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Sustainability Statement

Combating environmental contamination necessitates sensing technologies that are not only effective but also aligned with sustainability goals. This study presents a green advancement in the form of magnetically recoverable electrochemical sensors for the selective detection and remediation of contaminants. These sensors minimize secondary pollution, reduce energy and reagent consumption, and offer reusability by integrating environmentally friendly materials such as magnetic nanoparticles, carbon-based substrates, and conducting polymers. The unified system enhances both operational efficiency and environmental safety. This work directly supports the UN Sustainable Development Goals, particularly SDG 6 (Clean Water and Sanitation), SDG 12 (Responsible Consumption and Production), and SDG 13 (Climate Action), showcasing the promise of sustainable sensor technologies in real-world environmental monitoring and restoration.

ARTICLE

Magnetically Recoverable Hybrid Materials for Electrochemical Monitoring of Hazardous Contaminants: A Review

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Abstract

Environmental contamination due to toxic chemicals, heavy metals, and organic pollutants poses a significant threat to public health and ecosystems. Traditional methods for detecting and removing these contaminants often face limitations in sensitivity, selectivity, and efficiency. Among the different methods, electrochemical methods have taken the front seat due to various advantages over other methods. Magnetic sensors, particularly those based on magnetically recoverable nanocomposites, offer unique advantages such as high surface area, catalytic properties, and ease of separation. Integrating electrochemical techniques with these sensors allows for precise detection and efficient remediation processes. This review focuses on the advancement of magnetic sensors for the electrochemical detection and remediation of environmental contaminants. Herein, we explore recent developments in sensor design, focusing on functional materials such as magnetic nanoparticles, carbon-based materials, and conducting polymers. Various electrochemical detection methods, including amperometry, voltammetry, and impedance spectroscopy, are discussed in terms of their performance metrics, such as sensitivity, selectivity, and detection limits. Beyond detection, this review demonstrates the potential of magnetic sensors in contaminant remediation, specifically through adsorption, photocatalysis, and electrochemical degradation. Furthermore, we provide a critical assessment of the field's current challenges, including sensor stability, scalability for real-world deployment, and the development of cost-effective, sustainable solutions. Finally, this review outlines the promising prospects for this technology, underscoring the expanding role of electrochemical magnetic sensors as vital instruments in addressing environmental pollution.

Keywords: Magnetic nanoparticles; Electrochemical Sensing; Magnetic Sensors; potentially toxic metal ions and dyes; Contaminants.

1. Introduction

The quality of water has an impact on the overall health of all life on the planet. The increase in global population, associated with urbanisation, industries, and chemically modified agriculture, further harms the fragile water supplies ¹. Access to clean drinking water is becoming increasingly challenging for many people worldwide ². Therefore, it is urgently necessary to develop materials and technologies that are economically viable, easily accessible, ecologically sound, lightweight, thermally efficient and chemically robust to meet the growing demand for clean water throughout the world^{3, 4}. Numerous potentially harmful compounds are used, transferred, and handled daily in a range of contexts, from everyday activities to

industrial procedures. Such activities often release pollutants as groundwater or surface water via explicit or implicit pathways⁵. Recent years have experienced an enormous increase in public interest in hazardous pollutants due to the severe environmental risks they pose and the adverse impact their presence has on human health (Figure 1)⁶. Some synthetic organic colors and pigments are perceived by some as water contaminants. These colors are frequently used in a wide range of industries, such as textiles, tanneries, cosmetics, foods and in the treatment of humans and ⁷. Natural artificial colorants contaminated many areas of water and soil habitats due to their widespread use and production. Previous research has revealed the presence of dyes in ecological specimens such as fish species, soil, suspended fine particles, and water 8. Therefore, in the words of Tkaczyk et al. (2021) 9, they are categorised as micropollutants that affect aquatic ecosystems. Most of the dyes belong to a dangerous family of water pollutants

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and have a large impact on the ecology. Specific dyes, such as methylene blue, rhodamine B, methyl violet, and congo red, fall into this category and are known to be toxic for life.

Recently, pharmaceuticals have been identified as environmental pollutants that pose an increasing threat to ground water and surface water in industrial and residential areas ¹⁰. In general, drugs reach water sources primarily through discharge from pharmaceutical industries, municipal wastewater, and hospital effluents ¹¹. They are classified as stubborn bioaccumulative compounds and, because they

contaminate aquatic and terrestrial environments, they are considered dangerous chemicals ¹². Unfortunately, many of these pharmaceutical substances are not completely removed by wastewater treatment plants (WWTPs), and as a result, they have been found in drinking water (ground and surface) and WWTP effluents all over the world ¹³. Therefore, it is necessary to chemically or physically treat water that contains pharmaceuticals and their transformation products (TPs) to protect the environment and public health from potential toxicity and other negative effects ¹⁴.

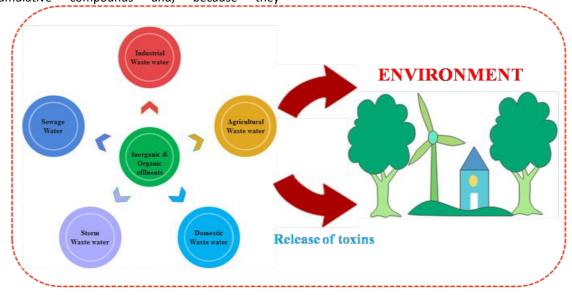


Figure 1: Diagram illustrating various sources of environmental pollutants. Illustrations shown in the figure are partly created with Wondershare Edraw Max. All text and scientific analyses are the author's own work and undertaken without the use of any Al tools.

Similarly, when organisms directly or indirectly ingest nutritional metals in large quantities, they suffer acute or chronic poisoning that cannot be reversed 15. For example, exposure to cadmium (Cd) (II) significantly alters plant growth and leads to metabolic failure ¹⁶. Consuming Cd (II) contaminated water and food is known to cause cancer and possesses the ability to develop hazardous lesions in human bones, liver and kidneys ^{17, 18}. Lead (Pb) (II) is an additional toxic metal ion that can compete with calcium, iron, and zinc for binding sites in mammals, causing fatal adverse effects on healthy organisms ¹⁹⁻²¹. The functioning of the heart and circulatory organs is adversely affected by Pb (II), which causes prolonged hypertension and myocardial dysfunction ²². Although vegetable crops are a safe source of nourishment for humans, the presence of contamination with Cd and Pb in these crops exposes individuals to serious dangers ²³. In light of the discussion above, it is essential to create a reliable and

precise method for analysing the presence of Cd (II) and Pb (II) in food materials and monitoring the extent to which these metal ions are ingested by the body. Inhalation of mercury vapor can cause fatal harm to the lungs, kidneys, mental health, digestive, and immune systems. Consumption of inorganic mercury salts can cause renal toxicity and damage to the skin, eyes, and digestive system ²⁴.This makes it essential that the contaminants present in wastewater are detected and removed urgently. Efficacy in the remediation of naturally occurring pollutants can be improved by employing a wide range of techniques ²⁵. The rapid development of highly specific and reliable analytical techniques in recent times that allow us to identify, monitor, and remove pollutants within complex environmental matrices has enriched understanding of the newly identified pollutants ²⁶. This review brings together the literature and discusses in detail the current status of the electrochemical

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detection based on magnetic materials. A vast variety of research articles were screened while preparing this review. We cited only those articles that are more

relevant, provide in-depth investigations on sensing mechanisms, follow greenness principles, and have excellent reusability.

2. Electrochemical sensors

Electrochemical sensors constitute an essential subgroup of chemical sensors. A chemical sensor is an apparatus that continuously reports information on the chemical composition of its surroundings²⁸. The amount of a specific analyte is directly correlated with the selective response produced by a chemical sensor 28. The sensor element is the core component of a sensor²⁹. It directly interacts with the detector and is responsible for identifying and establishing a connection with the target species in a complex environment. The detector then transforms the chemical signals produced by the sensor element for the species of interest into an output signal that can be measured. The working electrode (sensing electrode) and the counter electrode of an electrochemical sensor are typically separated by an electrolyte layer²⁸⁻³⁰. Typically, electrochemical sensors function by redox reaction of the species on the electrode surface, which produces an electrical signal according to the concentration of analyte species 30. To maintain a constant working electrode potential in a sensor, a reference electrode is utilised. Essentially, an excellent sensor should have the qualities listed below, (Figure 2):

- A. The signal output should be equivalent to the kind and number of target species.
- B. The sensor should have a high sensitivity to the species and be extremely particular.
- C. It should possess both high resolution and selectivity.
- D. It should be highly accurate and repeatable.
- E. It should have an immediate reaction time.

Electrochemical sensors have advanced to such an extent that they are now widely used, surpassing other categories of sensors, such as optical sensors. The prime example is of glucose meters using electrochemical sensors, which measure the blood glucose in an enzyme reaction.

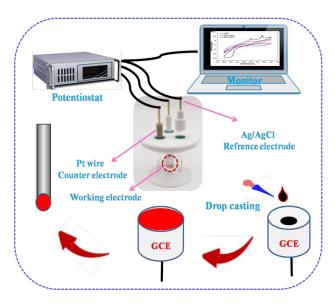


Figure 2: Diagram illustrating the working principle of an electrochemical sensor.

Furthermore, the cost-effectiveness, economical approach, and user-friendly properties electrochemical methods have attracted the attention of researchers to the detection of toxic contaminants over the last few years 31. Cyclic voltammetry, linear voltammetry (LSV), differential voltammetry (DPV) and stripping voltammetry are some of the prominent electrochemical techniques for the detection of toxic contaminants in water bodies 32. For the efficient detection of any contaminant, the conductive property and surface structure of the electrode material are the key factors that play a crucial role in their sensing ability. In this context, diverse materials, including graphene, carbon nanotubes conducting (CNTs), polymers, metal frameworks, metal oxides modified with polymers, etc., have been designed and developed as electrochemical sensors for the detection of heavy metal ions and other contaminants 15,33. Magnetic compounds such as zerovalent iron (nZVI), magnetite (Fe₃O₄), greigite (Fe₃S₄), and their composites are being used extensively due to their high surface area, excellent magnetic properties, ease of separation and therefore reduced post-treatment costs as illustrated in Figure 3.10,34-38. Moreover, the sensing performance of hybrid and magnetically recoverable nanomaterials outperforms pristine magnetic oxide nanoparticles

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carbonaceous materials such as carbon nanotubes, graphene, biochar, metal organic framework and conducting polymers³⁹. This overview will pay particular attention to the use of magnetic metal oxides as sensors for detecting heavy metal ions, dyes, medicines, and pesticides from aqueous systems.

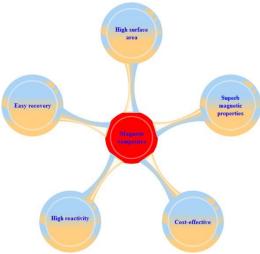


Figure 3: Key properties of magnetic composites in a cyclic representation. Illustrations shown in the figure are partly created with Wondershare Edraw Max. All text and scientific analyses are the author's own work and undertaken without the use of any Al tools.

3. Magnetic materials

A huge number of novel materials have been fabricated for the detection and decontamination of potentially toxic wastes ³⁹. However, the majority of them face significant challenges mainly due to their poor recovery and reusability from the sorption medium. Recovery and renewal characteristics are essential properties of effective functional adsorbent materials ⁴⁰. Design and preparation of nanocomposite materials from suitable superparamagnetic substances, such as nanozerovalent iron (nZVI), magnetite (Fe₃O₄), or greigite (Fe₃S₄), constitute one quick and efficient way to overcome this problem ⁴¹⁻⁴⁴.



Figure 4: Types of magnetic composites and their key components

Due to their superparamagnetic properties, biocompatibility, promising adsorption capacity, ease of recovery and reuse, magnetic nano-adsorbents have rapidly acquired popularity in the heavy metal contamination scenario ⁴⁵. The nanocomposite material consists of two or more materials where one material is of nanoscale range that provides properties unique to those of the other materials. The nanocomposite materials are viewed as the next-generation materials with the ability to detect and remove significant quantities of contaminants from wastewater. The following is a list of some magnetic substances that are used throughout the environmental detoxification process (Figure 4);

3.1. Magnetite (Fe₃O₄)

Fe₃O₄ crystallises in the inverse spinel structure (**Figure 5**). Fe₃O₄ has been extensively studied for its potential to immobilise heavy metal atoms and other toxicants ⁴⁶. The pristine Fe₃O₄ nanoparticles could be applied for the detection and removal of toxic waste; however, their agglomeration, low selectivity and low surface area restrict their application in the native form⁴⁷⁻⁴⁹. Therefore, the chemical stability and sorption efficiency can be enhanced by either capping the pristine Fe₃O₄ and/or by forming composites of Fe₃O₄ nanoadsorbents with various functional materials. ⁴⁷⁻⁴⁹

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Figure 5: A three-dimensional view of magnetite (Fe₃O₄)

Our research group capped Fe₃O₄ nanoparticles with sodium citrate, and the findings show enhanced sorption ability (Figure 6) 50. The existence of multiple functional groups of citrate on the surface of Fe₃O₄ nanoparticles improves the electrostatic attraction between positively charged metal ions and negatively charged citrate-coated magnetite. This electrostatic interaction enhances the electrochemical sensing of heavy metal ions. The glassy carbon modified with citrate-capped magnetite exhibited an outstanding electrochemical response to millimolar concentrations of Pb²⁺, Cd²⁺, and Zn²⁺ aqueous ions, with a response in the sequence of Pb $^{2+}$ > Cd $^{2+}$ > Zn $^{2+}$.

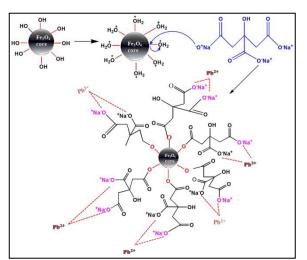


Figure 6: Mechanistic illustration of removal of positively charged heavy metal ions by which citratecoated magnetite nanoparticles.

3.2. Zerovalent iron (nZVI)

Zero-valent iron nanoparticles have undergone extensive studies for environmental cleanup due to

their various unique properties, such as strong reduction power, high surface area, earthly abundance, economic viability, biocompatibility, etc. However, these spherical nZVI particles exhibit a core-shell type structure (Fe⁰ Fe (ox hydroxide) ⁵¹. The presence of the Fe(0) core and outer oxidised layer produces a unique reactive surface that allows the initial adsorption of contaminants and their subsequent modification on the surface via reduction or oxidation pathways 52. Moreover, these nZVI particles are prone to agglomeration and oxidation, which consequently reduces their catalytic activity. Therefore, it becomes very important to modify these nanoparticles to inhibit their aggregation and increase their stability so that the catalytic activity of these nanoparticles is enhanced. One of the most efficient strategies is to immobilise such nanoparticles on a carbon-based solid support. Several carbonaceous substances, including biochar, graphene oxide, carbon nanotubes, metal-organic frameworks, activated carbon, and clay minerals, have shown a strong propensity to stabilise iron-based nanoparticles. In general, the FeO and iron hydroxides that make up the surface layer/shell and metallic core of the nZVI are negatively charged. The adsorption and quick reduction of metal ions are made possible by the role of the nZVI shell as an electron transport material⁵³. In addition, the nZVI-based material is being used for heavy-metal ion sensing by a series of chemical reactions by the zero-valent iron core and subsequent adsorption of metal ions on the shell of the material. Bare nZVI corrodes and progressively loses its efficiency. Therefore, attempts were made to address this challenge. A common procedure is to decorate nZVI on the functional support material, such as a carbonaceous framework. Bao et al. developed a highly economical electrochemical sensor using polyphenols in combination with nZVI and reduced graphene oxide for the determination of Hg²⁺ in water (Figure 7) 54.



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The rGO-ZVI-P composite exhibits a distinctive attraction for aqueous Hg²⁺as a result of a synergy between ZVI and rGO. Using square-wave anode stripping voltammetry (SWASV), the rGO-ZVI-P electrochemical sensor works exceptionally well for measuring Hg²⁺ in a pH 5.00 phosphate buffer over a 180-second accumulating period at 1.2 V accumulation potential. Under these conditions, the Hg²⁺ detection susceptibility and limit of Hg²⁺ detection are shown to be 41.42 A/M and 1.2 nM, respectively.

3.3. Greigite (Fe₃S₄)

Of the other iron-sulfur nanoparticles, Fe₃S₄ stands out as a top magnetic material, especially in gas detection and heavy metal decontamination. Fe₃S₄ has a saturation magnetism of 59 emu/g and is a semimetallic, commercially sustainable magnetic material that is similar to magnetite. Although Fe₃S₄ is a preferred material for energy storage uses, there are not many reports that can explain how well Fe₃S₄ nanoparticles bind to surfaces. Its metastability and aggregation potential may be the cause of the low surface binding efficiency of Fe₃S₄ nanoparticles. The prevention of agglomeration requires the impregnation of Fe₃S₄ nanoparticles onto the surface of various functional materials such as biochar, graphene, and polymers. For example, scientists studied the ability of magnetic graphite/biochar composites (MGB) to remove Cr (VI) from wastewater 55, which had been contaminated with heavy metals (Figure 8).

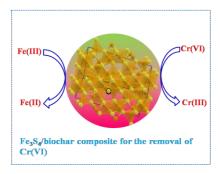


Figure 8: Greigite-based composites for the removal of chromium ions.

Our research team achieved a successful integration of mechanically exfoliated g- C_3N_4 nanosheets with purephase Fe₃S₄ nanoparticles ⁵⁶. During the solvothermal

synthesis, the Fe₃S₄ nanoparticles were wrapped in g-C₃N₄, giving them a spherical shape. The material exhibits and performs a wide range of electrochemical sensing and detoxification functions for pollutants in contaminated water. The soft sulfur centres in Fe₃S₄-g-C₃N₄ nanocomposite were identified as crucial for the interaction and capture of soft cationic contaminants such as Pb2+ and UO22+ ions. Charge transport at the electrode-electrolyte interface of the GCE-modified electrode, with KCl as the supporting electrolyte, was investigated by CV and DPV techniques. The electrochemical response of the modified glassy carbon electrode was highly responsive to micromolar concentrations of Pb2+ and uranyl ions with a limit of detection 0.71 and 0.22 μM for Pb²⁺ and UO₂²⁺ respectively. The material is superior and sustainable in action because of its reusability and magnetic recovery without any appreciable loss in activity⁵⁶.

3.4. Zincferrite (ZnFe₂O₄)

Zinc ferrite, typically ZnFe₂O₄, belongs to the family of spinel ferrites and finds applications in photocatalysis, sensing, and adsorption of contaminants. ZnFe₂O₄ depict a good electrochemical sensing performance against various pollutants in addition to its ease of preparation and magnetic recovery⁵⁷. For example, ZnFe₂O₄ exhibited 70% photocatalytic degradation diclofenac⁵⁸. Kumar drop-casted the glassy carbon electrode with a thin film of zinc ferrite⁵⁹. The particle size and surface shape of the nanoparticles were determined using FT-IR, XRD, BET, FE-SEM, and TEM techniques (Figure 9a). The concentrations of Pb (II) and Cd (II) ions were detected simultaneously at trace levels using differential pulse anodic stripping voltammetry (DPASV) (Figure 9b). The sensor's detection limits for Pb (II) and Cd (II) metal ions were 1.12 and 2.52 ppb, respectively. These detection levels are well below the drinking water permissible limits set by WHO: 10 ppb for Pb(II) and 3.0 ppb for Cd(II) ions. The use of bare ZnFe₂O₄ is limited due to its agglomeration, low sensitivity and stability as an electrode material. This can be avoided by integrating the ZnFe₂O₄ with carbonaceous materials to develop a more sustainable material⁶⁰. For example, the magnetic composite of ferrite / chitosan-curdlan (ZNF / CHT-CRD) was developed as a cost-effective adsorbent for the effective elimination of tetracycline from wastewater 61.

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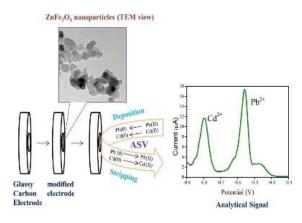


Figure 9: (a) Crystal structure of greigite (Fe₃S₄) (b) DPV analysis of Cd2+ and Pb2+ ions on ZnFe2O4 modified electrode ZnFe₂O₄. Source: Reproduced with permission from Kumar et al. (2018) J Environ Chem Eng.

3.5. Hematite

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Alpha hematite $(\alpha\text{-Fe}_2O_3)$ is a low-cost and sustainable semiconducting material with a wide range of applications in photocatalysis and electrochemical sensing. α-Fe₂O₃, like other iron oxide nanomaterials, is more promising when integrated with other functional support materials. B.Fall et al. modified a glassy carbon electrode with a ternary composite of α -Fe₂O₃, viz,rGO@CNT@Fe₂O₃. The modified electrode serves a a scaffold for the electrosynthesis of polypyrrole(PPy)⁶³. The thus formed quaternary magnetic composite depicts a strong electrochemical response to the trace detection of Pb(II) ions with a detection limit of 0.1nM, well below the WHO set permissible levels. The amino groups on the PPy chains can chelate Pb2+, creating a highly sensitive interface for Pb²⁺ detection. Additionally, rGO@CNT provides an enlarged surface area and promotes efficient electron transport, further improving the sensing performance. Therefore, the modified GCE rGO @ CNT @ Fe₂O₃ / PPy was expected to provide greater performance and enhance the signal for trace detection of Pb2+ ions (Figure 10b).

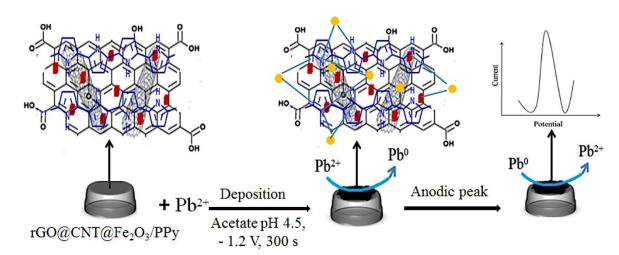


Figure 10: (a) Crystal structure of hematite (b) Detection of Pb2+ ions on the surface of rGO@CNT@Fe2O3/PPy electrode. Source: Reproduced with permission from Fall et al. (2021) Mater. Today Common

4. Electrochemical detection of contaminants by magnetic sensors

4.1 Electrochemical Sensing of Heavy Metal Ions by Magnetic **Metal Oxides**

Potentially toxic metal ions such as Pb(II), Hg(II), Cd(II), Cr(VI/III), As(III/V), and Zn(II) pose a threat to humanity. They are persistent and accumulate in the biosystem. Table 1 lists the WHO-recommended permissible levels of such metal cations in drinking water. The electrochemical detection technique is an ideal choice to detect the trace levels of potentially toxic metal cations well below their permissible levels.

Table 1: Maximum Concentration level of potentially toxic metal ions in drinking water

S.No	Metal ion	MCL(WHO)	MCL(WHO)	
		mg/L	μМ	
1	Pb(II)	0.01	0.048	

6

As

Cd(II) 0.05 0.445 3 Hg(II) 0.001 0.005 4 Zn(II) 3.0 45.88 5 Cr 0.003 0.057 0.01 0.133

Fe₃O₄ is the most common form of magnetic oxide used to detect toxic metals ⁶⁴. Furthermore, Fe₃O₄ has been reported to act as a good adsorbent for heavy metal ions. However, they have certain shortcomings, e.g., they are susceptible to aerial oxidation and agglomeration that decrease their conductive behaviour and limit their use as sensors 65. Therefore, the capping of these iron oxide nanoparticles with some functional moieties is essential for the rapid detection of heavy metal ions. Our research group reported the synthesis of citrate-coated magnetite for the sensing of Zn (II), Pb (II), and Cd (II) by differential pulse voltammetry. The best electrochemical response was observed for Pb (II) with a detection limit equal to 0.3 μM, followed by Cd (II) and Zn (II)⁵⁰. Though the material offers good sensitivity and stability, the detection levels for all three metal cations are above their maximum permissible limits. This marks the limitation of such materials. W. Wu et. al synthesised low-cost Fe₃O₄ nanoparticles crowned on fluorinated multi-walled carbon nanotubes for the sensitive detection of Cd (II), Pb (II), Hg (II), Zn (II) and Cu (II) ions⁶⁶. Square wave stripping voltammetry was used for the simultaneous detection of Cd (II), Pb (II), Hg (II), Zn (II) and Cu (II) ions, with a detection limit equal to 0.014, 0.0084, 0.0039, 0.012, and 0.0053 µM, respectively. The limit of detection is well below the maximum permissible levels for all the tested metal cations. In another such study M. Sedki et. al demonstrated an environmentally friendly approach synthesise linker-free to Fe₃O₄-Au nanoparticles (Figure 11) 67. Au nanoparticles with a 70nm diameter were decorated with small Fe₃O₄ nanoparticles of 10nm diameter that provide a large surface area for Fe₃O₄-Au nanoparticles. The prepared Fe₃O₄-Au modified glassy carbon electrode was used for the detection of Arsenic (III). The detection limit was found to be 0.22 μ g/L (0.0029 μ M) by using squarewave anodic stripping voltammetry. The detection level is again well below the permissible level.

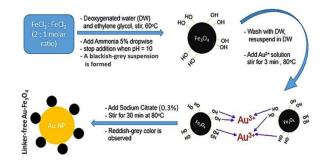


Figure 11 Diagram illustrating the synthesis and formation mechanism of Fe₃O₄-Au nanoparticles. Source: Reproduced with permission from Sedki et al. (2021) Sensors.

To detect even smaller concentrations of potentially toxic metal ions down to the nanomolar range, various studies were carried out. For example, M. Dib et.al modified the glassy carbon electrode using a Fe₂O₃@Ni/Al-LDH magnetic nanocomposite for the electrochemical detection of Hg (II) 68. The prepared electrode acts as an excellent sensor for Hg (II) ions due to its mesoporous nature (SEM images, Figure 12 a1, a2), which enhances the catalysis and adsorption of Hg (II) ions. The obtained voltammograms depict the reversible process in line with the Hg (II)/ Hg couple. The detection limit was found to be $0.00046 \mu M (0.46 nM)$.

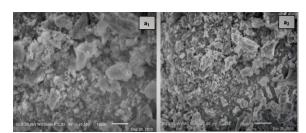


Figure 12: SEM images (a₁ and a₂) of the Fe₂O₃@Ni/Al-LDH sample. Source: Reproduced with permission from Mustapha et al. (2021). Copyright Inorganic Chemistry Communications.

Another work was carried out on some iron-based spinel magnetic oxides (CoFe₂O₄) by Shiguan ⁶⁹. In this work, a CoFe₂O₄-reduced graphene oxide composite was fabricated and tested for the nanomolar detection of Cu(II) ions. The addition of polyethyleneimine (PEI) or ethylenediamine (EDA) resulted in the size reduction of CoFe₂O₄ nanoparticles vis-à-vis enhanced adsorption /electrochemical detection of Cu(II) ions through its amine coordination. The modified electrodes were used for the electrochemical detection of Cu (II) ions by cyclic and square wave anodic stripping voltametric analysis with a detection limit down to picomolar level,

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ca.0.02nM (20pM). From cyclic voltammograms, the reduction peak around -0.19V increases with the increase in scan rate, which is typical of an irreversible reaction (Figure 13). Moreover, the polyethyleneimine (PEI)-modified electrode showed the best stripping response, indicating a higher sensitivity for Cu (II).

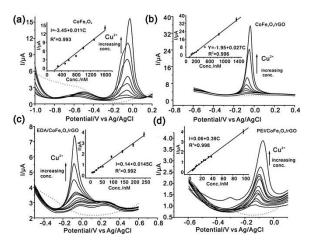


Figure 13: Typical SWASV responses of (a) CoFe₂O₄ (b) EDA/CoFe₂O₄/rGO, $CoFe_2O_4/rGO_4$ (c) PEI/CoFe₂O₄/rGO. Source: Reproduced with permission from Shiquan et al. (2016). Copyright Electrochimica Acta.

Similarly, the one-step hydrothermal method was adopted to prepare zinc ferrite, (ZFO)nanoparticles with a relatively large surface area of 54.1 m²/g ⁷⁰. The electrochemical properties of ZnFe₂O₄ were examined using CV and DPSAV techniques for Hg (II), Pb (II), and Cd (II). From CV, three oxidation peaks were observed at 0.3V, -0.52V, and -0.03V for the above three metal ions(Figure 14). Finally, the ZnFe₂O₄modified electrode was initially used for the concurrent detection of Hg (II), Pb (II), and Cu (II) with a limit of detection (LOD) down to the nanomolar range viz, 1.61, 7.38, and 12.03 nM, respectively. The mechanism of sensing is believed to proceed in two distinctive steps: (a) fast adsorption of metal ions at the surface of the modified electrode material, driven by the fast diffusion coefficient. Thereafter, the metal cations are transferred to the surface of the glassy carbon electrode for the stripping process. We expect the surface area of the modified electrode to largely influence the preconcentration of metal ions, and the same was observed as well. (b) During the stripping process, the metal ions are stripped off the electrode, wherein the electrochemical signal could be observed by the DPASV technique. The signal is very sensitive to the concentration of the metal ions. Both events are shown as an inset to Figure 14b.

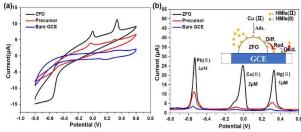
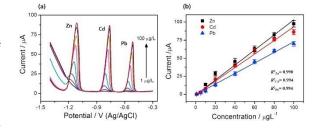


Figure 14: CV curves (a) and DPASV curves (b) of 2.0 μM Pb(II), 2.0 μ M Cu(II), and 1.0 μ M Hg(II) for different electrodes in 0.1 M HAc-NaAc buffer solution (pH = 5.0). Source: Reproduced with permission from Changchun et al. (2021) Copyright ACS Applied Nano Mater.

undoubtedly, the inorganic metal oxide nanoparticles demonstrated an excellent electrochemical performance with ultra-low detection limits well below their WHO-set permissible levels. The use of pristine inorganic electrode material is limited by its agglomeration and disintegration under working conditions. Such limitations can be avoided by the fabrication of composite materials^{71,72}. Lee et al. provide a quantitative analysis of a novel and highly efficient electrochemical sensor designed for the detection of trace levels of Zn2+, Cd2+, and Pb2+ ions. A strongly electroactive, ternary electrode material, (Fe₂O₃)/graphene/Bi nanocomposite, was fabricated by a solventless thermal decomposition technique⁷¹. Differential pulse voltammetry was employed to analyse Zn²⁺, Cd²⁺ and Pb²⁺ ions. The sensor successfully achieved the simultaneous detection of metal ions at concentrations ranging from 1 to 100 g L⁻¹, with detection limits down to the nanomolar range ca.0.11 $\mu g L^{-1}$ (1.68nM)for Zn²⁺, 0.08 $\mu g L^{-1}$ (0.71nM) for Cd²⁺, and 0.07 $\mu g L^{-1}$ for Pb²⁺(0.33nM)(Figure 15 a-d). Furthermore, the Fe₂O₃/graphene composite was applied to detect these heavy metal ions in real samples, producing results consistent with those obtained using ICP-MS.



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Figure 15: (a) Stripping voltammograms for varied Zn²⁺, Cd²⁺, and Pb²⁺ ion concentrations on the Fe₂O₃/G/Bi composite film electrode. (b) Calibration plot for the detection of Zn²⁺, Cd²⁺, and Pb²⁺. (c) DPV voltammograms of Zn²⁺, Cd²⁺, and Pb²⁺ ions on the Fe₂O₃/G/Bi modified electrode. (d) LOD plots for the same metal ions. *Source: Reported with permission from Lee et al. (2016), Talanta*

The formation of Fe_3O_4 @MPC, or mesoporous carbon nano chains of the core-shell Fe_3O_4 nanospheres (MPC), as an electrode-active composite for the detection of heavy metal ions,

In this arena, mesoporous carbon is another greener alternative to address the disintegration of metal oxide nanoparticles vis-à-vis its ability to capture a range of metal cations owing to its peculiar properties, such as high surface area and ease of synthesis. Y.Liu et.al synthesised a core-shell nanocomposite composed of magnetite core and mesoporous carbon shell, hereafter Fe₃O₄@MPC, with resorcinol-formaldehyde resin as a source of mesoporous carbon shell(MPC). The coreshell composite was prepared using a simple emulsion-induced interface assembly technique (**Figure 16**) ⁷².

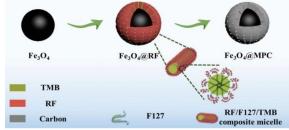


Figure 16: Mechanism of preparation of magnetic Fe₃O₄@MPC core-shell nanospheres. *Source:* Reproduced with permission from Liu et al. (2022) Copyright Adv. Mater. Interfaces.

Four different samples of $Fe_3O_4@MPC$ were prepared (numbered as 1-4) by just using different volumes of NH_3 - H_2O . Among them, $Fe_3O_4@MPC$ -2 depict the excellent performance where 1.0 mL has been used during the synthesis. In the sample, 280 nm size Fe_3O_4 nanospheres were present, together with an MPC shell that was approximately 150 nm thick and mesopores of approximately 40 nm (**Figure 17 a-h**).

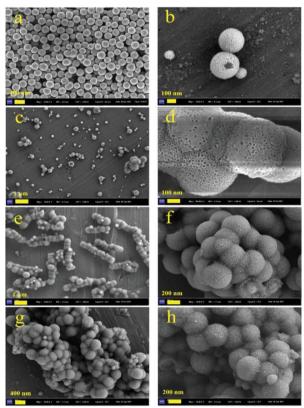


Figure 17: SEM images of (a) Fe₃O₄, (b,c) Fe₃O₄@MPC-1, (d, e) Fe₃O₄@MPC-2, (f, g) Fe₃O₄@MPC-3, and (h) Fe₃O₄@MPC-4. Source: Reproduced with permission from Liu et al. (2022) Copyright Adv. Mater. Interfaces.

Fe₃O₄@MPC demonstrated an efficient electrocatalytic response for the detection of Hg (II) and Pb (II) with LOD down to the nanomolar range, ca.7.8 and 12.1nM, respectively (Figure 18a-c). Moreover, the material demonstrated a good magnetic recovery with excellent reusability without an appreciable loss in activity. The Fe₃O₄ @ MPC-modified electrode demonstrated electrochemical sensing performance compared to pure Fe₃O₄, which was brought about by the cooperative action of numerous functional components in the core-shell-nanochain structure. While Fe₃O₄ nanospheres speed up the ion enrichment step, the conductivity of the mesoporous carbon layer encouraged electron transport during electrochemical analysis phase. The method for creating mesoporous carbon-coated magnetic nano chains is new and is presented in this paper. This coreshell arrangement improves both the durability of the chains and the effectiveness of the electrochemical detection of metal ions.

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Figure 18: Cyclic voltammograms of various modified electrodes in (a, b) 5 mm Fe $(CN)_6^{3-/4-}$ redox probe containing 0.1 m KCl. (c) Nyquist plots of PCM/GCE,

Fe₃O₄@MPC-2/GCE and Fe₃O₄. Source: Reproduced with permission from Liu et al. (2022) Copyright Adv. Mater. Interfaces.

Many such other studies are available in the literature. For better understanding, we have tabulated the results of metal oxide-based electrode materials for the detection of metal cations in Table 2.

Table 2: The limits of detection of various modified electrochemical magnetic sensors for the detection of heavy metal ions

Modified Electrode	Method adopted	Target Metal ions	Limit of detection	References
Fe ₂ O ₃ /Bi graphene oxide electrode	DPV	Zn ²⁺ , Cd ^{2+,} Pb ²⁺	0.11μgL ⁻¹ ,0.08 μgL ⁻¹ ,0.07 μgL ⁻¹	72
GO-Fe ₃ O ₄ -PAMPAM	SWASV	Pb ²⁺ , Cd ²⁺	130ngL ⁻¹ ,70ngL ⁻¹	73
MnFe ₂ O ₄ /GO	SWASV	Pb ²⁺	0.0883 μΜ	74
Co ₃ O ₄ -CeO ₂ -ZnO	SWASV	Pb ²⁺ , Hg ²⁺	0.054nM, 0.097nM	75
Amino-functionalized MgFe ₂ O ₄ /reduced graphene oxide (rGO)	SWASV	Cu ²⁺	0.2nM	76
Fe ₃ O ₄ /SiO ₂ /CS/Nafion/G CE	DPASV	Cu ²⁺	5n mol L ⁻¹	77
NiWO ₄ nanoparticles	CV, DPV	Hg ²⁺	2.25nM	78
Fe₃O₄@citrate/GCE	CV/ DPASV	Pb ²⁺	0.0061 μg·L ⁻¹	77
GSH@Fe₃O₄/MGCE	SWASV	Pb ²⁺	0.182 μg·L ^{−1}	77
		Cd ²	+ 0.172 μg·L ⁻¹	
Fe ₃ O ₄ @SiO ₂ -NH ₂	DPV	Pb ²⁺ and Hg ²⁺	6.06 and 9.09 nmol/L	79

4.2. Electrochemical Sensing of Dyes by Magnetic Metal Oxides

Not only the sensing of nanomolar concentration of metal ions, magnetic oxide nanomaterial can effectively be used to detect other classes of contaminants such as dyes, pharmaceuticals, and pesticides. Magnetic oxides due to their remarkable properties in terms of easy preparation, large surface area, and excellent catalytic, conductive, and electronic properties have been recently explored for the electrochemical detection of dyes/food colorants that are extremely toxic to

mankind ⁸⁰. For example, Sudan I is an orange-red coloured organic azo dye typically used as a colouring agent in hydrocarbon solvents and chilli powder. It is a potential carcinogen and has been banned for use in many countries. Moghaddam et. al fabricated an electrode material using the nano cubical La³⁺ doped Co₃O₄ for the trace analysis of Sudan I ⁸¹. The nanotube-like morphology of La³⁺ doped Co₃O₄ was determined from SEM (**Figure 19a**). The electrochemical behaviour of this prepared screen-printed electrode (SPE)was found to be suitable for Sudan I (**Figure 19b**). As can be seen from the cyclic voltammogram, no significant

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oxidation peak was found in the absence of the La³⁺ doped Co₃O₄ electrode, but in the presence of the La³⁺ doped Co₃O₄, the maximum current increases considerably, which supports the detection of Sudan I. Furthermore, the LSV and DPV studies showed a linear change in current with a detection limit equal to 0.05μM (**Figure 19 c,d**).

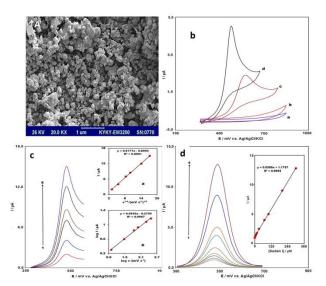


Figure 19: (a) SEM of (A) La³⁺⁻doped Co₃O₄.(B) Inset showing spherical images of La3+-doped Co3O4 nanotube.(b) Cyclic voltammograms of (a) pristine, (b) La³⁺-modified Co₃O₄ nanocubes/SPE without Sudan I, and (c) bare SPE, (d) La³⁺-modified Co₃O₄ nanocubes/SPE.(c) LSV plots of La3+doped Co3O4 nanocubes/SPE in 150.0 μM Sudan.(d)DPV of La³⁺doped Co₃O₄ nanocubes in different concentrations of Sudan. Source: Reproduced with permission from Hadi et al. (2019) Copyright Food Chem.

The use of screen-printed electrodes is appreciated for the on-site detection owing to the low cost; however, their non-homogeneous surface and relatively less reproducibility demand the use of a glassy carbon electrode for the detection of potentially toxic dyes. H. Yin et. al modified the glassy carbon electrode by superparamagnetic Fe₃O₄ nanoparticles for the nanomolar detection of Sudan I in food samples⁸². A prominent oxidation peak was seen on the surface of the bare electrode with a potential value of around 0.67 V when Sudan I was introduced to Phosphate-Buffered Saline(PBS). Under the aforementioned experimental circumstances, the electrochemical oxidation of Sudan I was completely irreversible on the bare electrode since there was no such decline peak for the reverse scan from 1.0 to 0 V. For Fe₃O₄/GCE, the oxidation peak potential (0.643 V) decreased while the oxidation peak current increased dramatically. This behaviour should be attributed to immobilised Fe₃O₄ nanoparticles, which can enhance the oxidative current and facilitate the catalysis of Sudan I, thus reducing the oxidation potential by adsorbing more Sudan I molecules on the electrode surface. Similar to the bare GCE, the oxidation peak observed at Fe₃O₄/GCE electrode surface is irreversible, indicating the irreversible oxidation of Sudan I under current experimental conditions. Furthermore, the modified electrode of Fe₃O₄ was devised to identify Sudan I by DPV. The manufactured electrode had a greater linear range between 0.01 and 20 μM . The detection limit for Sudan I was 0.001 μM . The interference effect due to the co-presence of chemical entities other than the target analyte is very common in the electrochemical detection, especially in real water samples. Such effects should be minimised for better results. One common method is to fabricate a highly target analyte-selective electrode material. For instance, a 500-fold concentration of Na⁺, Ca²⁺, Mg²⁺, Fe³⁺, Al³⁺, Zn²⁺, Cu²⁺, and Cl⁻, did not influence Sudan I determination, speaking of the superior selectivity of Fe₃O₄ nanoparticles for the Sudan I ⁸².

Artificial food and pharmaceutical colouring agents such as Tartrazine (a synthetic lemon-yellow azo dye) and sunset yellow (orange-red azo food dye) are potentially toxic due to their persistence. Therefore, their early detection is essential to avoid their accumulation. found in various products like candies, drinks, and cosmetics. M. Arvand et. al fabricated a carbon paste electrode functionalized with a core-shell Fe₃O₄@SiO₂/MWCNT nanocomposite, used for the simultaneous detection of sunset yellow and tartrazine 83. Square wave voltammetry was performed, which showed a linear response for both sunset yellow and tartrazine, having a very low detection limit ca. 0.05 and 0.04 µmol/L, respectively.

There is always a search for a more robust electrode material that can analyse the nanomolar concentration of tartrazine. One such material is hydrothermally grown Fe₂O₃ nanoparticles by Chen and his co-workers with three distinct morphologies: nanoplates(p-Fe₂O₃), nanorods(r-Fe₂O₃), and a three-dimensional flower(f-Fe₂O₃)⁸⁴. The research group developed the composite electrode material using expanded graphite (EG) and Fe₂O₃ nanoparticles. The typical micrographs are shown in Figure 20. The composite material composed of flower-shaped Fe₂O₃ (f-Fe₂O₃) exhibited superior electrochemical detection of sunset yellow and tartrazine dye with a limit of detection down to the nanomolar range, ca. 0.89 nM and 2.17 nM,

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respectively. This research is crucial because it reveals the relationship between the morphology of nanomaterial and the electrocatalytic activity of Fe_2O_3 nanoparticles (and other similar nanomaterials), allowing the creation of novel electrochemical uses for these materials and their composites ⁸⁴.

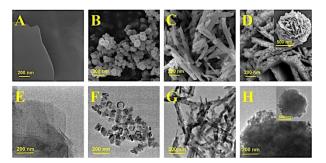


Figure 20: SEM (A–D) and TEM (E–H) images of EG (A, E), p-Fe₂O₃ (B, F), r-Fe₂O₃ (C, G) and f-Fe₂O₃ (D, H). The insets in (D) and (H) show the low-magnification SEM and TEM images, respectively. Source: *Reproduced with permission from Chen et al.* (2019)

In another study, R. Darabi et al reported the development of an electrode modified with 1-ethyl-3methylimidazolium chloride as an ionic liquid (IL) and a NiFe₂O₄-rGO nanocomposite (IL/NiFe₂O₄/rGO/CPE). modification resulted in а promising electrochemical sensor for monitoring sunset yellow 85. The electrochemical properties were analysed by CV and DPV techniques. Higher catalytic activity and increased surface area of the prepared electrode promote better electron transfer, resulting in efficient sensitivity. DPV studies were analysed to determine the LOD value for sunset yellow, and it was found to be $0.03\mu M$. Compared to $Fe_3O_4@SiO_2/MWCNT$, the IL/NiFe₂O₄/rGO offers more selective detection of sunset yellow dye in addition to a lower detection limit. In the interference study, the sunset yellow analysis was carried out in a ternary mixture containing Allura red and Tartrazine. The DPV showed distinct anodic peaks for the three dyes and the sunset yellow, and a higher sensitivity was observed for the sunset yellow (Figure 21).

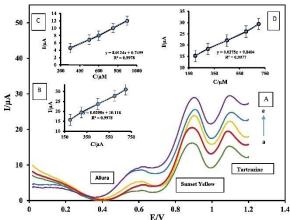


Figure 21: DPV of IL/NiFe₂O₄/rGO/CPE in pH 3 with varying concentrations of Allura Red, Sunset Yellow, and Tartrazine DPVs; Insets: linear ranges for Allura Red (B), Sunset Yellow (C), and Tartrazine (D). Source: Reproduced with permission from Rozhin et al. (2021) Copyright Food Chem.

Now we will introduce how a novel molecularly imprinted electrochemical sensor (MIES) was created using magnetic field-directed self-assembly (MDSA). A magnetite-reduced oxide composite (Fe₃O₄@rGO) was synthesised for the detection of amaranth anionic azo The pre-assembly solution that dye (Figure 22). included amaranth, aniline and Fe₃O₄@rGO was electropolymerized with the help of MDSA to create the imprinted membrane. By changing the ratio of Fe₃O₄ and rGO in Fe₃O₄@rGO composites, one can easily alter the thickness and shapes of the doped molecularly imprinted electrochemical electrodes 86. The doped patterned membrane and Fe₃O₄@rGO composites were characterised using spectral and electrochemical methods. Additionally, the manufactured demonstrated high sensitivity and outstanding selectivity when used to detect amaranth in fruit beverages.

We have highlighted in this section the use of magnetic metal oxide nanomaterials in the electrochemical detection of trace amounts of food colouring dyes. The results significantly support the use of a nanocomposite material for the best electrochemical results. This paves the way for future investigations to delve more into such materials to develop a realistic and sustainable material.

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Figure 22: Figure illustrating the process of creating a Fe₃O₄@rGO doped molecularly imprinted polymer membrane that uses a magnetic field to detect amaranth, Source: Reproduced with permission from Han et al. (2014) Copyright Talanta.

4.3. **Electrochemical** detection of pesticides/pharmaceuticals by magnetic metal oxides

Modernisation has introduced new challenges in food analysis due to the influx of various pollutants, including residual pharmaceuticals and pesticides, which are increasingly entering our ecosystem. In response, the detection of pesticides has become crucial, necessitating continuous advancements in detection methodologies^{87,88}. Researchers have developed an enzyme-based biosensor for the rapid electrochemical detection of organophosphorus pesticides, specifically dimethoate 89. This biosensor involves the immobilisation of acetylcholine esterase (AChE) on a Fe₃O₄/Au magnetic nanoparticle (MNP) electrode $(Fe_3O_4/Au-AChE)$. The Fe₃O₄/Au-AChE absorption is facilitated by a composite membrane of CNT/nano-ZrO₂/Prussian blue (PB)/Nafion (NF) on a screen-printed carbon electrode (SPCE) under an external magnetic field. The detection limit for dimethoate was determined to be 5.6×10^{-3} ng/mL, with a linear relationship between the degree of enzyme inhibition and pesticide concentration ranging from 1.0 \times 10⁻³ to 10 ng/mL. Another group of researchers has synthesised Fe₃O₄@ZrO₂ carbon-based magnetic coreshell nanoparticles, employing them electrochemical biosensor for methyl parathion (MP) detection 90. These Fe₃O₄@ZrO₂ nanoparticles were deposited on a glassy carbon electrode to construct a magnetic electrochemical sensor. The voltammetry analysis of MP on the modified electrode revealed that ZrO₂ exhibited a significant affinity for sensing phosphoric moieties of organophosphate compounds. Under optimal conditions, the simple square wave voltammetry technique was used to quantify methyl parathion, with the cathodic peak current exhibiting a linear increase with MP concentrations ranging from 7.60×10^{-8} M to 9.12×10^{-8} 5 M, and a detection limit of 1.52 × 10 $^{-8}$ M. Additionally, for the ultrasensitive detection of methamidophos or omethoate in vegetable samples, a biomimetic electrochemical sensor (BECS) has been developed. This sensor incorporates a Fe₃O₄/reduced graphene oxide nanocomposite (Fe₃O₄/rGO) integrated with a metal organic framework MIL-101(Cr) coated molecularly imprinted polymers film as a recognition element. The BECS sensor recorded strong responses for ultra-low concentrations of methamidophos or omethoate and the limit of detection of the BECS method was 2.67 ×10⁻¹³mol/L for methamidophos and 2.05×10⁻¹⁴ mol/L for omethoate, respectively 91. Qinghua designed an innovative biomimetic electrochemical sensor for the rapid detection of acephate, using a Fe₃O₄@carboxylfunctionalized multiwalled carbon nanotube/chitosan nanocomposite 92. The electrochemical performance of the fabricated electrode was evaluated using cyclic voltammetry and differential pulse voltammetry. The electrode exhibited linear current responses for acephate and trichlorfon concentrations ranging from $1.0 \times 10^{-4} \,\mathrm{M}$ to $1.0 \times 10^{-10} \,\mathrm{M}$ and $1.0 \times 10^{-5} \,\mathrm{M}$ to $1.0 \times 10^{-5} \,\mathrm{M}$ ¹¹ M, respectively. The sensor was again found to have ultra-low detection limits of 6.81 \times 10⁻¹¹ M and 8.94 \times 10⁻¹² M for acephate, and trichlorfon, respectively. Aptamers are tailor-made functional oligonucleic acids with a great affinity for the target analyte. In the arena of pesticide detection, aptamers were found very promising. For example, Yancui et al. developed a simple and highly sensitive sensor for the electrochemical detection of chlorpyrifos using a novel composite film with carbon black (CB) and GO @ Fe₃O₄ with the chitosan chlorpyrifos aptamer (Apt) as one of the bio-recognition elements 93. The developed aptasensor, Apt / GO @ Fe₃O₄ / CB / GCE provided a linear range of 0.1–10⁵ ng mL⁻¹ with a lower LOD ca. 0.033 ng mL⁻¹. The other properties that make this aptasensor useful are its good selectivity, stability, and reproducibility. Furthermore, a practical use of this aptasensor is to monitor the presence of chlorpyrifos residues in actual samples of vegetables such as cabbage, lettuce, leeks, and pakchoi.

Non-enzymatic detection of pesticides using electrode materials such as metal oxides, graphene oxide, and metal-organic frameworks (MOFs) is emerging because of its increased stability, simplicity, and costeffectiveness. Georgette et al. have employed a facile one-pot co-precipitation method to develop a nonenzymatic electrochemical sensor, CuFe₂O₄ and reduced graphene oxide nanocomposites for the

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detection of Malathion 94. It has been observed that the highest inhibition was observed at optimised conditions corresponding to 15% rGO loading, with a linear detection range of 0.5-8 ppm. Tadele et al. devised a Fe₃O₄ NP-modified graphite pencil electrode (GPE) sensor to detect chlorpyrifos pesticide with a detection limit of 2.8×10^{-6} mol L⁻¹ 95. Raymundo-Pereira et.al demonstrated the flexible, selective and stable on-site detection of carbendazim and paraquat using sustainable sensors printed on cellulose acetate (CA) substrates⁹⁶. The sensor is classified as a wearable sensor, which is its ability to non-invasively detect analytes in real time by integrating sensor material with everyday items. The sensor exhibited a very low detection limit, ca. 54.9 and 19.8 nM for carbendazim and paraguat, respectively. More importantly, the ecofriendliness of the developed sensor film was evaluated using the Analytical Greenness Metric Approach (AGREE)⁹⁶. Briefly, AGREE is based on 12 green analytical chemistry principles in the form of a colour pictogram wherein the values range from 0 to 1, with 1 as most environmentally friendly. In the present case, the sustainability index using AGREE is 0.79, signifying eco-friendly detection of carbendazim paraquat(Figure 23).

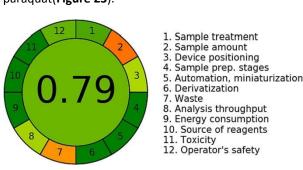


Figure 23: Greenness assessment of the developed voltammetric method with a sustainable sensor strip employing AGREE metric assessment tool. *Source: Reproduced with permission from* Raymundo-Pereira et.al (2023) Biomaterials Advances.

In another study, a magnetic metal-organic framework (MOF) was synthesised by impregnating magnetite nanoparticles into the MOF matrix. The as-synthesised nanocomposite offers an enormous surface area for the electrochemical detection of methyl parathion (**Figure 24**). Electrochemical analysis revealed that the multifunctional molecular organic framework (MOF) composite served as a superb matrix for the simultaneous adsorption of methyl parathion. The structured arrangement, expansive surface area, excellent compatibility and magnetic characteristics of

the material facilitated efficient accumulation, separation, and direct detection of methyl parathion in the solution with remarkable sensitivity and a low detection limit of 3.02×10^{-6} g L-1 97 .

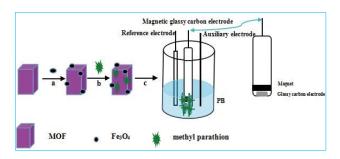


Figure 24: Schematic representation of Fe₃O₄/MOF synthesis. *Source: Reproduced with permission from Hu et al. (2014) Anal. Sci.*

Kulkarni describes a quick and easy process to create a highly associated nickel cobaltite (NiCo2O4) singlewalled carbon nanotube (SWCNT) hybrid for the electrochemical detection of pesticide paradoxes ethyl (PEL), which is very commonly found 98. Raman spectroscopy and X-ray photoelectron spectroscopy studies revealed a significant electronic interaction between the two components (Figure 25). The NiCo₂O₄-**SWCNT-modified** glassy carbon electrode demonstrated an exceptional detection limit of 2.03 pM, which is two orders of magnitude lower than that of bare NiCo₂O₄ (33.7 nM) and significantly outperforms other metal oxide-based composites reported in the literature. This remarkable performance photoelectrochemical (PEL) detection is attributed to the strong coordination between the two components. Furthermore, the sensor exhibited repeatability and stability with the potential to simultaneously detect paraoxon ethyl and dopamine.

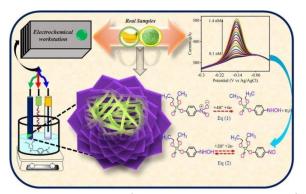


Figure 25: Synthesis of NiCo₂O₄-SWCNT composites for the detection of Paraoxon Ethyl. *Reproduced with*

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Similarly, the phosphonate herbicide glyphosate [N-(phosphonomethyl)glycine] is frequently used for a variety of agricultural uses. It is one of the most toxic and hazardous chemicals found in drinking water, because of its wide distribution, probable toxicity, and widespread bioaccumulation. This requires accurate detection and removal of glyphosate contaminated water. In this work, Altaf et al. describe how nano zero-valent iron (nZVI) nanoparticles were used to decorate new, highly porous biochar to create a useful electrochemical sensor for glyphosate trace detection. Modern instrumental techniques were used to fully define the composite as it was produced 97. Electron micrographs revealed the presence of a cavitylike structure and a substantial loading of nanoscale zero-valent iron (nZVI) particles in the composite materials (Figure 26). The existence of Fe (0) and oxygen-rich functionalities in composite the nanostructure was confirmed by FTIR and XPS studies. An electrochemical investigation suggested a detection limit as low as 0.13 ppm using CV, LSV, and DPV methodologies. The chronopotentiometry response additionally indicated good and stable long-term usage



Figure 26: Synthesis of nZVI@Biochar.Source: Reproduced with permission from Qureashi et al. (2023) Copyright Nanoscale Advances

Pharmaceuticals are another important class of compounds that need immediate quantification in the drinking water samples owing to their biological threat. Halligudra successfully developed a durable, highly efficient, magnetically recoverable, and reusable Fe₃O₄-MoS₂ heterogeneous catalyst using a cost-effective and environmentally friendly hydrothermal method ¹⁰⁰. This catalyst was used for the reduction of p-nitrophenol and organic dyes using NaBH₄ as a hydrogen source in an ethanol medium at room temperature. The presence of MoS₂ and Fe₃O₄ co-catalyst enabled the Fe₃O₄-MoS₂ catalyst to achieve efficient catalytic removal of organic pollutants, including p-nitrophenol (p-NP), methylene blue (MB) and methyl orange (MO). After the reaction, the catalyst was easily and effectively recovered using a magnetic field and reused in subsequent reactions. Up to nine rounds of reuse and recycling were demonstrated by the catalyst, with nearly unaltered efficacy. Additionally, paracetamol, ascorbic acid, hydrogen peroxide, and tetracycline could be detected using the synthesised material, as demonstrated by the specified cyclic voltammetry and impedance approaches (Figure 27a-d).

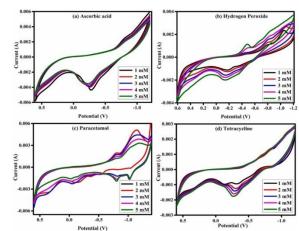


Figure 27: Cyclic voltammograms of Fe₃O₄–MoS₂ for the detection of a) AA b) HP c) PCM and d) TC concentration 1-5 mm Fe₃O₄ supported ranges (Magnetic MoS₂ nanoflowers as a catalyst for the reduction of pnitrophenol and organic dyes and as an electrochemical sensor for the detection of pharmaceutical samples. N Source: Reproduced with permission from Halliqudra et al. (2022) Copyright Ceram. Int.

In another classic example, Raymundo-Pereira et.al hydrothermally synthesise nitrogen-doped carbon spherical shells (N-CSSs) for the effective and sensitive quantification of diclofenac in saliva, urine, water, and tablets with a low limit of detection ca.0.36 μ M¹⁰¹. The material offers a huge specific surface area for the interaction of trace levels of diclofenac in addition to its superior AGREE greenness index of 0.75¹⁰¹. The results are highlighted in Figure 28. Furthermore, the material offers promising sensitivity and selectivity for diclofenac in the presence of various competing organic and biologically inorganic ions, including relevant molecules.

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Figure 28: Electrochemical quantification of diclofenac using N-CSSs. Source: Reproduced with permission from Raymundo-Pereira et.al (2024) Copyright ACS Applied Nanomaterials.

Such studies pave the way for the design and fabrication of effective electrode materials for the guick detection of pharmaceuticals.

5. Mechanism of interaction of contaminants with magnetic sensors

5.1 Mechanism of interaction of toxicants with magnetic

Hydrophobic, electrostatic, hydrogen bonds, 140 interactions, and covalent bonding can all be used to categorize how contaminants adhere to the surface of carbon nanomaterials (CNMs). Sorption of both organic and inorganic substances is a component of the hydrophobic interaction/sorption mechanism. Instead of their total surface area, the surface functional group density of CNMs is what primarily determines the inorganic sorption capability of these materials. The primary reason for electrostatic interaction, a key factor in the removal of metal ions and dyes, is the contact between contaminants and oxygen-containing particles of carbon nanotubes (CNT) that possess a negative residual charge. It has been proposed that the adsorption of organic contaminants onto the surfaces of carbon nanomaterials is attributed to hydrogen bonding, hydrophobic, electrostatic, and π -interactions. For instance, contaminants with functional groups (e.g., amine, hydroxyl, and carboxyl groups) can be absorbed through hydrogen bonding interactions, wherein the oxygen-rich groups of CNPs interact with the hydroxyl groups of the contaminants.

The presence of environmental pollutants including heavy metal ions, dyes, and pharmaceutical ingredients significantly contributes to environmental contamination, which directly affects the water bodies ¹⁰¹. The recent developments in the contamination mitigation approaches, processes and materials lead

the scientific community to directly address the problems through the mechanistic approach. The mechanism of adsorption varies from system to system. The driving forces responsible for the adsorption of toxins are those processes that include hydrogen bonding, charge transfer nature, van der waals, electrostatic interaction, complexation, etc. Based on the systems used for environmental mitigation purposes, the mechanistic details fall under various categories, which are discussed briefly in the subsequent sections. Magnetic materials, consisting of multi-walled carbon nanotubes combined with magnetite, emerge as promising candidates for the adsorption of heavy metal ions from wastewater. The removal of heavy metal ions such as Pb(II), Cu(II) and Cd(II) from wastewater using the 60-MWCNTs@Fe₃O₄ involves the interaction between empty d orbitals of metal ions and lone pair of electrons on oxygen atoms, which are present on the surface of multi-walled carbon nanotubes in the form of carboxyl and hydroxyl groups thus forming a complex 102. In addition, the presence of hydroxyl and carboxyl groups leads to deprotonation at certain pH values, rendering the surface more negative, which interacts with positively charged metal ions through electrostatic interactions as well. In addition to this, the magnetic material composed of graphene oxide varies in its adsorption capacity of heavy metal ions as well drugs, specifically tetracycline, depending upon the type of variant used which may include magnetic graphene oxide (MGO), magnetic chemicallyreduced-graphene (MCRG) and magnetic annealingreduced-graphene (MARG). The adsorption capability is dependent on the change in the pH value in the vicinity of the chemical environment. The three variants eliminated toxins that include tetracycline and arsenate [As(V)]. The predominant interaction driving the adsorption varies based on the surface functionalities of each variant. In the case of magnetic graphene oxide, the enhanced adsorption is ascribed to its high dispersibility, the presence of thin nano-sheets, and the existence of oxygen-containing functional groups on its surface. These functional groups coordinate more effectively with tetracycline through hydrogen bonding as well as π - π interactions. Furthermore, in the case of chemically reduced magnetic graphene (MCRG) and magnetic annealing-reduced magnetic graphene (MARG), the predominant functionality is attributed to oxygen-containing groups, with the primary π - π interaction between tetracycline and these variants ¹⁰³.

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In another work, poly-(m-phenylenediamine) in combination with MnO₂@Fe₃O₄ serves as a potential adsorbent to capture heavy metal ions as explained by Xiong 104. Incorporation of the magnetic composite enhances the surface area and alters surface electronegativity. The greatest capacities for magnetic materials to remove heavy metals were 438.6 mg/g for Pb (II) and 121.5 mg/g for Cd (II), respectively. Heavy metals and MnO₂ @ Fe₃O₄ / PmPD exhibit mainly attraction through electrostatic forces, ion exchanges, and coordinated contact. Due to its rich amine and imine functional groups, poly-(m-phenylenediamine) (PmPD) possesses outstanding redox capabilities, chelation abilities, and large-scale pollutant adsorption sites (Figure 29).

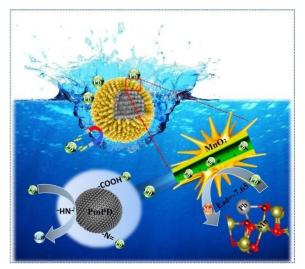


Figure 29: Mechanism of interaction of magnetic composites with environmental contaminants Source: Reproduced with permission from Xiong et al. (2020) Copyright Ecotoxicology. Env

Similarly, the development of pesticides is effective in controlling the pests that harm crop plants ¹⁰⁵. However, their widespread use leads to contamination in the food chain and subsequently in water bodies leading to their pollution. The accumulation of pesticides in water bodies in quantities more than the permissible limits poses a serious problem that needs to addressed before its accumulation. development of various materials, mostly magnetic composites, is fruitful in combating this problem. Many magnetic materials incorporate carbon-based magnetic composites, such as metal-organic frameworks (MOFs), and covalent organic frameworks (COFs), and their various combinations. These materials also include pristine carbon nanotubes (CNTs) or CNTs enhanced with other substances, as well as graphene and functionalized graphene. These advanced materials have been utilized to help mitigate pesticide pollution. Specific magnetic compounds were developed from iron oxide and carbon nanotubes (Fe₃O₄-MWCNTsOH@poly-ZIF67) for the detection of aromatic acids 106. Basic mechanistic details related to the adsorption behaviour involve the π - π interaction between the adsorbent and the hydroxyl groups enhanced by carbon nanotubes ¹⁰⁷. Similarly, one of the main advantages of magnetic separation of dangerous contaminants, which is emerging as a method for wastewater purification, is the removal of colors more effectively than conventional treatment methods. In addition to having negative impacts, dye compounds have an unsightly appearance in water. Dyes used in the textile industry encompass a wide range of structural types, including metal complex dyes, as well as acidic, reactive, basic, disperse, azo, and diazo dyes. Limin explored the use of ethylenediamine-modified magnetic chitosan nanoparticles for adsorbing Acid Orange 7 and Acid Orange 10 from aqueous solutions. The adsorption process is attributed to ionic interactions between dye ions and amino groups of the ethylenediamine-modified magnetic chitosan nanoparticles 108. The adsorbed dyes were desorbed using an NH₄OH/NH₄Cl solution at pH 10.

Zheng Yong synthesized magnetic Fe₃O₄/C core-shell nanoparticles using a straightforward method and demonstrated their application as adsorbents to remove organic dyes from aqueous solutions 109. The functional groups (such as -OH and CO) present on the surface of the composite, combined with the negative potential of the magnetic nanocomposites, promote a mild electrostatic attraction between the cationic dyes and the nanoparticles, leading to an increased adsorption capacity, particularly for methylene blue (MB). Drugs, essential for both humans and animals, play a crucial role in the combating of various diseases. The consumption of drugs leaves residues that pose a threat to the environment through the dumping of garbage and waste products into the water bodies, and also have a strong tendency to enter the food chain through meat, milk, eggs, etc., into humans 110. The challenge for the scientific community is to adopt and design certain materials that take care of their recovery ¹¹¹. Magnetic composites have been designed from time to time, which leads to an improvement in their recovery and hence leads to a decrease in their level of contamination. The porphyrin-functionalized Fe₃O₄graphene oxide composite (TCPP/ Fe₃O₄-GO) has a strong adsorption capacity for certain drugs¹¹². Pi-pi stacking interactions and the electrostatic attraction between the positive charge of sulfonamides and the

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negative charge of porphyrins are the mechanistic insights that cause drug molecules to interact with magnetic composites. The probable mode of interaction of magnetic composites with environmental toxicants is shown in the following figure 30. The illustrations shown in figure 30 are partly created with Wondershare Edraw Max. We have used Edraw Max only for the template drawing, without the use of any Al functionality. All the text and scientific analyses are the author's own work and undertaken without the use of any Al tools

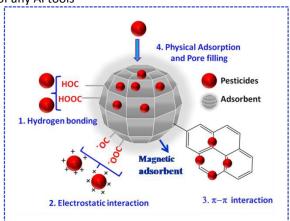


Figure 30: Mechanism of interaction of magnetic composites with environmental toxicants. Illustrations shown in the figure are partly created with Wondershare Edraw Max. All text and scientific analyses are the author's own work and undertaken without the use of any Al tools.

6. Conclusions and Future Perspectives

Rapid industrialisation, urbanisation, and population explosion result in the discharge of potentially toxic pollutants such as heavy metal ions, hazardous synthetic dyes, pharmaceuticals, pesticides and pesticide residues in the water bodies. This has a potential impact on human and aquatic lives and is thus a serious global environmental concern. WHO has set the maximum permissible limits for all the categories of contaminants in the drinking water; the majority of them fall within the micromolar concentration range. Therefore, electrochemical sensing emerges as a superior approach to selectively detect the contaminants up to the picomolar range. The most important component in the electrochemical sensing is the electroactive electrode material that could sense trace levels of the analyte. An ideal electrode material exhibits high electrical conductivity, possesses a large active surface area, excellent stability, sensitivity and reusability, minimum interference due to co-existing

ions, and low toxicity. In this arena, magnetically recoverable iron oxide and iron oxide-based nanocomposite materials have received considerable attention. There is still a need for comprehensive review articles that provide an in-depth overview of the uses of iron oxide-based nanomaterials for the picomolar/nanomolar detection of heavy metal ions, hazardous food colouring agents, pharmaceuticals and pesticides. In this review, we have highlighted the role played by Fe₃O₄, Fe₃S₄, Fe₂O₃, nano-zerovalent iron, and ZnFe₂O₄ nanoparticles in the electrochemical analysis of a range of contaminants. The results demonstrate excellent sensitivity, selectivity, and reusability of the electrode material when the iron oxide nanoparticles were integrated with strongly conducting non-metallic functional support, especially with carbonaceous materials. We have schematically shown the magnetic hybrid sensors in scheme 1. Among various carbonaceous materials reported in this review, graphene oxide, biochar, metal organic frameworks, and mesoporous non-metal doped carbon depict superior performance in terms of selectivity, sustainability, very low limit of detection and reusability. We also highlight the greenness index of some of the electrode materials according to the Analytical Greenness Metric Approach (AGREE). Additionally, we have provided an in-depth mechanistic detail of sensing and uptake of the toxicants on such oxide-based nanocomposite fabricated electrodes. Thereafter, we investigated and highlighted optimum parameters for the effective analysis of contaminants not only in the water samples but also in the real food samples. The integration of nanotechnology, functionalized magnetic materials, and advanced sensing mechanisms has significantly enhanced their performance in environmental applications. However, challenges such as sensor stability, interference from complex matrices, and large-scale deployment need to be addressed to ensure their widespread adoption.

Scheme 1: The outcome of this review article highlights the role of iron oxide nanoparticles when integrated with carbonaceous functional support for fabricating a nanocomposite electrode material.

Perspectives

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We highlight in this review some examples of electrode fabrication using a collaborative effect of carbon nanomaterials with magnetically recoverable iron oxide nanoparticles. However, there is always scope for advancement in terms of specificity, cost of electrode fabrication, sustainability and on-site detection. In this context, we will provide a future perspective as follows:

- Real-Time, In-Situ Detection: Most of the reported studies rely on the electrochemical detection of contaminants on a laboratory scale. Though the results were intriguing, the scenario in the real water samples is quite different. For example, the electrode may face interference due to the presence of co-existing species, fouling of the sensor surface by organic matter, and the limited stability for long-term monitoring. Such limitations demand fabrication nanostructured. miniaturised and screen-printed electrodes with antifouling properties(use of antifouling biopolymers-integrated oxide nanomaterials) and extended stability for more robust electrodes.
- Selective and Targeted Pollutant Identification -The selectivity of the sensor material is very crucial for the on-site and practical application for the detection of a particular toxin. More research is needed with much emphasis on design and fabrication of non-enzymatic electrode materials, molecularly imprinted polymers, low-cost and environmentally relevant nanocomposite materials based on carbonaceous materials and metal

- organic frameworks containing specific metal/ analyte trap centres. This will certainly help us in achieving targeted and selective detection of target analyte in nano/femto scale concentration range.
- Scalability and Practical Implementation Most of the electrode fabrication relies on the bench-level application. A range of contaminants and the level of pollution demand pilot-scale monitoring and removal of the contaminants. In this arena of research, 2D and 3D printing strategies facilitate the precise and scalable fabrication of miniaturised and portable electrode arrays. This will improve the reproducibility of the field-deployable sensor.
- Sustainable Material Design In this review, we highlight the iron oxide-based sensor materials for the detection of nanomolar concentrations of contaminants. However, the classical synthesis of iron oxide nanoparticles requires a huge chemical Such electrode fabrication undoubtedly increase the cost of the electrode for market use. Moreover, the majority of the studies used a modified glassy carbon electrode, which again adds to the cost of sensor setup. Scalable green synthesis of oxide nanomaterials using biomaterials is an alternative for the synthesis of low-cost, reproducible and sustainable oxide materials. Moreover, the limitations of using glassy carbon modified electrodes will be controlled by fabricating molecularly imprinted miniaturised electrodes and integrating the electrode material with portable detection devices.

continued innovation, magnetic nanomaterials have the potential to revolutionise environmental monitoring and remediation strategies, contributing to a cleaner and more sustainable future.

Conflicts of Interest

The authors declare no conflict of interest for this work.

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Data Availability

Most of the data supporting this article have been included. Any data not included is available on request from the corresponding author.

Author contributions

Aaliya Qureashi,: Writing, editing and original draft, and reviewing. Arshid Bashir: Review and editing.
Irfan Nazir and Firdous Ahmad Ganaie: writing. Kaniz Fatima, Zia-ul-Haq, and Lateef Ahmad Malik: editing. Abdullah Yahya Abdullah Alzahrani: Investigation, Methodology. Altaf Hussain Pandith: Conceptualization, Supervision, Review, and editing.

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Magnetic Hybrid Materials for Electrochemical Monitoring of Hazardous Contaminants: A Review

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Data Availability Statement

Most of the data supporting this article has been included. Any data not included is available on request from the corresponding author.