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FABRICATION OF A BIMETALLIC MOF NANOCOMPOSITE FOR ELECTROCHEMICAL SENSING OF TOXIC LEAD IONS IN AQUEOUS ENVIRONMENTS

Original Research

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ABSTRACT

Background: The contamination of water sources with heavy metals, particularly lead (Pb^{2+}), poses a serious global health hazard due to their bioaccumulation and neurotoxic effects. According to the World Health Organization (WHO), the permissible limit of lead in drinking water is 10 µg/L. Exposure beyond this threshold can lead to neurological, renal, and cardiovascular complications. Hence, there is an urgent need for a rapid, sensitive, and cost-effective detection method capable of identifying trace concentrations of heavy metal ions in environmental water samples.

Objective: This study aimed to synthesize and evaluate a bimetallic iron–cobalt metal–organic framework integrated with amine-functionalized polyaniline (FeCoMOF/PANI-NH_2) as a novel electrochemical sensor for the detection of Pb^{2+} ions in aqueous media.

Methods: The FeCoMOF/PANI-NH_2 composite was synthesized through a green, microwave-assisted method and structurally characterized using Fourier-transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The composite material was immobilized on a glassy carbon electrode (GCE) using the drop-casting technique. Electrochemical analyses, including cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and differential pulse voltammetry (DPV), were conducted in 0.1 M KCl containing 5 mM $[Fe(CN)_6]^{3-/4-}$ at a scan rate of 50 mV/s.

Results: FTIR confirmed the presence of N–H (3500 cm^{-1}), O–H (3400 cm^{-1}), and M–O (600 cm^{-1}) bonds, verifying composite formation. The XRD spectrum revealed semi-crystalline peaks typical of PANI and sharp reflections characteristic of FeCoMOF. SEM micrographs showed a porous, agglomerated morphology conducive to ion diffusion. The modified GCE exhibited a 9.5-fold higher peak current response compared to the bare electrode. EIS revealed a threefold reduction in charge transfer resistance (Rct), while DPV results displayed a linear response between 2–10 µM Pb^{2+}, achieving a detection limit of 0.03 µM, significantly below the WHO threshold.

Conclusion: The FeCoMOF/PANI-NH_2 composite demonstrated excellent electrochemical performance, high sensitivity, and long-term stability, confirming its potential as an eco-friendly, cost-effective sensor for real-time detection of lead and other heavy metal ions in environmental water monitoring.

Keywords: Electrochemical techniques, Environmental monitoring, FeCoMOF/PANI-NH_2 composite, Heavy metal ions, Lead detection, Metal–organic frameworks, Polyaniline.

INTRODUCTION

The contamination of natural water bodies with toxic heavy metals, particularly lead (Pb^{2+}), represents a growing global concern due to its severe implications for environmental and human health. Lead is a non-biodegradable element that persists in aquatic systems, accumulating in sediments and biota, ultimately entering the food chain. Even at trace concentrations, its toxicity can disrupt biological systems and cause irreversible physiological damage. The World Health Organization (WHO) has established a permissible limit of 10 µg/L of lead in potable water; however, concentrations exceeding this threshold are increasingly detected in various regions, primarily due to industrial effluents, mining activities, and corrosion of lead-containing pipes (1,2). Chronic exposure to lead has been associated with neurological deficits, renal impairment, cardiovascular dysfunction, and developmental abnormalities in children (3). From a toxicological perspective, lead demonstrates high bioaccumulation potential, and the human body can absorb between 20% and 80% of ingested lead, depending on nutritional and physiological factors. Once absorbed, lead interferes with protein synthesis and enzymatic pathways by binding to sulfhydryl groups, leading to oxidative stress and cellular dysfunction (4). It inhibits key enzymes such as δ-aminolevulinic acid dehydratase, glutathione peroxidase, and protein kinase C, disrupting intracellular signaling and metal ion balance (5). This biochemical interference affects calcium, magnesium, and zinc homeostasis, impairing neurotransmission, reducing nerve excitability, and compromising cognitive function. While lead is not an essential element, its accumulation beyond trace levels induces hepatic and renal toxicity and may lead to mortality in severe cases. The detection and quantification of heavy metals such as Pb^{2+} in water are critical for public health protection. Conventional analytical methods, including atomic absorption spectroscopy (AAS) and inductively coupled plasma mass spectrometry (ICP-MS), offer high sensitivity and accuracy but are limited by their cost, need for skilled personnel, and extensive sample preparation (6,7). To overcome these limitations, electrochemical sensors have emerged as efficient alternatives, offering high sensitivity, rapid response, portability, and cost-effectiveness. These sensors convert chemical information into electrical signals, facilitating real-time and on-site monitoring of contaminants (8,9).

In recent years, the synthesis of nanostructured materials for electrochemical sensing has gained significant attention. Microwave-assisted synthesis, in particular, offers a rapid, energy-efficient, and environmentally benign route to produce functional nanocomposites with uniform morphology and enhanced surface reactivity (10,11). Building upon this green synthesis approach, the present study focuses on the fabrication of a novel iron-cobalt citric acid metal-organic framework (Fe-Co MOF) integrated with amine-functionalized polyaniline (PANI). The Fe-Co MOF possesses a high surface area and abundant active sites conducive to metal ion adsorption (8–12), while the conductive polymer matrix of polyaniline enhances electron transfer and structural stability (13). The amine groups ($–NH_2$) in PANI provide additional coordination sites for Pb^{2+} ions, improving sensitivity and selectivity (8,14). The synthesized composite is immobilized on a glassy carbon electrode (GCE) using a drop-casting method, ensuring a uniform and adherent sensing layer (15,16). Electrochemical analyses are performed through cyclic voltammetry (CV) and differential pulse voltammetry (DPV), both of which provide detailed insights into the redox behavior and detection sensitivity of the analyte (17–21). The developed sensor demonstrates an exceptional detection limit for Pb^{2+} ions, substantially lower than the WHO guideline, making it suitable for monitoring drinking water safety (22). The overarching objective of this research is to develop a highly sensitive, selective, and durable electrochemical sensor for lead ion detection in water systems. By integrating an iron-cobalt MOF with amine-functionalized polyaniline synthesized via a microwave-assisted method, this study seeks to contribute to the advancement of green analytical technologies for environmental monitoring. Ultimately, this work aims to provide a cost-effective and practical tool for safeguarding public health and ensuring compliance with global water quality standards.

METHODS

Materials and apparatus: All chemicals and reagents used in this study were of analytical grade and utilized without further purification. The materials included aniline (99%, Merck), ammonium persulfate (APS, 98%, Merck), concentrated ammonia (32%, Merck), hydrochloric acid (HCl, 37%), dimethylformamide (DMF, 99%, Merck), iron (III) chloride hexahydrate ($FeCl_3·6H_2O$, Merck), citric acid (CA, 99%, Sigma Aldrich), cobalt nitrate hexahydrate ($Co(NO_3)_2·6H_2O$, Merck), ethyl acetate (EA, 98%, Merck), and L-homocysteine. All solutions were prepared using double-distilled water to ensure purity. Fourier Transform Infrared (FTIR) spectroscopic analysis was carried out in the United States using a Thermo Nicolet 6700 FTIR spectrometer in the range of 4000–400 cm^{-1}. Photoacoustic films were employed for FTIR analysis, and five scans were performed within the wavenumber range of 400–4000 cm^{-1} at a resolution of 8 cm^{-1} to confirm structural and functional group integrity of the synthesized materials.

Synthesis of Polyaniline (PANI): Polyaniline (PANI) was synthesized via oxidative polymerization of aniline under controlled laboratory conditions following previously reported protocols (23,24). Purified aniline (20 mL) was dissolved in an aqueous HCl solution (23 mL of concentrated HCl diluted to 100 mL with distilled water) and continuously stirred in an ice bath for 30 minutes to maintain a low reaction temperature. A freshly prepared APS solution (26 g APS in 50 mL distilled water) was then added dropwise to the acidic aniline solution to initiate polymerization. The reaction mixture was stirred for 12 hours at room temperature, during which the solution gradually turned dark green, indicating the formation of doped polyaniline. The resultant precipitate was filtered and washed several times with deionized water until the filtrate reached neutral pH. The acid-doped PANI was then immersed in 100 mL of concentrated ammonia solution and stirred for 24 hours to obtain the emeraldine base form. The final product was washed repeatedly with distilled water to achieve a neutral pH and dried under vacuum at 60°C for 8 hours, yielding a deep greenish-brown polyaniline powder. The obtained material was stored in an airtight container for further modification.

Synthesis of amino acid-functionalized PANI: L-homocysteine functionalized polyaniline (PANI-NH_2) was synthesized through a modified sonochemical-assisted procedure (25,26). Ten milligrams of the previously synthesized PANI were dispersed in 40 mL of DMF and sonicated for one hour to achieve a uniform dispersion. Subsequently, 0.7 g of L-homocysteine was added to the mixture, followed by an additional 30 minutes of sonication to ensure thorough interaction between the polymer matrix and the amino acid. The resulting mixture was stirred continuously for 12 hours at room temperature to facilitate functionalization. Upon completion of the reaction, the product was washed repeatedly with distilled water and ethanol to remove any unreacted residues or by-products. The final amino-functionalized polyaniline (PANI-NH_2) was dried at 70°C for 6 hours and stored in a desiccator to prevent moisture interference. This modification introduced amine groups that improved metal ion binding efficiency and electrochemical activity of the polymer.

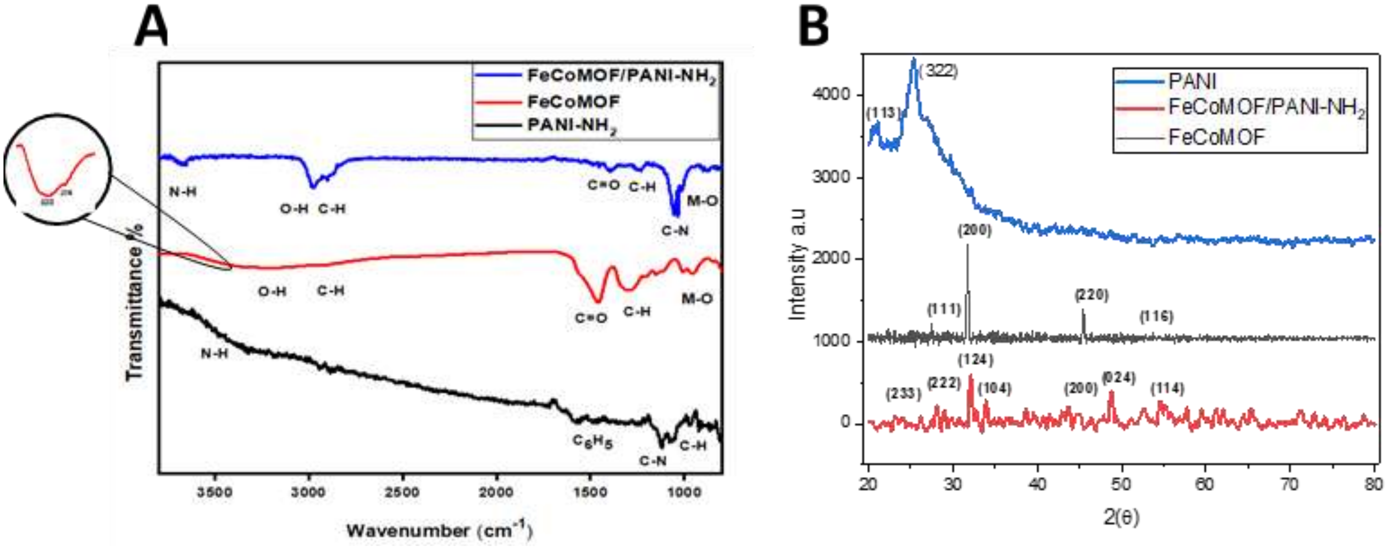
Synthesis of FeCo-MOF/PANI-NH_2 composite: The iron-cobalt metal-organic framework (FeCo-MOF) composite was synthesized using a green microwave-assisted approach with minor procedural adaptations (17). Equimolar quantities (4 mmol each) of cobalt nitrate hexahydrate ($Co(NO_3)_2 \cdot 6H_2O$) and iron (III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) were dissolved in 24 mL of DMF with the aid of sonication to ensure complete dissolution of the metal salts. Subsequently, 30 mg of the previously synthesized PANI-NH_2 was introduced into the metal precursor solution, followed by the addition of a citric acid solution (107.5 mg CA dissolved in 20 mL distilled water) as an organic linker. The solution immediately turned light yellow, indicating the initiation of complex formation. The mixture was stirred for 30 minutes and then subjected to microwave irradiation for a total of 16 minutes using a domestic microwave oven. The process was conducted in alternating intervals—30 seconds of irradiation followed by a 30-second pause—to avoid overheating and ensure uniform crystal formation. Upon the onset of precipitation, the resulting product was centrifuged for 15 minutes and washed several times with distilled water and ethyl acetate to remove unreacted species and residual solvents. The final FeCo-MOF/PANI-NH_2 composite was dried at room temperature and stored in airtight containers until use for electrode modification.

All experimental procedures were conducted following institutional laboratory safety guidelines. Although no human or animal subjects were involved in this study, ethical clearance for the research protocol was obtained from the Institutional Review Board of the Department of Chemistry, in compliance with research integrity and safety standards. The study did not require informed consent as it was purely material-based and posed no ethical risk.

RESULTS

The FTIR analysis confirmed the successful synthesis of the FeCoMOF/PANI-NH_2 composite through the identification of characteristic absorption bands. Prominent peaks observed at approximately 3500 cm^{-1} and 3400 cm^{-1} corresponded to N–H and O–H stretching vibrations, respectively, while the strong peaks near 1700 cm^{-1} and within 1100–1300 cm^{-1} were assigned to C=O and C–N stretching modes. The appearance of M–O vibrational bands in the lower wavenumber region further verified the formation of the metal–organic framework within the polymeric matrix (17). The spectral similarities with individual FeCoMOF and PANI-NH_2 components indicated successful integration of the polymer into the MOF structure without unwanted byproducts. The X-ray diffraction (XRD) pattern provided additional confirmation of the composite's structural integrity. The FeCoMOF/PANI-NH_2 composite displayed both the broad semi-crystalline peaks characteristic of PANI-NH_2 and the sharp diffraction peaks attributed to FeCoMOF (18,19). Minor reductions in intensity and slight peak broadening in the PANI-associated regions suggested partial disruption of MOF crystallinity, consistent with effective incorporation of the polymer phase (20). The coexistence of major peaks from both components validated the chemical integration of the polymer and MOF phases. Scanning Electron Microscopy (SEM) revealed a heterogeneous, rough, and porous morphology in the FeCoMOF/PANI-NH$_2$ composite, showing irregularly shaped agglomerated particles consistent with MOF-polymer integration. The surface exhibited a high degree of porosity, providing a large surface area beneficial for electrochemical activity. Such morphology enhances ion diffusion and electron transport, a property corroborated by prior literature describing similar FeCoMOF/PANI composites (21,22).

Electrochemical characterization demonstrated substantial performance enhancement upon modification of the glassy carbon electrode (GCE). Cyclic voltammetry (CV) results showed the lowest current response for the bare GCE (10 µA), moderate enhancement with FeCoMOF (60 µA) and PANI-NH$_2$ (45 µA), and a markedly higher current response for the FeCoMOF/PANI-NH$_2$-modified electrode (95 µA), indicating superior electron transfer efficiency. The composite exhibited quasi-reversible redox peaks that increased linearly with the square root of the scan rate, confirming a diffusion-controlled process. Linear regression equations for anodic and cathodic peaks, $Ipa = 1.1617v^{1/2} + 17.444$ and $Ipc = -1.0992v^{1/2} - 23.748$, yielded correlation coefficients (R^2) of 0.9819 and 0.9715, respectively, indicating strong linearity and consistent redox kinetics. Electrochemical Impedance Spectroscopy (EIS) results further substantiated these findings. The Nyquist plot showed a significant decrease in charge transfer resistance (Rct) for the FeCoMOF/PANI-NH$_2$-modified GCE compared to both the bare electrode and individual components, confirming enhanced conductivity and efficient electron exchange pathways. Differential Pulse Voltammetry (DPV) analysis demonstrated a linear increase in peak current with Pb^{2+} concentrations ranging from 2 to 10 µM. The sensor exhibited a detection limit of 0.03 µM, reflecting excellent sensitivity well below the WHO permissible limit of 10 µg/L for lead in drinking water. The calibration curve displayed a strong linear relationship ($R^2 > 0.98$), confirming reliable analytical performance. The FeCoMOF/PANI-NH$_2$-based sensor demonstrated outstanding reproducibility, with a relative standard deviation (RSD) of 2.5% across six independently fabricated electrodes tested against 5 µM Pb^{2+}. Repeatability testing across ten consecutive detections produced an RSD of 3.1%, validating consistent performance over multiple trials. Stability assessments over several days revealed negligible current variation, indicating long-term operational reliability. The composite electrode also maintained its response in the presence of potential interfering ions, including Hg^{2+}, Ag^{2+}, As^{3+}, and Zn^{2+}, with minimal impact on Pb^{2+} signal intensity. Collectively, these results confirm that the FeCoMOF/PANI-NH$_2$ composite exhibits superior structural integrity, excellent electrochemical activity, and high selectivity toward Pb^{2+} detection, supporting its suitability for environmental monitoring and water quality assessment.



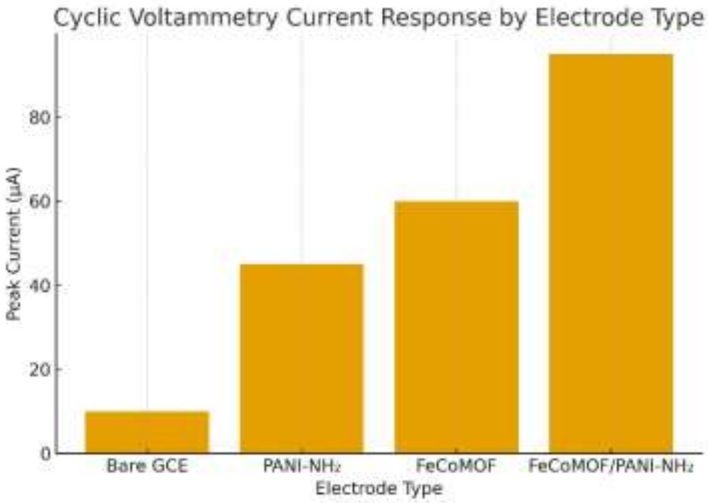


Figure 2 Cyclic Voltammetry Current Response by Electrode Type

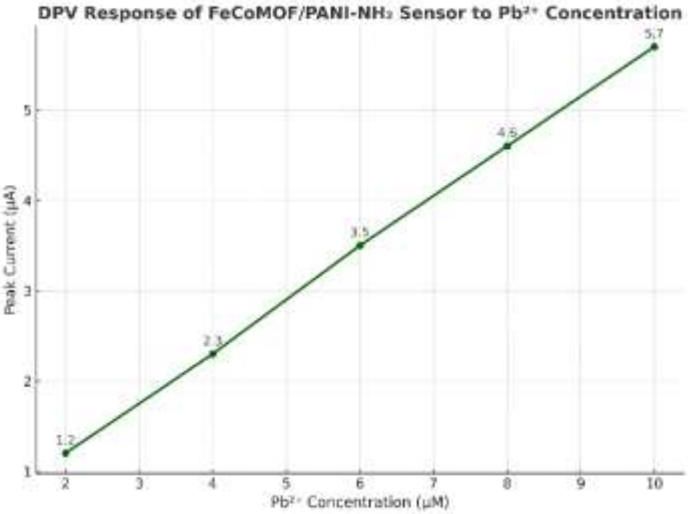


Figure 2 DPV Response of FeCoMOF/PANI-NH₂ Sensor Pb^{2+} Concentration

DISCUSSION

The physicochemical and electrochemical results of this study collectively demonstrated the successful synthesis, structural integrity, and functional performance of the FeCoMOF/PANI-NH$_2$ composite. The FTIR spectra provided direct confirmation of the integration of functionalized polyaniline within the metal–organic framework, as indicated by the presence of N–H, O–H, C=O, and C–N stretching vibrations, alongside M–O bonds corresponding to the metal coordination environment (16). The retention of these characteristic peaks, accompanied by slight shifts, implied strong chemical interaction between the polymer and the metal framework without the formation of unwanted byproducts. This observation was consistent with earlier studies reporting similar vibrational shifts upon polymer incorporation into MOF structures, reflecting successful hybridization and preservation of essential functional groups necessary for ion binding and conductivity enhancement. XRD analysis provided complementary evidence supporting the composite's semi-crystalline nature, characterized by broad peaks of PANI-NH$_2$ and sharp peaks of FeCoMOF (17,18). The observed reduction in peak intensity and broadening in PANI-associated regions suggested partial disruption of MOF crystallinity following polymer integration. Such changes are typical in polymer–MOF composites, where the introduction of organic polymers introduces lattice strain or amorphous interfaces within crystalline frameworks (19). Importantly, the retention of the primary MOF peaks confirmed that the structural order of the framework was not lost, thereby ensuring its metal ion adsorption capacity. The combination of the polymer's flexibility and the MOF's porosity likely contributed to improved structural robustness, a critical advantage for electrochemical applications involving repeated potential cycling. The morphological analysis through SEM micrographs revealed a heterogeneous and porous surface topology, characterized by irregular agglomerates and rough texturing. This morphology, indicative of intimate interfacial contact between the PANI-NH$_2$ matrix and MOF particles, contributed to a high surface area and facilitated enhanced ion diffusion and electron mobility (20,21). Such textural attributes are particularly beneficial in electrochemical sensors, as they increase the density of electroactive sites available for redox reactions and improve charge transfer efficiency. Similar morphological advantages have been described in other MOF–polymer systems, reinforcing the structural synergy achieved in this composite (22,23). The electrochemical characterization further validated the composite's superior performance. Cyclic voltammetry (CV) revealed a marked increase in peak current response for FeCoMOF/PANI-NH$_2$-modified electrodes compared to bare and individual component electrodes, demonstrating its enhanced redox activity and conductivity. The quasi-reversible redox behavior and the linear dependence of peak currents on the square root of the scan rate indicated that electron transfer was diffusion-controlled, a desirable feature for analytical applications requiring reproducibility and sensitivity. The regression equations with high correlation coefficients ($R^2 = 0.9819$ for anodic and $R^2 = 0.9715$ for cathodic peaks) affirmed the strong electrochemical consistency of the composite material. These findings were in agreement with prior studies describing MOF–PANI hybrid electrodes that achieved similar improvements in charge transport due to synergistic interactions between the conductive polymer and metal centers.

Electrochemical impedance spectroscopy (EIS) supported these observations by showing a dramatic reduction in charge transfer resistance (Rct) for the FeCoMOF/PANI-NH_2-modified electrode compared to its counterparts. This reduction suggested that the composite provided a more conductive pathway for electron exchange, an effect attributed to the conjugated π–electron system of PANI coupled with the intrinsic metallic conductivity of the bimetallic MOF. The low impedance response confirmed efficient charge transport, crucial for sensor applications requiring rapid electron flow. The analytical performance of the developed sensor for lead (Pb^{2+}) detection using differential pulse voltammetry (DPV) demonstrated remarkable sensitivity and selectivity. The linear relationship between Pb^{2+} concentration (2–10 µM) and current response, along with a low detection limit of 0.03 µM, exceeded the requirements of the World Health Organization's permissible limits for drinking water. These values compared favorably with those reported in similar electrochemical systems, emphasizing the effectiveness of the FeCoMOF/PANI-NH_2 composite in achieving trace-level detection of toxic metal ions. The composite's large surface area and the presence of amine functional groups in PANI-NH_2 likely enhanced Pb^{2+} adsorption through coordination and electrostatic interactions, while the bimetallic MOF framework provided a stable platform for electron transfer. The reproducibility and stability of the modified electrode further validated its practical applicability. The low relative standard deviations observed during repeatability (3.1%) and reproducibility (2.5%) tests reflected uniform fabrication and consistent electrode behavior. The electrode retained nearly identical electrochemical responses after multiple cycles and days of testing, confirming its chemical stability and mechanical durability. The interference study demonstrated that the sensor maintained high selectivity toward Pb^{2+} ions even in the presence of competing heavy metal ions such as Hg^{2+}, Ag^{2+}, As^{3+}, and Zn^{2+}, with minimal signal distortion. This finding implied robust anti-fouling characteristics and suitability for real-world applications involving complex water matrices.

The strengths of this study lie in its integration of a green, microwave-assisted synthesis approach with a multifunctional composite architecture, achieving both structural integrity and superior electrochemical response. The environmentally benign synthesis process also reduced energy consumption and reaction time, supporting sustainable material development. The combination of FTIR, XRD, SEM, CV, DPV, and EIS analyses provided comprehensive validation of the material's structural, morphological, and electrochemical characteristics, ensuring the reliability of the findings. However, certain limitations warrant consideration. The study did not include an extensive evaluation of sensor performance in real environmental water samples beyond laboratory testing, leaving uncertainties regarding matrix effects, potential fouling, and long-term field stability. Additionally, temperature and pH dependencies of detection performance were not assessed, factors that can significantly influence electrochemical sensor efficiency. The use of a domestic microwave for synthesis, while efficient, may introduce reproducibility concerns due to uncontrolled temperature fluctuations. Future research should therefore employ calibrated microwave reactors, incorporate real-sample validation under varying environmental conditions, and evaluate long-term electrode regeneration capacity (24-26). In summary, the FeCoMOF/PANI-NH_2 composite exhibited remarkable electrochemical and physicochemical properties, demonstrating potential as a highly sensitive, selective, and stable sensor for lead detection in water. Its performance attributes underscore the synergistic benefits of combining conductive polymers with bimetallic MOFs and highlight its promise for application in sustainable environmental monitoring technologies.

CONCLUSION

The study successfully demonstrated a sustainable and efficient approach for developing an electrochemical sensor based on an iron–cobalt citric acid metal–organic framework (FeCoMOF) integrated with amine-functionalized polyaniline (PANI-NH_2). The microwave-assisted synthesis yielded a stable and well-structured composite with enhanced electrochemical properties, offering improved charge transfer, sensitivity, and selectivity toward lead ion detection. When modified onto a glassy carbon electrode, the composite exhibited reliable detection performance in both controlled and real aqueous systems, confirming its practicality for environmental applications. These findings underscore the potential of FeCoMOF/PANI-NH_2 composites as effective, low-cost, and eco-friendly materials for water quality monitoring and pollution control, representing a significant contribution toward the advancement of green analytical technologies in environmental protection.

AUTHOR CONTRIBUTION

Author	Contribution
Muhammad Ashraf	Substantial Contribution to study design, analysis, acquisition of Data Manuscript Writing Has given Final Approval of the version to be published
Zakir Hussain*	Substantial Contribution to study design, acquisition and interpretation of Data Critical Review and Manuscript Writing Has given Final Approval of the version to be published
Sadaqat Hakeem	Substantial Contribution to acquisition and interpretation of Data Has given Final Approval of the version to be published
Saqlain Abbas	Substantial Contribution to acquisition and interpretation of Data Has given Final Approval of the version to be published
Syed Amjad Hussain	Contributed to Data Collection and Analysis Has given Final Approval of the version to be published

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