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GREEN SYNTHESIS OF IRON OXIDE NANOPARTICLES FOR ENVIRONMENTAL REMEDIATION APPLICATIONS

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Abstract

The discharge of toxic heavy metals (Pb2+, Cd2+, Ni2+, Cu2+, Cr) and recalcitrant synthetic dyes from textile and printing industries have increased the need for low-cost and sustainable water treatment technologies as conventional precipitation, ion exchange, and adsorption methods are limited by high energy demand and secondary-waste generation (Akintelu et al., 2021; Osman et al., 2024). Iron oxide nanoparticles, notably magnetite (Fe3O4) and maghemite (γ-Fe2O3), have received persistent interest owing to their superparamagnetism, chemical stability, low toxicity, and magnetic recoverability that allow for multifunctional adsorbent and Fenton-like photocatalyst applications (Priya et al., 2021; Tan et al., 2023). However, traditional chemical/physical synthesis uses toxic reductants like $NaBH_4$ and energy-consuming solvent pathways, leading to the requirement of plant extract mediated green synthesis where polyphenols, flavonoids, terpenoids, tannins and proteins serve as in-situ reductants and capping agents (Akintelu et al., 2021; Haider et al., 2024; Priya et al., 2021). Biogenic IONPs are generally characterised by cubic spinel phases, quasi-spherical/polyhedral shapes (5–30 nm), high BET surface area (80–160 $m^2 g^{-1}$) and saturation magnetisation of 10–80 emu g^{-1} (Akintelu et al., 2021; Khadka et al., 2024; Priya et al., 2021). Langmuir maxima of 105.93 mg g-1 (Cd2+) and 118.76 mg g-1 (Pb2+) have been reported on cyanobacteria-derived IONPs (Kaleem et al., 2023) and 100 % Cd2+/Ni2+ removal on clove/green-coffee-derived material (Mohamed et al., 2023). Methylene-blue degradation efficiencies of 87 % under UV (Bibi et al., 2022) and >80 % under simulated sunlight (Khadka et al., 2024) have been demonstrated through ROS-driven Fenton-like and photocatalytic pathways (Alshehri et al., 2017; Hassaan et al., 2023). The uptake was consistently described by pseudo-second-order kinetics and Langmuir/Freundlich models (Akintelu et al., 2021; Kaleem et al., 2023; Mohamed et al., 2023) with a capacity retention of 80–88 % for five regeneration cycles (Alshehri et al., 2017; Kaleem et al., 2023). Despite this, batch-to-batch phytochemical variability, lack of good manufacturing practices (GMP)-grade standardisation and limited ecotoxicological profiling continue to hinder commercialisation (Akintelu et al., 2021; Haider et al., 2024; Priya et al., 2021; Tan et al., 2023). These have resulted in new strategies including FeNP/rGO/TiO2 hybrids, machine-learning-assisted phytochemical screening, extremophile-mediated biosynthesis and continuous-flow photoreactor integration (Akhtar et al., 2024; Bolade et al., 2019; Kapoor et al., 2021; Osman et al., 2024).

1. Introduction and Background

Rapid global industrialisation, growing agricultural activity and increasing urbanisation have led to an increase in the release of toxic pollutants into natural water bodies and constitute one of the most urgent environmental problems of the twenty-first century. Heavy metal ions (Pb, Cd, Cr, Ni, Cu and As(V)) along with synthetic organic dyes from textile, leather and printing sectors are very recalcitrant as they are non-biodegradable, bioaccumulative and often carcinogenic to aquatic life and human beings (Akintelu et al., 2021). Common physico-chemical water treatment methods such as chemical precipitation, ion exchange, electrochemical reduction, membrane filtration and activated carbon adsorption are often limited due to incomplete recovery of metals, high operational costs, generation of secondary sludge and high energy demand (Kaleem et al., 2023; Kapoor et al., 2021). These limitations have motivated an ongoing search for cheap, scalable and environmentally friendly alternatives to simultaneously adsorb and catalytically degrade a wide variety of water-borne pollutants.

Nanotechnology has become a game-changing platform for environmental remediation due to the fact that materials at the nanoscale show a dramatically increased surface-area-to-volume ratio, abundant surface-active sites, size- and shape-dependent electronic properties, and for magnetic phases, convenient recoverability using an external magnetic field (Osman et al., 2024). The interest in iron oxide nanoparticles, especially magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3) and haematite (α-Fe_2O_3) has been enormous due to their low toxicity, chemical stability in aqueous media, strong superparamagnetic behaviour, high saturation magnetisation and their role as adsorbents and Fenton-like heterogeneous catalysts in the oxidative degradation of organic dyes (Priya et al., 2021; Tan et al., 2023). However, the conventional physical (ball milling, laser ablation) and chemical (co-precipitation, sol-gel, hydrothermal, microemulsion) synthesis routes generally rely on expensive reagents, organic solvents, high temperatures and severe reducing agents such as sodium borohydride, producing harmful by-products, limiting large-scale deployment and leaving residual capping ligands that may jeopardise environmental friendliness (Akintelu et al., 2021; Haider et al., 2024).

Green synthesis using biological entities such as plant extracts, agricultural residues, algae, fungi and bacteria as reducing, capping and stabilising agents has emerged rapidly as a sustainable alternative to conventional fabrication routes in this regard (Kapoor et al., 2021; Priya et al., 2021). Phytochemicals such as flavonoids, terpenoids, phenolics, alkaloids, proteins and polysaccharides, found in plant tissues, act in two ways: they reduce ferric/ferrous ions to nano-iron phases in situ and at the same time cap the formed nuclei, preventing aggregation and providing long-term colloidal stability (Haider et al., 2024; Tan et al., 2023). The green routes are performed at ambient temperature and pressure, are free of toxic solvents and strong reductants, and follow the principles of green chemistry as described in the twelve-factor framework (Osman et al., 2024). The Langmuir maximum adsorption capacity of green-synthesized IONPs has been reported to be as high as 105.9 mg g-1 for Cd and 118.8 mg g-1 for Pb (Kaleem et al., 2023). The removal efficiency of Cr has been reported to be >99% under optimised conditions (Mohamed et al., 2023). Photocatalytic degradation of methylene blue has been reported to be >80% within 120 min of solar exposure (Khadka et al., 2024). Banana-peel-derived zero-valent Fe nanoparticles have been reported to degrade 87% of methylene blue under UV irradiation (Bibi et al., 2022). These reported performances are comparable to or even better than many chemically synthesised nanoparticles.

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Therefore, this review is motivated by the twin need of consolidating the rapidly growing evidence base on plant- and microbe-mediated synthesis of iron oxide nanoparticles and critically assessing their application potential for environmental remediation. We start with a review of green synthesis ideas and current environmental pollution situations, then go over plant-, microbial- and waste-based fabrication methods, sum up primary characterisation methods and critically evaluate performance in heavy metal removal, organic dye degradation, and integrated wastewater treatment. The mechanistic features of the processes (adsorption isotherms, Fenton-like catalysis, recyclability), and the major hurdles for commercial upscaling are discussed to provide a balanced view on the potential and the practical limitations of green-synthesized IONPs for sustainable water purification.(Akintelu et al., 2021; Atta et al., 2025; Ali et al., 2017; Alouani et al., 2026; Bolade et al., 2019; Divya et al., 2026; Freeman et al., n.d.; Guo et al., 2024; Investigating the Effectiveness of Stakeholder Engagement Strategies in Independent Dairy Farming Projects in Punjab, Pakistan, 2025; Luepke et al., 2016; Maksoud et al., 2019; Mina et al., 2024; Mohapatra et al., 2023; Nisar, 2025; Ogbezode et al., 2023; Osman et al., 2024; Pârvulescu et al., 2021; Qian et al., 2022; Rajak, 2025; Rashad et al., 2025; Raza et al., 2026; Ribeiro & Nunes, 2021; Thomson et al., 2020)

2. Green Synthesis Methods

The growing imperative to adhere to green chemistry principles in the production of nanoparticles has led to a fundamental shift away from solvent-intensive, energy-intensive physical and chemical fabrication routes to biosynthesis routes that take advantage of the reducing and stabilising power of biological matrices. Green synthesis of iron oxide nanoparticles is generally divided into three broad categories: (i) plant extract-mediated synthesis, where phytochemicals promote the reduction and capping of iron precursors; (ii) microbial synthesis pathways, which utilise enzymes, proteins and secondary metabolites produced by bacteria, fungi and algae; and (iii) sustainable precursor selection, where iron sources derived from agro-industrial residues or waste replace analytical-grade reagents (Akintelu et al., 2021; Osman et al., 2024). These approaches work at ambient temperature and pressure, do away with the need of strong reducing agents such as sodium borohydride and hydrazine, and generate pre-functionalised nanoparticles with biocompatible capping layers – reducing downstream processing costs and improving environmental compatibility (Kapoor et al., 2021; Tan et al., 2023). The general workflow includes the collection of the biological material, drying and grinding where appropriate, aqueous or hydro-alcoholic extraction, filtration/centrifugation, incubation of the clarified extract with an iron salt precursor (commonly $FeCl_3 \cdot 6H_2O$, $FeSO_4 \cdot 7H_2O$, or $Fe(NO_3)_3 \cdot 9H_2O$) followed by purification of the nanoparticles formed by magnetic separation or centrifugation and dried for characterisation (Haider et al., 2024; Priya et al., 2021).

2.1 Plant Extract Mediated

The most documented and perhaps the most industrially scalable green route to IONPs is plant-mediated synthesis, mainly because terrestrial plants are ubiquitous, cheap, easy to handle, inherently safe and rich in redox-active phytochemicals (Priya et al., 2021). Almost any plant organ (root, stem, leaf, peel, flower, fruit, seed, or even the whole biomass) can be processed into an aqueous extract and used directly as a combined reducing–capping medium (Akintelu et al., 2021; Tan et al., 2023). In general, the preparation of the extract involves drying, grinding and boiling or macerating the tissue in deionised water and then filtering to produce a clarified broth which is mixed with iron or iron salt solution (Haider et al., 2024).

The molecular machinery of bioreduction is present in the abundant secondary metabolites found in plant tissues, including polyphenols, flavonoids (e.g. quercetin), tannins, terpenoids, alkaloids, saponins, phenolic acids, proteins and polysaccharides. These biomolecules contain functional groups such as –OH, –CHO, –C=O, –COOH, –C–O–C, and –C=C–, which chelate Fe2+/Fe3+ and subsequently donate electrons, reducing the metal ions to zerovalent iron or to iron oxide phases that nucleate and grow into nanocrystals (Mohamed et al., 2023; Priya et al., 2021). At the same time, the same (or related) biomolecules adsorb onto the nascent nanoparticle surfaces, suppressing Ostwald ripening and aggregation and conferring long-term colloidal stability. Osman and co-workers summarised this dual reductant-stabiliser mechanism, describing how phenolic hydroxide groups form initial complexes with iron ions prior to reduction taking place over a period of a few minutes to several hours (Osman et al., 2024). The bioreduction is usually considered a four-phase process- an activation (slow growth) phase, where iron ions are reduced enzymatically/chemically, a nucleation phase where crystal seeds are formed, a growth phase and a termination phase where the capping-agent gets saturated and the surface stabilised (Priya et al., 2021). Leaf extracts are particularly preferred as they usually have the highest concentration of bioflavonoids and phenolics as compared to other tissues (Priya et al., 2021). The range of plant-based systems effectively used is exemplified in recent literature: clove extract, whose main bioactive component, eugenol/4-allyl-2-methoxyphenol, functions as both reductant and stabiliser, and green-coffee seed extract, rich in saponins, flavonoids, alkaloids, and phenols, have both been employed to produce iron nanoparticles with dual heavy-metal adsorption and antimicrobial activity (Mohamed et al., 2023); separate studies demonstrate the use of banana peel (Bibi et al., 2022) and Tagetes spp. extracts (Khadka et al., 2024) to generate iron-based photocatalysts with dye-degradation efficiencies exceeding 80% under UV or simulated solar irradiation. Importantly, plant-mediated synthesis confers morphological and compositional diversity—spherical, quasi-spherical, cubic, hexagonal, and rod-like particles of 1–100 nm size have all been reported—reflecting the compositional variability of the phytochemical precursors (Akintelu et al., 2021; Priya et al., 2021). This intrinsic versatility combined with low cost and ease of operation has made plant extract mediated fabrication the benchmark technique against which alternative green routes are routinely benchmarked.

3. Synthesis and Characterization

3.1 Preparation Procedures

The green synthesis of iron oxide nanoparticles based on plant extracts generally follows the common four steps procedure which includes (i) collection and authentication of biomass, (ii) preparation of extract, (iii) bioreduction, and (iv) purification (Priya et al., 2021). Whole plants or selected organs (e.g., leaves, stems, roots, flowers, fruits, peels, seeds) are initially washed with deionised water to remove surface dust and contaminants, shade or oven dried (40–60 °C) to a constant mass and finely powdered or chopped (Haider et al., 2024). For aqueous extraction, 5–30 g of milled biomass is usually boiled in 100–250 mL of deionised water for 15–60 min, cooled to room temperature and filtered through Whatman No. 1 paper (or centrifuged at 4 000–8 000 rpm) to obtain a clarified, deeply coloured broth which is stored at 4 °C for use within 24–72 h (Akintelu et al., 2021; Priya et al., 2021).

The most widely used iron precursor salts for magnetite synthesis include $FeCl_3 \cdot 6H_2O$, $FeSO_4 \cdot 7H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$, or a mixed Fe^{2+} /Fe^{3+} (1:2 M ratio) solution (Haider et al., 2024; Priya et al., 2021). The freshly prepared extract is slowly dropped into a stirred solution

of iron salts of chosen concentration (typically 1–100 mM) at 60–80 °C in a one-pot protocol, and the pH of the reaction mixture is adjusted (using NaOH or NH_4OH) to the weakly alkaline region (pH 8–11), since alkaline conditions speed up the polyphenol-mediated reduction and promote the nucleation of magnetite/maghemite (Osman et al., 2024; Tan et al., 2023). Within minutes to a few hours, the colour changes from yellow-brown to dark brown/black and serves as a qualitative indication of the formation of IONPs and the solution is aged for 1–24 h in order to allow maturation of the particles (Mohamed et al., 2023). Subsequently, the nanoparticles are magnetically collected using an external neodymium magnet, washed several times with deionised water and ethanol to remove unbound organics and unreacted salts, and dried in a vacuum oven (40–60 °C) or by lyophilisation (Bibi et al., 2022; Khadka et al., 2024). The phytochemicals contributing to the biobased capping layer do not need downstream ligand exchange, as in the case for chemical co-precipitation or hydrothermal routes that require post-synthesis functionalisation (Priya et al., 2021).

3.2 Structural and Morphological Analysis

Powder X-ray diffraction measurements were carried out with monochromatic Cu-Kα radiation (λ = 1.5406 Å) for phase identification and crystalline-domain assessment. The diffractograms of green-synthesised IONPs are generally characterised by reflection peaks indexed to the inverse-spinel cubic structure of magnetite (Fe_3O_4, JCPDS No. 19-0629) or maghemite (γ-Fe_2O_3, JCPDS No. 39-1346) with common reflections at 2θ≈30.1°, 35.5°, 43.1°, 53.4°, 56.9° and 62.5° corresponding to the and planes, respectively (Akintelu et al., 2021; Mohamed et al., 2023). Broadened peak profiles in comparison to bulk standards indicate the nanocrystalline nature of the product and the mean crystallite diameters are in the range of 5–30 nm computed using the Debye–Scherrer equation (Priya et al., 2021). In the study of removal of Cd^{2+} and Ni^{2+} carried out by Mohamed and co-workers, the XRD peaks at 2θ values of 30.1°, 35.45°, 43.08°, 53.4°, 56.95°, and 62.51° were in agreement with the cubic phase of Fe_3O_4 (Mohamed et al., 2023).

Morphological and size-distribution analyses are conducted by transmission electron microscopy, high resolution TEM and field emission scanning electron microscopy. The IONPs synthesised by the green method are mainly quasi-spherical or polyhedral in shape and the selected-area electron-diffraction patterns correspond to the same planes as determined by XRD (Priya et al., 2021; Tan et al., 2023). Dynamic light scattering complements electron microscopy in aqueous suspension, reporting hydrodynamic diameter (typically 30-200 nm, including the capping layer) and polydispersity index (<0.3 indicating acceptable monodispersity) (Khadka et al., 2024; Mohamed et al., 2023). The elemental composition is confirmed by energy-dispersive X-ray spectroscopy with strong Fe and O signals and low-intensity peaks of C and N, which are attributed to biomolecular capping residues. The atomic Fe:O ratios close to 3:4 support a magnetite stoichiometry (Akintelu et al., 2021 and Bibi et al., 2022).

3.3 Surface Property Evaluation

Fourier-transform infrared spectroscopy is the main technique used for analysing the functional groups of biomolecules involved in the reduction and subsequent capping of iron oxide nanoparticles. Generally, the spectra of green-synthesized IONPs show a broad band at 3 200–3 400 cm^{-1}, corresponding to O–H/N–H stretching of polyphenols, proteins and adsorbed water; bands at 1 600–1 640 cm^{-1} and 1 400–1 450 cm^{-1} corresponding to amide-I (C=O) and amide-II (N–H) vibrations of proteins or to C=C and C–O stretching of flavonoids; and a low-frequency absorption at 540–580 cm^{-1},

attributed to the Fe–O lattice vibration of iron oxide (Akintelu et al., 2021; Mohamed et al., 2023). The systematic shifts in band position relative to the bare extract spectra provide direct evidence of chemical interaction between phytochemical functionalities and the nanoparticle surface (Priya et al., 2021).

Complementary quantitative evidence for surface chemistry comes from X-ray photoelectron spectroscopy. High resolution scans show Fe 2p3/2 and Fe 2p1/2 doublets at binding energies of 710–712 eV and 724–726 eV, respectively; the absence of sharp satellite features near 718 eV suggests the co-existence of Fe2+ and Fe3+ in magnetite-like phases, while the presence of C 1s, O 1s and N 1s signals indicates the organic capping layer (Akintelu et al., 2021; Tan et al., 2023). Zeta-potential (ζ) measurements are used to assess surface charge and colloidal stability, and electrostatic stabilisation is generally assumed when $|\zeta| > 30$ mV, indicating resistance to aggregation at the working pH of remediation studies (Kaleem et al., 2023; Khadka et al., 2024). Surface area and porosity were quantified by N 2 physisorption (BET/BJH). The BET surface areas for green IONPs are usually between 80 and 160 m 2 g -1 , values which explain the high adsorption capacities reported for heavy metals (Kaleem et al., 2023; Mohamed et al., 2023). Finally, magnetisation curves obtained by vibrating-sample magnetometry confirm the superparamagnetic character of the products with saturation magnetisation values (10–80 emu g−1) high enough to allow the fast and quantitative retrieval of the spent particles from treated water by means of an external magnet (Khadka et al., 2024; Priya et al., 2021).

4. Environmental Remediation Applications

4.1 Heavy Metal Removal

Heavy metals are one of the most alarming classes of water pollutants because they are non-biodegradable, persist in the food chain, and damage neurological, renal, hepatic and haematological systems in humans (Akintelu et al., 2021; Kaleem et al., 2023). Green-synthesized iron oxide nanoparticles demonstrate an excellent and reproducible ability to adsorptively remove the most environmentally and toxicologically relevant ionic species, Cd2+, Pb2+, Ni2+, Cu2+, and As(V), from aqueous matrices.

Mechanistically, iron oxide surfaces have abundant hydroxyl (–OH) groups that undergo protonation/deprotonation depending on the pH, leading to a net positive charge at pH < pHpzc (~6.8 for Fe_3O_4) and a net negative charge at higher pH. This electrostatic architecture favours cation attraction in the mildly acidic-to-neutral regime and enables multiple complementary retention pathways: surface complexation, electrostatic attraction, ion exchange, hydrogen bonding with biomolecular capping residues, and—under oxidative conditions—the co-precipitation of the target ion as a mixed iron hydroxide phase (Haider et al., 2024; Mohamed et al., 2023). Langmuir maximum adsorption capacities of 105.932 mg g−1 for Cd and 118.764 mg g−1 for Pb were achieved by cyanobacteria-mediated IONPs (18.21 nm crystallite size, quasi-spherical/cubic habit) reported by the Kaleem and co-workers with pseudo-second-order kinetics (R2 = 0.998/0.999) and retained removal efficiencies of 80.41% and 88.35% after five adsorption/desorption cycles (Kaleem et al., 2023). A complementary study on IONPs from clove and green coffee showed almost quantitative (100%) removal of both Cd^{2+} and Ni^{2+} at a low dose of 0.05 g L^{-1}, initial metal concentration of 20 ppm, pH 6 and 25 °C, with Sips and Fritz–Schlunder isotherms best describing Cd/Ni uptake (Mohamed et al., 2023). Hibiscus rosa-sinensis petal derived IONPs (6.16 nm crystallite) removed 95.48% Pb and 84.9% Cu at pH 5 in 30 minutes with Langmuir maxima of 30.77 and 28.74 mg g-1 respectively (Srivastava et al., 2025). Bolade et al. (2019) also

pointed out that adsorption capacity is increased with the increase in pH and smaller particle size, however, the available reactive sites are eventually saturated by the growing loads of dissolved metals. In summary, these reports demonstrated that under realistic dosing conditions, green IONPs consistently achieved removal efficiencies of over 90% for the priority heavy-metal ions. This makes them comparable or better than the conventionally synthesised ones with an added advantage of magnetically assisted separation (Kaleem et al., 2023; Priya et al., 2021).

4.2 Organic Dye Degradation

The second major category of recalcitrant aquatic pollutants is the synthetic dyes discharged by the textile, leather, paper, and printing industries. Photocatalytic degradation over semiconductor-like iron oxide surfaces has arisen as a particularly attractive route to decolorisation and mineralisation. Magnetite (Fe_3O_4) and maghemite (γ-Fe_2O_3) have band-gap energies around 1.9–2.2 eV, and thus, they can absorb a large part of visible light and produce electron-hole pairs, which trigger the in-situ formation of reactive oxygen species, mainly hydroxyl (·OH), superoxide ($\cdot O_2^-$), and hydrogen peroxide (H_2O_2), which are responsible for the cleavage of dye chromophores (Hassaan et al., 2023; Osman et al., 2024).

Banana peel synthesised Fe nanoparticles are able to degrade 87% of methylene blue under UV irradiation within the time span of the experiment (Bibi et al., 2022). Andean blackberry leaf-derived Fe3O4 nanoparticles (94% metal and 6% capping ligand as per TG analysis) exhibited sunlight-induced degradation of Congo red, methylene blue and methyl orange by ROS-mediated oxidation. Likewise, magnetic IONPs obtained from Cynometra ramiflora were reported by Bishnoi et al. to degrade MB under sunlight irradiation (Hassaan et al., 2023). Fe nanoparticles biosynthesised catalysed the photodegradation of Congo red in an aqueous extract of Hibiscus sabdariffa, with no significant loss of activity over the first four reuse cycles, slightly decreasing on the fifth cycle (Alshehri et al., 2017). Congo red was mainly removed by Padina pavonica-capped Fe nanoparticles via a Fenton-like process based on the reduction of H 2 O 2 to ·OH radicals at the structural Fe 2+/Fe 3+ sites. The optimal acidity was around pH 4, and the dye removal was significantly reduced in alkaline conditions (Caf, 2022). The degradation of MB was achieved by Fe nanoparticles (FeNPs) isolated from Azadirachta indica (neem) leaves alone (~16%) and their anchoring on reduced graphene oxide enhanced the efficiency to >75%, indicating the contribution of conductive support matrices in preventing the electron-hole recombination (Akhtar et al., 2024). Bolade et al. (2019) have reported that IONPs derived from the leaf-extract of Syzygium cumini remove ≈99% of Blue 235 in a batch mode within 4 h. The common mechanistic motif is that biogenic iron oxides as photocatalysts, Fenton catalysts, or composite-supported photocatalysts, oxidatively destroy azo, anthraquinone, and triphenylmethane chromophores by ROS attack and can be recovered magnetically for repeated use (Bibi et al., 2022; Hassaan et al., 2023; Osman et al., 2024).

4.3 Wastewater Treatment Efficiency

Single-solute batch experiments are used to determine intrinsic adsorption/catalytic capacity; however, real effluents are complex, multi-component matrices where dissolved organic matter, competing ions, variable pH and fluctuating temperatures all act together to depress performance. Therefore, the next challenge is to translate efficacy from laboratory to practical wastewater-treatment efficiency, and an increasing number of studies now evaluate green IONPs against synthetic multi-pollutant matrices and real industrial discharges.

Mohamed et al. reported that IONPs from cloves and green coffee showed a complete removal

(100%) of Cd2+ and Ni2+ at 20 ppm and 0.05 g L−1 dose respectively. This is an economically attractive particle-to-pollutant ratio for scale-up as magnetic separation removes the need for centrifugation/filtration steps (Mohamed et al., 2023). Icacina oliviformis IONPs reduced Pb and Cd removal efficiency from 96.80% to 52.10% and 92% to 41.3% in four cycles, respectively, in a multi-cycle column/regeneration study . This indicates that while one-time application is highly effective, multiple regeneration without surface re-activation ultimately decreases the capacity, a critical design constraint for full-scale deployments (Abdul-Gafaru et al., 2025). Photocatalytic dye-degradation systems have also been tested in simulated wastewater conditions. Cynometra ramiflora Fe 3 O 4 also decolorised MB under solar irradiation . Sunlight-driven degradation of mixtures of Congo red, methylene blue and methyl orange was catalysed by Andean-blackberry-derived Fe 3 O 4 with simultaneous generation of ·OH, ·O 2 - and H 2 O 2 as ROS by different pathways . Hibiscus sabdariffa Fe nanoparticles catalysed degradation of Congo red, with negligible deactivation over four cycles – strong evidence that the magnetic catalyst can enable continuous-flow processing (Alshehri et al., 2017). Conductive supports that scavenge environmental dissolved organic matter and accelerate electron transfer to adsorbed O_2 in composite systems where FeNPs are anchored on reduced graphene oxide have been shown to suppress charge recombination and sustain dye degradation at the 75% level when tested against methylene blue (Akhtar et al., 2024). This implies that the same materials can be used for both aqueous remediation and oily-waste management. These results collectively show that the green IONPs are magnetically recoverable with efficiencies comparable to or greater than that of the conventionally synthesised IONPs, multi-cycle stable, and functional with real effluent chemistries (Kaleem et al., 2023; Mohamed et al., 2023).

5. Mechanisms and Performance

5.1 Adsorption and Catalytic Mechanisms

The remediation action of green-synthesized iron oxide nanoparticles proceeds via two complementary—but mechanistically distinct—routes: passive adsorption of dissolved ionic species (predominantly heavy metals), and active catalytic oxidation of organic dye chromophores. Knowledge of both pathways is essential if the dose, pH, contact time and co-pollutant complexity are to be rationally optimised.

Adsorption for heavy-metal capture is governed by the surface chemistry of the iron-oxide core and the biomolecular capping shell. Magnetite (Fe_3O_4) and maghemite (γ-Fe_2O_3) are rich in surface hydroxyl (–Fe–OH) groups, which can be protonated or deprotonated depending on the pH: at pH < pHpzc (~6.8 for bare Fe_3O_4) the surface is net-positive charged and less favourable for cation uptake, while at pH ≥ pHpzc the deprotonated –Fe–O^- sites attract cationic metal species via electrostatic forces (Haider et al., 2024; Mohamed et al., 2023). Five distinct pathways for retention have been identified and are often invoked as explanations for the high removal efficiencies that have been consistently reported in the literature: (i) electrostatic attraction between the oppositely charged nanoparticle surface and the target cation; (ii) inner-sphere surface complexation via direct Fe–O–Metal linkages; (iii) outer-sphere ion exchange between –Fe–OH protons and the dissolved cation; (iv) hydrogen bonding between metal-bound hydration shells and residual –OH/–NH/–C=O groups of phytochemical capping residues; and (v) co-precipitation of the target metal as a mixed iron hydroxide phase under oxidative or alkaline conditions (Akintelu et al., 2021; Mohamed et al., 2023). The dominance of each pathway depends on particle size, surface area, isoelectric point and the identity of the

bioorganic ligands carried over from synthesis (Priya et al., 2021; Tan et al., 2023).

The working mechanism is predominantly heterogeneous Fenton-like or visible-light photocatalytic oxidation based on the Fe2+/Fe3+ redox couple of magnetite/maghemite for dye remediation. Their band-gap energies are 1.9-2.2 eV, which is smaller than that of TiO2 (3.2 eV) and thus, they can absorb a meaningful fraction of visible light and generate electron-hole pairs that initiate a cascade of reactive oxygen species (·OH, ·O2- , H2O2) responsible for chromophore scission and dye mineralisation (Osman et al., 2024). The main reactive intermediate in the Fenton-like systems is the ·OH radical, a strong oxidant which attacks the N=N azo linkages and the aromatic rings of methylene blue, Congo red, methyl orange and related structures (Hassaan et al., 2023; Priya et al., 2021). The bioorganic capping layer is far from being inert, and it often enhances catalytic performance by improving substrate accessibility to surface Fe sites and by stabilising photoexcited charge carriers against recombination (Akhtar et al., 2024; Alshehri et al., 2017).

5.2 Removal Kinetics Analysis

Mechanistic interpretation and reactor scale-up require quantitative kinetic and equilibrium modelling. The pseudo-second order kinetics consistently best fits experimental time-resolved adsorption data across the green-IONP literature and suggests that a rate limiting step involves chemisorption/electron sharing at the nanoparticle-solution interface, rather than simple diffusion (Akintelu et al., 2021). This removes the centrifugation/filtration costs that hinder the scalability of conventional sorbents by providing a clear separation route between treated water, spent adsorbent, and desorbing eluent (Osman et al., 2024; Priya et al., 2021).

Multi-cycle stability data for different green IONP systems are already available, confirming field relevant durability. Fe3O4 mediated by Cyanobacteria showed 80.41% removal efficiency of Pb2+ and 88.35% removal efficiency of Cd2+ after five consecutive adsorption–desorption cycles. The adsorbed metal was successfully eluted by diluted acid and the regenerated sorbent recovered near-performance on each subsequent cycle (Kaleem et al., 2023). On the other hand, leaf-mediated magnetic IONPs with Icacina oliviformis showed more significant loss, where Pb^{2+} removal efficiency decreased from 96.80% to 52.10% and Cd^{2+} removal from 92% to 41.3% over four consecutive cycles. This implies progressive surface fouling or partial capping-layer removal requiring periodic surface re-activation during long-term deployments (Abdul-Gafaru et al.,., 2025).

5.3 Recyclability and Stability Tests

The long-term operational viability of any remediation nanomaterial is contingent upon the retention of removal/degradation capacity over multiple adsorption–desorption cycles and chemical robustness within the working effluent matrix. This provides an economic solution by eliminating the centrifugation/filtration costs that limit the scale-up of traditional sorbents through providing an elegant separation route between treated water, spent adsorbent and desorbing eluent (Osman et al., 2024; Priya et al., 2021).

The durability relevant to the field is confirmed by the availability of multi-cycle stability data for different green IONP systems. Fe3O4 mediated by Cyanobacteria showed 80.41% removal efficiency of Pb2+ and 88.35% removal efficiency of Cd2+ after five consecutive adsorption–desorption cycles. The adsorbed metal was successfully eluted by diluted acid and the regenerated sorbent recovered near-performance on each subsequent cycle (Kaleem et al., 2023). On the other hand, leaf-mediated magnetic IONPs with Icacina oliviformis showed more significant loss, where Pb^{2+} removal efficiency decreased from 96.80% to 52.10% and Cd^{2+}

removal from 92% to 41.3% over four consecutive cycles. This implies progressive surface fouling or partial capping-layer removal requiring periodic surface re-activation during long-term deployments (Abdul-Gafaru et al.,., 2025).In photocatalytic dye-degradation systems, Fe nanoparticles from Hibiscus sabdariffa catalysed the degradation of Congo red, without significant deactivation over the first four reuse cycles, with only a slight decrease on the fifth cycle; suggesting that the biogenic capping layer does not degrade substantially under repeated illumination, and that minimal mechanical attrition occurs during magnetic recovery (Alshehri et al., 2017). Composite systems with Fe nanoparticles anchored on reduced graphene oxide have shown that conductive supports not only stabilise the catalytic phase but also enhance the interfacial electron transfer, with methylene blue degradation efficiency close to 75 % over several reuse cycles (Akhtar et al., 2024). Superparamagnetic character confirmed by vibrating-sample magnetometry (saturation magnetisation in the 10–80 emu g−1 range) ensures that spent particles can be quantitatively recovered from treated water within seconds with a handheld magnet, an operational feature that materially reduces the techno-economic footprint relative to non-magnetic sorbents and photocatalysts (Khadka et al., 2024; Priya et al., 2021).

6. Challenges and Future Perspectives

6.1 Scalability and Commercial Barriers

Despite the exponential increase in research at the laboratory scale on green-synthesised iron oxide nanoparticles, commercial deployment has yet to follow, due to a coherent set of technical, economic and regulatory barriers. Batch-to-batch reproducibility is one of the most important challenges. The composition of phytochemical in plant extracts is dependent on plant species, chosen organ, harvest season, soil chemistry, geographical origin, and extraction protocol, such that measurable differences in particle size, morphology, surface charge, and capping chemistry may be observed between nominally identical syntheses (Akintelu et al., 2021; Haider et al., 2024; Priya et al., 2021). Unlike the carefully controlled chemical co-precipitation or hydrothermal routes, which use analytical grade precursors and well-defined temperature–pH ramps, plant-mediated routes rely on a heterogeneous biological matrix, which makes standardisation and quality assurance difficult at industrial throughput. Another practical challenge is the scale-up of raw material supply; laboratory studies generally use 5–30 g of milled biomass per 100–250 mL of extract, while kilogram production of consistent extract for tonne-scale nanoparticle manufacture strains agricultural supply chains, drying infrastructure, and cold-chain logistics, particularly when seasonal or wild-harvested species are involved (Osman et al., 2024; Priya et al., 2021). The cost of the precursor is also non-trivial, although $FeCl_3 \cdot 6H_2O$ and $FeSO_4 \cdot 7H_2O$ are inexpensive, the energy costs associated with pH adjustment, ageing, washing and magnetic separation steps still increase the unit cost of green IONPs compared to large-scale commodity iron oxide pigments (Akintelu et al., 2021). These are further compounded by the absence of harmonised standardisation protocols, GMP-equivalent quality-control frameworks and regulatory pathways specifically tailored to biosynthesised nanomaterials, which currently prevent many green IONP formulations from entering into mainstream commercialisation (Osman et al., 2024, Tan et al., 2023). Overcoming these barriers will require high-throughput screening of plant matrices, standardised protocols for extract conditioning, validated batch-release criteria and lifecycle-cost analyses comparing green routes to both conventional chemical synthesis as well as

emerging electrochemical and continuous-flow biosynthesis platforms.

6.2 Environmental and Safety Assessment

It is a common assumption in the green-synthesis literature that the use of biological reductants translates directly into environmental and toxicological benignity, but this premise deserves systematic scrutiny. Iron oxide nanoparticles are generally considered as one of the less toxic inorganic phases due to the abundance of iron in nature and the relatively low residual solubility of Fe_3O_4 and γ-Fe_2O_3 in neutral aqueous environments (Priya et al., 2021; Tan et al., 2023). However, the capping layer adds another and less characterised biological element. Phytochemical residues like flavonoids, tannins, terpenoids, alkaloids and residual proteins may be bioactive per se and their persistence on the nanoparticle surface after deployment, their behaviour in soils and sediments and their trophic transfer through aquatic food webs are not fully understood (Akintelu et al., 2021; Osman et al., 2024). Systematic ecotoxicity assay batteries to probe acute and chronic effects on algae, daphnids, fish embryos, soil microbes, and higher plants against green IONPs of defined composition are still relatively sparse, and the existing studies cover only a small fraction of the formulation space defined by plant species, extract dose and synthesis pH (Haider et al., 2024; Tan et al., 2023). Secondary concerns involve accidental releases during manufacturing, unintentional ingestion or inhalation by workers, and photo-induced generation of reactive oxygen species, which are critical for dye remediation but may also lead to collateral oxidative stress on non-target biota upon prolonged environmental exposure (Hassaan et al., 2023; Osman et al., 2024). Moreover, the fate of spent IONPs at the end of their life and their degraded capping residues, e.g. post-adsorption loaded metal ions which are prone to be released in sediments under variable pH/redox conditions, have not been quantitatively tracked in most published studies (Akintelu et al., 2021). Therefore, a prerequisite for safe, large-scale deployment is a thorough life-cycle and ecotoxicological assessment, ideally performed on the most promising formulation candidates using standardised OECD-style protocols.

6.3 Emerging Research Directions

Research frontier of green-synthesized iron oxide nanoparticles is rapidly expanding towards several high-value directions. One major thrust is the rational design of hybrid and composite materials combining the green iron oxide core with high-surface-area supports or co-catalytic phases – notably reduced graphene oxide, carbon nanotubes, TiO_2, ZnO, and conducting polymers – to suppress charge-carrier recombination in photocatalytic dye degradation and broaden absorption into the visible region (Akhtar et al., 2024; Osman et al., 2024). A second emerging direction is the application of computational and machine-learning frameworks, including DFT-based prediction of adsorption energetics and QSAR-style models linking phytochemical descriptors to nanoparticle size and surface chemistry, to de-risk the experimental screening of plant species and accelerate optimisation (Akintelu et al., 2021). Microbial synthesis using extremophilic bacteria, cyanobacteria, marine algae, and fungal consortia is gaining attention as a complementary route that may offer better batch-to-batch reproducibility under controlled fermentation conditions and may produce surface chemistries not accessible through plant extracts (Bolade et al., 2019; Kapoor et al., 2021). The convergence of water-treatment, public-health and resource-recovery objectives is increasingly reported for multifunctional platforms that integrate simultaneous heavy-metal adsorption, dye degradation and antimicrobial activity within a single magnetic-recoverable particle system (Haider et al., 2024; Mohamed et al., 2023).

Lastly, the incorporation of green IONPs into continuous flow reactors, membrane filtration modules and photocatalytic membrane reactors is a hot topic in engineering research to bridge the lab-to-pilot gap and to unlock the techno-economic potential of this otherwise promising class of sustainable nanomaterials (Abdul-Gafaru et al., 2025; Kaleem et al., 2023; Osman et al., 2024).

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