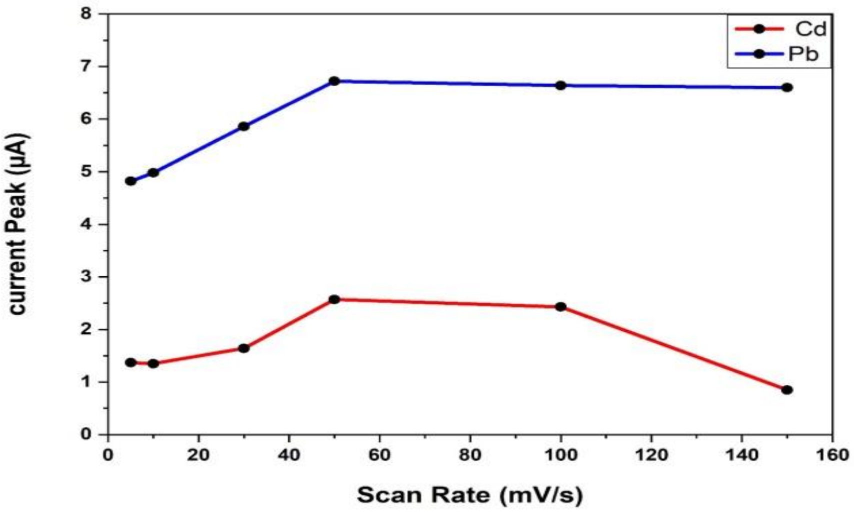

Figure.5. Effect of Scan Rate on the peak Current of Pb^{+2} and Cd^{+2}ions.

Interferences

One of the most significant advantages of the modified electrode in metal ion determination is its high selectivity. Electroactive species that bind to graphene oxide could potentially interfere with the analysis; however, no notable changes in the voltametric signals of Pb^{2+} or Cd^{2+} were observed in the presence of various additional ions, including K^{+}, Mg^{2+}, Ca^{2+}, Mn^{2+}, Zn^{2+}, Co^{2+}, and Fe^{2+}. However, in presence Hg^{+2} and Cu^{+2} ions; the signal to be matched is greater and the peak current of lead and cadmium has increased.

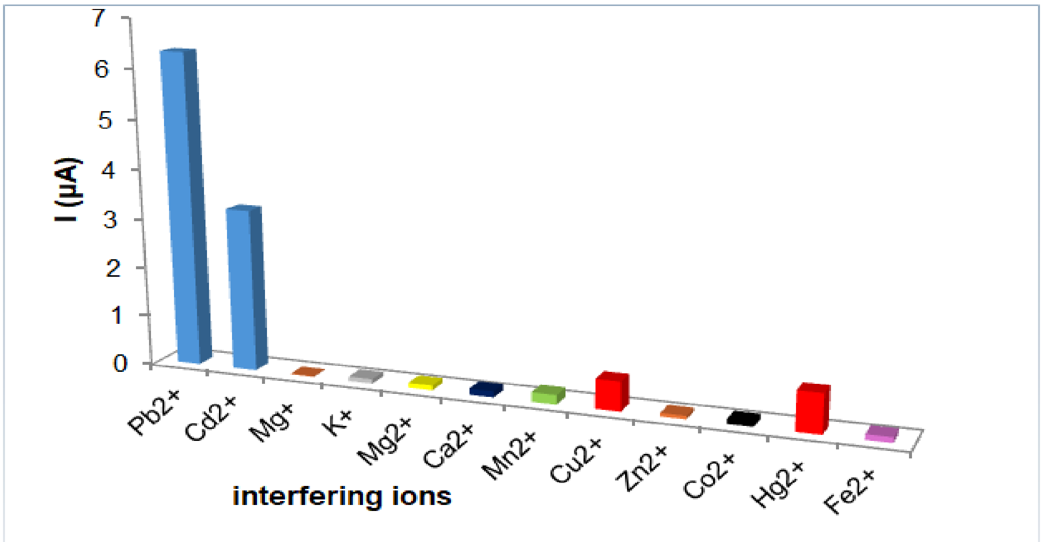

Figure.6. Effect of Various interfering ions on the electrochemical current response (I, µA) during the detection of lead (Pb^{+2}) and cadmium (Cd^{+2}) ions.

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Analytical applications

Optimal conditions for SWASV (Simulated Wave Amplitude Square Voltammetry) determination of Pb and Cd on GO/GCE were found in the construction of calibration curves. Fig. 7 displays these calibrations curves. The equations relating current to concentration together with correlation coefficients are I=0.2396C+0.6687, I=0.0874C+0.1294 where $r =$ 0.9991 and 0 .9989 for Pb and Cd respectively (C is concentrations $\mu g L^{-1}$, I current µA). Over a range from 0 .5 -50µgL-1 both plots showed perfect linearity(. Detection limits based on three times background noise were calculated as being equal to 0.09 and 0.23 $\mu g L^{-1}$). Reproducibility was checked by making ten consecutive measurements of Cu at GONPs/GC E Relative standard deviations came out as being equal t o1.84% and 1. 90% for 20 $\mu g L^{-1}$ of Pb an cd respectively (This means that this modified electrode improves not only sensitivity but also reproducibility when compared with another type).

Application of the new Pb and Cd modified electrode in real samples was one of the

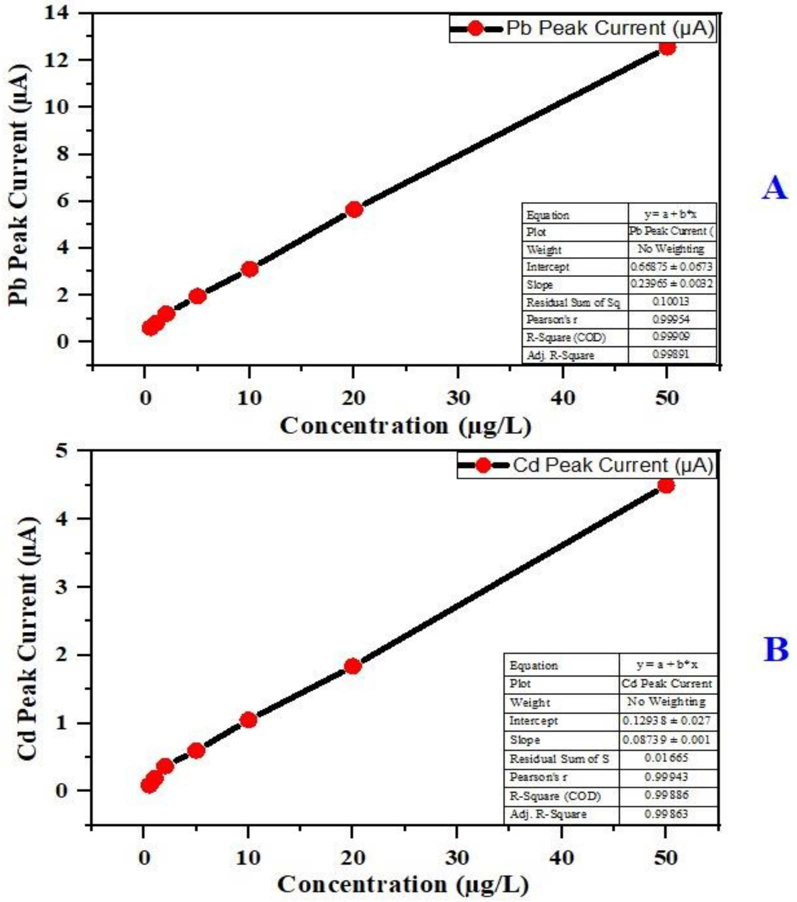

Figure.7. calibration Curve illustrating the relationship between ion concentration (µg/L) and electrochemical peak current (µA) obtained by anodic stripping

voltammetry (ASV) for lead (Pb^{+2}) and cadmium (Cd^{+2}) ions .

The first step in verifying this is the attainment of the primary requirements listed below. Standard additions of 5 μgL^{-1} Pb and Cd increased the current at the sample location; this made it possible to determine Pb and Cd in the water table and industrial water.

The concentration was determined by ASV from the calibration graph. data for the proposed lead and cadmium altered electrodes in Fig. 7. The fidelity of the accuracy was determined with experiments that fed the food back.(Table 1). It was found that the concentrations of lead and Cadmium in five different experiments were all consistent with the intended solution.

Table 1. Results of determination of Lead and Cadmium in samples of water

Sample	Sample cone. X^{30} µgL^{-1}	Pb-added X^{30} µgL^{-1}	Pb-found X^{30} µgL^{-1}	Recovery %
1	ND	5	4.91	98.2
2	0.03	5	5.04	100.8
Sample	**Sample cone. X^{30} µgL^{-1}**	**Pb-added X^{30} µgL^{-1}**	**Cd- found X^{30} µgL^{-1}**	**Recovery %**
1	0.02	5	5.01	100.2
2	0.04	5	5.06	101.2

- ❖ Average of three determinations (n=3)

Table 2. Several modified GO electrodes for determination of lead and cadmium ions

Electrode	Technic.	Linear range (µg L^{-1})		LOD (µg L^{-1})		Sample	Ref.
		Pb^{+2}	Cd^{+2}	Pb^{+2}	Cd^{+2}		
Bi/NA/rGO-AuNPs/GCE	SWASV	1-90	1-90	0.12	0.08	Soil	[22]
(BiO)2CO3/rGO/NA/GCE	SWASV	1 – 60	1 – 60	0.24	0.16	Water	[23]
BOC/GCE	DPASV	0 –10	0 – 5	4.30	3.97	Water	[24]
rGO/Sb/GCE	SWASV			9.427	8.218	chamomile tea	[25]

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Electrode	Technic.	Linear range (μg L^{-1})		LOD (μg L^{-1})		Sample	Ref.
Bi2O3/CS/GCE	DPASV	108 – 758	89.93 – 629.5	31.05	5.62	Water	[26]
SnO2/rGO/GCE	SWASV	0 – 269.36	0 – 146.133	0.0381	0.0114	water	[27]
rGO/NiWO4/C paper	DPASV	20.72 – 414.4	11.24 – 224.82	0.041	0.012	milk and fruit juices	[28]
Electrode Nafion/SnNPs WPVP/ITO	SWASV	10-100	10-100	1.97	1.44	Water	[29]
N-rGO@ppy/GCE	SWASV	1 – 500	1 – 500	0.080	0.029	drinking water, milk,	[30]
G-COOH-MWCNTs / ZnO on GCE	DPV	25 – 450	25 – 450	0.535	0.354	seawater samples	[31]
rGO/NDs/Bi	DPASV	10 - 100	10 - 100	2.4	0.61	Fish, shellfish, vegetables	[32]
LC-rGO-Bi	DPASV	1 – 30	1 – 30	0.08	0.10	Decorative Materials	[33]
GONPs/GCE	SWASV	0.5-50	0.5-50	0.09	0.23	water	This work

CONCLUSIONS

In this study, a facile and effective method was successfully developed for the highly sensitive and simultaneous determination of Pb and Cd using a GCE modified with Graphene Oxide Nanoparticles (GONPs). The integration of GONPs with the GCE provided a working electrode with a large surface area and enhanced properties, which significantly improved the accumulation and stripping efficiency of the metal ions. Optimized experimental parameters included a pH of 7, an accumulation time of 50 s at -1.2 V, and a 0.1 mol/L KNO_3 electrolyte concentration. The developed GONPs/ GCE exhibited a linear detection range from $0.5-50\ \mu g L^{-1}$ and achieved low detection limits of $0.09\ \mu g L^{-1}$ (Pb) and $0.23\ \mu g L^{-1}$ (Cd), which are competitive with established methods. The high reproducibility and excellent

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recovery rates demonstrated the reliability and practical applicability of the modified electrode for analyzing heavy metal contamination in real water samples. Therefore, the GONPs/GCE combined with SWASV is confirmed as a robust, sensitive, and effective tool for monitoring environmental Pb^{+2} and Cd^{+2} concentrations.

Author Contributions:

- **Lazim Abdullah Salih:** Data curation, Formal analysis, and Investigation, Writing,Methodology,Conceptualization, Writing Software, and original draft.
- **Nashwan H. Ali:** Review and editing, Visualization, Resources, Project administration, Validation and Supervision.

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