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Systematic Review

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Magnetic Iron Oxide and Ferrite Nanomaterials for Electrochemical Sensing and Remediation of Hazardous Water Contaminants

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Abstract

Toxic metals, metalloids, pesticides, drug residues, dyes and pathogens occur together in natural and waste waters, and the health-based guideline values for several of them sit at the µg/L level. Meeting such limits calls for sensitive measurement and for treatment that can be recovered and reused, yet the two are usually studied apart. Magnetic iron oxides, spinel ferrites and their hybrids can serve both purposes: a tunable surface supplies recognition or binding, and magnetism allows preconcentration and recovery. This critical narrative review sets reported performance against drinking-water guideline values. An Au-decorated $MnFe_2O_4$ electrode, for example, detected As(III) at 3.37 µg/L against a 10 µg/L guideline, and PEI-functionalised Fe_3O_4 adsorbed up to 60.98 mg/g Pb(II). We relate material design (Fe_3O_4/γ-Fe_2O_3, MFe_2O_4, core–shell and carbon hybrids) to electrochemical transduction and to removal mechanisms, and we draw on characterisation data (SEM, TEM, EDS, XRD, FTIR) to show what the materials actually are. In the six primary studies examined closely, only one used real water, none reported ecotoxicity, one measured metal leaching, and regeneration was followed for five cycles at most. We propose a sensor-guided, recoverable workflow and minimum reporting standards. Magnetic nanomaterials look most credible as application-specific, recoverable components; sustainability claims need direct evidence.

Keywords Magnetic nanomaterials; Spinel ferrites; Electrochemical sensing; Water remediation; Magnetic separation; Environmental safety

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1 Introduction

Water pollution comes from many places: mines and smelters, textile dye houses, farms, hospitals and pharmaceutical plants, chemical works, and sewage that has not been properly treated. What ends up in a river or an aquifer is rarely a single compound. Metal ions turn up beside dyes, antibiotics, pesticides, humic material and salt, and any analytical or treatment

method has to cope with that mixture. The limits it must meet are strict. The World Health Organization gives guideline values of 10 µg/L for arsenic (provisional) and for lead, 3 µg/L for cadmium, 50 µg/L for total chromium (provisional), 6 µg/L for inorganic mercury and 6 µg/L for the organophosphorus pesticide dimethoate [1]. Arsenic alone is thought to affect 225–500 million people who depend on contaminated groundwater [2], and lead, cadmium, mercury, chromium and arsenic rank among the most toxic heavy-metal ions [3]. Pharmaceuticals and pesticides add to the burden as emerging micropollutants, and regulation for them is still taking shape [4].

Limits of this kind create two practical needs. The first is a measurement that can resolve a target at or below its guideline value despite interferents. ICP-MS, atomic absorption spectrometry and chromatography coupled to mass spectrometry are the reference methods, but they need expensive instruments, careful sample preparation and a central laboratory. Electrochemical techniques are attractive for the opposite reasons: they are sensitive, cheap and, in principle, portable enough for on-site monitoring [3]. The second need is treatment that takes the contaminant out of the water without simply moving it into a new waste stream.

Magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3) and the spinel ferrites (MFe_2O_4) suit both jobs. They respond to a magnet, their surfaces can be modified to bind or recognise a target, and many of them take part in redox or catalytic reactions. A magnet can hold particles on a screen-printed electrode [5], gather nanospheres during synthesis [6], or pull a spent sorbent out of treated water without filtration. The literature is large. Reviews cover magnetic nanoparticles for pollutant detection [7], nanomaterial-based sensors in general [8, 9], ferrite sensors [10], magnetically recoverable hybrids for electrochemical monitoring [11, 12], ferrite-based remediation [13–15] and iron-oxide sorbents [16–19].

The sensing results deserves a closer look. Mesoporous $MnFe_2O_4$ nanospheres decorated with gold nanoparticles, cast on a glassy carbon electrode, detected As(III) by square-wave anodic stripping voltammetry with a sensitivity of 0.315 µA/ppb and a detection limit of 3.37 ppb (µg/L); the measurements were made in 0.1 M NaAc–HAc at pH 5.0 after a 150 s deposition at −0.9 V [6]. That limit is about three times below the 10 µg/L guideline [1]. Bismuth nanoparticles on CoFe2O4 have been used for heavy-metal detection by the same technique, with acceptable reproducibility and stability [3]. A $NiFe_2O_4$–$CoFe_2O_4$ nanocomposite has been applied to Cu, Cd and Mn together [20], and a carbon paste electrode modified with $Mn_{0.5}Zn_{0.5}Fe_2O_4$ to Pb^{2+} and Cd^{2+} [21]. For organic targets, a Nafion/Fe_3O_4 nanocrystal electrode detected bisphenol A by electrochemiluminescence over 0.01–5.0 mg/L, with a

detection limit of 0.66 µg/L and recoveries of 96.0–105.0% in river and tap water [22]. An acetylcholinesterase biosensor built on Fe_3O_4/Au particles held on a screen-printed electrode by a magnet detected dimethoate from 1.0×10^{-3} to 10 ng/mL, with a limit of 5.6×10^{-4} ng/mL [5]. That is roughly four orders of magnitude below the 6 µg/L (6 ng/mL) guideline, but it was measured in pH 7.5 buffer, so it says more about the format than about field performance. Magnetic nanoparticle conjugates have also been used for electrochemical detection of waterborne bacteria [23].

On the treatment side, polyethyleneimine-functionalised Fe_3O_4 nanoparticles reached a maximum Pb(II) capacity of 60.98 mg/g (Langmuir fit) at 25 °C and pH 5.0; capacity rose with temperature and pH, fell when competing electrolytes were added, and the particles kept working through five regeneration cycles [24]. A $CoFe_2O_4$@methylcellulose photocatalyst removed metronidazole under UV-C light, reaching 85.3% at pH 11 after 120 min. The authors separated the contribution of dark adsorption (23.7%) and photolysis (16.9%) from that of photocatalysis (81.59%), and found no Fe or Co in solution after four runs [25]. Green magnetite made with onion-peel and corn-silk extracts removed arsenic from real groundwater, with maximum capacities of 1.86 and 2.79 mg/g against 1.30 mg/g for conventionally precipitated magnetite [26]. Other studies used $CoFe_2O_4$ for photocatalytic removal of azithromycin from simulated effluent [27], $NiFe_2O_4$ for Cr(VI) and As(V) [28], functionalised magnetic nanoparticles for agricultural wastewater carrying antibiotics, pesticides and toxic metals [29], and magnetic nanostructured adsorbents for heavy metals in textile wastewater [30].

Existing reviews tend to treat sensing [7–11] or remediation [13–19] on its own, and the numbers quoted above show why a joint view is useful. They come in µg/L, ng/mL, mg/L, mg/g and per cent, under different pH values, doses and water matrices, so they cannot simply be ranked. Safety evidence is thin. In the six primary studies we examined in detail (Section 7), one used real water, none reported ecotoxicity, one measured metal leaching, and regeneration was followed for five cycles at most. It also helps to keep three ideas apart. A sensor signal shows that something responded, not that anything was removed. Adsorption moves a contaminant onto a solid, and the loaded solid is still a hazardous material. Catalytic degradation can leave toxic fragments of the original molecule, and reducing Cr(VI) to Cr(III) lowers toxicity but leaves Cr(III) to be captured. The strongest systems pair measurement with verification, recovery and a plan for the spent material.

The aim of this review is to evaluate how magnetic iron oxides, spinel ferrites and their hybrids work in electrochemical sensing and in remediation of hazardous water contaminants, to set reported performance against health-based guideline values, and to define the evidence needed before claims of removal, detoxification or sustainability can be accepted. Five objectives follow: to relate material design (composition, shell, support, recognition layer) to function; to compare sensing and treatment mechanisms with a common set of metrics; to ask how sensing and treatment might be combined in a sensor-guided, recoverable workflow; to identify what the primary studies report and leave out about matrix, leaching, ecotoxicity, transformation products and regeneration; and to set out research priorities.

The scope is deliberately limited. We cover Fe_3O_4, γ-Fe_2O_3, spinel ferrites and magnetic hybrids with carbon, polymers, biopolymers, biochar, metals and recognition layers, applied to toxic metals and metalloids, dyes and phenols, pesticides, pharmaceuticals and microbial hazards in surface water, groundwater and wastewater. The sensing techniques are voltammetry, amperometry, impedance and electrochemiluminescence; the treatment mechanisms are adsorption, complexation, redox conversion, Fenton-like and photocatalytic oxidation, and coupled electrochemical treatment. Biomedical uses, gas sensing, soil remediation and purely optical sensors are outside the scope, and non-magnetic materials appear only as comparators. Section 2 describes how the literature was handled; Sections 3 to 5 deal with materials, sensing and remediation; Section 6 considers their integration; Sections 7 and 8 cover reporting standards and environmental safety; Sections 9 and 10 give research gaps and conclusions.

2 Review methodology

This is a critical narrative review, not a systematic one. Its evidence base is a set of 170 records assembled by the authors. We removed duplicates by DOI, which left 128 unique sources, and kept those that bear directly on magnetic iron oxides, spinel ferrites or magnetic hybrids used for electrochemical sensing or for remediation of water contaminants. 90 of these sources are cited. The original database search (databases, search strings, dates) was not recorded, so we cannot report it, and we make no claim of exhaustive or reproducible coverage. The conclusions are a critical reading of selected literature.

Quantitative statements come only from primary research articles. The evidence matrix in Section 7 covers six such studies. For each one we checked the bibliographic record against Crossref-indexed or publisher pages and took the sample matrix, test conditions and

performance values from the abstract and open-access page. If a metric did not appear there, we wrote “not stated” and did not estimate it. No numerical values were taken from review articles, and we applied no formal quality-appraisal tool; the matrix should be read as a set of worked examples, not as a systematic comparison.

3 Magnetic nanomaterial families and structure–property relationships

3.1 Iron oxides

Magnetite and maghemite are the usual starting points. They can be made in water, coated with silanes or polymers, and combined with carbon, biopolymers, metal oxides or recognition layers [17–19]. Magnetite holds both Fe(II) and Fe(III) and can take part in redox reactions, whereas maghemite is mostly Fe(III). How a given particle behaves depends on crystallite size, oxidation state, aggregation, coating thickness and the water around it. A large nominal surface area means little if the particles clump together or a coating blocks the active sites.

3.2 Spinel ferrites

Spinel ferrites have the formula MFe_2O_4, with M = Mn, Co, Ni, Zn, Cu or Mg. The way the cations are shared between tetrahedral and octahedral sites shapes the magnetic response, conductivity, redox behaviour and surface reactivity [14, 15, 31]. Synthesis route, annealing temperature and defects alter the crystallite size, the degree of cation inversion, the number of oxygen vacancies and the number of accessible surface sites. Ferrites offer magnetic recovery and catalytic activity, but their conductivity is often too low for a good electrode [10, 32, 33]. Pairing them with graphene, reduced graphene oxide, carbon nanotubes, biochar, conductive polymers or metal particles improves electron transport and limits aggregation. Bi-loaded $CoFe_2O_4$ [3], a $NiFe_2O_4$–$CoFe_2O_4$ composite [20] and a ferrite/N-doped reduced graphene oxide hybrid [34] are examples. A support should be chosen to fix a named weakness; it is not a generic booster.

3.3 Magnetic hybrids and core–shell systems

In core–shell designs each part has a job. The magnetic core handles recovery, and the shell or attached phase supplies selectivity, conductivity, catalysis or protection. Examples include Fe_3O_4@SiO_2, polymer-coated magnetite [35, 36], chitosan- or graphene oxide-modified iron oxide [37, 38], ferrite–carbon composites [39, 40] and ferrite–metal hybrids such as Au-decorated mesoporous $MnFe_2O_4$ nanospheres [6]. Shell quality matters. A thin or porous shell exposes active sites, a thick insulating one can block electron transfer and slow mass transport,

and a shell with gaps lets metals leach and unwanted reactions occur. Surface charge and functional groups should be measured at the pH and ionic strength of the intended use.

The $MnFe_2O_4$/Au spheres reported by Zhou and co-workers [6] show how much can be read from a good characterisation set (Fig. 1). In the scheme, the ferrite spheres are first covered with an aminosilane (3-aminopropyltriethoxysilane, the amine-terminated chain in the top panel), and gold nanoparticles are then attached to the amine-functionalised surface. SEM and TEM show near-spherical particles of sub-micrometre size (scale bars, 500 nm), and the selected-area diffraction pattern of a single sphere gives rings indexed to the (220), (311) and (440) planes of the spinel lattice. A high-resolution image resolves a (311) lattice spacing of 0.257 nm. After gold loading the surface of the spheres becomes visibly rougher with small bright particles, and the EDS spectrum gains Au peaks alongside the Mn, Fe and O signals. Together, these data show that the magnetic spinel phase is intact and that gold sits on the surface, where it can act as an electrocatalyst.

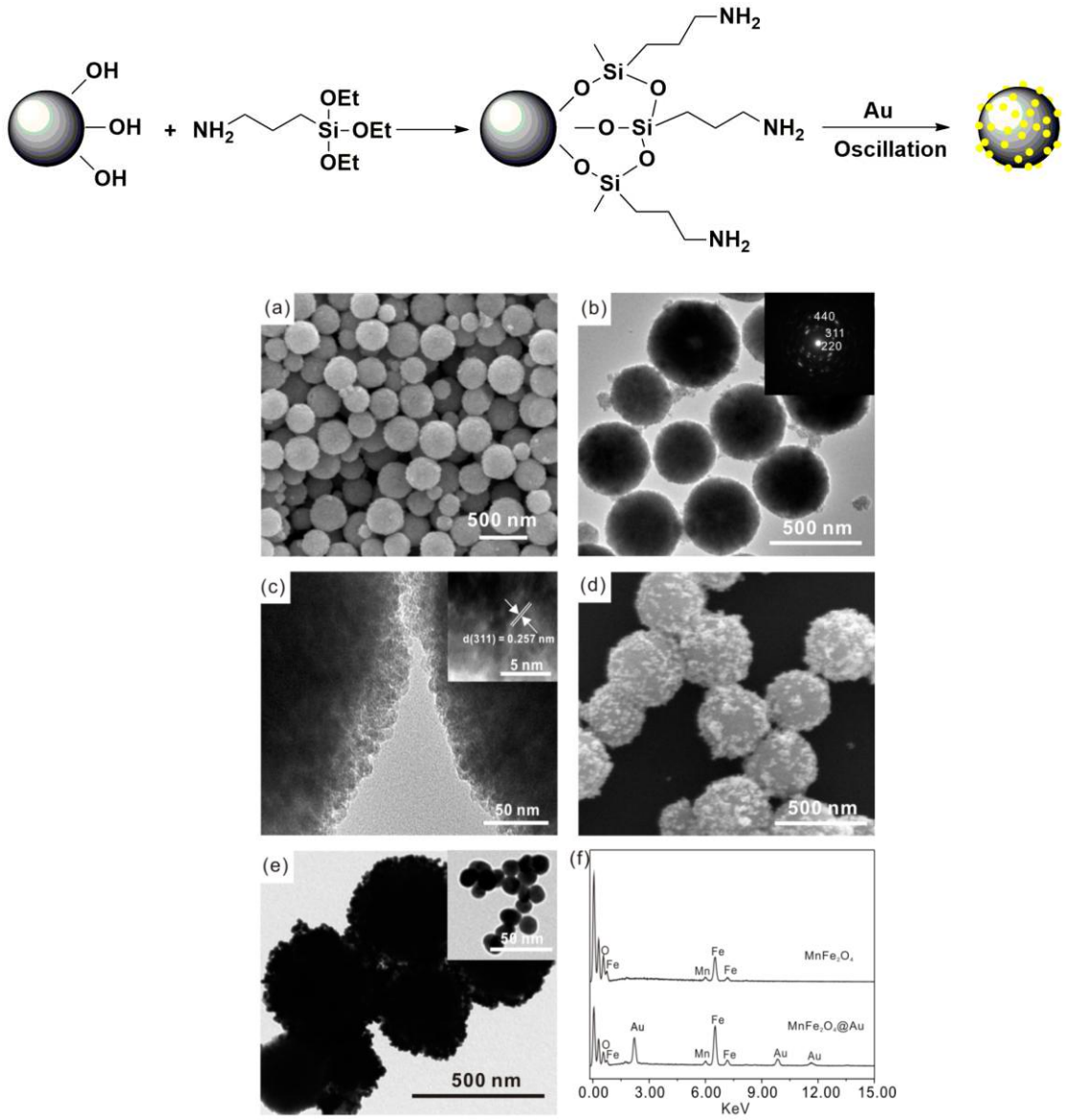


Fig. 1 Au-decorated mesoporous $MnFe_2O_4$ spheres for electrochemical sensing of As(III). Top: scheme of surface amination with an aminosilane followed by Au deposition. Bottom: (a) SEM and (b) TEM of the $MnFe_2O_4$ spheres, with the selected-area diffraction rings indexed to (220), (311) and (440); (c) high-resolution TEM, lattice spacing d(311) = 0.257 nm; (d) SEM and (e) TEM of the Au-decorated spheres; (f) EDS spectra before and after Au loading. Scale bars, 500 nm (a, b, d, e) and 50 nm (c). Reproduced from [6] under a CC BY licence

3.4 Synthesis and quality control

Co-precipitation scales well and is simple, but it gives broad size distributions and agglomerates. Hydrothermal and solvothermal routes give better crystallinity and morphology control, at the price of pressure equipment and more energy. Sol–gel routes give uniform composition but need solvents and heat treatment [16, 17, 41]. Green synthesis with plant

extracts or biological agents can avoid some hazardous reagents [26, 42, 43], but extract variability, residual organics and life-cycle impacts have to be measured, not assumed. A minimum characterisation set for environmental use includes XRD for phase, TEM or SEM for size and morphology, hydrodynamic size and zeta potential where relevant, FTIR or XPS for surface chemistry, VSM for magnetic response, impedance for charge transfer, and leaching and stability tests. Surface area alone predicts neither sensing nor remediation performance.

The green route of Nikić and co-workers shows both the appeal and the limits of the approach [26]. Onion-peel and corn-silk extracts reduced Fe3+ in the presence of sodium acetate. The mechanism proposed is that quercetin, the main flavonoid in onion peel, chelates Fe^{3+} and transfers an electron, which gives Fe^{2+} and oxidises the flavonoid to quercetin quinone; Fe_3O_4 then forms from the Fe^{2+}/Fe^{3+} mixture (Fig. 2). SEM images show rough, aggregated particles of irregular size, and EDS gave mass fractions of 57.1% Fe and 40.3% O for the onion-peel material and 55.6% Fe and 40.9% O for the corn-silk material (Fig. 3). In the XRD patterns (Fig. 4) the peaks between $2\theta = 18.7°$ and 62.5° match the magnetite planes from (111) to (440). Debye–Scherrer crystallite sizes were 26 nm (onion peel), 28 nm (corn silk) and 12 nm for the co-precipitated reference, while the BET areas of the two green materials were 243 and 261 m²/g. The green materials are therefore not smaller crystallites than the conventional one; the advantage claimed lies in the reagent chemistry and in the arsenic capacities discussed in Section 5.1.

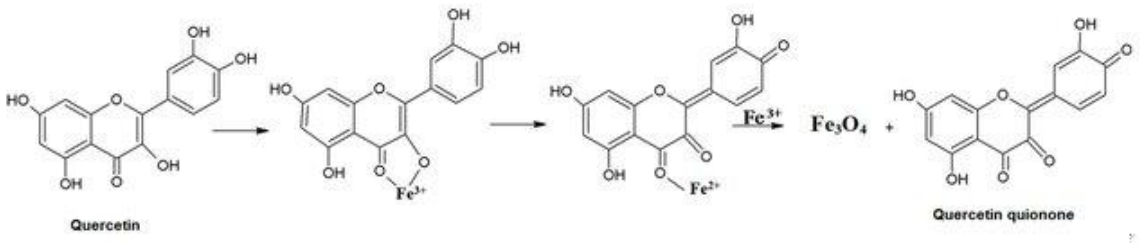


Fig. 2 Proposed mechanism of magnetite formation in the onion-peel route: quercetin chelates Fe^{3+}, transfers an electron to give Fe^{2+} and quercetin quinone, and Fe_3O_4 forms from the Fe^{2+}/Fe^{3+} mixture. Reproduced from [26] under a CC BY licence

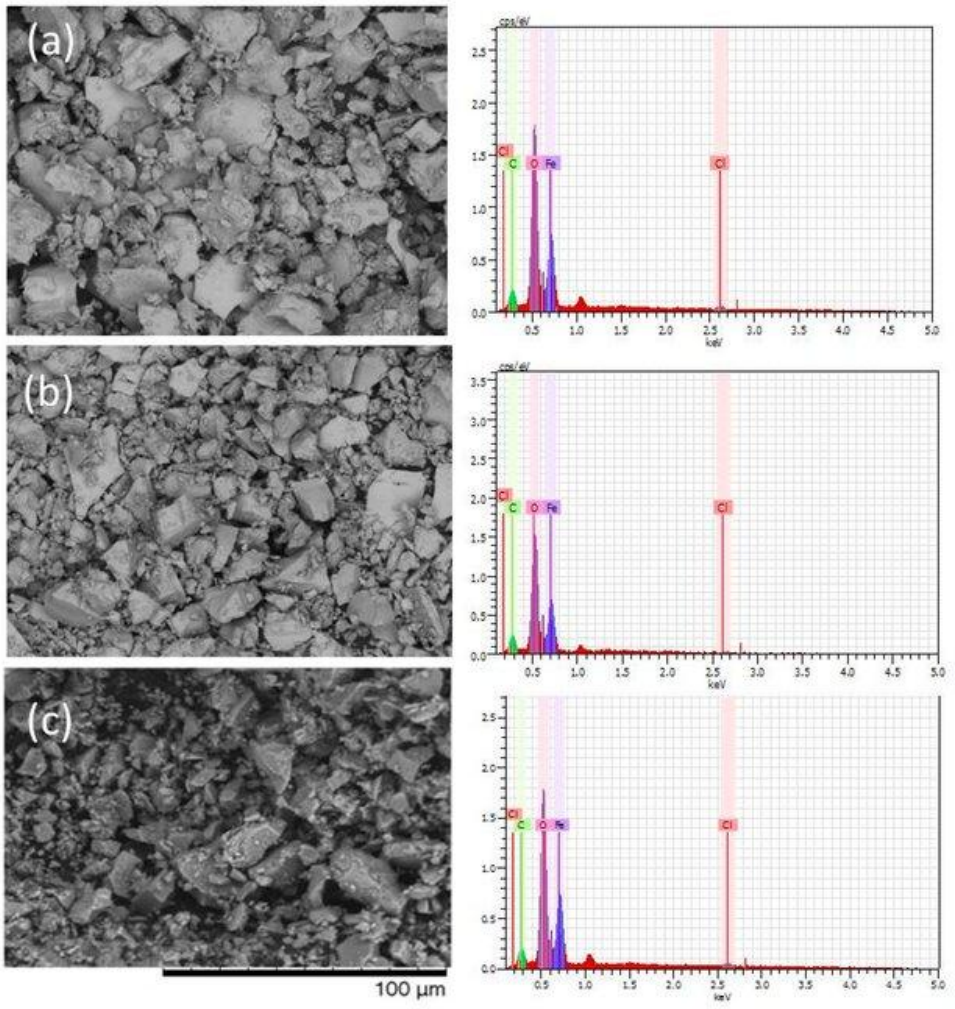


Fig. 3 SEM images with EDS spectra of magnetite made with onion-peel extract (a), corn-silk extract (b) and conventional co-precipitation (c). The EDS mass fractions were 57.1% Fe and 40.3% O (a) and 55.6% Fe and 40.9% O (b). Reproduced from [26] under a CC BY licence

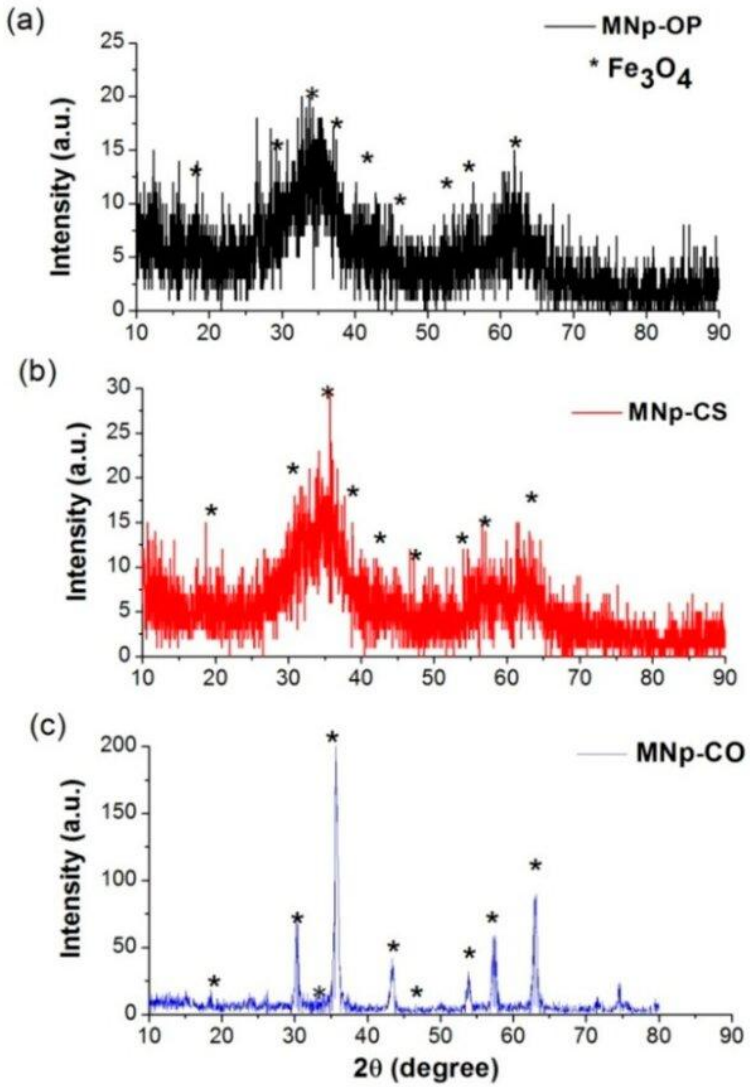


Fig. 4 XRD patterns of the three magnetite samples of Fig. 3. The peaks between 2θ = 18.7° and 62.5° are assigned to magnetite, (111) to (440); Debye–Scherrer crystallite sizes were 26 nm (a), 28 nm (b) and 12 nm (c). Reproduced from [26] under a CC BY licence

A second worked example is the PEI-functionalised Fe_3O_4 spheres of Tao and co-workers [24] (Fig. 5). SEM gave sizes of 100–350 nm (mean about 209 nm) for the coated particles and 100–450 nm (mean 256 nm) for the uncoated ones, so the polymer coat did not enlarge the particles, and an amine band in the FTIR spectrum confirmed the PEI. FTIR spectra were also recorded after Pb(II) adsorption, so the same panel allows the binding step to be checked. Table 1 summarises the main material classes.

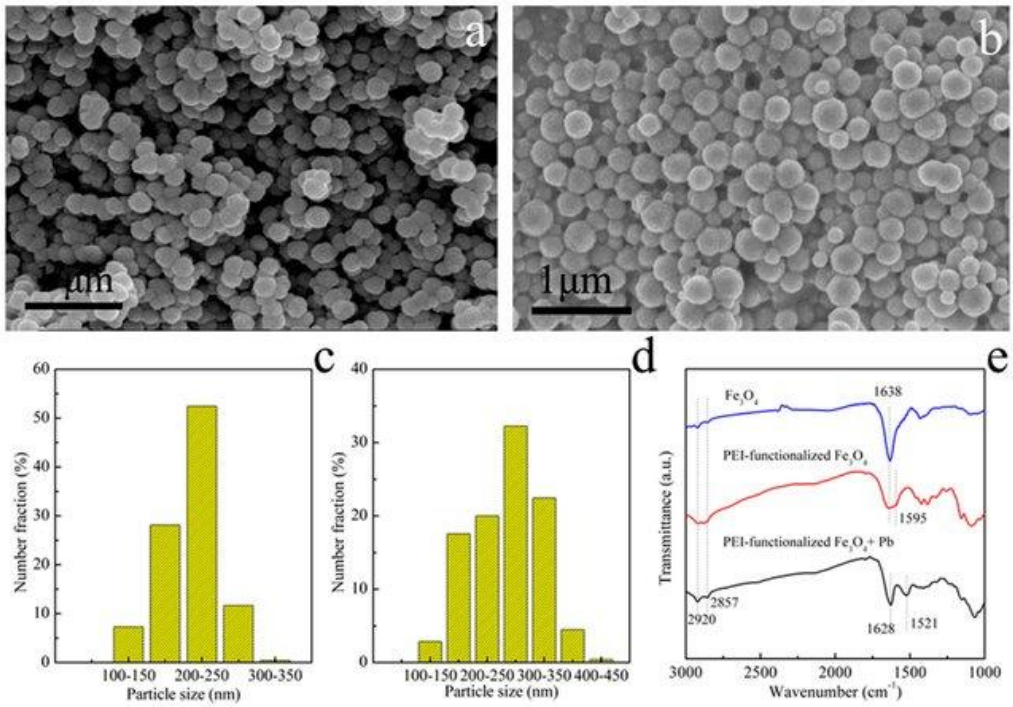


Fig. 5 PEI-functionalised Fe3O4 nanoparticles: SEM images of the coated (a) and uncoated (b) particles, size distributions (c, d; coated, mean about 209 nm; uncoated, mean 256 nm) and FTIR spectra of uncoated particles, PEI-functionalised particles and PEI-functionalised particles after Pb(II) adsorption (e). Reproduced from [24] under a CC BY licence

Table 1 Magnetic material classes, properties, limitations and best-fit roles

Material class	**Useful attributes**	**Common limitations**	**Best-fit role**
Fe_3O_4 / γ-Fe_2O_3 [17–19]	Magnetic recovery; adaptable surface chemistry; potential redox activity	Oxidation, aggregation, limited conductivity, iron leaching under harsh conditions	Magnetic preconcentration, sorption, support for functional coatings
MFe_2O_4 spinel ferrites [14, 15, 31]	Composition-tunable magnetism and redox/electronic properties	Composition-dependent toxicity; modest conductivity; cation leaching	Electrode modifiers, catalytic sorbents, magnetically recoverable catalysts
Magnetic carbon hybrids [34, 39, 40]	Improved conductivity, accessible surface, π	Complex synthesis; carbonaceous	Voltammetric electrodes; adsorption of aromatic organics

	interactions and adsorption	residues; uncertain long-term stability	
Magnetic polymers / MIPs [35, 36, 44]	Functional groups and molecular selectivity; convenient handling	Swelling, fouling, slow diffusion, polymer ageing	Selective extraction; antibiotic and pesticide recognition
Magnetic metal / ferrite hybrids [3, 6]	Catalytic or electrocatalytic enhancement; multiple active sites	Noble-metal cost or base-metal leaching; complex interfaces	Trace-metal sensing and catalytic transformation
Magnetic biochar / biopolymer composites [37, 38, 40]	Potentially low-cost feedstocks; functional groups; sorption	Feedstock variability; possible organic matter release	Bulk treatment and low-cost polishing after validation

4 Electrochemical sensing: mechanisms and architectures

4.1 Voltammetric and stripping methods

Cyclic voltammetry is best for probing redox behaviour and electrode kinetics. Differential-pulse and square-wave voltammetry suit trace quantification because they suppress the capacitive background. In anodic stripping voltammetry, metal ions are first accumulated on the electrode and then oxidised off it, which builds a preconcentration step into the measurement. Magnetic particles can sit in an electrode film or collect analyte-bearing phases before transfer to the electrode. The signal depends on active area, mass transport, binding chemistry, accumulation time, deposition potential, electrolyte and the state of the electrode. Ferrite and magnetic-particle electrodes have been reported for As(III) [6], Pb(II) and Cd(II) [21], Cu, Cd and Mn [20] and other ions [3, 34, 45], and reviews of nanocomposite trace-metal sensing are available [32, 46–48].

Iron oxide can also take part in the signal chemistry. In the As(III) sensor of Fig. 6 [47], two routes run in parallel. On gold, As(III) adsorbs and is reduced to As(0) with electrons from the electrode, a catalytic route. On the iron oxide, surface Fe(II) is oxidised to Fe(III) while As(III) is reduced during adsorption, and the cycle reverses during the stripping scan, when Fe(III) is reduced back to Fe(II) as As(0) is oxidised to As(III). The Fe(II)/Fe(III) couple therefore acts

as a mediator that is regenerated on every scan, and the scheme shows both routes drawing on the same concentration gradient of As(III) at the surface.

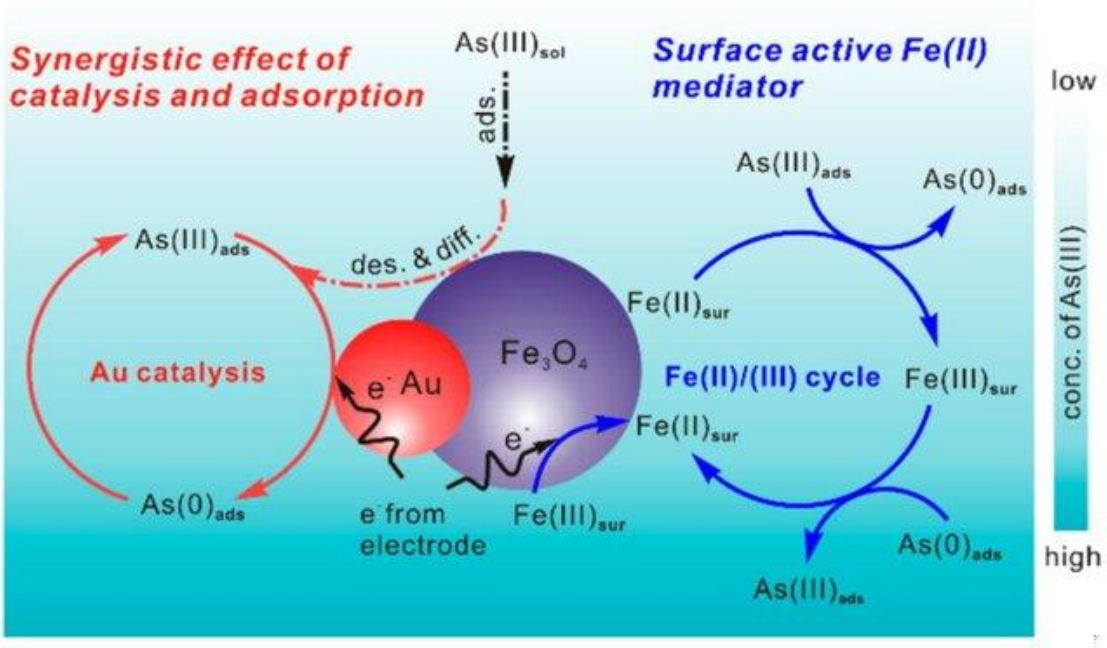


Fig. 6 Two coupled routes in stripping detection of As(III) at Au/Fe_3O_4 nanoparticles: Au-catalysed reduction of adsorbed As(III) to As(0), and a surface Fe(II)/Fe(III) cycle that mediates deposition and stripping. Reproduced from [47] under a CC BY licence

A sensitivity measured in clean buffer cannot be set beside a detection limit measured in river water. A fair comparison states the calibration range, how the limit of detection was calculated, the sample volume, the preconcentration protocol, the matrix, the number of replicate electrodes, recovery, selectivity tests and agreement with a reference method. The 3σ/slope estimate means little unless blank variability and calibration assumptions are described, and a limit of quantification is not a limit of detection.

4.2 Amperometry, impedance and electrochemiluminescence

Amperometric sensors read current at a fixed potential. They suit targets with a defined electrode reaction, or ones coupled to an enzyme, aptamer or catalytic label [5, 23]. Electrochemical impedance spectroscopy follows changes in interfacial resistance and capacitance. It pairs well with affinity recognition but responds to non-specific adsorption, ionic strength and electrode-to-electrode variation. Electrochemiluminescence has a low optical background and has been used for bisphenol A with Fe_3O_4-modified electrodes [22]. In each case the magnetic phase may be the active material, a preconcentration support, a label or a recoverable carrier, and papers should say which.

4.3 Recognition layers and selectivity

Bare magnetic oxides adsorb broadly and recognise little. Selectivity comes from ion-imprinted or molecularly imprinted polymers, aptamers, antibodies, enzymes, chelating ligands or tailored surface groups. Examples include a magnetic ion-imprinted membrane sensor for uranyl ion [44] and aptamer sensors for emerging pollutants [49, 50]. Recognition layers sharpen discrimination but can reduce electron transfer, slow diffusion or lose activity in storage. For metals, amine, thiol, carboxylate and phosphate ligands support binding [24], but natural organic matter and other ions compete for them, and that competition has to be tested. For drugs and pesticides, imprinting or affinity recognition improves selectivity, and electroactive aromatics may also be read directly from their oxidation or reduction peaks.

4.4 Antibiotics, pesticides and emerging contaminants

Antibiotics and other drug residues are hard targets. Related compounds coexist at low concentrations, and losing the parent compound does not mean the hazard is gone. Nanomaterial-based electrochemical antibiotic sensing, including magnetic variants, is growing quickly [51–54], and magnetic Fe_3O_4 has been used to detect ibuprofen in saline media [43]. Pesticide sensors use enzyme inhibition, imprinting or direct oxidation [55–57]. The enzyme biosensor for organophosphorus pesticides on screen-printed carbon, with Au-coated Fe_3O_4 particles held on the electrode by a magnet [5], shows how a disposable format can work. Real-water tests of such sensors need transformation products, dissolved organic matter, pH and salinity effects, and cross-reactivity with similar molecules.

4.5 Sensor validation and reporting

A field sensor has to be judged on sensitivity, selectivity, precision, reproducibility, response and recovery time, operational and storage stability, fouling tolerance, throughput and drift (Fig. 7). Sensitivity is the slope of the response against concentration, $S = \Delta Y/\Delta X$, linearity is usually checked against $Y = mX + c$, and the limit of detection is taken as 3 SD/S, where SD is the standard deviation of the response (typically of blank measurements) [47]. Validation should use certified reference materials where they exist, matrix spikes, standard addition and a recognised laboratory method, and field tests should span independent sites and seasons. A low detection limit does not help if the device gives false positives, cannot be regenerated or needs frequent replacement of expensive reagents. Disposable screen-printed formats [5] and lab-on-a-chip integration of magnetic-particle electrodes [58] show how some of these requirements can be met.

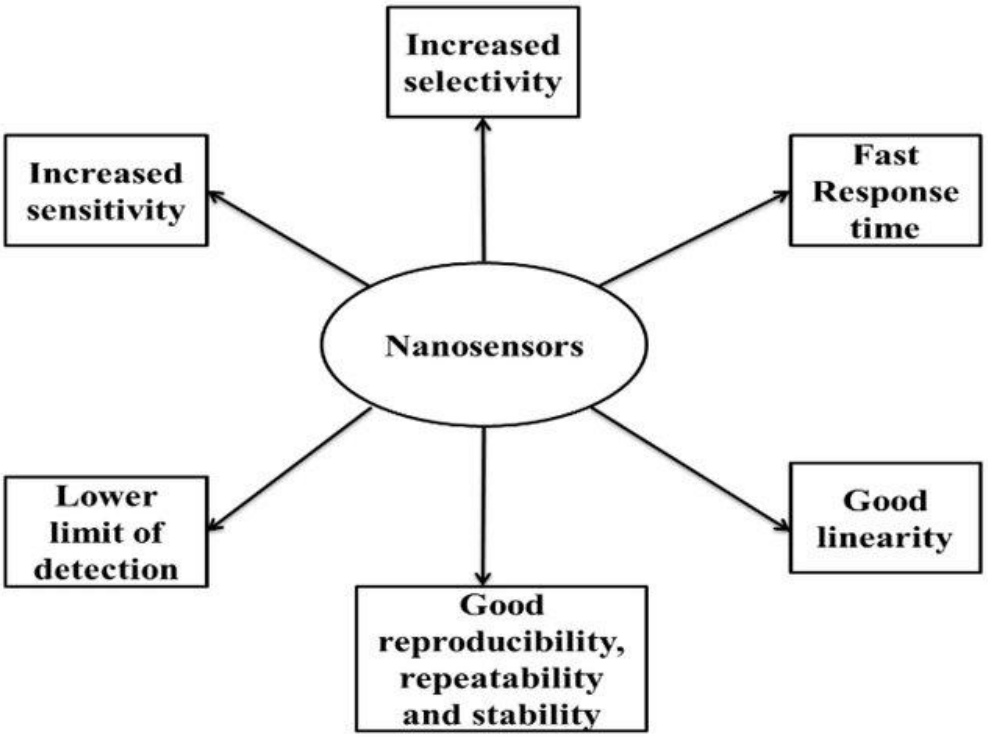


Fig. 7 Performance parameters used to judge nanomaterial-based sensors: sensitivity, selectivity, response time, linearity, reproducibility and stability, and detection limit. Reproduced from [47] under a CC BY licence

Fig. 8 puts three of the detection limits quoted in this review next to the relevant WHO guideline values [1]. The As(III) limit of 3.37 µg/L [6] is only about threefold below the 10 µg/L guideline, which leaves little margin once matrix effects and calibration uncertainty are included. The dimethoate limit of 5.6×10^{-4} ng/mL [5] is about four orders of magnitude below the 6 µg/L guideline, which looks comfortable but was obtained in buffer. The bisphenol A limit of 0.66 µg/L [22] is shown without a comparison because we did not use a guideline value for it. The chart makes a simple point: a detection limit far below a guideline is only reassuring if it survives real water, and Table 2 links each sensing strategy to its main validation check.

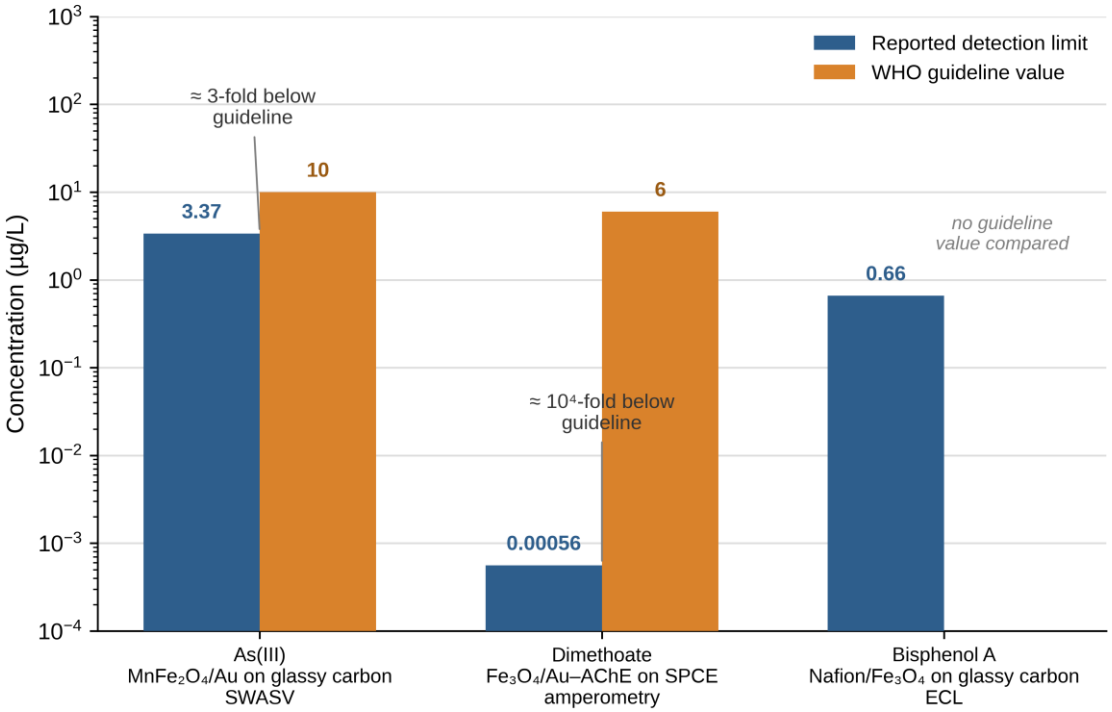


Fig. 8 Reported detection limits (blue) compared with WHO guideline values (orange) on a logarithmic scale: As(III) at an $MnFe_2O_4$/Au electrode, dimethoate at an Fe_3O_4/Au–acetylcholinesterase biosensor and bisphenol A at a Nafion/Fe_3O_4 electrochemiluminescence sensor (no guideline compared). Data from [1, 5, 6, 22]; the chart was prepared by the authors

Table 2 Electrochemical sensing strategies and critical validation points

Sensing strategy	**Typical target / use**	**Role of magnetic nanomaterial**	**Critical validation point**
Anodic stripping voltammetry [6, 47]	Pb, Cd, Cu, As-related workflows	Preconcentration; electrode catalysis; surface-area enhancement	Deposition conditions, intermetallic effects, matrix spikes, reference method
Differential-pulse / square-wave voltammetry [51–53]	Electroactive drugs, phenols, pesticides	Catalytic interface and electron-transfer enhancement	Peak overlap, fouling, selectivity against analogues

Amperometry [5, 23]	Enzyme/affinity assays; electroactive analytes	Magnetic label, carrier or catalytic phase	Potential selectivity; long-term stability of the recognition element
EIS [49, 50]	Affinity recognition and interfacial binding	Magnetic capture or functionalised interface	Equivalent-circuit justification, ionic-strength effects, nonspecific binding
Electrochemiluminescence [22]	Selected phenolic/organic pollutants	Electrode modification and signal enhancement	Luminescence quenching; optical and electrochemical controls
Magnetic extraction + electroanalysis [7, 58]	Low-level contaminants in complex samples	Enrichment followed by physical separation and measurement	Extraction recovery, desorption efficiency, mass balance

5 Remediation mechanisms and target contaminants

5.1 Adsorption, complexation and ion exchange

Adsorption is popular because it works under mild conditions and combines easily with magnetic recovery. Metal ions bind by electrostatic attraction, surface complexation, ion exchange or precipitation, and organic molecules by hydrogen bonding, hydrophobic interaction, π–π stacking and pore filling. Which mechanism dominates changes with pH, ionic strength, speciation and surface groups. A drop in dissolved concentration should be confirmed by mass balance and not by a single spectroscopic reading. Capacities measured in clean single-solute systems tend to overstate real-wastewater performance, because competing species take up the sites [30, 59].

For arsenic, oxidation state and pH matter, and phosphate and silicate compete for iron-oxide sites [2, 26]. For Cr(VI), reduction to Cr(III) can accompany adsorption, so the final oxidation state has to be measured [28, 42]. Pb(II), Cd(II), Cu(II) and similar cations bind to amine- or carboxyl-functionalised materials [24, 29, 60, 61], although Ca^{2+}, Mg^{2+} and dissolved organic

matter change the apparent capacity. Magnetically separable metal sorbents have been reviewed at length [62, 63]. The PEI-Fe_3O_4 data in Fig. 9 illustrate several of these points together [24]: capacity increases with temperature, which points to an endothermic uptake, and with pH, which fits the deprotonation of amine groups that then bind Pb(II); added Ca^{2+}, K^+ and Na^+ lower the capacity, as competition for binding sites would predict; and the regenerated particles (washed with 1.0 mol/L HCl) kept a high capacity across five cycles, with the bars in Fig. 9d staying close to the first-cycle value.

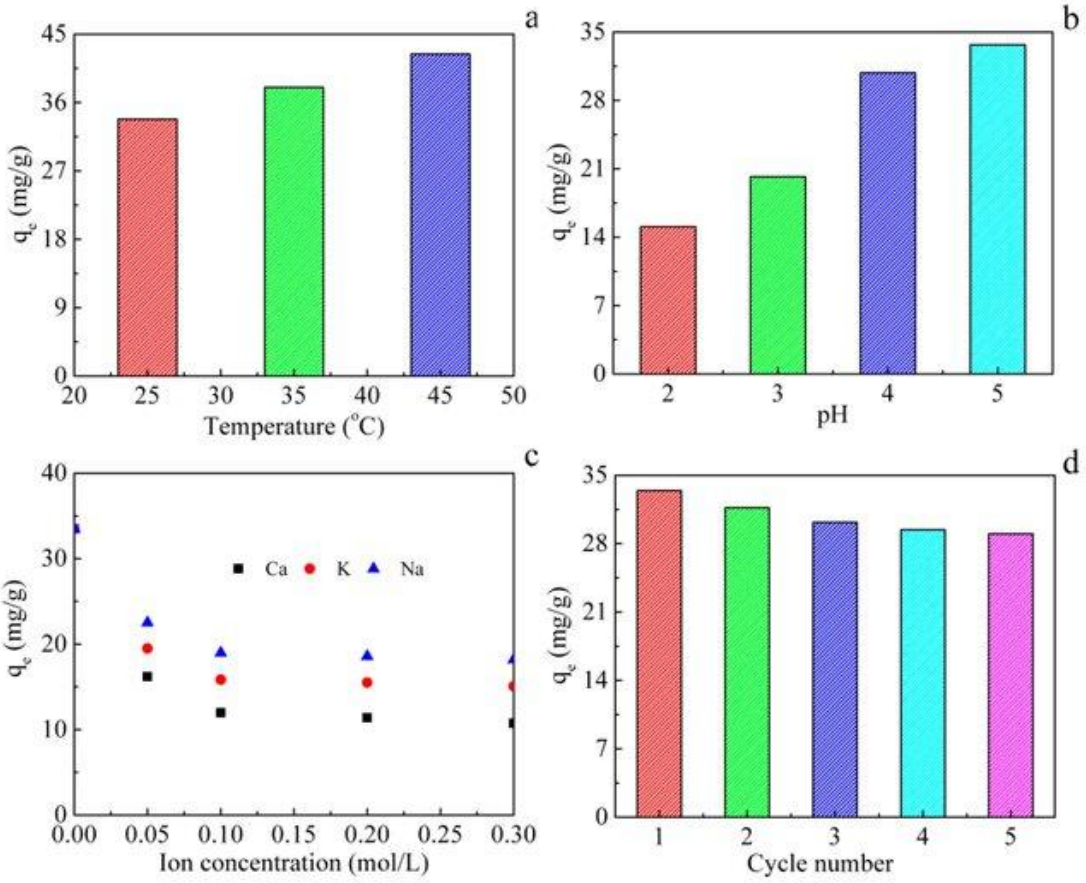


Fig. 9 Pb(II) adsorption capacity of PEI-functionalised Fe_3O_4 nanoparticles as a function of temperature (a), pH (b), competing cations (c) and number of regeneration cycles (d). Reproduced from [24] under a CC BY licence

5.2 Dyes, phenols and persistent organic pollutants

Magnetic polymers, chitosan, graphene oxide, activated minerals, biochar and ferrites have been tested on cationic dyes, Congo red, methylene blue, crystal violet and phenols [35–38, 64–72], and on polychlorinated biphenyls [73]. Electrostatic attraction and aromatic interactions usually act together, and for ionic dyes the pH relative to the point of zero charge is decisive. For hydrophobic compounds such as PCBs, partitioning into polymeric or

carbonaceous domains can outweigh electrostatics. Adsorption data should include initial concentration, dose, volume, temperature, pH, ionic strength and equilibrium time.

5.3 Pharmaceutical residues and antibiotics

Drug residues are chemically varied and may be positive, negative or neutral depending on pH. Magnetic carbon–ferrite composites [39, 40], zinc ferrite [74] and zwitterionic nanomagnets [75] have been tested on antibiotics and other pharmaceuticals, and reviews approach the topic through adsorption [76–79]. Several interactions can operate at once: electrostatics, hydrogen bonding, π interactions and hydrophobic partitioning. If a study claims degradation, it needs transformation-product and toxicity data as well as parent-compound analysis. Regeneration matters too, because replacing sorbent can dominate treatment cost and environmental burden [80].

5.4 Catalytic oxidation, Fenton-like chemistry and photocatalysis

Iron-containing magnetic materials can activate oxidants or carry photoinduced charge, which drives oxidation of organic pollutants. In Fenton-like systems, iron redox cycling produces reactive oxygen species, and efficiency depends on pH, oxidant dose, surface chemistry and scavengers [14]. Spinel ferrites and ferrite–TiO_2 or carbon composites have been studied for photocatalytic degradation [81–83], for example Zn–Cu ferrites [84], $CoFe_2O_4$ for azithromycin [27] and spinel ferrites for a model organic pollutant [85].

The $CoFe_2O_4$@methylcellulose study of Nasiri and co-workers [25] is useful because it includes the controls that many papers leave out (Fig. 10). In the comparison experiments, metronidazole removal was 23.7% by dark adsorption, 16.9% by UV photolysis alone and 81.59% by photocatalysis, so most of the loss is genuine photocatalytic degradation and not adsorption. The coated ferrite (85.30% at pH 11, 5 mg/L metronidazole, 0.2 g catalyst, 120 min) outperformed uncoated $CoFe_2O_4$ (55.13%). When the catalyst was recovered with a magnet, washed with alcohol and water and reused, removal fell from 81.59% to 78.42% in the second run and then stayed close to that level, at 77.58% in the fourth run. The authors attribute the small decline to intermediates adsorbing on active sites. They also measured Fe and Co in solution after the fourth run and did not detect them, and the XRD pattern changed only in intensity. Disappearance of the parent molecule is still not mineralisation, and this study, like the others, would need total organic carbon, intermediate identification and toxicity assays to support a claim of detoxification.

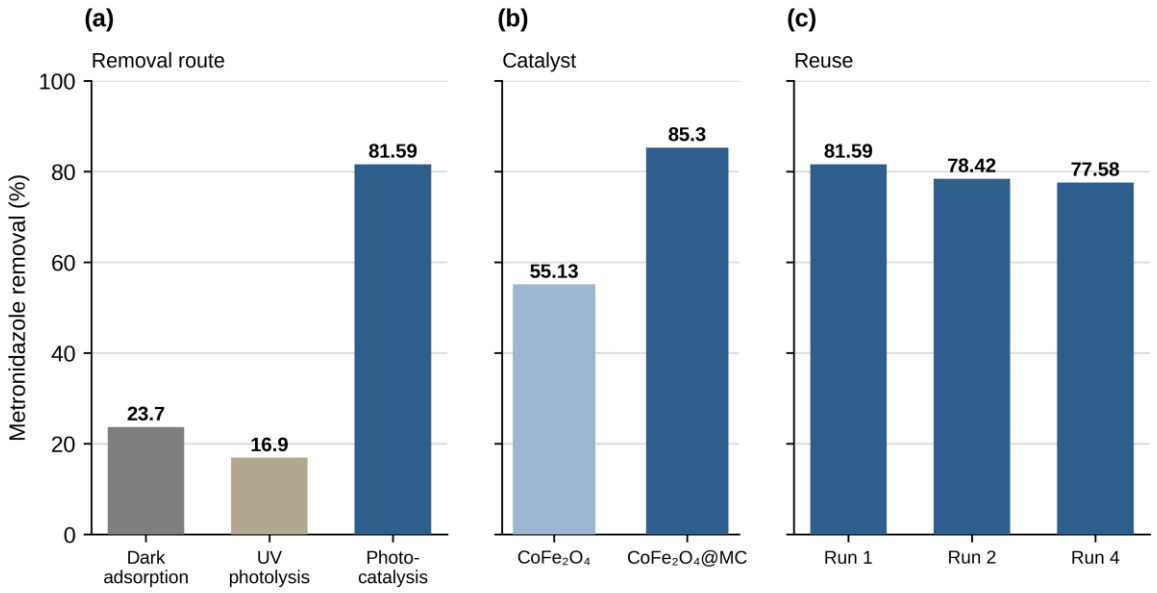


Fig. 10 Photocatalytic removal of metronidazole by $CoFe_2O_4$@methylcellulose under UV-C: (a) contribution of dark adsorption, photolysis and photocatalysis; (b) uncoated $CoFe_2O_4$ compared with the coated catalyst at pH 11, 5 mg/L, 0.2 g catalyst and 120 min; (c) removal in the first, second and fourth runs after magnetic recovery. Data from [25]; the chart was prepared by the authors

5.5 Coupled electrochemical treatment

Electro-oxidation, electro-reduction and electro-Fenton processes can be combined with magnetic catalysts to give treatment that is controlled by the applied potential. The attractions are local generation of reactive species and less bulk chemical dosing; the difficulties are fouling, energy demand, electrolyte use, by-products and catalyst stability. A magnetic catalyst should be compared with a non-magnetic analogue and a catalyst-free control, otherwise it is unclear whether magnetic recovery improves the process at all. Among the sources cited here, none addresses this coupling directly, while adsorption and photocatalysis are covered widely.

5.6 Microbial hazards and biological indicators

Magnetic nanoparticle conjugates have been used to capture and electrochemically detect waterborne bacteria and biohazard agents [23, 86]. The magnetic part concentrates the target and simplifies washing, while antibodies, aptamers or other ligands give recognition. Validation has to deal with non-specific binding, viable versus total cells, biofilm formation, interference from natural microbial communities and biosafety. A signal from captured cells

does not show that they are infectious. Table 3 summarises the contaminant classes covered in this section.

Table 3 Contaminant classes, remediation mechanisms, required controls and caveats

Contaminant group	**Representative mechanisms**	**Critical controls / measurements**	**Important caveat**
Toxic metals/metalloids [2, 24, 26]	Complexation, ion exchange, electrochemical deposition, redox conversion	Speciation, competing ions, leaching, total mass balance	Metals are transferred, not destroyed
Dyes and phenolic compounds [64, 65, 67]	Electrostatic attraction, π–π interaction, adsorption, oxidation	UV–Vis plus chromatographic confirmation; TOC for mineralisation	Colour loss can occur without complete detoxification
Antibiotics / pharmaceuticals [25, 27, 74]	Hydrogen bonding, electrostatics, hydrophobic interactions, catalytic oxidation	Parent compound and transformation products; bioassay where possible; dark and photolysis controls	Removal does not automatically prevent antimicrobial-resistance selection
Pesticides [5, 55, 56]	Hydrophobic partitioning, imprinting, enzyme inhibition, oxidation	Matrix recovery, analogue selectivity, degradation products	Food and environmental matrices can behave differently
Arsenic and chromium species [6, 28, 42]	Surface complexation and/or redox conversion	Oxidation-state-specific analysis; pH and competing oxyanions	Total concentration alone can conceal speciation changes

Microbial hazards [23, 86]	Affinity capture, magnetic concentration, electrochemical readout	Positive/negative controls, viability assay, non-target organisms	Capture or signal is not necessarily inactivation

6 Integrating sensing and remediation in one workflow

The most useful idea in this field may be to treat sensing and treatment as one workflow. A field sample is screened electrochemically. A validated result decides whether treatment is needed and under what conditions. A functionalised magnetic sorbent or catalyst is added, recovered with a magnet and checked for residual contaminant. The spent material is regenerated or sent to a controlled waste route, a post-treatment measurement confirms the endpoint, and periodic laboratory checks catch calibration drift.

The integration is not automatic. A material tuned for fast electron transfer may lack the capacity for bulk sorption, and a high-capacity sorbent may be too insulating or too slow for direct electroanalysis. A modular design is often more defensible: one formulation for sensing, another for treatment, sharing magnetic recovery or compatible surface chemistry. When one material is said to do both, each function must be validated on its own, and the effect of regeneration on sensor calibration must be reported. Some studies already combine detection and removal in a single material [87], but among the sources cited here it is the only one that treats the two together.

In practice the chain has seven steps: identify the target and its speciation; calibrate the sensor and check for interferences; choose a sorbent or catalyst for the water chemistry; contact and separate magnetically; quantify after treatment; evaluate regeneration and leaching; and manage the waste. The framework suits decentralised treatment, industrial pretreatment, groundwater remediation and episodic contamination events.

7 Comparative performance: reporting evidence responsibly

Ranking papers against each other is hard. Studies differ in initial concentration, sample volume, pH, dose, electrode geometry, accumulation time and the definition of the detection limit. The highest capacity in one paper should not be set against a removal percentage in another. Sensing metrics and treatment metrics belong in separate tables, each saying whether

the data came from model solutions, spiked environmental samples or untreated real water. Table 4 defines the metrics. Table 5 records, for the six primary studies discussed here, the target, material, matrix and test conditions as given in the article.

Table 4 Recommended definitions and reporting of sensor and remediation metrics

Metric	**Recommended definition / reporting**	**Frequent source of misleading comparison**
LOD [47]	3 × SD of the blank ÷ calibration slope; state blank replicates and units	Comparing different statistical definitions or sample preparation
Sensitivity [47]	Slope $\Delta Y/\Delta X$ over a stated concentration range, with units	Using peak current without electrode area or concentration basis
Recovery	Measured / spiked concentration × 100%; state matrix and n	Reporting recovery only in clean buffer
Removal efficiency	$(C_0 - C_t)/C_0 \times 100\%$, with sampling time and analytical method	Equating colour loss with complete degradation
Adsorption capacity	Mass-balance-based uptake per mass of dry sorbent	Using different concentration ranges or assuming equilibrium
Reusability	Capacity or signal retained per cycle, with regeneration method	Reporting only the first few cycles or omitting leaching
Energy / cost [80]	Energy per treated volume or mass removed; include consumables	Ignoring regeneration, separation, disposal and instrument cost

For sensing, report the target, electrode configuration, linear range, limit of detection and how it was calculated, sensitivity units, response time, recovery, precision, interference tests, matrix and reference-method comparison. For remediation, report initial and residual concentration, removal percentage, contact time, dose, capacity basis, pH, temperature, regeneration protocol, capacity retained over cycles, metal leaching, transformation products and toxicity. If a field-relevant metric is missing from a study, mark it “not reported”.

Table 5 Evidence matrix for six primary studies discussed in this review. "Not stated" means the item was not found in the abstract or open-access text examined for this review

Ref.	Target	Material and format	Sample matrix and test conditions	Performance and safety data
[6]	As(III)	$MnFe_2O_4$/Au hybrid nanospheres on glassy carbon; square-wave anodic stripping voltammetry	0.1 M NaAc–HAc, pH 5.0; deposition 150 s at −0.9 V; Cd(II) and Hg(II) tested as interferents	Sensitivity 0.315 µA/ppb; LOD 3.37 ppb; reproducibility and stability reported; recoveries, leaching and toxicity not stated
[22]	Bisphenol A	Nafion/Fe_3O_4 nanocrystals on glassy carbon; electrochemiluminescence with luminol	River water and tap water; anodic signal at +1.6 V; Fe_3O_4 amount, luminol concentration and pH optimised	Linear 0.01–5.0 mg/L (r = 0.9972); LOD 0.66×10^{-3} mg/L; recovery 96.0–105.0%, RSD < 4.8%; leaching and toxicity not stated
[5]	Organophosphorus pesticide (dimethoate)	Fe_3O_4/Au particles carrying acetylcholinesterase, with CNT/ZrO_2/Prussian blue/Nafion on screen-printed carbon; amperometric inhibition	pH 7.5 KNO= solution; particles held on the electrode by an external magnet; real-sample matrix not stated	Linear 1.0×10^{-3} to 10 ng/mL; LOD 5.6×10^{-4} ng/mL; disposable format; leaching and toxicity not stated
[24]	Pb(II)	Polyethyleneimine-functionalised Fe_3O_4 nanoparticles; adsorption	Aqueous Pb(II); 25 °C, pH 5.0; temperature, pH and added electrolytes varied	q max 60.98 mg/g (Langmuir; pseudo-second-order); capacity rose with temperature and pH and fell with electrolytes; "good performance" after

				five cycles (no percentage stated); toxicity not stated
[27]	Azithromycin	$CoFe_2O_4$ nanoparticles; UV photocatalysis	Simulated effluent; azithromycin 20–60 mg/L, 30–90 min, pH 6–10, catalyst 20–60 mg/L	Removal efficiency not stated in the text examined; leaching and toxicity not stated
[25]	Metronidazole	$CoFe_2O_4$@methylcellulose; UV-C photocatalysis (microwave-synthesised, magnetically recovered)	Simulated aqueous solution, 300 mL photoreactor; pH 11, 5 mg/L, 0.2 g catalyst, 120 min; HPLC analysis; no real water	Removal 85.30% (uncoated $CoFe_2O_4$, 55.13%); dark adsorption 23.7%, photolysis 16.9%; 81.59%, 78.42% and 77.58% in runs 1, 2 and 4; Fe and Co not detected after run 4; toxicity not stated

8 Environmental safety, sustainability and scale-up

Magnetic recovery reduces filtration and simplifies handling, but it does not guarantee that every particle is caught. Nanoparticles can stay dispersed, pass through a separator or be released during regeneration. Stability should be tested across realistic pH, salinity, organic matter and redox conditions, dissolved iron and other metals should be measured, and ecotoxicity should be tested for both fresh material and leachate. For cobalt-, nickel- or copper-containing ferrites the hazard assessment has to be specific to the composition; a "magnetic" or "green-synthesised" label is no substitute for toxicity data.

The primary studies we examined are uneven on this point. None of the six in Table 5 reported ecotoxicity. Only the $CoFe_2O_4$@methylcellulose study measured metal release directly, and it found no Fe or Co in solution after four runs [25]. Leaching can also depend on dose: in a groundwater study with green magnetite, residual iron in the treated water rose with the sorbent dose and exceeded the 0.1 mg/L drinking-water limit above 2.5 g/L [26]. We therefore present the safety points in this section as evidence requirements and not as established properties of the materials.

Life-cycle assessment should cover precursor synthesis, solvent and energy use, coating agents, magnetic separation, regeneration chemicals, electrode fabrication, sensor replacement and final disposal. Plant-extract synthesis can cut some reagents but adds batch variability and may need a lot of water. Reuse needs a complete material balance, not just a cycle count. Metal-laden sorbents may allow metal recovery, but regeneration eluates need treatment of their own. Catalytic processes should be compared with conventional ones at the same treatment endpoint for energy and oxidant use. Cost analyses of magnetic nanoadsorbents [80], assessments in real water [59] and the regulatory context for emerging micropollutants [4] are useful starting points.

Scale-up raises its own problems: consistent synthesis, storage stability, powder handling, separator design, pressure drop in continuous flow, magnetic field gradients and particle escape. Fixing particles in beads, membranes or porous supports eases handling but can cut the accessible surface and slow diffusion [88]. Continuous-flow studies should report flow rate, residence time, breakthrough curves, pressure drop, magnetic capture efficiency and behaviour under variable influent. Table 6 lists the evidence each readiness dimension needs and what the primary studies reported.

Table 6 Translational readiness matrix: evidence required before field deployment and what the primary studies in Table 5 reported

Readiness dimension	**Evidence required before deployment**	**Found in the primary studies of Table 5**
Synthesis reproducibility	Batch-to-batch size, phase, magnetisation and surface chemistry	Not stated in the abstracts examined
Real-water validation	Spiked and untreated real samples; reference-method comparison	River and tap water for a bisphenol A sensor [22]; not used in the other five
Continuous-flow performance	Breakthrough curves, residence time, pressure drop	Not stated; all six studies describe batch or single-electrode tests
Recovery efficiency	Measured magnetic capture and particle release	Magnet used to collect particles [6], hold them on an electrode [5] or recover the catalyst [25]; capture efficiency not quantified
Metal leaching	Residual metal in treated water compared with the drinking-water limit	Fe and Co not detected after four runs [25]; not stated in the other five. In a further groundwater study, residual iron exceeded 0.1 mg/L above a sorbent dose of 2.5 g/L [26]
Ecotoxicity	Fresh material and leachate; transformation products	Not stated in any of the six studies
Regeneration	Capacity retained per cycle; leaching per cycle	Five cycles, “good performance” [24]; 81.59% to 77.58% over four runs [25]
Cost and life-cycle assessment	Cost per treated volume; LCA including regeneration and disposal	Not stated; cost context is reviewed in [80]

9 Research gaps and future directions

Eight priorities stand out. First, standardised reporting and independent replication of the best sensor and sorbent formulations. Second, a move from model solutions to real wastewater and groundwater across seasons and industries [59]. Third, materials designed from explicit structure–function hypotheses, with controls that isolate the role of the magnetic phase. Fourth, data-driven optimisation, which will only help if the training data are comparable, traceable and come with uncertainty. Fifth, sensors judged as measurement systems and not only as modified electrodes, since calibration transfer, drift, reference electrodes, temperature, packaging, sample conditioning and maintenance all decide field usefulness [89, 90]. Sixth, toxicity and transformation-product data for pharmaceutical and pesticide remediation. Seventh, direct measurement of magnetic recovery efficiency and particle release. Eighth, life-cycle and techno-economic evidence behind sustainability claims.

One further step ties these together: closed-loop tests in which a measured residual concentration sets the contact time, regeneration or treatment switching. A modular platform with a magnetic preconcentration stage, a disposable or regenerable sensor, a recoverable catalytic sorbent and post-treatment verification is a plausible target. It should be judged against conventional alternatives on cost per sample, cost per treated volume, energy, residual toxicity, material loss and reliability, not on peak sensitivity or maximum capacity.

10 Conclusions

Magnetic iron oxides and spinel ferrites contribute three separable functions to water monitoring and treatment: magnetic handling, surface binding or recognition, and redox or catalytic activity. The most convincing systems give each function to a defined component, for instance a conductive support for electron transport, an imprinted or ligand layer for selectivity, a protective shell against leaching and a magnetic core for recovery. Characterisation data show what is actually present: the indexed spinel rings and 0.257 nm (311) lattice fringes of the $MnFe_2O_4$/Au spheres [6], the magnetite XRD pattern and crystallite sizes of 12–28 nm of the green and co-precipitated materials [26], and the PEI band in the FTIR spectrum of the amine-coated Fe_3O_4 [24]. Each added component brings its own cost, stability and leaching question, so design should start from a named limitation and not from a larger surface area.

Reported figures of merit are competitive with health-based limits but are not directly comparable. An Au-decorated $MnFe_2O_4$ electrode detected As(III) at 3.37 µg/L, about threefold below the WHO guideline of 10 µg/L [1, 6]. A Nafion/Fe_3O_4

electrochemiluminescence sensor reached 0.66 µg/L bisphenol A with 96.0–105.0% recovery in river and tap water [22]. PEI-functionalised Fe_3O_4 adsorbed up to 60.98 mg/g Pb(II) [24], and a $CoFe_2O_4$@methylcellulose photocatalyst removed 85.3% of metronidazole while photocatalysis, not adsorption or photolysis, accounted for most of the loss [25]. These values come from different units, matrices and test conditions, which is why Tables 4 and 5 define the metrics and record the conditions.

The evidence for safety and durability lags behind the evidence for performance. Among the six primary studies examined in detail, one used real water, none reported ecotoxicity, one measured metal leaching, none reported continuous-flow tests, and regeneration was followed for four or five cycles. Removal of metronidazole dropped from 81.59% to 77.58% between the first and fourth run [25], a small loss but a short test. In the text examined, loss of the parent compound was not accompanied by transformation-product data. Claims that these materials are green, sustainable or detoxifying are therefore hypotheses until leaching, ecotoxicity, transformation-product and regeneration data support them.

Three recommendations follow. First, sensing and treatment should be validated separately even when one material does both, and every study should state the matrix, the definition of the detection limit, the capacity or removal basis, the number of regeneration cycles, metal leaching and toxicity, or report them as not measured. Second, a modular sensor-guided workflow, in which an electrochemical measurement selects the treatment and a magnet recovers the spent material, is a practical route to decentralised monitoring and treatment. Third, closed-loop tests in untreated real water, with continuous flow and life-cycle assessment, are the next evidence step before deployment. Magnetic nanomaterials will be most useful as tunable, application-specific, recoverable components and not as universal solutions.

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Data availability. Not applicable.

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