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Design of Iron Oxide Nanoparticle Sensors for Electrochemical Detection of Lead and Cadmium in Waste water samples

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Abstract

The present articlestudies the successful development and detailed characterization of a novel electrochemical sensor based on a carbon paste electrode (CPE) modified with iron oxide nanoparticles (Fe_2O_3-NPs) for detecting toxic metal ions in water samples. The iron oxide nanoparticles were thoroughly characterized using several techniques, including Fourier-transform infrared spectroscopy (FT-IR), field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD), and UV-Visible spectroscopy, to investigate their structural and morphological properties. The fabrication of the Fe_2O_3/CPE sensor was accomplished by homogeneously blending Fe_2O_3 nanoparticles with graphite powder and paraffin oil. This mixture was then compacted into a Teflon tube, with a copper wire serving as the electrical contact. Prior to use, the sensor surface was polished and rinsed with double-distilled water to ensure optimal performance.

Keywords: Iron oxide nanoparticles, electrochemical sensor, water samples, toxic metal, Pb(II), Cd(II)

Introduction

Water pollution is a major global issue affecting ecosystems and human health. It occurs when harmful substances like chemicals, heavy metals, and waste enter water bodies, degrades water quality [1]. Common sources include industrial discharges, agricultural runoff, and untreated sewage. Polluted water harms aquatic life and can spread diseases or toxic chemicals to humans through contaminated drinking water and food. Addressing water pollution is essential to protect both the environment and public health.

The increasing contamination of water sources by toxic heavy metals, such as lead (Pb) and cadmium (Cd), has become a significant environmental and public health concern [2-6]. These metals, even at trace levels, pose serious risks to human health and ecosystems due to their high toxicity, persistence, and tendency to bio-accumulate. As a result, the development of effective, reliable, and sensitive detection methods for monitoring heavy metal ions in water [7-8] has gained considerable attention. Traditional detection techniques, while accurate, often require expensive equipment, complex sample preparation, and lengthy analysis times.

Water pollution has become a pressing environmental issue in many regions, including Panipat, Haryana, which is known for its thriving industrial sector, particularly in textile manufacturing. The unchecked discharge of untreated industrial effluents into local water bodies has led to the widespread contamination of these water sources with hazardous chemicals, including heavy metals and synthetic dyes [9-13]. This pollution has not only degraded aquatic ecosystems but also raised serious public health concerns for local communities who rely on these water sources for drinking, irrigation, and other daily activities. Exposure to these pollutants can lead to long-term health issues, including skin diseases, respiratory problems, and other toxic effects.

While various wastewater treatment methods such as ion exchange, adsorption, photocatalytic degradation, sorption, advanced oxidation processes, nanotechnology, andInternet of Things(IoT) have been employed to mitigate pollution [14-23], many of these methods are either costly, energy-intensive, or limited in their scope of application. As a result, there is an urgent need for more efficient, cost-effective, and environmentally sustainable solutions to tackle water pollution in industrial areas like Panipat. The development of such technologies could significantly reduce pollution levels while protecting both the environment and public health.

In recent years, electrochemical sensors [24-26] have emerged as promising tools for environmental monitoring because of their sensitivity, simplicity, low cost, and potential for on-site analysis. The integration of nanomaterials [27-28], such as iron oxide nanoparticles (Fe_2O_3-NPs), into electrochemical sensors has significantly enhanced their performance. Metal oxides have attracted considerable attention among the various materials utilized in electrochemical sensors [29-35].Iron oxide nanoparticles [36-39] are particularly attractive due to their high surface area, excellent conductivity, and ease of modification, making them ideal candidates for detecting metal ions in aqueous environments.

The current study presents the development and characterization of a novel electrochemical sensor based on a carbon paste electrode (CPE) modified with Fe_2O_3-NPs. The sensor was designed to detect trace amounts of lead and cadmium in water samples, offering a cost-effective and efficient solution for environmental monitoring. Comprehensive characterization of the Fe_2O_3 nanoparticles was conducted to assess their structural and morphological properties, and the sensor's fabrication process was optimized to ensure high sensitivity and reliable performance.

ExperimentalMaterials used

All chemicals employed in the study were of analytical grade and were utilized as received, without undergoing any additional purification.Deionized water was used for all experimental procedures.

Instruments used

Fourier-transform Infrared Spectroscopy (FTIR) was conducted using a spectrometer with a wide-range infrared source and detector to identify chemical bonds and functional groups. Field Emission Scanning Electron Microscopy (FESEM) was employed to visualize the morphology and structure of nanoparticles at low voltages using a high-resolution electron microscope. Additionally, UV-Visible Spectroscopy was performed with a spectrophotometer capable of analyzing both UV and visible light spectra to investigate electronic transitions and optical properties. Electrochemical characterizations were carried out using a Potentiostat/Galvanostat (AUTOLAB-PGSTAT 302 N, Metrohm). The electrochemical cell was configured with three electrodes: a modified carbon paste electrode as the working electrode, a platinum wire (Pt) serving as the counter electrode, and a reference electrode.

Synthesis of the nanoparticles

Iron oxide nanoparticles were synthesized using a straightforward co-precipitation method [37], which is known for its simplicity and effectiveness. Initially, 10 grams of iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) was dissolved in 150 milliliters of deionized water, creating a clear solution while being stirred continuously at room temperature. To initiate the precipitation of iron oxide, 2 milliliters of ammonium hydroxide (NH_4OH) solution was added dropwise to the stirring mixture, at a rate of 1 milliliter per minute. Throughout this process, the pH was carefully maintained at 1 to ensure optimal conditions for nanoparticle formation.

Following the addition of the ammonium hydroxide, the resulting black dispersion was stirred continuously for one hour at room temperature to promote thorough mixing and particle formation. After this initial stirring period, the mixture was heated at 80 °C for two hours to facilitate the evaporation of excess water, resulting in the formation of a brown powder. Once the heating was completed, the product was allowed to cool to room temperature. Finally, to enhance the structural properties of the nanoparticles, the brown powder was calcined at 500 °C for four hours. This calcination process not only helps in achieving the desired crystalline structure but also improves the thermal stability of the synthesized iron oxide nanoparticles.

Characterization of nanoparticles

The synthesized iron oxide (Fe_2O_3)nanoparticles have undergone characterization through various analytical techniques to assess their properties comprehensively. Fourier-transform infrared spectroscopy (FT-IR) was employed to identify specific functional groups and analyze the chemical composition of the nanoparticles. Field emission scanning electron microscopy (FESEM) provided high-resolution images of the nanoparticle surfaces, enabling detailed observation of their morphology and size distribution. Furthermore, UV-Visible spectroscopy was utilized to examine the optical properties and electronic structure of the nanoparticles, aiding in the determination of their absorption characteristics and bandgap energies. These characterization methods collectively contribute to a deeper understanding of the synthesized iron oxide nanoparticles and their potential applications.

Fabrication of electrochemical sensors from Fe_2O_3NPs

The fabrication of an iron oxide-based electrochemical sensor involves several key steps to ensure optimal performance in detecting target metal ions. Initially, a carbon paste electrode (CPE) is prepared by mixing conductive graphite powder with a suitable binder, such as paraffin oil, in an optimized ratio to achieve the desired mechanical properties and electrical conductivity.Iron oxide nanoparticles (Fe_2O_3) are produced through a co-precipitation technique [37], wherein a 0.5 M solution of $FeCl_3 \cdot 6H_2O$ is reacted with a 1 M NaOH solution at a pH range of 9–11, thereby ensuring that the nanoparticles exhibit the requisite dimensions and structure. Subsequently, these nanoparticles are integrated into the carbon paste formulation at a predetermined concentration, generally varying from 5% to 20% by weight, to augment the electrochemical characteristics of the electrode. The resulting carbon paste containing iron oxide nanoparticles is packed into a Teflon tube, forming the working electrode, with a copper wire inserted to serve as the electrical connector. Before use, the surface of the electrode is polished to ensure a smooth and clean surface for reliable electrochemical measurements, followed by rinsing with deionized water to remove any loose particles. The electrochemical cell is then assembled with the modified CPE as the working electrode, a platinum wire as the counter electrode, and a reference electrode Silver/Silver Chloride (Ag/AgCl) to maintain a stable reference potential during measurements. Prior to conducting experiments, the sensor is calibrated using standard solutions of target heavy metals, to establish a relationship between current response and concentration. The performance of the sensor is evaluated through electrochemical techniques, such as cyclic voltammetry, assessing its sensitivity, selectivity, and detection limits. This systematic approach enables the development of a reliable and efficient tool for detecting toxic metal ions in environmental samples, ultimately contributing to the improved water quality monitoring and public health safety.

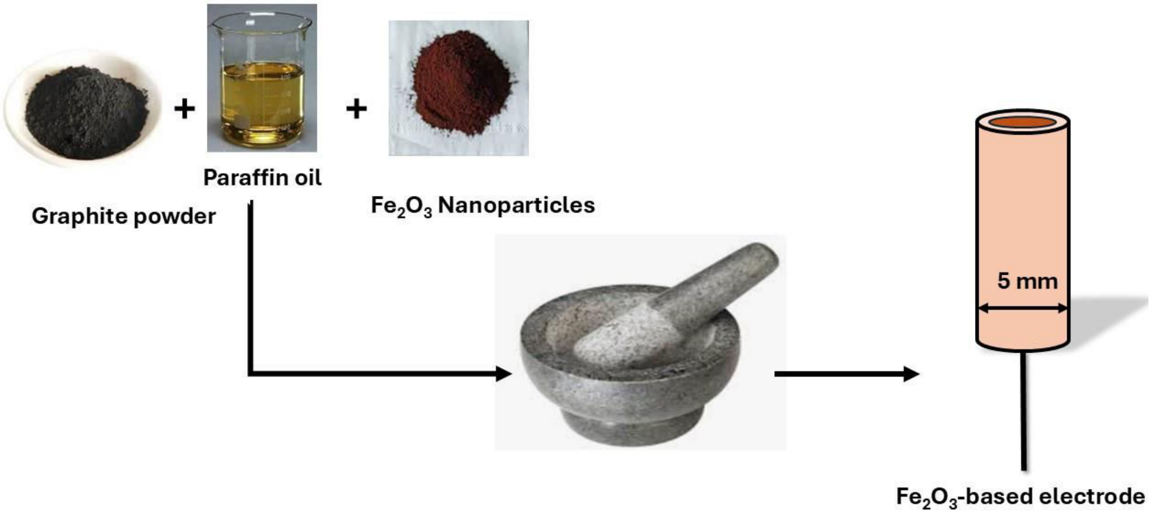

Figure 1: Schematic representation of the fabrication process for the Fe_2O_3based electrode.

Electrochemical study

Differential Pulse Voltammetry (DPV) measurements were conducted in 0.1 mol/L sodium acetate/acetic acid(NaAc/HAC) buffer (pH 5) containing Cd^{2+} and Pb^{2+}, selected as the optimal electrolyte, within a potential range of -0.1 to -0.4 V. All electrochemical responses were recorded at room temperature (25±2 °C). After stirring and pre-concentrating the solution at a constant potential for a specified duration, the mixture was allowed to rest for 10 seconds before being analyzed. The potential scanning range was set from -1.1 V to -0.4 V, with an amplitude of 25 mV, a potential increment of 4 mV, and a frequency of 15 Hz.

Real water sample analysis

Water sampleswere collected from the vicinity ofNauhra-AssanKalan,National fertilizer limited, Sat kartarnagar, Industrial area (Sector-29),Dadlana of Panipat, Haryana in the pre-cleaned water bottles. This sample was tested for lead and cadmium using the studyed electrochemical sensor. Simultaneously, a portion of the same sample was sent to an outsourced agency for lead and cadmium testing. The results are presented in Table 1 and discussed in the results and discussion section.

Results and Discussion

Fe_2O_3nanoparticles were synthesized through the chemical co-precipitation method described in the experimental section and characterized using Fourier Transform Infrared Spectroscopy (FTIR), as illustrated in Figure 2. The FTIR spectrum of Fe_2O_3nanoparticles exhibits several characteristic peaks, offering valuable insights into the chemical bonds and functional groups within the material.

The spectrum [40-41] shows distinct peaks at 536.45 cm^{-1} and 698.15 cm^{-1}, which correspond to the Fe-O bond stretching and bending vibrations, confirming the presence of iron oxide. The peak at 1487.55 cm^{-1} is associated with the bending vibrations of hydroxyl groups (-OH). The peak at 1695.35 cm^{-1} corresponds to C=O stretching, which may indicate the presence of carbonyl-containing groups, potentially from surface-bound functional groups formed during the synthesis process. Finally, the broad peak at 3385.35 cm^{-1} is attributed to O-H stretching vibrations, reflecting the presence of surface-bound water or hydroxyl groups. These features highlight the chemical composition and surface interactions of the Fe_2O_3 nanoparticles.

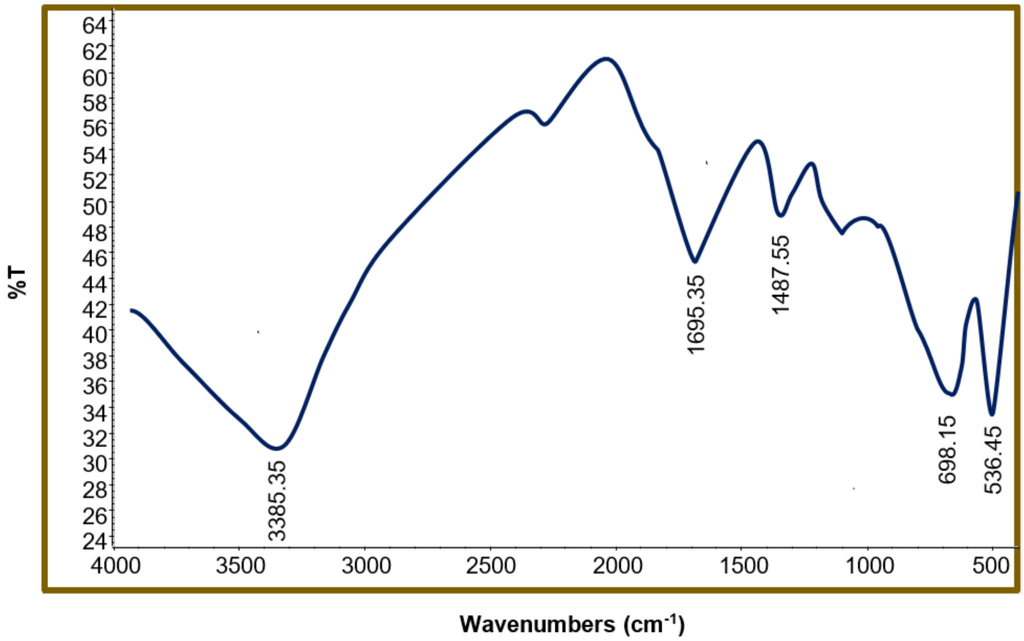

Figure 2: FTIR spectra of Fe_2O_3nanoparticles.

The SEM analysis of Fe_2O_3nanoparticles (revealed in Figure 3), conducted at an accelerating voltage (HV) of 25 kV, with a magnification of 35 KX and a resolution of 10 nm, offers detailed insight into the morphology and size distribution of the nanoparticles. At this magnification and resolution, the SEM image reveals a uniform and spherical shape of the Fe_2O_3nanoparticles, with relatively smooth surfaces. The nanoparticles appear well-dispersed with minimal agglomeration, indicating good synthesis quality. The average particle size observed is within the nanometer scale, typically ranging between 10-20 nm. The high-resolution imaging highlights the clear boundaries of individual particles, which helps in understanding their surface characteristics. The detailed structural visualization at 25 kV also provides insights into the porosity and surface area of the nanoparticles, which are critical for applications like catalysis, sensing, and environmental remediation. These findings confirm that the synthesized Fe_2O_3nanoparticles are of high quality, exhibiting desirable size and morphology for nanotechnology applications.

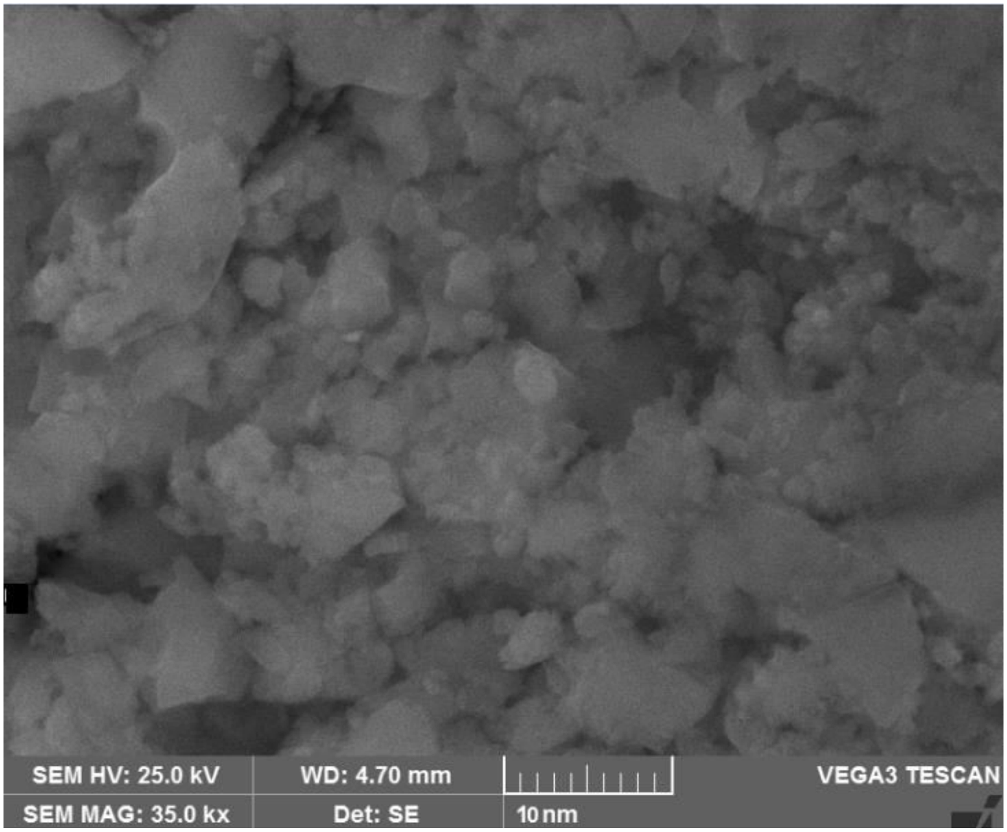

Figure 3: SEM image of Fe_2O_3nanoparticles.

The UV-Visible (UV-Vis) spectrum of Fe_2O_3nanoparticles, depicted in Figure 4, reveals a distinct absorption band characterized by a single peak at a wavelength of 250 nm. This peak indicates electronic transitions within the material and corresponds to the bandgap energy of the Fe_2O_3nanoparticles, signifying the movement of electrons from the valence band to the conduction band. Additionally, the specific wavelength of 250 nm suggests that the Fe_2O_3nanoparticles has semiconductor properties, highlighting its potential applications, and suggests a high degree of purity and uniformity in the synthesized nanoparticles.

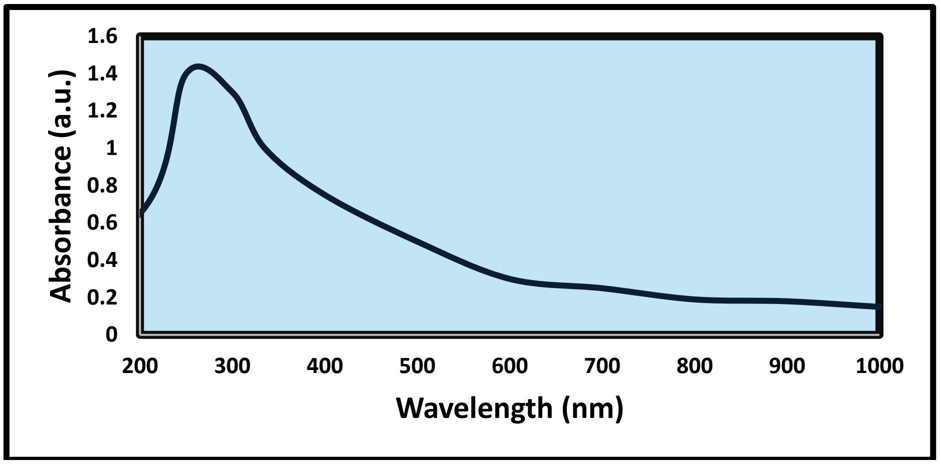

Figure 4: UV-Visible spectra of Fe_2O_3nanoparticles.

The X-ray diffraction (XRD) pattern of Fe_2O_3nanoparticles reveals prominent peaks at the (012), (024), (104), (113), and (214) planes, indicating a well-crystallized rhombohedral structure [37] typical of hematite (α-Fe_2O_3). The (012) and (024) peaks correspond to the basal planes of the crystal, confirming strong crystallographic alignment, while the (104) peak is a significant indicator of the hematite phase, validating the material's composition. The presence of peaks at (113) and (214) further supports the high crystallinity of the nanoparticles. These diffraction patterns align with previously studyed values for α-Fe_2O_3, confirming the successful synthesis of high-purity, well-ordered iron oxide nanoparticles. The crystalline nature of these nanoparticles is essential for their application in areas such as catalysis and sensing, where the structure influences performance.

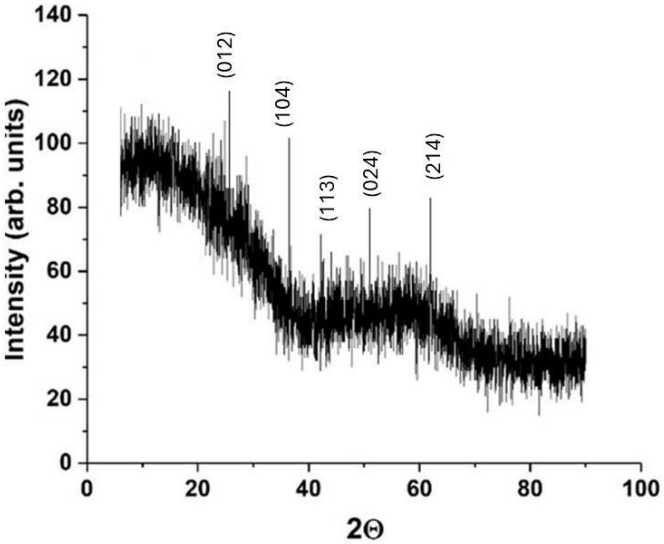

Figure 5: XRD spectra of Fe_2O_3nanoparticles.

The sensor has been successfully fabricated and is currently undergoing testing for the detection of toxic metal ions in real-time water samples collected from Panipat, Haryana. This testing is crucial, given the region's high industrial activity, particularly in the textile and chemical sectors, which can lead to the contamination of local water sources with hazardous metal ions. Such contamination poses significant health risks to the local population. The development and evaluation of this sensor aim to tackle these environmental and public health issues by offering a reliable method for monitoring water quality and safeguarding community health.

The Differential Pulse Voltammetry (DPV) measurements of Fe_2O_3-modified electrodes for the detection of Cd^{2+} and Pb^{2+} ions exhibit distinct peaks at -0.51 V and -0.79 V, respectively, as depicted in Figure 6. These peaks correspond to the reduction potentials of cadmium and lead ions, which occur due to the electrochemical reduction of these metal ions on the modified electrode surface. The appearance of these peaks at designated potentials indicates the successful modification of the electrode with Fe_2O_3nanoparticles, enhancing its sensitivity towards the detection of these toxic metal ions.

The peak at -0.51 V is attributed to the reduction of Cd^{2+} to Cd^0, while the peak at -0.79 V corresponds to the reduction of Pb^{2+} to Pb^0. The well-defined separation of these peaks reflects the electrode's ability to distinguish between the two metal ions in a mixture, indicating high selectivity. The Fe_2O_3-modified electrode provides improved electrochemical activity, offering a stable and reproducible platform for detecting Cd^{2+} and Pb^{2+} even at trace levels in water samples. This enhanced sensitivity is critical for environmental monitoring applications, particularly in the detection of heavy metal contamination in water sources.

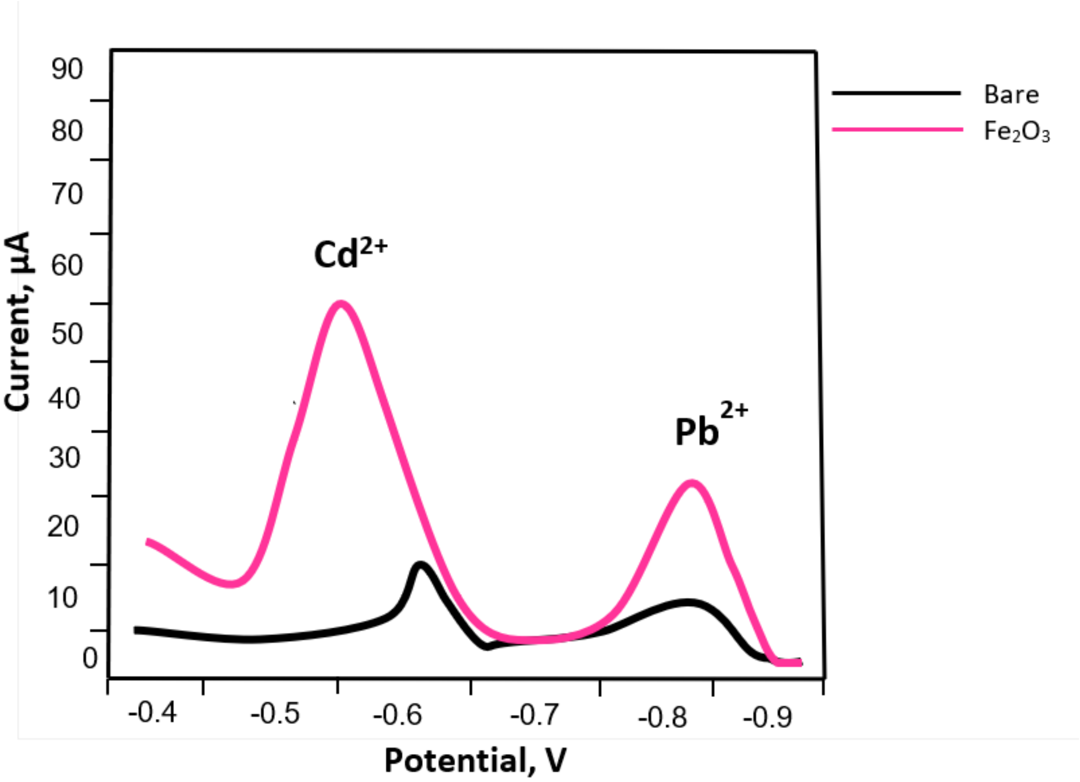

Figure 6:DPV signals of the Fe_2O_3CPE electrode, compared with the bare electrode for $Cd^{2+}$and Pb^{2+} ions.

Here is the line chart depicting the concentrations of Cd(II) and Pb(II) ions in real water samples based on our data. It compares the values detected by the present study with those obtained via the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) method [43].

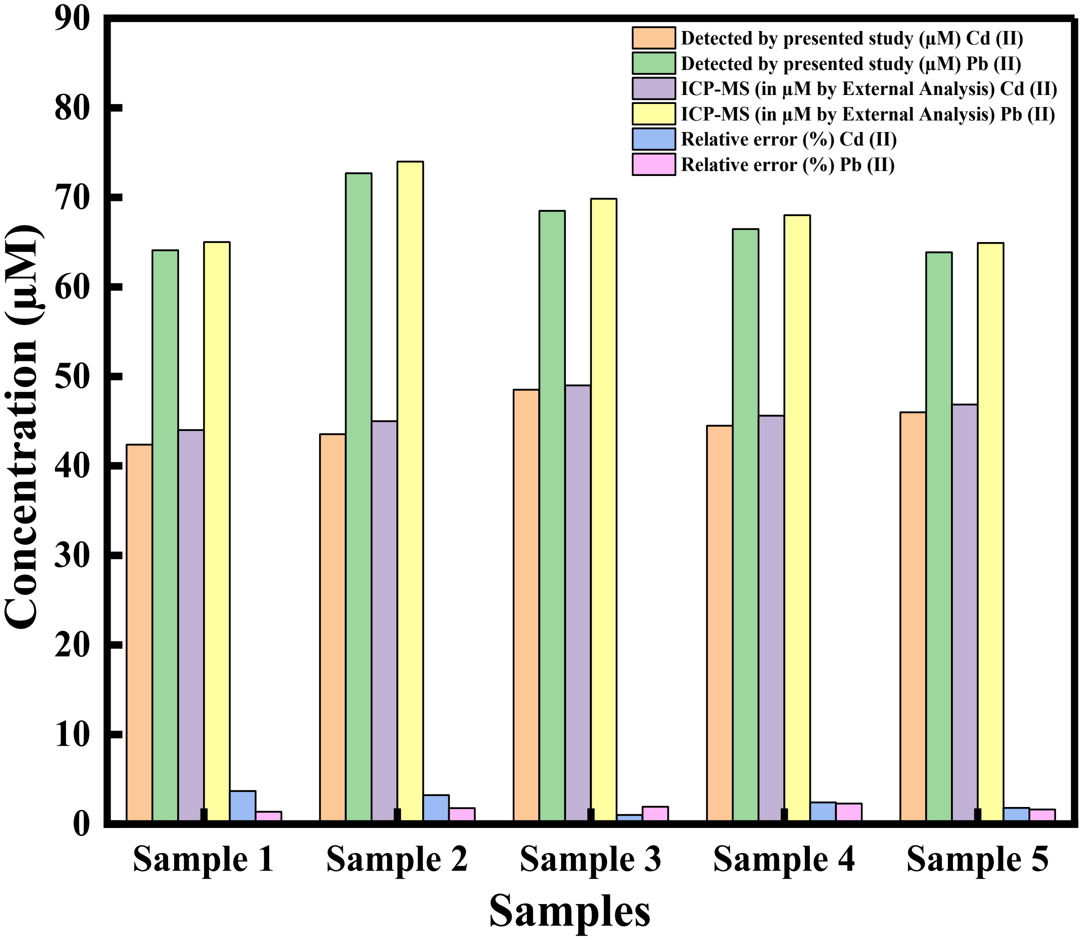

Figure 7: Concentrations of Cd(II) and Pb(II) ions in real water samples with relative errors.

The practical evaluation of the modified electrodes was conducted using thereal water samples from Panipat, Haryana. Before measurement, the samples were filtered to remove large particulates. The results obtained with the modified electrodes were compared to those from ICP-MS (shown in Table 1). The Differential Pulse Voltammetry (DPV) measurements for the Fe_2O_3-modified electrode demonstrated strong accuracy in detecting Pb(II) and Cd(II) concentrations in water samples. For Pb(II), the concentrations measured were approximately 65 µM, 74 µM, 69.85 µM, 68 µM, 64.9 µMforfive samples, with relative errors of 1.38%, 1.78%, 1.93%, 2.28% and 1.62% respectively, when compared to the Inductively Coupled Plasma Mass Spectrometry (ICP-MS) results. Similarly, for Cd(II), concentrations of 44 µM, 45 µM, 49 µM, 45.6 µM and 46.85 µM were detected, with relative errors of 3.68%, 3.22%, 1.02%, 2.41% and 1.81%, respectively compared to the ICP-MS results as shown in figure 7,8. These low relative errors suggest that the Fe_2O_3-modified electrode offers a high degree of accuracy and reliability. This consistency between the DPV and ICP-MS results indicates that the modified electrode holds significant potential for practical applications in environmental monitoring, specifically for the detection of toxic metal ions such as lead and cadmium in water samples.

Table 1: Comparison of Fe_2O_3-modified electrodes and ICP-MS methods for determining Cd(II) and Pb (II) ions in a real water samples.

Ions, to be detected	Detected by present study (μM)		Detected by ICP-MS (μM) [43]		Relative error (%)	
	Cadmium (II)	Lead (II)	Cadmium (II)	Lead (II)	Cadmium (II)	Lead (II)
Sample 1	42.38	64.10	44	65	3.68	1.38
Sample 2	43.55	72.68	45	74	3.22	1.78
Sample 3	48.5	68.5	49	69.85	1.02	1.93
Sample 4	44.5	66.45	45.6	68	2.41	2.28
Sample 5	46	63.85	46.85	64.9	1.81	1.62

Conclusion

The Fe_2O_3-modified electrochemical sensor has shown promising results for the detection of Cd^{2+} and Pb^{2+} ions in real wastewater samples from Panipat, Haryana. The sensor demonstrated high sensitivity and selectivity, with low relative errors when compared to ICP-MS measurements, confirming its practical utility for environmental monitoring. Extensive characterization of the Fe_2O_3nanoparticles, using techniques such as XRD, SEM, FTIR, and UV-Vis spectroscopy, confirmed the crystallinity, morphology, functional groups, and optical properties of the synthesized nanoparticles, contributing to the sensor's excellent performance. These findings suggest that the Fe_2O_3-modified electrode can be effectively applied in real-world wastewater treatment scenarios, offering a reliable and cost-efficient solution for detecting toxic metal contamination in industrial regions.

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