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Nature Environment & Pollution Technology

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Review Paper

A Comprehensive Review on Iron Oxide and Iron oxide-based Nanomaterials for Wastewater Treatment

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Key Words	Iron oxide, Nanoparticles, Wastewater treatment, Nanosorbents, Photocatalysts
DOI	https://doi.org/10.46488/NEPT.2026.v25i02.B4336 (DOI will be active only after the final publication of the paper)
Citation for the Paper	Mankad, S., Chaudhari, S., Ghodmare, S., Mahajan, R., Susladkar, S., Topare, N., Choudhari, V., Khedkar, S. and Thorat, P., 2026. A comprehensive review on iron oxide and iron oxide-based nanomaterials for wastewater treatment. *Nature Environment and Pollution Technology*, 25(2), B4336. https://doi.org/10.46488/NEPT.2026.v25i02.B4336

ABSTRACT

The growing need for water and the increase in the generation of wastewater globally demands efficient water treatment processes. Conventional water remediation efforts shortfall to cater to current water treatment requirements. Nanomaterial water remediation has shown promising results and needs to be explored at a greater scale to address these issues. The most recent focus has been on composite nanomaterials made of iron oxide magnetic and superparamagnetic NPs, which have attracted a lot of interest owing to a number of desirable characteristics, including excellent after-use recovery, targeted quality, and affordability. Iron oxide-based nano-materials have multiple ways for removing organic and inorganic contaminants. In addition, several nanocomposites are employed to improve performance and incorporate novel, advantageous characteristics. Chemical, green, and biological processes are employed in the production of these materials. Synergistic properties for water cleanup have been demonstrated by various iron-based nanocomposites. Various mechanisms of pollution removal such as adsorption, desorption, photocatalysis, and flocculation/coagulation targeted while designing NPs. Types of water pollutants, choice of remediation methods, and various methods required for QC and efficiency of these nanomaterials were reviewed. The review discusses various methods of preparation of magnetic nanoparticles, their composite materials, the mechanism of pollutant removal, and recent applications exploring synergistic behavior for the removal of pollution for efficient water remediation. Issues such as safety toxicity, removal after use, and disposal of these materials were also discussed. The manuscript provides a quick overview of iron oxide nanomaterials as a reference for advancing further studies in the areas. It is planned to make contributions to the production bibliography regarding various aspects of iron-based nanomaterials for water and wastewater treatments.

INTRODUCTION

Water covers more than 70% of the earth's surface, merely 2.5 percent of it can be consumed by humans. Many people do not have access to potable water of the world's population, almost 2 billion people, or 26%, do not have access to clean drinking water. Conversely, two to three billion people globally experience water shortages for at least a month out of the year. Two to three billion people globally experience water shortages over a few weeks out of the year. The number of people experiencing water problems is predicted to rise from approximately 930 million in 2016 to 1.7 to 2.4 billion by 2050, raising concerns about water scarcity. The environment as well as human livelihoods are significantly impacted by the scarcity of water. The depletion of freshwater resources due to pollution is a major contributor to water shortage, a pressing problem on a global scale. When water sources are polluted, they become unusable for human consumption, agricultural irrigation, and even industrial processes. Reuse and treatment of wastewater, as well as other clean water reuse programs, can help lessen the effects of pollution and water scarcity (Bonazzi 2023; Borah and Chetia 2023). Various types of water pollution sources and iron oxide nanomaterials (IONMs) and methodologies used for W&WT are presented in Fig. 1 a and b.

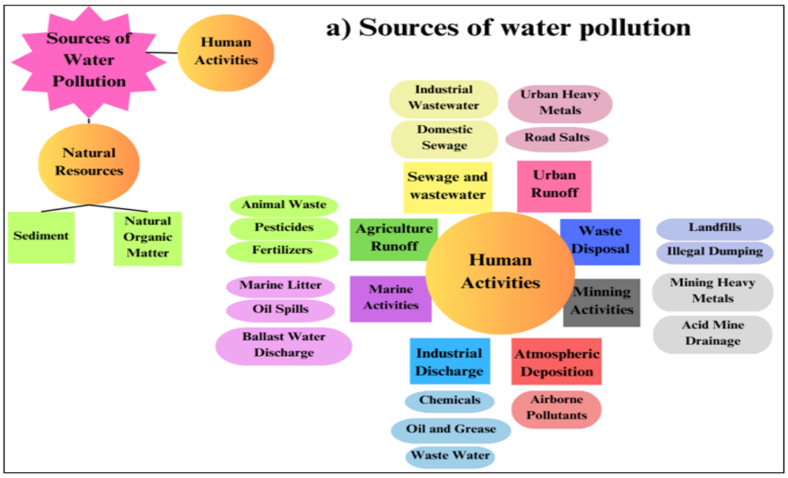

Fig.1 (a): Various types of water pollution sources

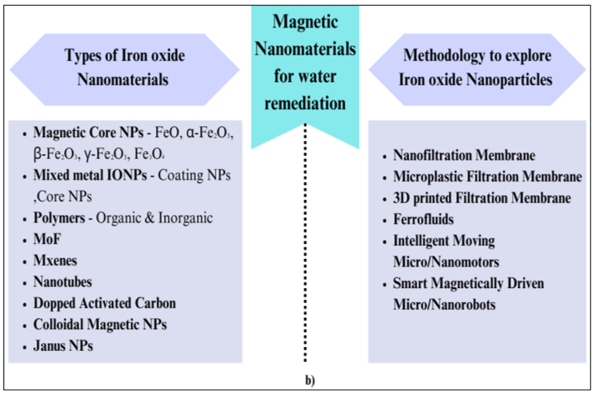

Fig.1 (b): Various types of iron oxide nanomaterials and their methodologies used for W&WT

Traditionally, liquid distillation, anion exchange, ultrafiltration, reverse osmosis, activated carbon adsorption, deionization, and ultraviolet (UV) filtration are typical techniques for treating water and wastewater. These traditional wastewater treatment methods are not only costly but also energy-intensive, consuming approximately 2-3% of the country's energy used in water treatment, particularly in biological wastewater treatment facilities. This has created an urgent need for low-cost, more sustainable, robust, and efficient solutions for wastewater treatment which should involve minimal use of energy resources, and chemicals and have low direct or indirect effects on environmental and human life (Borah and Chetia 2023).

Nanomaterials have shown a great deal of promise in addressing water and problems for wastewater treatment because of their capacity to eliminate a variety of contaminants. Numerous nanomaterials have been studied and found to be useful in removing a range of contaminants from water, such as heavy metals, organic pollutants, inorganic anions, and microorganisms. Photocatalytic degradation and adsorption are the most preferred water and wastewater treatment methods (W&WT) due to their low operating costs, environmentally friendly nature, and efficacy. It was discovered that the most adaptable, durable, simple to prepare, and highly stable NPs investigated for W&WT are metal oxide NPs (MONPs). Iron oxide MNOPs have been studied the most for this purpose and have a wide range of uses in this regard (Campos et al. 2015). Magnetite (Fe_3O_4), hematite (α-Fe_2O_3), β-Fe_2O_3, maghemite (γ-Fe_2O_3), and wustite (FeO) are among the several nanocrystal forms of iron oxide. Hematite (α-Fe_2O_3) is the most durable state of iron oxide under ambient conditions among various crystalline formations.

The wide applications of α-Fe_2O_3 are derived from their superior physical and chemical properties, further, it can be tailored to various morphologies, particle dimensions, and heterostructures (Santhosh et al. 2019a). The nanomaterials based on Fe_2O_3 include nanoparticles, nanotubes, nanosponges, nanocomposites with organic and inorganic materials, MXenes etc, and metal oxide frameworks (MOFs). These nanomaterials work by the mechanisms of adsorption, photocatalysis, coagulation, flocculation, etc. These are fabricated using various chemical, hydrothermal, sol-gel methods, etc. methods to improve their properties (Tao et al. 2021). Iron oxide nanoparticles explored as a basis for Magnetic Iron-based Nanomaterials (MINMs) are summarised in Table 1.

Table 1: Most used IONPs, their properties and applications. (Jojoa-Sierra et al. 2022; Jjagwe et al. 2023; Keshta et al. 2024; Muthukumar et al. 2024; Lin et al. 2018; Mohamed et al. 2017; Razzouki et al. 2015)

Chemical formula	**Common name**	**Color**	**Crystal system**	**Type of magnetism**	**Water remediation application of magnetic iron-based Nanomaterials (MINMs)**
α- Fe_2O_3	Hematite	Red	Rhombohedral hexagonal	Weakly ferromagnetic	(i) Adsorptive removal of organic and inorganic pollutants, microorganisms, oils, etc. (ii) Catalytic degradation of organic pollutants (dyes, chemicals, pharmaceuticals, oil spills, etc) and anti-biofouling effect. (iii) Coagulation /flocculation-based removal of microbes, oil droplets, etc. (iv) Recovery and biodegradation of crude oils. (v) Pollutant separation by membrane filtration process assisted by MINMs.
γ- Fe_2O_3	Maghemite	Red-brown	Cubic or tetragonal	Ferrimagnetic	
Fe_3O_4	Magnetite	Black	Cubic or tetragonal	Ferrimagnetic	
FeO	Ironmonoxide, wustite	Back	Black pigment	Weakly ferromagnetic	
FeOOH	Iron oxy-hydroxide	Yellow	Hexagonal, orthorhombic, and cubic	Weakly ferromagnetic or superparamagnetic	
α-FeOOH	Goethite	Yellow	Orthorhombic	Ferrimagnetic	
$Fe(OH)_3$	Iron(III) hydroxide	Reddish-brown	Different crystalline structures	Paramagnetism	

The unique characteristics of nanoparticles influence their selection, and the current state of nanoparticle research takes into account all of their potential uses. By filling a gap in our understanding and pushing the boundaries of W&WR, this work is innovative because it uses a novel strategy to precisely regulate nanoparticles. The study also summarizes the applications of MINMs in contemporary nanotechnology and delves deeper into the knowledge gap by examining current trends in nano-synthesis. Furthermore, the restrictions on the use of iron-based nanoparticles for the elimination of contaminants are brought to light. The study on the use of nanotechnology in water purification is thoroughly examined in this review, with a focus on iron oxide nanoparticles. Despite this, owing to their desirable qualities, iron-based nanoparticles require extensive research and evaluation. In this review, we take a look at iron-based nanoparticles and assess their key features, manufacturing methods, and ability to remove various contaminants from wastewater. In this era of prioritizing sustainable development and environmental protection, the application of nanomaterials will be crucial to ensuring a safer, more sustainable, and cleaner future in industrial wastewater treatment.

2. METHODS OF SYNTHESIS OF IRON OXIDE-BASED NANOMATERIALS

High-quality iron oxide nanoparticles can be synthesized using various techniques, such as co-precipitation, hydrothermal synthesis, microemulsion, sonochemical synthesis, thermal decomposition, and many more

(Teja and Koh 2009). Considering modern scientific advancements selection of the suitable method and relevant process parameters for getting IONPs of desired characteristics viz. crystallinity, size, morphology, colloidal stability, polydispersity, porosity, zeta potential, and morphology is possible. IONP qualities are significantly impacted by the selected method of preparation and the reaction parameters. The three main categories of IONP preparation techniques are physical, chemical, and biological (Fig. 2). Methods of preparation of Iron oxide nanomaterials (IONM) and their merits and demerits are summarized in Table 2.

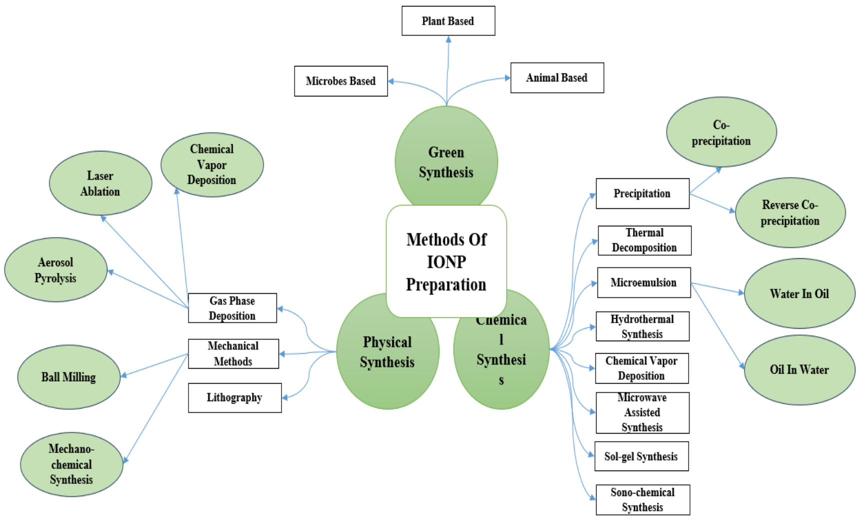

Fig. 2: Methods of Iron Oxide Nanoparticle Synthesis

Table 2: Methods of preparation of Iron oxide nanomaterials (IONM) and their merits and demerits

Preparation Method	General properties of prepared IONMs	Iron oxide nanomaterials (IONM) preparation method	
		Merits	**Demerits**
Co-precipitation	Size- 3 to 100nm; Shape- Spherical	• High yield of IONPS • Approved by FDA as a contrast agent for MRI • Homogenous distribution	• Difficult to control size of NPs due to agglomeration • pH has to be maintained high
Reverse co-precipitation	Size- <100nm; Shape- Spherical	• Control over particle properties • Precipitation occurs in nano-drops	• Agglomeration • Environmental concerns
Microemulsion	Size – 4 to 50 nm; Shape- Spherical and Cubic	• The initial reagent conc. and drop size can be utilized to control the IONPS size. • Nanoparticles have homogenous shapes and sizes	• Low yields obtained in comparison with co precipitation • Purification Is complicated.
Sol-Gel method	Size- Less than 50 nm; Shape: Various Shapes	• High purity of nanoparticles • Scalability	• Time consuming • Residual solvents • Crack formation • High permeability

Sono-chemical method	Size- 20 to 80 nm; Shape- Variable	• Fast reaction rate • Reduced solvents • Simple set up	• Scale-up challenges • Heat generation
Microwave-assisted synthesis	Size- 4 to 50 nm; Shape- Spherical	• Control over properties such as temperature, and irradiation time which can influence size and morphology • Rapid and uniform particles having high magnetic saturation	• Chances of non-uniform heating • Limited reaction monitoring
Green Synthesis	Size- 10 to 50 nm; Shape- Spherical	• Ecofriendly • Nontoxic safer end products • Cost-effective good scalability	• Standardization inconsistency • Lower yield and purity • Unknown mechanism
Hydrothermal	Size – 2 to 1000 nm; Shape- Various shapes	• Good crystallinity • Controlled agglomeration • Medium to high yield	• Scale-up challenges • Equipment maintenance

2.1. Co-precipitation and reverse co-precipitation

These are among the most widely utilized methods of synthesis for IONPs. Usually, it is performed by using $FeCl_2$ and $FeCl_3$ along with a base such as NaOH or KOH, ammonium hydroxide, etc. at a higher temperature. Here, the oxide is formed by the dehydration of hydroxides which later form a precipitate (Rangarajan et al. 2014). Ammonium hydroxide, deionized water, and hydrated ferric chloride are the chemical reagents used in co-precipitation to create iron oxide nanoparticles. The process of ionizing the chemical reagents at different temperatures while vigorously churning them in the presence of nitrogen gas yields magnetite crystals. Therefore, the co-precipitation method's simplicity, quick synthesis at low temperatures, and energy efficiency make it superior to alternative biochemical methods for synthesizing iron oxide nanoparticles.

2.2. Reverse co-precipitation technique

Making use of an active reduction reagent as a stabilizer, (Estelrich et al. 2015) effectively carried out a reverse chemical co-precipitation synthesis on magnetite (Fe_3O_4) NPs. The characteristics of the uncoated and dimethyl sulfoxide (DMSO)-coated Fe_3O_4 and Fe_3O_4 NPs were contrasted. XRD and SEM were used to examine the microstructural and mineralogical characteristics of both samples. The findings showed that DMSO-coated sample particles outperformed untreated Fe_3O_4 nanoparticles in Fourier analysis and SEM inspection. When using base materials with a varied range of reactant rates, reverse co-precipitation and co-precipitation procedures have consistency problems, require a long time, and have the capability to introduce foreign particles into the metal matrix structure of the IONPs result. When using base materials with different reactant rates, co-precipitation and reverse co-precipitation techniques have challenges with repeatability and can introduce foreign particles into the metal matrix structure of the iron oxide nanoparticle result (Ogbezode et al. 2023).

2.3. Microemulsion

This procedure uses a surfactant, a polar phase, and a non-polar phase. are used to prepare a microemulsion. The polar phase consists of metal salts whereas the non-polar phase consists of hydrocarbons. The growth of NPs can be well regulated by this method. Microemulsions can be of mainly two types i.e. oil in water and water in oil (Darbandi et al. 2012) (Table 2).

2.4. Biological / Green Synthesis

Due to chemical methods being thought to be as hazardous and energy-intensive. There has been an increase in the demand to create nanoparticles with an eco-friendly method. Here, plants or microbes could be used to synthesize nanoparticles along with the precursor of the metal. Karpagavinayagam and Vedhi 2019 used Avicinnia marina flowers for the synthesis of IONPs. $FeCl_3$ was added to the flower extract and then centrifuged to obtain the nanoparticles. The particle size was measured to be around 45 nm. Baaziz et al. 2014 used a biochemical strategy to develop iron oxide nanoparticles in coated sand; the study's goal was to solve water treatment-related difficulties by biochemically producing and characterizing iron oxide nanoparticles from coated sands. (El-Sheekh et al. 2021a) used extract of different algae to biosynthesize IONPs using $FeCl_3$ as an iron precursor. The size range of these IONPs prepared from different extracts was found to be between 5 to 35 nm.

2.5. Microwave-assisted synthesis

Aivazoglou et al. (2018) performed IONP synthesis using microwave radiation, where the metal precursors are heated along with a base that produces metal oxides such as NaOH or NH_4OH. This method produces a high concentration of nanoparticles at a faster rate.

2.6. Heat treatment/thermal decomposition technique

In general, iron oxides can be heated to temperatures higher than 250°C, and nanomaterials can be produced using the thermal breakdown heat treatment approach (Ogbezode et al. 2023).

2.7. Lithographic technique

The lithographic process is a physical technique employed for nanoparticle production, known for being both costly and energy-intensive. Despite these challenges, it is widely used to create nanomaterials for various applications in electronic devices and computer accessories (Seyedi et al. 2015). As a top-down approach, lithographic synthesis is suitable for generating micro- and nanoparticles. Various methods including fusion-ion, nano-imprint, dip-print, electron beam lithography, and photolithography are used in this process. These techniques have broad applications, notably in additive manufacturing and the semiconductor industry (Kayani et al. 2014).

2.8. Large-Scale Synthesis of IONP-based nanomaterials

The key problems in large-scale Fe_3O_4 nanoparticle production can be stated as follows: The degree of uniformity and repeatability between batches, expenses, efficiency, environmental concerns, and sorption capability. To address the problem of reproducibility across batches and determine the ideal conditions for the effective removal of pollutants particle size distribution and purity must be closely tracked, and reaction parameters such as pH, temperature, and concentrations of reactants must be precisely controlled. However, there are major obstacles to the large-scale manufacturing of these nanoparticles, which limits their economic viability and real-time applications. On the other hand, typical materials for Fe_3O_4 production might be expensive and inefficient for large-scale applications. In addition, several synthesis processes utilized to make high-quality magnetite nanoparticles are expensive and time-consuming. For example, utilizing surfactants, organic solvents, and other additions to promote uniform particle formation and reduce agglomeration has been shown in numerous studies to significantly increase the total cost of synthesis. To address this issue, green synthesis is a workable method for creating Fe_3O_4 nanoparticles from sustainable resources (Keshta et al. 2024a).

3. MAGNETIC IRON OXIDE NANOCOMPOSITES MATERIALS

Functionalizing iron oxide nanoparticles (IONPs) is vital to improving their applicability with wastewater treatment because of innate difficulties like rapid aggregation, poor dispersibility in water, and limited pollutant selectivity. Functionalization addresses these issues by modifying nanoparticle surfaces with chemical groups, polymers, and coatings. Chemical groups like EDTA, GSH, and PAA, and polymers such as chitosan and Poly(N-isopropyl acrylamide) (PNIPAM) enhance stability, dispersibility, and surface properties, while coatings like silica and graphene stabilize IONPs, prevent aggregation, and improve catalytic and adsorption capabilities in both homogeneous and heterogeneous systems. Surface functionalization of IONP and its composite materials is presented in Fig. 3a and GSH functionalization in Fig. 3b. Functionalized IONPs exhibit improved catalytic activity, effectively degrading pollutants and enhancing wastewater treatment efficiency. Functionalization offers tailored solutions to address the challenges associated with unmodified IONPs, making them indispensable tools in modern environmental remediation efforts. Surface functionalization can be achieved by in situ process and by post-synthesis functionalization of IONPs. Some frequently utilised magnetic sources are given in Table 3.

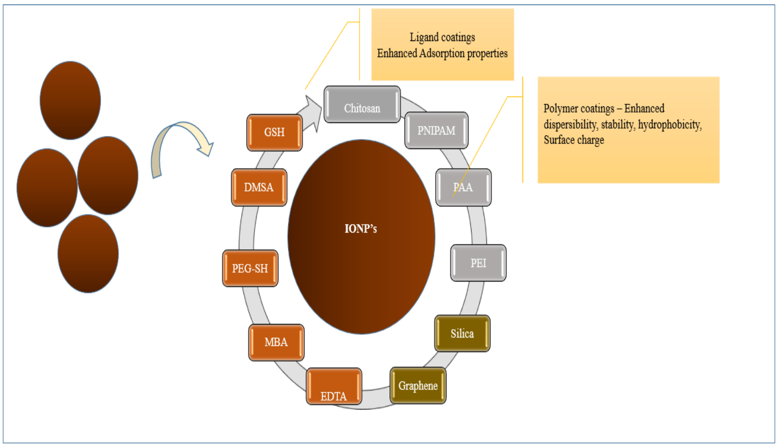

Fig 3 (a). Surface functionalization: Various surface functionalization agents used for IONP

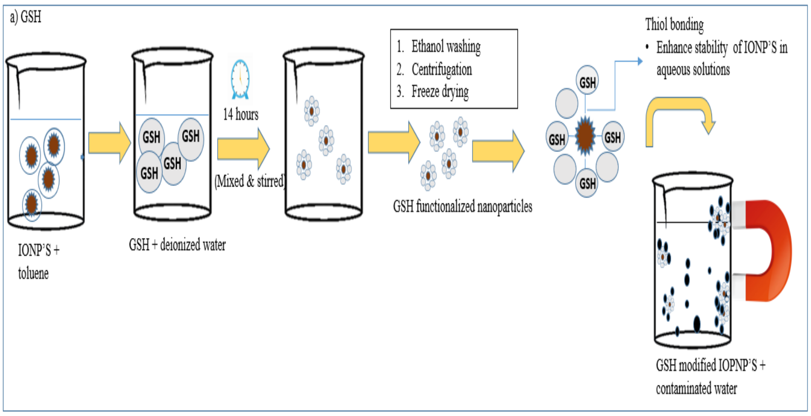

Fig 3 (b). Surface functionalization: Schematic of IONP functionalization with GSH

In situ surface functionalization of Fe_3O_4: It involves direct functionalization of groups during the synthesis. Depending on the intended use, this process can alter their chemical, physical, or biological characteristics. This contributes to advancement and broadens the applications of nanoparticles (Popescu et al. 2019). De Tercero et al. 2013a and Popescu et al. 2019 demonstrated in situ functionalization of IONP, where functionalization of alkyne groups was done in IONPs by a continuous process called click reactions. The process imparted the nanoparticles an inorganic core and an organic shell thus widening the doors for its applications in various fields. Karimzadeh et al. 2017 performed synthesis and surface coating of superparamagnetic nanoparticles with polyethyleneimine (PEI) and polyethylene glycol (PEG). The surface coating was done using the Cathodic Electrochemical deposition (CED) process. It was found that the surface coating enhanced the physicochemical properties as well as magnetic properties essential for biomedical applications.

Post-synthesis surface functionalization of Fe_3O_4: The post-synthesis surface functionalization is a process to modify already synthesized IONPs with different functional groups, chemicals, or coats. This will help to customize and impart a desired property to the nanoparticles thus increasing the range of its applications. In the process of post-synthesis functionalization, mesoporous guanidine functionalized Santa Barbara Amorphous-15 (SBA-15)/Fe_3O_4 i. e. (Fe_3O_4@SBA-15-Gd) was designed. In situ magnetization by Fe_3O_4 nanoparticles was achieved (Fe_3O_4@SBA-15). Composites were then modified by 3-aminopropyltriethoxysilane (APTES) to get Fe_3O_4@SBA-15-NH_2. This was followed by nucleophilic addition to cyanimide to get magnetic nano adsorbent NPs (Fe_3O_4@SBA-15-Gd) and used successfully for decontamination of aqueous solutions from Pb(II) and Cu(II) (Hassanzadeh-Afruzi et al. 2023; Rosaline et al. 2024).

Table 3: Some frequently utilized magnetic sources

Source	Iron oxide (type)	Particle size	Solvent/Temperature (°C) /Time (min.)	Value for magnetic saturation (emu/g)	References
$FeCl_3$, $FeSO_47H_2O$	Magnetite	13 nm	Water/80/20	74	(De Mello et al., 2019)
$FeSO_{47}H_2O$, $FeCl_{36}H_2O$	Magnetite	10-50 nm	Water/65/15	-	(Klencsar et al., 2019)
$Fe(NO_3)_{39}H_2O$	Hematite	24 nm	Ethanol/79/60	0.71	(Nguyen et al., 2020)
$FeCl_{36}H_2O$	Ferrite	20-40 nm	Water/--/60	148	(Peng et al., 2017)
$FeCl_3$, $FeSO_{47}H_2O$	Magnetite	25 nm	Water/40/120	14.46	(Sundararaman, et al., 2020)
$FeCl_3$	Magnetite	5 nm	Ethylene glycol/100/30	49.7	(Radon et al., 2020)
$FeCl_{36}H_2O$, $FeCl_{24}H_2O$	Magnetite	12.5 nm	Water/70/40	69.2	(Li et al., 2017)
$Fe_2(SO4)_{35}$ H_2O/ $FeSO_{47}H_2O$	Magnetite	50-75 nm	Water/70/30	10.40	(Andrade Neto et al., 2020)
$Fe_3(CO)_{12}$	Magnetite	3 nm	Water/450/40	-	(Vangelista et al., 2012)

3.1. Types of Functionalization

Functionalization is achieved by various chemical groups and ligands, polymers and inorganic materials. Conversely, IONPs can be doped on other materials such as activated carbon for improved performance.

3.1.1. Chemical Groups and Ligands

EDTA: Ethylene diamine tetra acetic acid (EDTA) functionalized iron oxide nanoparticles represent a significant advancement in wastewater treatment technologies, offering enhanced performance, ease of recovery, and sustainability, particularly in heavy metal ion removal (Kobylinska et al. 2020a; Ramos-Guivar et al. 2021; Fumis et al. 2022). EDTA modification enhances the nanoparticles adsorption properties by introducing additional chelating sites, thereby increasing affinity and selectivity regarding metals like Cr (III), Zn (II), Pb (II), Cu (II), and Cd (II), and boosting overall adsorption capacities. These nanoparticles exhibit rapid kinetics, efficiently removing contaminants from water within short contact times (Kobylinska et al. 2020a). EDTA-functionalized iron oxide nanoparticles have demonstrated robust stability due to a protective silica shell, enhancing thermal stability against oxidation processes that could compromise their effectiveness in water treatment applications (Fumis et al. 2022). This stability ensures prolonged performance and reliability during multiple adsorption-desorption cycles, making them economically viable and sustainable options for environmental remediation (Ramos-Guivar et al. 2021; Fumis et al. 2022).

These nanoparticles' intrinsic magnetic qualities make it simple to separate them from the water after adsorption using external magnetic fields, streamlining the recovery procedure and permitting repeated use without noticeably reducing their effectiveness. Experimental information from numerous studies has validated the efficiency of EDTA-functionalized IONPs in real-world scenarios, showcasing high removal efficiencies and accurate recoveries across various water samples. Advanced kinetic and adsorption isotherm models further elucidate the mechanisms behind their adsorption behavior, highlighting chemisorption as the predominant mechanism due to strong EDTA-metal ion interactions (Kobylinska et al. 2020a; Ramos-Guivar et al. 2021) underscoring their potential as effective Nano adsorbents for addressing water pollution challenges posed by heavy metal ions, supporting their integration into practical environmental remediation strategies (Ramos-Guivar et al. 2021).

Glutathione (GSH): The integration of magnetic separation with GSH-functionalized iron oxide nanoparticles provides significant advantages for water treatment applications. Superparamagnetic iron oxide nanoparticles (SPIONs) are first synthesized and coated with oleic acid in toluene for stability, GSH is dissolved in water separately, and Mixing SPIONs with the GSH solution displaces oleic acid via coordination bonds during stirring. Centrifugation is used to separate the GSH-coated SPIONs, after which excess GSH is removed using an ethanol wash and the GSH is freeze-dried into a powder for use in applications. By means of various mechanisms such as electrostatic attraction, hydrogen bonding, and π-interactions between GSH moieties and the contaminants, which promote their adsorption onto the nanoparticle surface, GSH functionalized nanoparticles eliminate pollutants from water, functionalisation of IONPs with GSH is presented in Fig. 3b.

The magnetic and adsorption properties of GSH-functionalized nanoparticles make them particularly effective for removing contaminants from water, making them efficient and cost-effective alternatives to conventional methods (Tariq et al. 2022). Additionally, the study Behera et al. 2022 demonstrated the enhanced sensitivity and stability of GSH-functionalized nanocomposites in detecting nitrophenol isomers. Characterization techniques confirmed the formation of structured α- Fe_2O_3 and underscored the nanocomposites' electrocatalytic activity, conductivity, and wide detection range. These findings highlight the pivotal role of GSH in augmenting sensor performance by facilitating improved electron transfer kinetics and providing a larger surface area for enhanced interaction with analytes signifying the benefits of GSH functionalization in enhancing the capabilities of iron oxide nanoparticles for wastewater treatment applications, particularly in improving sensing efficiency and overall effectiveness in environmental remediation efforts (Behera et al. 2022).

3.1.2. Polymer Coatings

In general, polymer Coatings of NPs offer various advantages such as improved W&WT efficacy, enhanced stability, and increasing the porosity and surface area of the IONPs.

Chitosan: Chitosan-coated nanoparticles improve wastewater treatment efficacy by enhancing the stability and increasing the porosity and surface area of the IONP. They are effective in the photocatalytic removal of pollutants, including dyes and mercury, from aquatic environments due to their high water permeability and antibacterial properties. Additionally, the ease of chemical modification and robust nature of chitosan enhance the stability and efficiency of these nanoparticles in wastewater treatment applications (Ismail et al. 2023). These properties facilitate strong electrostatic interactions with anionic dyes such as Evans Blue (EB) and Acid Yellow 25, resulting in high-capacity adsorption even under continuous flow conditions, as demonstrated in chromatography setups (Ismail et al. 2023). FeO_4 nanoparticles' magnetic characteristics make it simple to separate them from treated water using an external magnetic field, which makes the procedure effective and simple. Moreover, the biocompatibility and biodegradability of chitosan make it an environmentally sustainable choice for such applications, offering a promising, eco-friendly solution for removing pollutants from wastewater (Lee et al. 2019; Rehman et al. 2024).

The preparation methods for these functionalized nanoparticles typically involve chitosan dissolving in acetic acid, combined with iron oxide nanoparticles, stirred for an extended period, and then magnetically collected, washed, and dried (Gómez Pérez et al. 2020). These coatings significantly enhance the ability of nanoparticles to adsorb heavy metal ions as a result of chitosan's chemical and biological characteristics. Modifications of chitosan, such as introducing functional groups, further improve its adsorption capacity and selectivity for metal ions, making it highly effective for removing toxic substances like Cr (VI) from aqueous solutions (Khalil et al. 2020).

PNIPAM: PNIPAm is a thermoresponsive polymer known for its phase transition behavior triggered by temperature changes, shifting from a hydrophilic to a hydrophobic state around 32°C. This characteristic phase transition, where polymer chains collapse due to altered hydrogen bonding with water molecules within a cross-linked network, makes Poly-N-isopropyl acrylamide highly advantageous in various applications, including wastewater treatment. Its ability to undergo substantial volume phase transitions (VPTs) in response to minor temperature adjustments allows for precise control over the adsorption and removal of organic compounds from aqueous environments (Lu et al. 2016). PNIPAm-functionalized nanoparticles exhibit enhanced performance in treating emulsified oily wastewater by leveraging their thermoresponsive nature. This property enables the nanoparticles to adjust their hydrophobicity based on temperature variations, thereby optimizing the adsorption and separation processes for contaminants (Tao et al. 2020). By utilizing PNIPAm-grafted iron oxide nanoparticles, wastewater treatment systems can achieve improved efficiency and efficacy in pollutant removal, highlighting PNIPAm's potential as a versatile and effective material in environmental remediation applications. In wastewater treatment strategies, PNIPAm has been effectively utilized to functionalize iron oxide nanoparticles (MIO@SiO_2-PNIPAM) through a "grafting through" approach involving MPS-modified MNPs and NIPAM monomers (Tao et al. 2020).

PAA: Polyacrylic acid (PAA) considerably raises iron oxide nanoparticles' (IONPs') efficacy in wastewater treatment especially in the elimination of ions from heavy metals like Pb (II). Through surface adsorption and ligand exchange methods (Fig. 4a). Magnetic chitosan (MCS–PAA) nanoparticles were prepared by first activating polyacrylic acid (PAA) carboxyl groups with EDC and NHS in water, followed by conjugation with magnetic chitosan (MCS) through amide bond formation over 24 hours. After isolating the resultant nanoparticles with a magnet, washing them with deionized water, and drying them, a stable MCS–PAA composite appropriate for use in wastewater treatment applications was produced. (Fig. 4b), PAA functionalization effectively boosts the adsorption capacity of IONPs by chelating pollutants via its abundant carboxyl groups. This modification enhances the stability and dispersibility of IONPs in aqueous environments, ensuring their robust performance under acidic conditions typical of wastewater. Covalently bonding PAA to magnetic chitosan surfaces further enhances the composite material's durability and acid resistance, crucial for sustained pollutant removal processes (Mdlovu et al. 2020; Nordin et al. 2021). The successful application of PAA-functionalized IONPs demonstrates high adsorption capacities, efficient kinetics, and favorable isotherm models, highlighting their potential as advanced materials in wastewater treatment technologies.

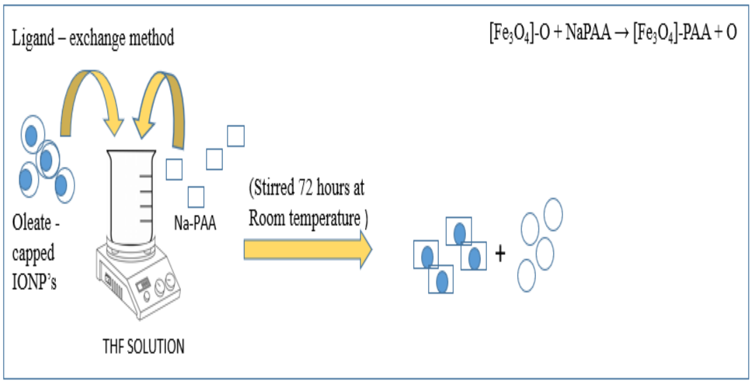

Fig. 4 (a). Polyacrylic acid (PAA) functionalization: Ligand exchange PAA activation method

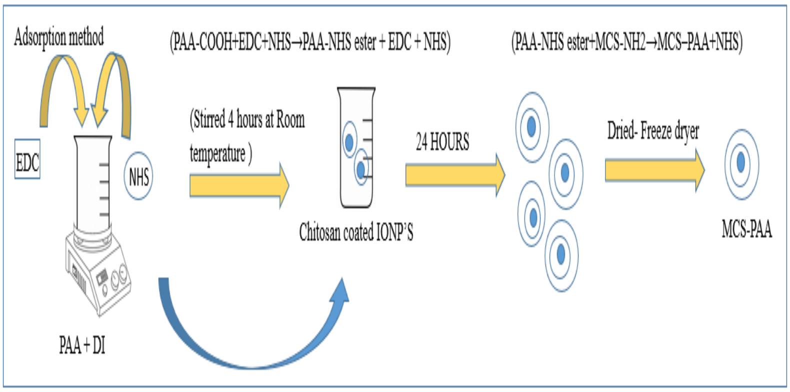

Fig. 4 (b). Polyacrylic acid (PAA) functionalization: Absorption PAA activation method

Moreover, PAA's compatibility with chitosan enhances the overall environmental applicability of these nanocomposites, supporting their role in sustainable water purification processes. This synergistic approach not only improves the performance and stability of IONPs as well as ensures their biocompatibility and recyclability, making them suitable for long-term use in treating contaminated water sources. By leveraging PAA's properties through methods like in situ synthesis and layer-by-layer assembly, researchers can tailor IONPs for specific pollutant removal applications, further advancing the field of wastewater treatment (Shair et al. 2021). Overall, PAA-functionalized IONPs represent a promising advancement in environmental remediation, offering efficient, reliable, and environmentally friendly solutions for addressing water pollution challenges globally.

PEI: Polyethyleneimine (PEI) functionalization has significantly enhanced the efficiency of adsorbents in wastewater treatment, improving their adsorption capacity, stability, and recyclability (Shair et al. 2021; Yan et al. 2023). PEI-functionalized magnetic nanoparticles (MNPs) have proven effective in removing heavy metals like Pb (II) under various conditions, with adsorption behavior fitting pseudo-second-order kinetics and Langmuir isotherm models (Shair et al. 2021). This indicates strong monolayer adsorption, and these nanoparticles maintain their performance after multiple reuse cycles, highlighting their durability and cost-effectiveness. In dye removal, PEI-functionalized cellulose with magnetic nanoparticles (MCPEI) shows high adsorption capacity for anionic dyes, offering a simpler and more efficient alternative to traditional activated carbon (Yan et al. 2023).

PEI-coated zero-valent iron nanoparticles are highly effective for treating hexavalent chromium (Cr(VI)) contaminated water, reducing Cr(VI) to Cr(III), which is less toxic efficiently while maintaining structural integrity. This makes them suitable for in-situ remediation of Cr-contaminated groundwater (Mardikar et al. 2021). PEI functionalization significantly enhances the adsorption properties of various nanoparticles, making them versatile and practical for diverse wastewater treatment applications. These advancements suggest that PEI-functionalized materials can play a vital function in improving the effectiveness and sustainability of wastewater treatment processes. PEI-functionalized cellulose with magnetic nanoparticles (MCPEI) exhibits high adsorption capacity for anionic dyes, presenting a simpler and more effective alternative to traditional activated carbon (Sachan et al. 2021). PEI-coated Fe_3O_4 with β-cyclodextrin (Fe_3O_4@PEI@β-CD) demonstrates high efficiency in demulsifying oily wastewater, achieving over 95% separation under various conditions and retaining its performance after multiple recycling cycles. These advancements indicate that PEI-functionalized materials are versatile, practical, and environmentally friendly solutions for diverse wastewater treatment applications, enhancing both efficiency and sustainability (Jain et al. 2018a; Seth et al 2019).

3.1.3. Surface Coating of IONPs with Inorganic Materials

IONPs are coated with inorganic materials like graphene and silica coatings, metal oxides, metal sulfides, clay nanocomposites, etc. These composite materials exhibit enhanced performance in W&WT by various mechanisms such as enhanced photoactivity under visible light, increased stability, dispersibility, and functionality.

Graphene: Graphene oxide (GO) has attracted noteworthy interest owing to its distinct characteristics, making it an ideal candidate for various applications, including wastewater treatment. When GO is combined with iron oxide nanoparticles, the resulting composite material exhibits enhanced performance in contaminant removal from water. The oxygen-containing functional groups present in GO, such as epoxy, hydroxyl, carboxyl, and carbonyl groups, facilitate the attachment of metal oxide nanomaterials, improving the adsorptive and photocatalytic properties of the composite (Maji et al. 2018). This functionalization also enhances the stability and dispersion of iron oxide nanoparticles, preventing agglomeration and increasing the surface area available for interaction with pollutants (Kumar et al. 2019). Additionally, the GO coating shifts the absorption threshold to the visible region, enhancing photoactivity and enabling effective photocatalytic processes under visible light (Wang et al. 2019). This modification not only improves the thermal and chemical stability of the composite but also enhances its reusability and longevity, making it a robust and efficient solution for industrial wastewater treatment (Khan et al. 2016). The synergistic effects of GO and iron oxide nanoparticles address the need for high-performance, cost-effective, and sustainable treatment technologies (Weng et al. 2018). These advancements underscore the potential of GO-based composites in revolutionizing wastewater treatment applications (Lingamdinne et al. 2019).

Silica: Functionalizing IONPs with silica significantly enhances their stability, dispersibility, and functionality for various applications, including catalysis and environmental remediation. The primary approaches to achieve this include the hydrolysis and condensation of silica precursors in sol-gel processes., and Stöber synthesis, which produces uniform silica coatings via controlled hydrolysis in an alcohol-water mixture. Additionally, surface modification techniques, such as salinization, attach silane coupling agents to the nanoparticle surfaces, creating a silica layer. These methods ensure a robust and uniform silica coating, improving the nanoparticles' resistance to aggregation, chemical stability, and compatibility with other functional groups for targeted applications. Andolsi et al. 2023 prepared and characterized surface-coated Fe_3O_4 nanocomposite and explored for crystal violet (CV) dye removal. In this work separately prepared Fe_3O_4 and SiO_2 NPs were prepared and finally, magnetite coating of silica Fe_3O_4 /SiO_2 was performed. Process optimization experimented for the removal of CV form solution; effects of solution pH, conc. of adsorbent, exposure time, and ionic strength were evaluated.

Metal nanoparticle coating: Fe_3O_4 surfaces are coated with metals to create an inert and protective layer. Fe_3O_4 surfaces as such are less amenable to functionalization compared to metal-coated surfaces. Gold nanoparticles (Au NPs) are commonly used to coat Fe_3O_4 due to their strong interaction with sulfur-containing compounds. One method involves reducing $HAuCl_4$ onto Fe_3O_4 nanoparticles, as reported. Xia et al. 2011prepared a Fe_3O_4@C@Ag composite for use in medication delivery, MRI applications, and antibacterial therapies.

Metal oxides/sulfides: Coating Fe_3O_4 with metal oxides or sulfides confers special chemical and physical characteristics. Fe_3O_4@ZnO nanoparticles were produced by Hong et al. 2008. via direct precipitation of zinc acetate and ammonium carbonate. These functionalized nanoparticles showed improved antioxidant properties compared to bare Fe_3O_4.

Magnetic iron oxide-clay nanocomposites (MICNCs): Mohammed and Samaka 2018 coated natural bentonite (NB) with Fe_3O_4 NPs to get coating (CB) and used for sequestering Cu(II) ions from the polluted solutions. The study revealed a substantial improvement in CB such as surface area, surface morphology, and mesoporous structure. A comparison analysis suggests CB as an excellent material for Cu(II) removal and practical applications for Cu(II) removal from contaminated wastewater

3.2. Activated Carbon Doped with IONPs

The production of iron oxide nanoparticles (Fe_3O_4) and their composite with activated carbon (Fe_3O_4/AC) was investigated by Jain et al. to remove harmful metal ions from water, including chromium (Cr(VI)), copper (Cu(II)), and cadmium (Cd(II)). The nanoparticles were synthesized using a straightforward chemical process and characterized using various techniques to examine their structure and properties. The study identified optimal conditions for removing these metals, noting that an acidic environment (pH 2) was ideal for chromium, while a neutral pH (6) was most effective for copper and cadmium. The Fe_3O_4/AC composite demonstrated particularly high efficiency, especially in removing chromium and copper, with the adsorption data fitting well with predictive models. The study also found that the adsorption process was more effective at higher temperatures, and the nanoparticles could be regenerated and reused multiple times after treatment with acid, highlighting their potential for water purification (Jain et al. 2018a).

4. USEFUL PROPERTIES OF IRON OXIDE NANOMATERIALS, MAGNETIC PROPERTY MEASUREMENT, AND MAGNETIC PROPERTY FACTORS

4.1. Useful Properties of Iron Oxide Nanomaterials

The properties of IONPs encompass a wide range, including magnetic properties (Vakili-Ghartavol et al. 2020a; Arumugam et al. 2021; Keshta et al. 2024b; Atabaki 2024), high surface area, surface chemistry, size, and shape (Baalousha et al. 2008; Lassoued et al. 2017a; Nguyen et al. 2020; Niraula et al. 2021a; Vo et al. 2024), biocompatibility(Kharey et al. 2023a), optical properties (Salakhitdinova et al. 2024), catalytic activity (Bouazizi et al. 2019; Rawat et al. 2021; Ammar et al. 2021), and photoelectrochemical properties (Ben Mammar and Hamadou 2023; Lys et al. 2024). The properties of the iron oxide nanomaterials are summarized in Fig. 5a and the equipment used for the measurement of magnetic properties is presented in Fig. 5b.

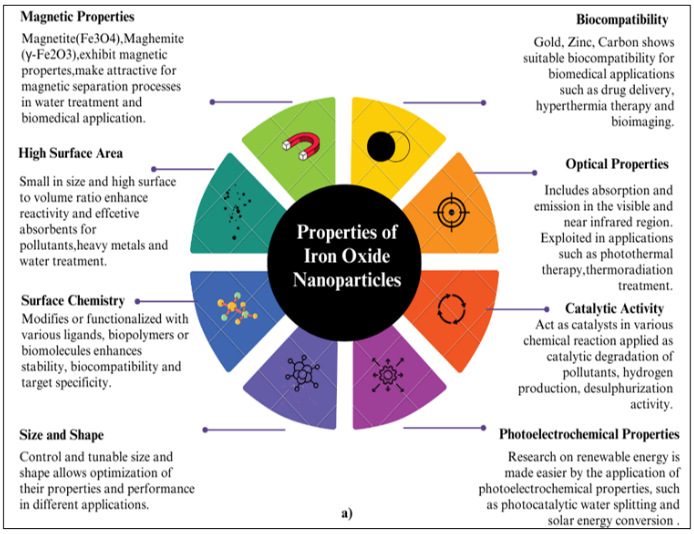

Fig. 5 (a). Useful properties of Iron oxide nanomaterials

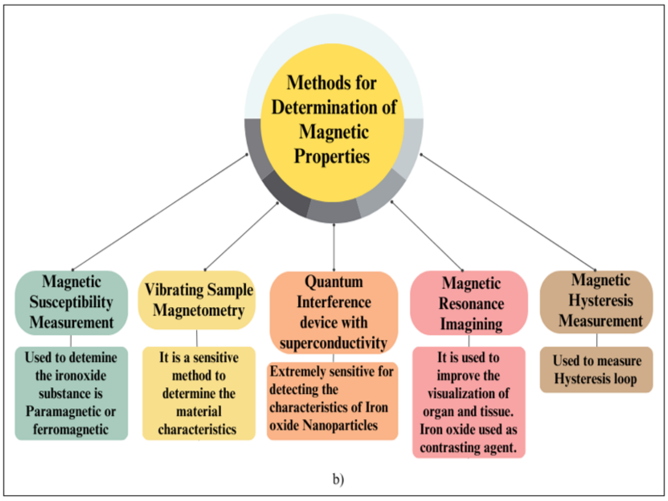

Fig. 5 (b). Methods for determination of their magnetic properties

Magnetic coercivity indicates the ability of the materials to resist changes in magnetization. It also indicates how the materials withstand external magnetic fields. This is the property used to classify material as hard, semi-hard, and soft. High saturation magnetization values enable quick response to external magnetic fields, facilitating fast separation from aqueous solutions containing heavy metals (Jain et al. 2018). Superparamagnetic materials align with an applied magnetic field but become randomized when the field is removed and do not retain magnetic memory. Ferromagnetic materials (γ-Fe_2O_3) have all their electrons aligned in one direction when a magnetic field is applied. Even after the field is removed, they remain aligned in the same direction. Ferrimagnetic materials have electrons arranged in an unequal alignment when a magnetic field is applied. An example is Fe_3O_4. Magnetic activity measurement is summarised as follows.

4.2. Devices for Measurement of the magnetic properties of the materials

Magnetic susceptibility measurement involves assessing a material's susceptibility to magnetization in an applied magnetic field. This test can determine if an iron oxide substance is paramagnetic or ferromagnetic. Vibrating Sample Magnetometry (VSM) is a highly sensitive method for determining a material's magnetic characteristics. In VSM, a sample is exposed to different magnetic fields to assess its magnetization. This method can be used to analyze the magnetic characteristics of iron oxide. Quantum Interference Device with Superconductivity (SQUID) Magnetometry is an extremely sensitive method for measuring the magnetic characteristics of materials with weak magnetic signals, particularly iron oxide nanoparticles. Magnetic Resonance Imaging (MRI) is a medical imaging method that utilizes the magnetic characteristics of certain atoms, such as those found in iron oxide nanoparticles, to improve organ and tissue visualization during imaging. Magnetic Hysteresis Measurement is a technique used to measure a material's magnetic hysteresis loop, which demonstrates the relationship between the material's magnetization and the applied magnetic field. This method can help identify and examine the distinctive hysteresis behavior of iron oxide materials.

4.3. Factors affecting magnetic properties of IONPs

Size and shape: The hematite nanoparticles displayed a diverse size range, spanning from 21 nm to 82 nm, with a gradual increment in particle size (Lassoued et al. 2017b). These nanoparticles exhibited diverse shapes such as nanotubes, nanodiscs, and nanorods (Niraula et al. 2021b). This variability in size and morphology is expected to impart distinctive properties to the nanoparticles, rendering them highly effective for applications in magnetic separation processes for water treatment and biomedical purposes.

Surface Coating: Iron oxide nanoparticles possess the capacity to be rendered biocompatible through an assortment of surface treatments, comprising the application of biopolymers and biomolecules. Interestingly, as the pH levels increase, the percentage of the process of adsorption also increases. It is important to remember that substances like phosphatidylcholine (PC) and humic acids (HA) are frequently employed to coat particular, iron oxide nanoparticles (Demangeat et al. 2020).

Composition of nanomaterials: Iron oxide nanoparticles, or IO-Au NPs, have a significant magnetism of 65 emu/g and exhibit amazing supermagnetic qualities. These nanoparticles have the potential to be used as theranostic agents for therapeutic and biomedical imaging purposes (Kharey et al. 2023b). Moreover, through atomic contact, carbon-modified iron oxide nanoparticle electrochemical properties improve molecular biocompatibility (Verma et al. 2021).

Numerous academic articles have put forward a scientific approach for converting standard iron oxide nanoparticles (IONPs) into superparamagnetic ones by employing surface functionalization techniques. Overcoming the challenges of loss of magnetism and inconsistency in magnetic properties, these strategies encompass various structural designs such as Janus-type formations, matrix dispersion, core-shell, and shell–core-shell. To be more precise, the Janus structure enhances the magnetic behavior of IONPs in a magnetic force field by using functionalized magnetic IONPs as stabilizing nanomaterials. He et al. 2020 found that the magnetic Janus nanoparticles are efficient in removing emulsified oily waste materials present in the wastewater. When subjected to an external magnetic field, the asymmetrical surface wettability of the Janus NPs anchors more actively to oil droplets and removes them efficiently from oily wastewaters with a uniform surface wettability. The Janus NPs show a higher removal efficiency of 91.5% as compared to magnetic carboxymethyl cellulose and ethyl cellulose nanoparticles [M-CMC-EC NPs] which was found to be 84.3% which makes Janus NPs suitable to use for oily waste removal. The study also highlighted the recyclability and efficiency of Janus NPs as they are easily recyclable and reusable for frequent use without undergoing any complex regeneration process (He et al. 2020b).

The chemical process involves the destabilization of ions due to the loss of magnetism. Various approaches are proposed in multiple articles to address issues such as structural instability, particle aggregation, and problems with their magnetic behavior (Zhao et al. 2020). The encapsulating of iron oxide microstructures with nano-coating materials is the core-shell structure. These materials stabilize the elemental core shells of iron oxide nanoparticles (IONPs) by occupying the voids in their crystal structure. The crystal is supported by functional materials placed adjacent to each other on magnetic IONPs, which also protect the materials' core from chemical reactions. The shells of IONPs keep unwanted chemical activity from entering the core. Another goal of the technique is to prevent the growth of small superparamagnetic nanoparticles into larger but less potent magnetic IONPs (Marcu et al. 2013; Siddiqi et al. 2016; Vakili-Ghartavol et al. 2020a).

5. CHARACTERIZATION OF IONPS AND NANOCOMPOSITES

Analytical studies including adsorption kinetics, exemplified by models like the Langmuir model, are pivotal in evaluating the efficiency of these functionalized IONPs. The Langmuir model helps understand the monolayer adsorption capacity (Qmax) and affinity (KL) of IONPs towards pollutants, providing insights into surface coverage and adsorption energy (Dehbi et al. 2020). Magnetic properties studies elucidate the nanoparticles' behavior under external magnetic fields, crucial for their recovery and reuse in water treatment processes (Aisida et al. 2021). These analytical approaches collectively assess the nanoparticles' stability, dispersibility, and pollutant adsorption efficiency, essential for designing effective wastewater treatment strategies and ensuring environmental sustainability. The comprehensive characterization of magnetic iron-based nanomaterials can be achieved through various instrumental techniques enlisted in Table. 3.

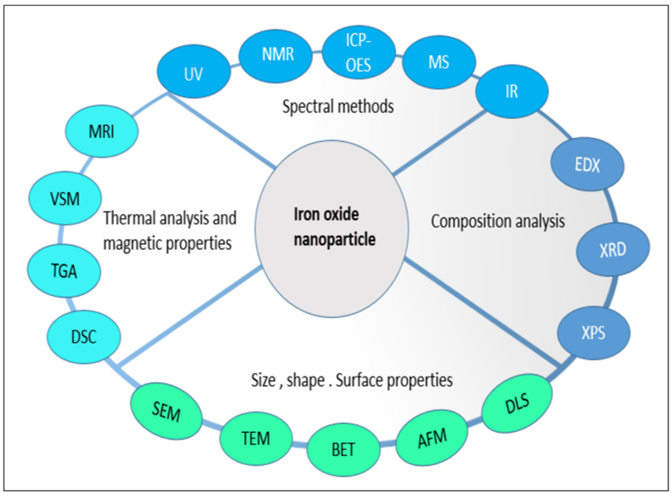

Fig. 6. Different IONP-based material characterization techniques

Spectral methods, including UV-visible spectroscopy, FT-IR, and ICP-OES, are crucial for identifying nanoparticle formation, functional groups, and elemental composition (Rajiv et al. 2017). X-ray diffraction (XRD) analyses crystal structure and phase composition, while X-ray fluorescence (XRF) offers detailed elemental analysis (Rajendran et al., 2023). High-resolution imaging techniques such as Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) reveal surface morphology, size, and dispersion (El-Sheekh et al. 2021b). The pH – Point Zero Charge (pHpzc) method assesses surface charge characteristics. (Ha et al. 2021) When combined, these methods offer vital details about the IONPs' magnetic, chemical, and physical characteristics. These details are crucial for maximizing their use in a variety of settings, such as industrial processes and environmental remediation. Different IONP-based material characterization techniques are presented in Fig. 6 and are summarised in Table 4.

Table 4. Methods for characterizing iron oxide nanomaterials

Characterization Technique	Primary Application of the Technique	Key Material Attributes Measured
UV-visible spectroscopy	Optical Analysis	Surface Plasmon Resonance, Absorption.
Infrared Spectroscopy (FT-IR)	Molecular Structure Analysis	Functional Groups, Bonding Types.
Scanning Electron Microscopy (SEM)	Morphology Study	Size, Shape, Aggregation, Surface Morphology.
Transmission Electron Microscopy (HRTEM)	Detailed Morphology Study	Size, Shape, Aggregation, Lattice Structure.
Dynamic Light Scattering (DLS)	Particle Size Distribution	Hydrodynamic Size, Polydispersity Index.
Atomic Force Microscopy (AFM)	Surface Topography	Structure, Geometry, Distribution.
Fluorescent Spectroscopy (FS)	Optical Properties	Emission, Wavelength, Optical Properties

Differential Scanning Calorimetry (DSC)	Thermal Analysis	Phase Transitions, Heat Capacity
Mass Spectrometry (MS)	Composition and Molecular Analysis	Surface Composition, Molecular Weight, Isotopic Distribution, Fragmentation Patterns
Nuclear Magnetic Resonance (NMR)	Structural and Purity Analysis	Purity, Composition, Chemical Shifts.
X-Ray Photoelectron Spectroscopy (XPS)	Surface Composition Analysis	Chemical Composition, Elemental Composition.
X-ray diffraction (XRD)	Crystallinity and Phase Analysis	Crystallinity, Phase Identification.

Spectral Methods: UV-visible spectroscopy, Fourier-transform infrared (FTIR) spectroscopy, and Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES) are crucial techniques for characterizing iron oxide nanoparticles (IONPs), each providing unique insights into their properties and applications. UV-visible spectroscopy identifies absorption peaks to determine the concentration, size, shape, and aggregation state of IONPs, elucidating surface Plasmon resonance (SPR) in metallic nanoparticles and confirming their formation and properties within the 200–800 nm range (Rajiv et al. 2017; Yadav et al. 2023).

FTIR spectroscopy reveals functional groups and bonding structures, highlighting characteristic bands for Fe-O-Fe vibrational modes and interactions between organic extracts and IONPs. It also confirms dye adsorption and the presence of magnetic cores in porous magnetite nanospheres (El-Sheekh et al. 2021b; Yadav et al. 2023; Alex Mbachu et al. 2023).

ICP-OES performs elemental analysis by utilizing high-temperature plasma to ionize samples, measuring emitted wavelengths to quantify elements like iron in various matrices, including plant tissues. This technique ensures accurate characterization and safety assessments in environmental, nanotechnology, and food science applications (Kobylinska et al. 2020b) Coupled with other characterization methods, ICP-OES helps optimize synthesis parameters and enhances the understanding of iron oxide nanoparticle (IONP) behavior. Collectively, these techniques provide comprehensive insights into IONP properties and applications, facilitating advancements in wastewater treatment and biomedicine.

Nuclear Magnetic Resonance spectroscopy is used to analyze the chemical structure and stability of functionalized iron oxide nanoparticles. To confirm the effectiveness of nanoparticle functionalization and guarantee the purity of the finished product, it offers comprehensive information on the molecular weights and composition of the stabilizing agents. NMR helps understand the effectiveness of different stabilization strategies by examining the structure and interactions of functionalized nanoparticles, confirming desired chemical modifications and overall quality. Mass spectrometry, particularly Electron Impact Mass Spectroscopy (EIMS), offers significant potential for the characterization of iron oxide nanoparticles. The ability to differentiate between compounds and identify their molecular fragments highlights the effectiveness of mass spectrometry in understanding the surface chemistry and composition of iron oxide nanoparticles (Willis et al. 2005).

Methods to Determine Physical and Particle Surface Properties: Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM), Brunauer– Emmett–Teller (BET) analysis, and pH point zero charge (pHpzc) measurements are essential techniques for characterizing iron oxide nanoparticles (IONPs). SEM provides detailed insights into surface morphology, aggregation state, and elemental composition, revealing spherical-shaped nanoparticles forming aggregates due to their magnetic nature (Alex Mbachu et al. 2023). It is indispensable for understanding structural and morphological features critical for IONP functionality in wastewater treatment applications. TEM offers high-resolution imaging, allowing observation of IONP size, shape, dispersion, and crystalline structure at the nanoscale (El-Sheekh et al. 2021b). BET analysis measures surface area and porosity by adsorbing and desorbing gas molecules, calculating specific surface area and pore volume to understand nanoparticle reactivity and surface properties, pH point zero charge (pHpzc) is crucial for characterizing iron oxide nanoparticles (IONPs), revealing their net surface charge and acidic or alkaline nature (Ha et al. 2021). It is essential for understanding adsorption processes, as it helps predict IONPs' interaction with pollutants and optimize their effectiveness in various pH environments. The pHpzc also indicates surface modifications and functional groups that enhance adsorption properties, making it vital for designing effective IONPs for environmental and industrial applications (Ha et al. 2021).

Atomic Force Microscopy (AFM) is a cost-effective and versatile tool for characterizing iron oxide nanoparticles (IONPs). It provides high-resolution three-dimensional visualization, allowing precise measurement of size, morphology, surface texture, and roughness. AFM operates in ambient air and liquid dispersions, is beneficial for biological studies, and offers a broad particle size range from 1 nm to 8 µm. Additionally, AFM requires less lab space and is simpler to operate than TEM/SEM, making it an ideal choice for nanoparticle characterization (Kumar et al. 2019).

Dynamic Light Scattering (DLS) is essential for characterizing the size and stability of iron oxide nanoparticles in solution. It provides insights into how the molecular weight of grafted polymers affects nanoparticle size and stability. DLS allows for real-time monitoring of particle size changes, crucial for understanding nanoparticle behavior under various conditions, such as in water or physiological buffers (Boyer et al. 2009).

Methods for Analysis of Composition of Iron Oxide Nanocomposite: Energy Dispersive X-ray (EDX), X-ray Photoelectron Spectroscopy (XPS), and X-ray Diffraction (XRD) are pivotal techniques for the characterization of iron oxide nanoparticles (IONPs), each offering unique insights into their composition and structure. EDX provides rapid elemental analysis and mapping, essential for confirming the synthesis and surface modification of IONPs (Majumder et al. 2019). XPS facilitates detailed chemical-state surface analysis, quantitative elemental composition, and determination of metal oxidation states, revealing the presence of functional groups and the chemical environment of atoms on the nanoparticle surface (Majumder et al. 2019). XRD is crucial for characterizing the crystal structure, phase composition, and crystallite size, identifying phases like magnetite, maghemite, and hematite by analyzing diffraction patterns (Yadav et al. 2023). The Debye-Scherrer equation estimates crystallite size, ensuring nanoparticle quality by detecting impurities and confirming phase composition against standard reference patterns (Bhutto et al. 2023). These techniques collectively enhance the understanding of IONP properties, optimizing their application in environmental remediation, biomedicine, and other fields (Rajendran et al. 2023).

Thermal Method for Characterization of Iron Oxide Nanocomposites: Thermogravimetric Analysis (TGA) is a critical technique for characterizing iron oxide nanoparticles (IONPs), offering valuable insights into their thermal stability and the presence of organic components. TGA measures weight changes of nanomaterials as a function of temperature, enabling the detection of thermal decomposition processes and the identification of any volatile or adsorbed substances (Kouhbanani et al. 2019). This method helps determine the extent of functionalization by assessing weight losses related to the removal of moisture and decomposition of organic molecules, confirming the presence and stability of surface modifications. By providing a detailed thermal profile, TGA supports the verification of synthesis processes and the quality of functionalized nanomaterials, making it an essential tool in the development and application of IONPs in various fields (Sandhya and Kalaiselvam 2020).

Determination of pH of Nanomaterials: PH point zero charge (pHpzc) is crucial for characterizing iron oxide nanoparticles (IONPs), revealing their net surface charge and acidic or alkaline nature (Ha et al. 2021). It is essential for understanding adsorption processes, as it helps predict IONPs' interaction with pollutants and optimize their effectiveness in various pH environments. The pHpzc also indicates surface modifications and functional groups that enhance adsorption properties, making it vital for designing effective IONPs for environmental and industrial applications (Ha et al. 2021). The pH of IONPs can affect their stability, surface charge, reactivity, and interactions with other molecules or substances. The pHzpc is an important factor in the absorption of heavy metals from wastewater (Mittal et al. 2023). The pH-sensitive magnetic nanofibers are used for thermos-chemotherapy (Vo et al. 2024). The term "point of zero charge" (PZC) signifies the pH value at which the net surface charge that results from the adsorption of the prospective ions H+ and OH- is zero. PZC is essential for the purification process of water, particularly for adsorption onto surfaces like activated carbon. It affects how organic pollutants, heavy metals, and other contaminants adsorb onto activated carbon surfaces (Nguyen et al. 2020). An examination was carried out to explore how the pH and SRHA (specific reactive surface area) impact the behavior of iron oxide nanoparticles. Both factors were found to be concentration-dependent, influencing the formation of open pores and aggregation (Baalousha et al. 2008)

6. ADVANTAGES OF IRON OXIDE NANOMATERIALS IN W&WT

The different advantages of Iron Oxide Nanomaterials in W&WT are summarised such as (a) Green synthesis of Iron oxide nanoparticles allows a greener approach for water purification (Reducing chemical intervention in water treatment), (b) Existing methods of water purification are effective, but some prevailing problems, like packed column plugging, membrane fouling, and high-pressure treatment streams, can be prevented by the use of INOPS owing to their high degree of dispersion and also its versatile ability to get deposited/combined with numerous technologies for enhancement of filtration activities (Razali et al. 2023), (c) The adsorption and catalytic conversion of pollutants into neutral components. Studies showed activities on polysaccharides and amino acids (Akhtar et al. 2024), Proteins (George and Kumar 2023), Polycyclic aromatic Hydrocarbon (Shanmuganathan et al. 2023), Pentachlorophenol (George et al. 2023) (d) IONP has shown high efficiency in the removal of inorganic substances like Zn (Ahmadi and Izanloo 2023), AsIII&AsIV (Priyadarshni et al. 2020;

Torasso et al. 2021), Triclosan (Dhasan et al. 2023), (e) Another useful function that can be made possible (by exposing the nanoparticles to a magnetic field) is site-specific targeting and in-situ applications, (f) INOP provides a high surface area-to-volume ratio, which when considered with the high number of active sites, provides a high capacity and adsorption affinity for pollutants and high reaction kinetics (Priyadarshni et al. 2020; Ahmadi and Izanloo 2023), (g) Easy Recovery of nanoparticles can also be made, attributed to the magnetic properties of INOP (Powell et al. 2020) and (h) Nanoparticles can be used in a more synergistic approach enhancing a few properties like oil-water mixture separation (Khandan Barani et al. 2023) along with bacterial control due to the inherent properties of IONP.

7. SYNERGISTIC PROPERTIES OF IRON OXIDE NANOMATERIALS FOR W&WT

Currently, there are many reports of synergistic activities of iron oxide nanomaterials in W&WT applications which are summarised such as, Alabresm et al., (2018) described the use of IONP with several bacterial strains like Halomonas sp., Vibrio gazogenes, and Marinobacte hydrocarbonoclasticus these few bacterial strains along with the nanoparticles coated with Polyvinyl Pyrrolidone were successful in removing Lower-chain alkanes, and higher chain alkanes with a removal efficiency of about 70% and 65% respectively within one hour of incubation. NPs with bacterial strains showed no effects immediately but had a significant increase in the removal at about 80-90% after 24-48 hours. The study showed a synergistic effect of the combination of oil-degrading bacterial strains and surface functionalized iron NPs. The system effectively can remove essentially 100% of oil within 48 h or less.

Shukla et al. 2021 provide a brief background of the metal oxide for synergistic remediation of the pharmaceutical polluted wastewater by the effect of the photocatalyst and dopants simultaneously. It shows how the drawback of the metal oxide nanoparticles can be overcome by their doping; this specific doping enhances its photocatalytic rate. The literature also describes briefly about the metal oxides including, iron, copper, titanium, zinc, silver, tungsten, bismuth, and platinum.

Aboelfetoh et al. 2023 introduce a cost-effective and environmentally friendly approach for generating α-Fe_2O_3-CuO nanocatalyst. To create the α-Fe_2O_3-CuO nanocomposite, copper hexacyanoferrate was decomposed by mild heat oxidation. XRD, SEM, FTIR, EDX, TEM, XPS, and VSM were used to verify its structure and surface morphology. In the presence of H_2O_2, α-Fe_2O_3-CuO acts as a catalyst to promote thermal degradation of dyes such as direct violet 4, rhodamine B, and methylene blue. The nanocomposite's catalytic activity increased due to the synergistic action of Fe_2O_3 and CuO, surpassing that of the individual components. Incorporating inorganic anions such as chloride or nitrate accelerated the breakdown process. While sulfate and humic acid, especially in large amounts, delayed it. The mechanism of H_2O_2 activation on α-Fe_2O_3-CuO is investigated. Chemical oxygen demand and total organic carbon measurements reveal that all colors are significantly mineralized. This nanocomposite's capacity to effectively remove various colors makes it a viable solution for wastewater treatment.

Using a combination of ferrate (VI) and peroxymonosulfate, Feng et al., (2017) invented a method for efficiently breaking down four fluoroquinolone antibiotics in water. The combined method fared better than using each oxidant alone. The investigation, which used LC-HRMS analysis, found that the mixture of oxidants causes the antibiotics to undergo hydroxylation, ring cleavage, and defluorination. These results point to the method's potential as a cutting-edge water treatment technique for getting rid of organic contaminants. In another study water decontamination occurred through the Fenton reaction along with CO and Fe ions showing a synergistic effect in the removal mechanism (Wang et al. 2022).

8. MECHANISM OF WATER REMEDIATION BY IONP-BASED NANOMATERIALS (IONPNMS)

Adsorption and desorption of metal and organic pollutants, and photocatalysis of dyes are some widely used mechanisms for research with the addition of a few new methods like Fenton's oxidation and other electrochemical processes.

8.1. Adsorption and Desorption

As the name suggests this mechanism is based on the capacity of surface adsorption of the pollutants to remove them directly which can then be desorbed to obtain the iron oxide nanoparticle Solvents are required for the desorption process and it also has some major flaws like loss of activity of the iron oxide nanoparticle due to permanent adsorption on the surface Some benefits as they can be stated are as such that high surface area is present due to smaller size, presence of the negatively charged hydroxyl group (OH^-)imparts a slight negative charge to the nanoparticle making it effective in the removal of the cationic pollutants mainly heavy metals Physisorption and chemisorption play the major roles in the binding of the pollutants where in physisorption provides a weaker bond than that of chemisorption. Another important point to be noted is that as explained in the recovery of the nanoparticles the cycles before efficiency loss must be as maximum as possible which can be achieved by the composite formation (Hussen Shadi et al. 2020).

Talbot et al., 2018 studied the absorption/desorption cycles of pH-sensitive magnetic alginate/γ-Fe_2O_3 NPs using methylene blue dye. Reusability is a crucial factor in evaluating the performance of NPs. After exposure to dye, these NPs were magnetically separated, washed with solvent, and introduced to nitric acid for desorption. Before the next cycle pH of the particles was raised to 7, and the study performed adsorption and desorption cycles continuously. As investigated nitric acid at pH 2 was used as a desorption agent. The observed efficiency of desorption was 99%, and the adsorption capacity remained 100%, showing signs of no change even after 10 cycles. This study provided evidence in favor of the potential application of alginate magnetic nanoparticles in the water treatment procedure. Composite of iron oxide NPS with organic and inorganic materials explored for removal of metal ion pollutants are listed in Table 5; pollutant removal efficiency ranges from 96 % to 99 %. Micro boxes showed the removal of 97.30 % of Pb^{2+}, 96% of Cu^{2+}, 97.90% of Cd^{2+}, and 98.30% of Hg^{2+} contamination (Ravindranath et al. 2017). Multifunctional MIONPs (Fe_3O_4) modified with 2,3-dimercaptosuccinic acid/dopamine-s to remove heavy metal ions have shown 49.46 mg/g of Cd^{2+} and 87.62 mg/g of Pb^{2+} removal efficiency (Lei et al. 2023).

Table 5. Iron oxide nanocomposite and inorganic pollutant adsorption efficiency

Nanoparticles	Composite designed with	Application for metal ion Pollutant	Pollution removal Efficiency	References
FeO	-	Zn^{2+}	99%	(Ahmadi and Izanloo 2023)
FeO and CuO	Stabilized on biochar (rice husk)	As^3 As^5	>95%	(Priyadarshni et al. 2020)
Fe_2O_3 and Al_2O_3	Microboxes	Hg^{2+} Pb^{2+} Cu^{2+} Cd^{2+}	98.30% 97.30% 96.00% 97.90%	(Ravindranath et al. 2017)
Fe_3O_4	Dopamine-Dimercaptosuccinic acid	Pb^{2+} Cu^{2+} Cd^{2+}	187.62 mg/g 63.01 mg/g 49.46 mg/g	(Lei et al. 2023)

8.2. Catalysis

8.2.1. Photocatalysis

The photocatalytic process operates under the assumption that the energy absorbed from light radiation is sufficient to equal or exceed the band gap energy of the semiconductor demonstrating how the reactions occurring on a semiconductor material are photocatalytic (Topare et al. 2022). Electrons in the valence band of the semiconductor material will be stimulated to move to the conduction band and leave holes in the valence band when incident light energy exceeds the band gap. The excitation produces electrons (e^-) in the conduction band and holes (hvb^+) in the valence band in the fs time scale. These are then recombined in the range of 10–100 ns after being trapped in 100 ps (shallow trap) to 10 ns (deep trap). The photo-induced electrons react with adsorbed oxygen or oxygen dissolved in water as part of the reduction processes to generate $•O^{2-}$. Subsequently, O^{2-} may combine with H^+ to generate a hydroperoxyl radical, which may then react to make H_2O_2 or hydrogen peroxide. H_2O_2 can react with the electrons and become •OH. While this is happening, the photo-induced holes diffuse to the surface of TiO2 and produce •OH by reaction with the adsorbed water molecules in a neutral solution or the adsorbed hydroxyl group in an alkaline solution. •OH, can degrade most organic compounds with second-order rate constants of 108–1010 M^{-1} s^{-1}. Reactive oxygen species and holes have the potential to facilitate oxidative processes as explained by Khan et al., 2022.

Mansur et al., 2023 suggested that in addition to their photocatalytic activity, biocompatible and ecologically harmless inorganic nanomaterials, including heavy metal-free semiconductors, have drawn increased attention since they meet the majority of sustainability criteria. Because they are non-toxic and environmentally benign, zinc-based compounds, such as oxides (ZnO) and chalcogenides (ZnS, ZnSe), are chosen over traditional highly toxic quantum dots (QDs e.g. CdS, CdSe, CdTe, PbS, PbSe, etc.). This is the result of extensive research on binary semiconductor nanoparticles (II-VI NPs). In addition, Zn-based chalcogenide semiconductor nanoparticles, also known as QDs, experience the quantum confinement regime at minuscule sizes (< 5 nm), offering special optoelectronic characteristics for a variety of environmental and biological applications. They used carboxymethylcellulose (CMC) to stabilize the magnetic iron oxide (MION, Fe_3O_4). To create the hybrid multifunctional nanoplexes, this MION was further mixed with ZnS semiconductor quantum dots (ZnS QDs) that had been chemically biofunctionalized with epsilon-poly-l-lysine (εPL). The findings showed that strong cationic/anionic electrostatic interactions between the biomacromolecule capping ligands of the two nanoconjugates (polypeptide in ZnS@εPL and polysaccharide in Fe_3O_4@CMC) were responsible for the formation of supramolecular colloidal nanoplexes. These nanosystems demonstrated the photocatalytic degradation of methylene blue (MB), a common dye contaminant in water. MB was not the only dye utilized to test the selectivity of the photodegradation induced by the nanoplexes; methyl orange, congo red, and rhodamine were also checked. The antibacterial activity of the εPL biomolecule against drug-resistant bacteria as well as Gram-positive and Gram-negative bacteria was confirmed. The process of mechanism of water remediation with IONPs is presented in Fig. 7.

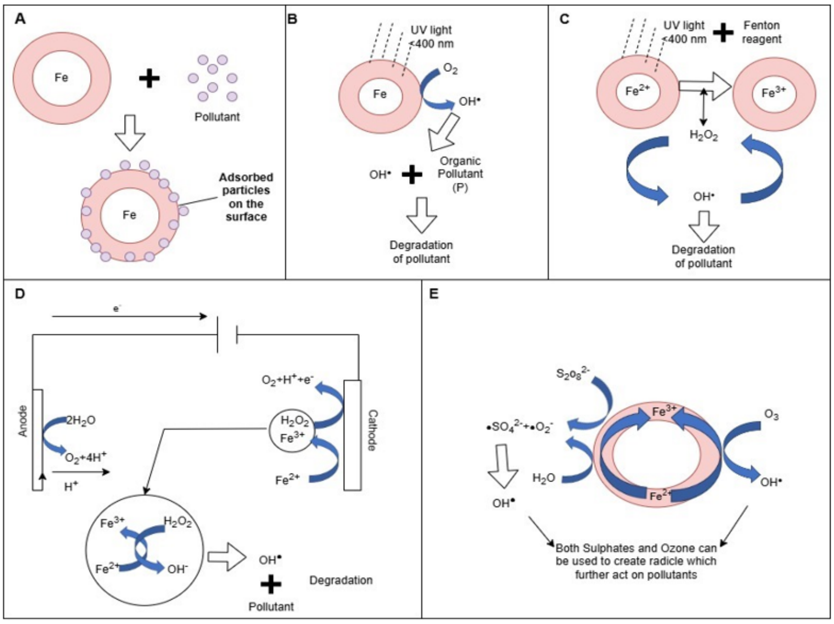

Fig. 7. Mechanisms of water remediation by IONP-based materials A) Adsorption, B) Photocatalysis, C) Photo Fenton process, D) Electro Fenton process E) Chemical oxidation process

8.2.2. Photo-Fenton processes

Photo-Fenton reactions use Fenton reagents and irradiation to produce HO˙ by photo-reduction of ferric to ferrous ions, followed by hydrogen peroxide photolysis (Han et al. 2020). Photochemistry's contribution to this process is to generate energy by utilizing ultraviolet (UV) or visible light to increase the system's catalytic activity while simultaneously reducing the amount of catalyst used. Han et al. 2020 studied the efficiency of the photo-fenton process in degrading pollutants like tetracycline and oxytetracycline. After studying different methods, it was concluded that the UV light-assisted Fenton reaction ($UV/H_2O_2/Fe^{2+}$) was the most effective technique to remove pollutants from water. This conclusion was based on a comparison of this procedure with H_2O_2, UV, and UV/H_2O_2 procedures. While incomplete antibiotic mineralization occurred, the resulting by-products were identified and assessed for toxicity using a bacterial model. This is a critical concern given the limited availability and energy-intensive production of UV light, commonly used in photo-Fenton processes. Visible light-driven photoreactions offer a more cost-effective alternative (Liu et al. 2014).

Iron oxides, such as hematite, are capable of potently absorbing visible light due to their electronic band structure; nevertheless, photocatalytic activity is limited because the e^-/h^+ couples recombine quickly (Santhosh et al. 2019b; Kumar et al. 2024). Photo-Fenton oxidation, which employs semiconductors with large bandgaps such as TiO_2, is an effective charge separation technique. Hassan et al. 2016 established a photocatalytic mechanism for methyl orange (MO) degradation using a Fe_2O_3/TiO_2 nanocomposite with visible light. Duca et al., 2023 examined the breakdown of toluene using IONP and H_2O_2 and found that benzaldehyde and bibenzyl were the predominant oxidation products. The investigations demonstrated that INOP could be reused without loss of efficiency, indicating the potential for heterogeneous photo-Fenton reactions to degrade aromatic contaminants such as toluene in water. This work demonstrates quicker reactions with Fe (II), but also the benefits of employing IONP which is less toxic and easy to reuse. pH was shown to have a substantial impact on the processes, with optimum degradation occurring at around pH 3.0.

8.3. Electrochemical Processes

Electrochemical oxidation is one of the most extensively studied electricity-driven advanced oxidation processes (EAOPs) for water remediation (Hodges et al. 2018). The pollutants can be turned into minerals by anodic oxidation, which transfers electrons directly to the electrode's surface, or by indirect electro-oxidation, which creates strong oxidants (Buthiyappan et al. 2016; M'Arimi et al. 2020). Although this process has some advantages, such as versatility, ease of operation, and the ability to adapt to variations in flow rate and influent water composition, concerns about the formation of toxic byproducts, further cost, and electrode performance issues can limit its applications (Radjenovic and Sedlak 2015). Magnetic iron oxides facilitate rapid electron transfer, making them useful for electrocatalytic applications (Kudr et al. 2018; Topare et al. 2022). Researchers have developed composite electrodes containing magnetic particles to enhance these properties. Ribeiro et al., (2017) created a layered electrode using iron oxide nanoparticles and carbon nanotubes to detect salicylic acid, a drug metabolite and environmental pollutant.

The electro-Fenton technique, on the other hand, is one of the most widely used E-AOP. In this method, in situ, generation of at least one ingredient for the Fenton reaction is achieved by utilizing electrons as reagents, where the following options might occur: Hydrogen peroxide (H_2O_2) is produced at the negative electrode (cathode) and oxygen (O_2) at the positive electrode (anode). Iron ions (Fe^{2+}) can either be added as a catalyst or generated from a dissolving iron anode. The final instance does not require the addition of chemicals, making the operation straightforward to carry out. The continual in situ synthesis of H_2O_2 overcomes the issues connected with its transportation and storage. The disadvantage observed for this procedure is the production of iron hydroxide sludge, which requires proper disposal. Ferrous sludge emission can be avoided by employing membranes (Muddemann et al. 2019).

8.4. Chemical Oxidation / Alternative Advanced Oxidation Processes

In search of alternatives to Fenton reactions, researchers have been exploring the activation of different oxidants to generate highly reactive radicals, leading to an interest in species like ozone and sulfates. Ozonation has been identified as a possible method for water cleanup. Pollutants can be degraded through two pathways: direct oxidation (molecular O3) or complicated chain reactions that produce HO radicals (Buthiyappan et al. 2016). Because direct oxidation of O3 is a selective technique, it produces partial mineralization at low rates. Furthermore, ozone's limited solubility in aqueous solutions limits its practical application (Yu et al. 2020). As a result, studies have used catalysts to increase ozone breakdown and ROS production. (Chen and Wang 2019) employed a Fe_3O_4/Co_3O_4 composite for catalytic ozonation of sulfamethoxazole (SMX), resulting in a 60% increase in pollutant mineralization. This finding was attributed to the catalyst's increased surface area and reactive sites, which may have accelerated the interaction between O_3-SMX and free radical production. Various factors were studied, including pH, catalyst concentration, ozone, and contaminants, to determine which parameters have the best catalytic activity and how they impact the reaction pathways.

8.5. Sulfate Radical-Based Advanced Oxidation Processes (AOPs)

This is a promising alternative to existing methods like Fenton and ozonation. It uses persulfates (PS) and peroxymonosulfate (PMS) to generate sulfate radicals for water disinfection. Sulfate radicals offer several advantages like, lower cost and higher stability compared to hydrogen peroxide and ozone, similar or higher oxidation power than hydroxyl radicals ($HO^{\bullet}$), wide effective pH range (2-8), and eco-friendly (Xiao et al. 2020). Iron oxide nanoparticles can activate peroxygen to form sulfate radicals, making them effective in this process. Research has shown that combining iron nanoparticles with PS or PMS enhances the degradation of pollutants compared to using them alone (Jegadeesan et al. 2019). The potential of IONP-based catalysts for ultrasound-assisted water treatment has been mentioned in the literature. These catalysts are a viable choice for effective and affordable water filtration since they are inexpensive, widely accessible, and safe (Zhang et al. 2018). Hassani et al. 2018 described the use of a heterogeneous Sono-Fenton method using Fe_3O_4 nanoparticles to remove the cationic dye violet 10. As predicted, the connection of both AOPs increased radical production under acidic circumstances, leading to dye degradation. However, given that the dye was not fully mineralized, the scientists emphasized the need for identifying the by-products generated throughout the operations and their possible delirious effect on the environment.

9. RECOVERY OF IRON OXIDE NANOMATERIALS

Iron oxide nanoparticles are valuable for water remediation due to their magnetic properties, enabling easy separation and reuse. However, current applications are limited to small-scale, handheld magnet setups, hindering their use in large-scale or continuous water treatment (Leonel et al. 2021a). In recent years, there have been a lot of advancements with the availability of statistically backed data on the in-line working of magnetic devices. Powell et al., (2020) investigated an in-situ approach where the installation of a specially made magnetic nanoparticle recovery device (MagNERD) under continuous flow. Its application was demonstrated for a smoother recovery of nanoparticles from the water flow in a process of water treatment. The device uses specific magnetic fingers to attract the nanoparticles and captures them inside stainless-steel wool which was wrapped around magnetic fingers. A unique approach was presented for separating the nanoparticles after their use in effluent treatment. Recovery of the IONPs was attributed majorly to their magnetic properties (Eldos et al. 2023).

In another recovery study IONPs were recovered by using acetone ethanol and distilled water, these NPs were able to retain their efficiency for around 5 cycles (Getahun et al. 2022). In a study IONPs were immobilized on the surface of expanded graphite where for the recovery of active surface 0.1 M NaOH was found to have the utmost efficiency in removal of the adsorbate and regenerating the adsorbent surface (Do et al. 2020). According to Shadi et al. 2020 recovery can also be facilitated with distilled water by varying the pH of the water, it is also mentioned that the bond between the pollutant and the nanoparticle is relatively weak and can be broken simply by using a concentration of base or acid.

10. IONPS-BASED METHODOLOGIES FOR W&WT

Over the past few years, there has been an introduction of IONPs for the purpose of water and wastewater treatment (W&WT) processes. The use of IONPs in such treatments has been the subject of much study and development, to offer safe, long-lasting, and reasonably priced water treatment options while protecting the environment. Undoubtedly, there exist certain limitations that impede the practical implementation of IONPs, such as diverse obstacles. These challenges and possible solutions to the challenges are summarised in Fig. 8 (Tai et al. 2023, Bhuiyan et al. 2020; Leonel et al. 2021b; Epelle et al. 2022). Recently iron oxide nanoparticles (Fe_3O_4) have sparked impactful interest in modified nanocomposites by fabrication of the membranes. This modification is certainly aimed at its intrinsic properties that could improve membrane surface hydrophilicity, antifouling, etc. properties leading to better removal rates and durability of these membranes, the advantages are presented in Fig. 9.

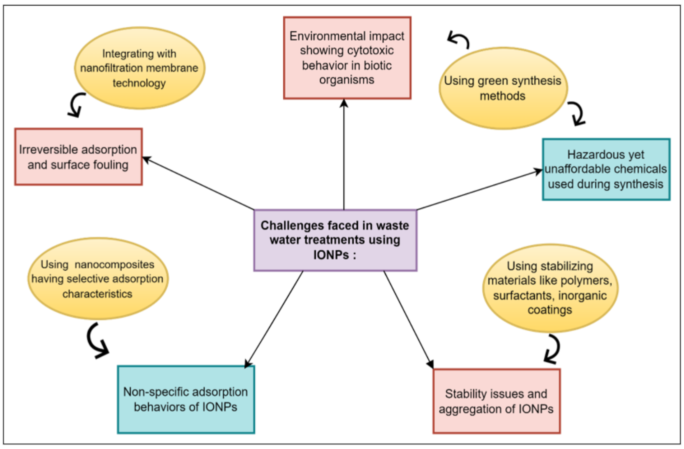

Fig. 8. Challenges in wastewater treatment using IONPs and their possible solutions (Bhuiyan et al. 2020; Leonel et al. 2021b; Epelle et al. 2022)

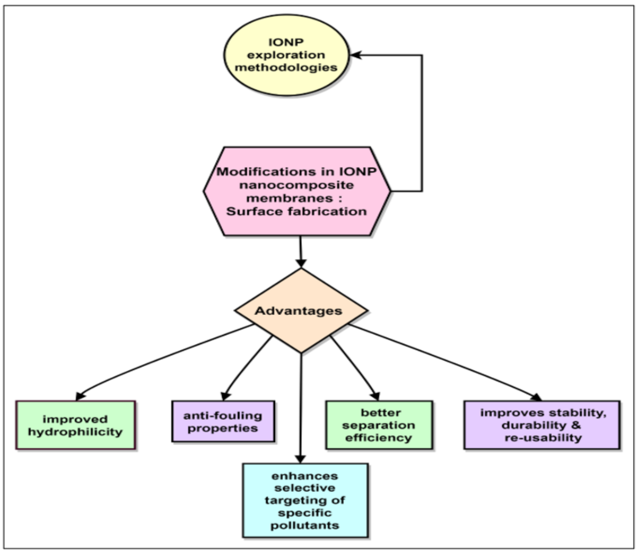

Fig. 9. Advantages of surface fabrication of IONP nanocomposite membranes

10.1. IONP-based Nano, Ultra, Reverse Osmosis (RO) filtration systems for W&WT

MIONPs embedded in nanofiltration (NF) membrane: Iron oxide (FeO) nanoparticles embedded in thin-film nanocomposite nanofiltration (NF) membrane for water treatment. Efficient nanofiltration mechanisms for dye removal are demonstrated by mixed matrix membranes. A special water flux enhancer called cobalt ferrite functionalized on bentonite ($CoFe_2O_4$@BT) and embedded in a polyethersulfone matrix. For Congo red, crystal violet, and humic acid, $CoFe_2O_4$@BT functions as a better flux enhancer and offers higher separation efficiencies of 95%, 94.69%, and 94.16%, respectively. Additionally, this kind of NF membrane is better at preventing fouling, which lowers the membrane's ability to separate and purify substances. As a result, it can retain its separation and purification capabilities longer. Increased water flux and separation efficiency are provided by this NF membrane, which also keeps dyes from passing through and produces purified water (Maraddi et al.

2024). The proposed mechanism of dye removal by $CoFe_2O_4$@BT with improved anti-fouling properties for purified water production is presented in Fig. 10.

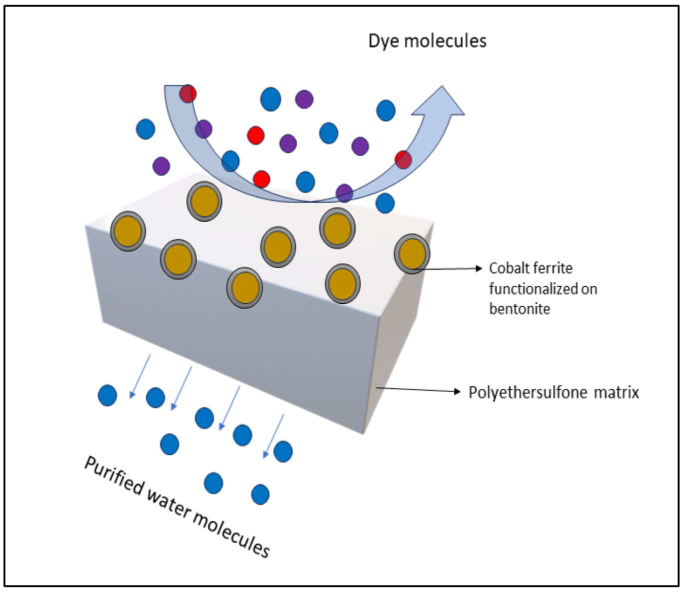

Fig. 10. Mechanism of dye removal by $CoFe_2O_4$@BT with improved anti-fouling properties

Iron Oxide Functionalized Membranes for Toxic Metal Removal from Power Plant Scrubber Water: Iron-functionalized lab-scale membranes were created to reduce and adsorb selenium from scrubber water from coal-fired power plants. Because iron-functionalized membranes prevent particle aggregation and dissolution, they are more effective than iron suspension in this regard. Polyvinylidene fluoride (PVDF) membranes were coated with polyacrylic acid (PAA) to prepare both lab-scale and full-scale membranes. This was followed by ion exchange of ferrous ions and reduction to zero-valent iron nanoparticles. The highest ion exchange capacity was achieved at 20% PAA with highly responsive pH pores, as the percentage of PAA functionalization increased while the water permeability of the membrane decreased (Gui et al. 2015).

IONP-based MXene Composite Material for Ultra and RO filtration: The MMM (mixed matrix membrane) was created using $ZnFe_2O_4$ and $Ti_3C_2T_x$ MXene composite i.e. $ZnFe_2O_4$@$Ti_3C_2T_x$ MXene with polyether sulfone (PES). The composite improved MMMs antifouling properties, and pore structure and improved hydrophilicity and surface polarity. The ultrafiltration membranes (UFM) maintained their flux for five cycles of bovine serum albumin (BSA) solution/backwashing at approximately 350 $L \cdot m^{-2} \cdot h^{-1}$, showcasing excellent stability. UFM exhibited the highest flow (324.56 $L \cdot m^{-2} \cdot h^{-1}$) and achieved high dye/salt separation (RCR = 97.3; RNa_2SO_4 = 8%). This highlights the potential of PES MMMs containing $ZnFe_2O_4$@MXene for separating dyes from high-salinity wastewater (Zhou et al. 2023). Sun et al. 2024 studied the process of creating a thin film by combining PVA with MXene@Fe_3O_4. This innovative combination enhanced the compaction of the coating materials and effectively prevented corrosive reactions on metal surfaces.

The inorganic corrosion inhibitor ferroferric oxide (Fe_3O_4) was electrostatically loaded onto Ti_3C_2 MXene nanofluids to obtain a hybrid material to improve its corrosion resistance. In another study, an innovative 2D sandwich-layer structure was meticulously crafted using MXene-iron oxide (MXI). These ultrafine nanocomposites have been discovered to be remarkably effective for sequestering phosphate in water purification processes. (Zhang et al. 2016). In one study polyamide commercial reverse osmosis (RO) membranes were coated with iron nanoparticles (FeNPs) and graphene oxide (GO) to prevent biofouling. Tests showed that the GO-FeNP coating reduced biofilm thickness, total cell count, optical density, and total organic carbon compared to uncoated membranes. Despite reduced permeance, the coated membranes exhibited larger fluxes after fouling than the fouled uncoated membrane (Armendáriz-Ontiveros et al. 2019).

10.2. IONP Nanocomposites

Another highly efficient technique to treat wastewater is preparing IONP nanocomposites. One of the studies suggests embedding IONP on chitosan-lignocellulose fiber nanocomposite. The chemicals selectively targeted are negatively charged compounds, particularly acidic dyes (AR-18). This type of dye along with (Acid Yellow: AY23, AY6) is excessively used in textile, cosmetic, and food industries where untreated water disposal poses a risk to aquatic animals and humans (Vargas et al. 2012). The separation takes place with a biosorption mechanism as adsorption of this model azo dye takes place on chitosan depending on temperature, pH, and ionic strength. The most suitable separation reaction conditions are low pH where protonation of amino groups occurs in chitosan which increases electrostatic interactions with anions in the AR-18 (Zhou et al. 2016). The nanocomposite's removal rates of acidic dyes remain at 99.68% through 10 consecutive cycles and can be easily recovered using magnetic fields and reused again. Hemoglobin iron oxide (Hb/Fe3O4) composite is an additional example of an iron oxide composite used to remove various dyes from aqueous solutions, including indigo carmine, naphthol blue-black, erythrosine, tartrazine, Eriochrome black T, and bromophenol blue. Instead of adsorption, it demonstrated the electrostatic interaction mechanism. The adsorption demonstrated removal capacities over a range of 80–178 mg/g, following a pseudo-second-order kinetic model and Langmuir isotherm. An additional benefit is that the composite could be separated from the aqueous solution using an external magnet (Essandoh and Garcia 2018). Various nanocomposites of iron oxide-based NPs discussed in section 3.

10.3. 3D Printed Wastewater Filtration System for Targeted Arsenic Removal

Arsenic is an excessively found metal in groundwater which is used for drinking purposes by many people, especially in rural areas. To prevent ingestion of this highly toxic metal, and to aim for cost-effective and large-scale treatment of groundwater bodies, an example involved a 3D printed water filtration system for Arsenic metal removal. The filters were developed by 3D printing technique using AutoCAD 2016, stereolithography, and finally printed using DeltaBot unit (Fig. 11). The heated nozzle melted and deposited Polylactic acid (PLA) filament to prepare filters with channel widths varying from 0.8- 4 mm. Further steps are followed by surface treatment of these PLA filters using 10M HCl to improve hydrophilicity and $Fe(OH)_3$ deposition by acid hydrolysis. Surface treatment was carried out by iron (III) hydroxide solution, as shown in Fig. 11, with consequent drying (60°C for 12 hours) to increase the bounding of Fe(OH)3 brown color particles in the 3D PLA filter. Arsenic removal involves adsorption on the iron (III) oxide particles deposited on the filter. An isotherm study determined that 95 % Arsenic removal rates were achieved by narrower channels (0.8-1mm) filter whereas rapid saturation and decreased Arsenic removal rates were observed in 1.8mm- 4mm filter channel sizes (Kim et al. 2020).

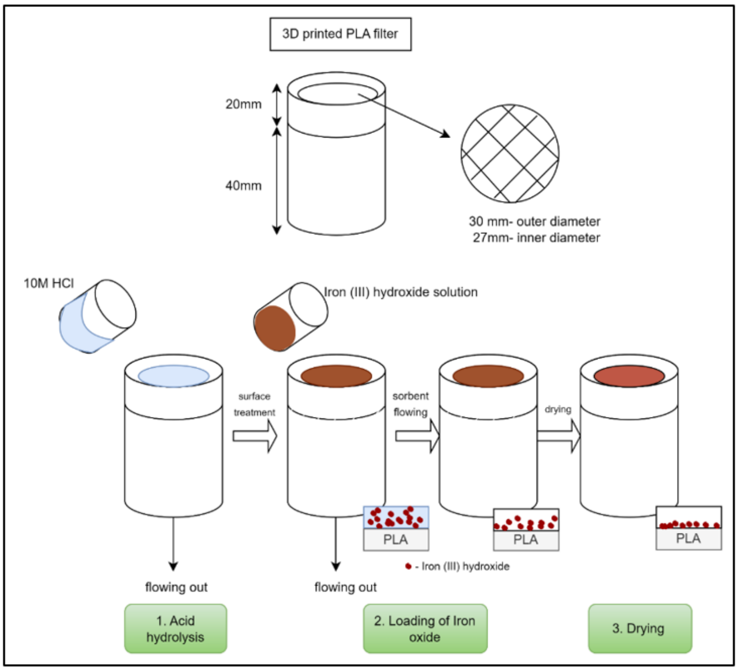

Fig. 11. Systematic steps to prepare 3D printed PLA filter

10.4. Use of micro/nanorobots for water remediation

Using micro/nanomotors (MNMs) has opened exciting new possibilities for water remediation due to nanotechnology. MNMs are a novel class of IONP-based materials that can move on their own, which gives them the ability to interact with pollutants in water systems in an efficient manner. These tiny motors transform energy into motion, enabling them to work together or independently to complete particular tasks. When it comes to environmental remediation, MNMs can be extremely helpful in treating water that has been contaminated by pathogens, organic compounds, toxic metals, and emerging pollutants. Because of their ability to move and interact with their surroundings on their own, MNMs can carry out tasks depending on the parts and designs they have. MNMs can interact with pollutants by stacking interactions between aromatic rings, capturing them inside their pores, or adsorbing them through electrostatic attraction forces (El-Naggar et al. 2024).

By using their sensitive components for oxidant activation and energy conversion to trigger oxidation reactions, they can also break down pollutants through oxidative processes. Additionally, MNMs are useful for detecting various types of pollutants because they can sense pollutants through colorimetric detection and fluorescence quenching. MNMs can fight harmful microorganisms and can also be used for bacterial disinfection. Moreover, they can disintegrate for the purpose of disinfecting water, and various methods can effectively expand their use for disinfection to address additional harmful microorganisms (El-Naggar et al. 2024). IONP can be incorporated with self-fuelled motors like ZIF-67 to give micromotors Fe_3O_4/ZIF-67. Compared to regular Fe_3O_4 nanoparticles, the autonomous motion of the micromotors improves the degradation and removal of Methylene blue dye.

11. CHOICE OF WATER REMEDIATION METHODS WITH IRON OXIDE NANOMATERIALS

The wastewater generated through various sources like agricultural practices, industrial practices, sewage, and wastewater sludge is remediated through many mechanisms like adsorption, centrifugation technique, coagulation and flocculation, gravity settling, and filtration. Industrial wastewater is the main source of contamination and poses serious environmental hazards among all the sources. Many recent advancements are focusing on the remediation of water from industrial sources such as Chemical oxidation, biological treatments, membrane bioreactors, Reverse osmosis, and adsorption techniques. Adsorption is the most frequently used technique for water remediation procedures. Activated carbon was used in the adsorption process by conventional techniques; however, the latest breakthrough in water treatment technologies is nanotechnology (Adegoke and Stenström 2019).

Magnetic nanoparticles and nanocomposites are found to be the most efficient way for water remediation processes. Nanoparticles are a promising method for water remediation because of their easy synthesis, low cost, high surface area to volume ratio, biocompatibility, catalytic activity, optical activity, and photoelectrical activity. The next section discusses the latest research on Iron Oxide Nanoparticles (IONPs) and offers information on the effective removal of various pollutants from industrial wastewater using this method.

12. METHODS FOR EVALUATION OF THE WATER TREATMENT EFFICIENCY OF NANOMATERIALS

12.1. General Methods

Total organic carbon (TOC): TOC As a carbon analysis tool, it calculates the total amount of organic carbon in the effluent. To evaluate the wastewater's strength by calculating the amount of carbon-based chemicals present Is measured by oxidizing organic carbon directly or indirectly to CO2 and water. Biological Oxygen Demand (BOD): Microorganisms will continue to decompose waste material aerobically in the presence of oxygen until it is completely utilized. Chemical Oxygen Demand (COD): The parameter calculates how much oxygen is needed in water to oxidize both organic and inorganic materials. Oil and Grease (O&G): O&G components in wastewater are derived from hydrophobic plants and animals that are poorly soluble in the water, which makes them less biodegradable by microbes. Removal of solids from water: The concentration of particle solids that can dissolve or suspend in wastewater was estimated using the terms total solids (TS), total suspended solids (TSS), total dissolved solids (TDS), total volatile solids (TVS), and total fixed solids (TFS). Nutrient removal efficiency: A measurement of the concentration of specific elements that can speed up eutrophication, the natural aging process of water bodies, such as phosphorus and nitrogen. Various elements determined in the sample for the purpose are ammonia (NH), total Kjeldahl nitrogen (TKN), nitrogen typically as nitrate or nitrite (N-N), total phosphorous (TP), etc. The general tests used to evaluate the water treatment efficiency of nano-materials are presented in Fig. 12.

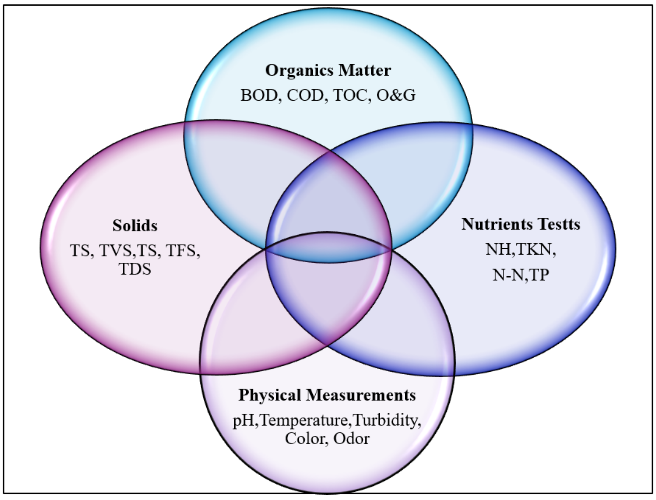

Fig. 12. General test used to evaluate water treatment efficiency of nanomaterials

12.2. Removal of organic pollutants

12.2.1. Dye Removal Efficiency from Effluents

Iron oxide nanoparticles are used to eliminate a variety of wastewater generated in industries and other sources. One of the most common wastes generated are dyes which can have harmful effects on the environment, humans, and aquatic animals. Dyes can impede the photosynthetic activity of aquatic plants. The most efficient way to remove the dye materials in the effluents is by adsorption mechanism (Kalaiyan et al 2025). A few applications of various modified IONPs for dye removal from wastewater are presented in Table 6.

Table 6. Applications of modified IONPs for dye removal from wastewater

Name of Adsorbent	Pollutant targeted	Removal efficiency	Adsorption isotherm fitting model	Reference
IONP embedded on chitosan-lignocellulose fiber nanocomposite	Acidic dyes [AR-18, AY23, AY6]	99.68%	Langmuir, Freundlich, Sips	(Zhou et al. 2016)
Fe_3O_4 and polyoxometalate hybrid nanocomposite	Methylene blue, Rhodamine B, Methyl Orange	97.84%, 91.22%,	Langmuir	(Li et al. 2019)

Fe_3O_4 modified with 3-glyciodoxypropyl trimethyoxysilane and glycine	Methylene blue, Orange I, Acid Red 10, Methyl Blue	90%	Langmuir	(Zhang et al. 2013)
Sodium dodecyl sulfate-modified Maghemite	Brilliant Cresyl Blue, Thionine, Janus Green B	93%	Langmuir	(Afkhami et al. 2010)
Carbon nanotubes and iron oxide-composed nanocomposite	Methylene blue, Neutral Red, Brilliant cresyl blue	99.16%, 98.33%, 98.8%	Freundlich	(Gong et al. 2009)
Fe_2O_3 cross-lined chitosan composite	Methyl Orange	98.25%	-	(Zhu et al. 2010)

12.2.2. Removal of drugs and Pharmaceuticals from Effluents

Many residual pharmaceutical materials, intermediates, and other products are sometimes left untreated in the effluent and discarded in the water bodies which can cause potential harm to the environment. This type of discharge can also happen via animal farms and human wastes. Many antibiotics, anti-inflammatory drugs, anti-leprotic, anti-viral, contraceptives, lipids, beta-blockers, and anti-cancer can form toxic derivatives in the post-chlorinated effluent (Adegoke and Stenström 2019). Excessive accumulation of antibiotics and antimicrobials has led to the development of antibiotic resistance in aquatic organisms and has promoted changes in the aquatic microbiome. Chen 2015 discussed the anti-biotic ofloxacin having neurotoxicological effects and effect on marine fish biomes. Anti-inflammatory -Ibuprofen had shown inhibition of growth of aquatic plants. Pascoe 2003 had shown Digoxin, a cardiac glycoside showing depletion of regeneration in aquatic animals.

In wastewater and water treatment applications, several methods, including photocatalysis, nanofiltration (NF), adsorption, and electrochemical oxidation, can be employed to further reduce or eliminate pharmaceutical pollutants. Pharmaceutical contaminants present in quantities below the capacity of the most advanced processes can be removed with metal oxide nanoparticles. The treatment processes involved depend on the phase of the contaminant (organic/inorganic), size exclusion, charge repulsion, hydrophobicity, hydrophilicity, dipole movement, and hydrogen binding capacity (Adegoke and Stenström 2019). Fe_2O_3-NPs show a wide range of anti-microbial properties (Dinesh et al 2024). This is due to the high isoelectric point – 7, hence having a higher affinity to bind to the microbial cell wall. The IONPs show anti-microbial properties by the oxidative stress generated by ROS. (Adegoke and Stenström 2019). There are various pharmaceutical pollutants targeted by the magnetic nanoparticles. Mahlaule-Glory et al. 2022discussed the preparation of Fe_3O_4 NPs by the green synthesis method from M. burkeana. It was found to have removal efficiencies of 99% for methylene blue dye and 60% for sulfisoxazole. Its action was more predominant towards Gram-positive bacteria in the water bodies.

Hussaini et al. 2023 prepared a magnetic nanocomposite using multi-walled carbon nanotubes and frankincense resin. They targeted amoxicillin, a broad-spectrum beta-lactam antibiotic, which can pose as an agent causing antibiotic resistance in aquatic microbial biomes. The group performed adsorption studies using Langmuir and Freundlich isotherms which showed that the nanocomposite was an excellent adsorbent for the remediation of amoxicillin-containing effluents having a maximum adsorption efficiency of 322.2mg/g. The Langmuir isotherm explained the adsorption process more efficiently, revealing that the process is endothermic, spontaneous, and follows a physisorption process. The nanocomposites proved to be effective for up to three cycles in adsorbing amoxicillin. Adel Naji and Tark Abd Ali 2023 developed a single-step method to prepare sand-coated magnetic nanoparticles and used the resulting nanocomposite to remove moxifloxacin and Cd^{2+} from the effluent. The coating method used green synthesis (vegetable peel extract) and the co-precipitation method. The study showed that the nanocomposite had a removal capacity of 94% for moxifloxacin and 80% for Cd^{2+} from the effluents.

12.3. Removal of Inorganic Pollutants

The rapid increase in population brings increased demand for materials for livelihood. This demand leads to increasing agricultural activities and other industrial manufacturing which may result in inappropriate waste management (Topare et al. 2023). The improper disposal of waste results in untreated water, waste, and pollutants being released into waterways, soil, and the air. Lead (Pb), mercury (Hg), silver (Ag), tin (Sn), platinum (Pt), gold (Au), copper (Ni), arsenic (As), molybdenum (Mo), nickel (Ni), vanadium (V), manganese (Mn), cobalt (Co), copper (Cu), zinc (Zn), and arsenic (As) are some of the most prevalent inorganic pollutants. Roy et al. 2021 performed a review on nanomaterials for the remediation of environmental pollutants that are dumped in water bodies causing the risk of ingestion and further complications for humans and aquatic life. Applications of modified iron oxide NPs for the removal of inorganic pollutants are presented in Table 7.

Table 6. Applications of modified IONPs for dye removal from wastewater

Adsorbent	Targeted pollutant	Mechanism	Reference
Iron oxide functionalized polyvinylidene fluoride (PVDF) membranes coated with 20% PAA	Selenium (Se), As, Ni, Hg, NO_3-	Nanofiltration	(Gui et al. 2015)
IONPs prepared from the steel pickling process	Pb $^{2+}$, Cr^{6+}	Adsorption	(Mwebembezi et al. 2024)
IONPs prepared from seed extract of Pheonix dactylifera	Cr^{6+}	Adsorption	(Kumar Chelike et al. 2024)
Zero valent IONPs derived from leaf extract of Green mulberry and Oak	As^{3+}	Adsorption	(Poguberović et al. 2016)

12.4. Antimicrobial efficiency of IONP-based materials

12.4.1. Minimum Inhibition Concentration (MIC)

To determine their lowest inhibitory concentration, samples of nanoparticles (Fe-NPs, Fe–Ag NPs, and Fe–Ag-CS NPs) that demonstrated antimicrobial activity during antibacterial screening underwent additional testing. Micro-broth dilution studies were carried out utilizing 96-well microtiter plates to assess the MICs. The MICs of NPs against the studied bacteria were calculated using the micro-diluted broth technique, as detailed by NCCLS. At 37 °C, the incubation period continued for twenty-four hours. After the incubation period, each well received 5 µl of Resazurin sodium salt dye solution (R7017 Sigma-Aldrich). The absence of microbial growth in microwells (column 11) containing only media certifies that plate contamination did not occur during dish preparation. On the other hand, additional columns 1 through 10 display the serial dilution of the NPs with medium, ranging from 1 mg/ml to 0.0019 mg/ml. This method addresses significant color and solubility issues that could impede growth evaluations for a variety of medications and helps to provide reliable MIC values. (Faiz Jaha et al. 2024).

12.4.2. Estimation of Minimal Bactericidal Concentration (MBC)

The least bactericidal concentration (MBC) is the lowest concentration of samples treated with Fe-NPs, Fe–Ag NPs, and Fe–Ag-CS NPs at which inoculation bacteria were killed. Next, 10 µl of the media from the microplate containing the MIC was spread out and incubated for 24 hours at 37 °C without showing any signs of bacterial growth. Subsequently, the nutrient agar plates were re-injected. The first well with colony counts of fewer than five, which was considered detrimental to growth, was the MBC(Faiz Jaha et al. 2024.

12.4.3. Disk Diffusion Method

Mueller Hinton Agar Media (MHA) was utilized as the growth media. MHA was supplemented with 106 colony-forming units per milliliter (CFU/ml) of several bacterial strains. Then, Fe-NPs, Fe-Ag NPs, and Fe-Ag-CS NPs were impregnated into 7-mm paper filter discs at a concentration of 1 mg/ml. The agar was then covered with these discs. The negative control used in the experiment was sterile distilled water (SDW). The nanoparticles were allowed to diffuse into the medium at room temperature for thirty minutes. After that, the plates were incubated for 24 hours at 37 °C. The average and standard deviation (SD) of three different trials were calculated to determine the zone of inhibition. (Faiz Jaha et al. 2024).

12.4.5. Biofilm formation measurement

The 96-well polystyrene plates were employed to evaluate biofilm formation by various strains of bacteria. Overnight bacterial cultures were standardized to an OD60 nm of 0.5 in LB medium and subsequently cocultured for 24 hours at 37 °C in the presence of varying concentrations (100, 50, 25, 12.5, 6.25, 3.12, and 0 μg/ml) of green-produced Fe_3O_4 NPs. A control group without MNPs was also included. Bacterial proliferation was monitored by measuring absorbance at OD600 nm using UV-Vis spectroscopy. Planktonic microorganisms were removed by repeated washing with water. Biofilms were stained with crystal violet (0.1%, v/v) for 30 minutes at 25°C, followed by washing and blotting. The extracted crystal violet was quantified at OD570 nm to assess total biofilm formation (Alavi and Karimi 2019). To ensure experimental reproducibility and statistical significance, all experiments were conducted in triplicate, with results reported as mean values ± standard deviations (SD) of three independent cultures. Tukey's test ($p \leq 0.05$) was employed to determine statistically significant inhibition of biofilm formation.

13. Safety of Iron oxide nanomaterials

A range of techniques, such as in vitro, in vivo, and ecotoxicological assays, are commonly used to evaluate iron oxide nanoparticles. Fig.13 provides a summary of these approaches, with detailed explanations in the preceding sections of this review, highlighting their role in evaluating nanoparticle toxicity

13.1. Biological Impact and Safety Risks of Engineered Iron Oxide Nanoparticles

Iron oxide nanoparticles (IONPs) are increasingly prevalent in the environment, found in various forms such as particulates from air pollution and volcanic eruptions. Their presence in emissions from industries, traffic, as well as synthetic Nano wastes has proven to have significant impact on the environment and ecosystems. As the use of IONPs grows, so does their exposure to water, air, and soil, necessitating thorough evaluation of their nanoecotoxicity in natural and experimental settings (Kalaiarasu et al 2025). Understanding the safety and environmental implications of IONPs is crucial due to their potential interactions with biological systems and the environment. Concerns regarding the interactions of synthetic iron oxide-based nanomaterials with the environment and their possible effects on aquatic ecosystems have been raised by the rising use of these materials. Though often considered non-toxic, these materials can change such as agglomeration, adsorption, dissolution, and redox reactions that alter their properties and toxicity.

Factors like pH, surface coatings, ionic strength, and natural organic matter (NOM) significantly impact nanoparticle behavior in water (Leonel et al. 2021c). For instance, pH affects the zeta potential (ZP) of NPs, influencing their stability and agglomeration tendency. Surface coatings and NOM can either stabilize or destabilize NPs depending on environmental conditions, for example, Uncoated SPIONs (Superparamagnetic Iron Oxide Nanoparticles) have low dispersibility, leading to high agglomeration rates, and appropriate coatings can stabilize the nanoparticles and prevent the release of toxic ions. Common SPION coatings include biological molecules, polyethylene glycol (PEG), dextran, and polyethylene oxide (PEO). PEG coatings reduce immunogenicity, but PEG-SPIONs have been proven to be more toxic than bare dextran SPIONs. Comparing bare

SPIONs with thin silica-coated SPIONs on A549 and HeLa cell lines showed that coated SPIONs reduce oxidative stress and lower iron ion release However, dextran-coated SPIONs can induce cell death similarly to uncoated SPIONs (Leonel et al. 2021b). When iron oxide NPs enter the environment, they engage with living things, influencing oxidation and reduction-related chemical transformation. These interactions can result in the formation of (ROS) i.e. reactive oxygen species, metal ion release, and cellular uptake, which may have cytotoxic effects. The exact mechanisms of these effects are complex and require further study (Leonel et al. 2021c). Different nanotoxicity evaluation assay methods used for Iron-based nanomaterials are presented in Fig. 13.

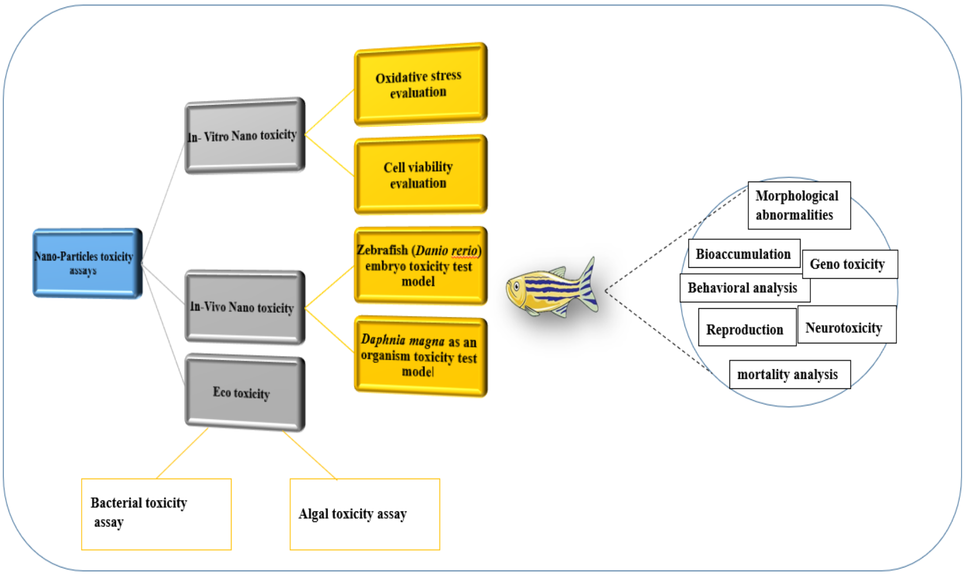

Fig.13. Different nanotoxicity evaluation assay methods used for Iron-based nanomaterials (Leonel et al. 2021c)

13.2. Assessment of in vitro Nanotoxicity

In vitro assessment of iron oxide nanoparticles (SPIONs) is critical for evaluating their uptake, biomedical fate, and cytotoxic effects. Reactive oxygen species (ROS) generation and cell viability are critical parameters for assessing cellular responses to various treatments, including nanoparticles and other stress-inducing agents. The studies highlighted demonstrate that the toxicity and cellular response to SPIONs are influenced by factors such as nanoparticle size, surface modifications, dose, and cell type. Various cytotoxicity assays on different cell lines reveal that the interaction and uptake of SPIONs depend significantly on surface charge, with positively charged SPIONs generally exhibiting higher toxicity due to greater cellular uptake. Furthermore, amine, carboxyl, and hydroxyl functional groups on the surface of nanoparticles can alter their toxicity, with hydroxyl-rich SPIONs showing lower toxicity. These in vitro assessments are vital for understanding the cellular fate of SPIONs, including their internalization, interaction with cellular organelles, and potential to induce reactive oxygen species (ROS) leading to cellular damage (Vakili-Ghartavol et al. 2020b).

13.2.1. Oxidative Stress Evaluation

Iron oxide nanoparticles (IONPs) enhance ROS production, leading to cytotoxicity and oxidative damage. This can be detected by measuring ROS, assessing oxidative damage to proteins, lipids, and DNA, and evaluating antioxidant status. IONPs induce oxidative stress in rat lymphocytes through ROS and glutathione depletion, causing dose-dependent cytotoxicity and reduced catalase activity. Thymoquinone, a natural antioxidant, was found to mitigate these harmful effects (Leonel et al. 2021b). The generation of ROS can be evaluated through multiple approaches. Gaharwar et al. 2017; Ansari et al. 2019 have demonstrated the measurement of enzymatic activity involving catalase (CAT), glutathione (GSH), and superoxide dismutase (SOD), which are crucial antioxidant enzymes. Lipid peroxidation, another indicator of oxidative stress, was assessed by (Ansari et al. 2019). Additionally, mitochondrial membrane potential changes, a marker of mitochondrial health and function, were examined by (Gaharwar et al. 2017). The use of DCF fluorescence intensity to measure intracellular ROS levels was detailed by (Leareng et al. 2020) and the MTT assay, which measures cellular metabolic activity, was utilized by Carvalho et al. 2019; Ansari et al. 2019. These varied methodologies collectively provide a comprehensive evaluation of ROS generation under experimental conditions.

13.2.2. Cell Viability Evaluation

Cell viability assessments of iron oxide nanoparticles (IONPs) often use colorimetric assays to evaluate potential toxicity. Techniques like the MTT assay measure mitochondrial activity by quantifying formazan crystals formed by viable cells, while trypan blue (TB) and lactate dehydrogenase (LDH) assays assess membrane integrity and cell damage. Although these methods provide valuable insights into nanoparticle-induced cytotoxicity, direct comparison across studies is challenging due to variations in experimental conditions, nanoparticle properties, and cell types (Deda et al. 2017; Leonel et al. 2021b).

13.3. Assessment of in vivo Nanotoxicity

Animal models are used in in vivo assessments of iron oxide nanoparticles (IONPs) in order to determine toxicity and possible health impacts. Typically, adult rats are exposed to IONPs through methods such as intratracheal instillation. The rats are divided into different groups, including untreated controls and various dose groups, with body weight and organ weights monitored throughout the study. Histopathological examinations, including organ weight measurements and tissue staining, are conducted to detect any abnormalities or damage. Intra-tracheal instillation of low-dose (LD) and high-dose (HD) iron oxide nanoparticles (IONPs) caused reduced body weight gain and significant lung weight decrease in rats, with inflammation and mild pulmonary fibrosis observed, especially in the HD group. Liver and kidney weights initially decreased but normalized over time, emphasizing the potential pulmonary toxicity of IONPs (Szalay et al. 2012). These studies help identify significant changes in body weight, organ weight, and tissue pathology associated with nanoparticle exposure.

Such evaluations are crucial to understanding the environmental risks and potential health impacts of IONPs (Szalay et al. 2012; Leonel et al. 2021b).

13.3.1. Zebrafish (Danio rerio) embryo toxicity test model

The zebrafish (Danio rerio) is highly suitable for evaluating iron oxide nanoparticle (IONP) toxicity due to its rapid development, large egg production, and genetic similarity to humans. Zebrafish are superior to mammalian models in terms of cost, time efficiency, and sensitivity, according to studies. The model has been used extensively for nanotoxicity assessment, including investigations into the effects of maghemite nanoparticles. Zebrafish embryos exposed to various concentrations of IONPs showed concentration-dependent mortality and indicated that both static and semi-static exposure conditions influence nanotoxicity. Despite high mortality rates at higher concentrations, IONPs did not affect the hatching rate, highlighting the need for specific guidelines for nanoparticle tests (Pereira et al. 2020a). In zebrafish, morphological abnormalities are assessed as indicators of developmental toxicity, as reported by Chakraborty et al. 2022. Bioaccumulation and biodistribution studies, which track the internal distribution and accumulation of nanoparticles, were detailed by Haque and Ward (2018). Behavioral analyses, including swimming kinetics and spatial recognition, provide insights into the neurobehavioral effects of nanoparticles and were covered by both Haque and Ward 2018; Chakraborty et al. 2022. Reproduction and mortality analyses offer critical data on the reproductive toxicity and overall survival impact, as highlighted in studies by Haque and Ward 2018; Chakraborty et al. 2022. Additionally, genotoxicity and neurotoxicity evaluations, such as those conducted by Pereira et al. 2020, and assessments of endocrine disruption, as reported by Pereira et al. 2020b; Chakraborty et al. 2022 provide further insight into the toxicological effects at the genetic and hormonal levels.

13.3.2. Daphnia magna organism toxicity test model

Daphnia magna, a tiny planktonic crustacean, serves as a crucial model for Eco toxicological research because of its regular parthenogenetic life cycle and the simplicity of its cultivation and handling. Research has shown that different iron oxide nanoparticles, such as magnetite and hematite, lead to distinct toxicity outcomes due to variations in their physicochemical properties. For example, Fe_3O_4 nanoparticles demonstrated higher dissolution and oxidation rates, causing significant metabolism disturbances within the organism. Additionally, the surface reactivity of nanoparticles can influence their toxicity, with effects linked to specific bio-interactions with reactive surfaces. These findings highlight the critical relevance of nanoparticle composition and surface chemistry in assessing Ecotoxicological risks in aquatic systems (Leonel et al. 2021b). Daphnia magna, bioaccumulation, and biodistribution studies by Kwon et al. 2014 help in understanding the internal concentrations of nanoparticles. Morphological changes, which can signal physical deformities or stress responses, were documented by Valdiglesias et al. 2016. Physiological changes, including swimming motility, vertical migration, and feeding rates, were examined by Prajitha et al. 2019to assess the impact on vital physiological functions. Reproduction and mortality analyses, which are essential for understanding the long-term effects on population dynamics, were also reported by Valdiglesias et al. 2016. These methods collectively offer a robust framework for assessing the in-vivo toxicity of iron oxide nanoparticles in aquatic models.

13.4. Assessment of Ecotoxicity

The use of iron oxide nanoparticles (IONPs) in water remediation has attracted a lot of interest; however, their potential Eco toxicity poses substantial risks to aquatic ecosystems. These manufactured nanoparticles can be introduced into freshwater and marine habitats. lead to adverse effects across various trophic levels due to processes such as bioconcentration and biomagnification. Once in the environment, IONPs can accumulate in organisms, potentially disrupting physiological processes and leading to toxicological outcomes. The persistence of these nanoparticles in aquatic systems can result in the gradual accumulation in higher trophic levels, amplifying their impact on the ecosystem. Consequently, assessing the Ecotoxicity of iron oxide nanoparticles is imperative to mitigate their environmental footprint and safeguard water resources. Understanding the interactions of IONPs with aquatic organisms and the broader ecosystem is essential to developing effective strategies for minimizing their adverse effects and ensuring sustainable use of these materials (Leonel et al. 2021b).

13.4.1. Bacterial toxicity assay

Bacteria are essential in aquatic ecosystems and are used to quickly screen for water contaminants due to their rapid response to environmental changes. Bacterial assays assess toxicity by measuring changes in growth or cell viability, often through absorbance analysis or agar plate inhibition zones. Studies have shown that metal oxide nanoparticles can inhibit bacterial growth, with the effect depending on nanoparticle-bacteria interactions (Jadhav et al. 2023). For example, chitosan-coated magnetite nanoparticles were more toxic to bacteria because the positive charge of chitosan attracted the nanoparticles more strongly to the bacterial surfaces, leading to increased bacterial death (Leonel et al. 2021b). The test used for the evaluation of iron-based nanomaterials in this regard was discussed in section 12.4 as the antimicrobial efficiency of IONP-based materials.

13.4.2. Algal toxicity assay

Algae, as primary producers in aquatic ecosystems, play a crucial role in assessing water quality due to their sensitivity to pollutants. Algal assays, which measure parameters like fluorescence and cell counting, are effective for evaluating the impact of iron-based nanoparticles on growth. Research has shown that factors such as nanoparticle size, crystal phase, and oxidation state significantly affect algal toxicity through mechanisms like oxidative stress and interactions with nanoparticles. Nanosized iron oxides, in particular, exhibit greater toxicity than bulk forms due to increased internalization and oxidative stress (Leonel et al. 2021b). Additionally, a study by Gambardella et al. 2014 found that metal oxide nanoparticles, including Fe_3O_4, caused significant toxic effects when marine microalgae (Cricosphaera elongata) contaminated with 5 mg L^{-1} of various NPs were fed to sea urchin larvae (Paracentrotus lividus). The study observed reduced larval survival and abnormal development, indicating that metal oxide NPs can adversely affect aquatic organisms and potentially enter the food chain.

13.5. Factors that Influence the toxicity of Nanoparticles

Size and shape: When the surface area rises and the particle size drops, there will be an increase in the reactivity of molecules, leading to unsatisfied bonding and faster diffusion in the gastrointestinal tract. Smaller particles can cause necrosis and apoptosis. Nanoparticles can easily enter the bloodstream and reach mucosal and lymphatic tissues (Suh et al. 2009). Sukhanova et al. 2018 show smaller nanoparticles can easily cross cell barriers. Particles smaller than 25 nm can cross membranes via pinocytosis/endocytosis (Zhang et al., 2015; Sahay et al., 2010; Cypriyana P J et al. 2021).

Surface chemistry: Different metals have various surface chemistry, which greatly influences the toxicity of their nanoparticles. For instance, the surface coating and charge of superparamagnetic iron oxide nanoparticles (SPIONs) affect their interaction with biological systems. (Shagholani et al., 2018). Coatings such as polyethylene glycol (PEG), dextran, and polyethylene oxide (PEO) are commonly used to stabilize SPIONs (Sadeghiani et al., 2005; Lacava, 2001; Mojica et al., 2014). However, different coatings can lead to varying toxic responses. It is important to conduct further studies to understand better the mechanisms underlying the toxicity of SPIONs (Berry et al., 2003; Cypriyana P J et al. 2021).

14. HANDLING AND DISPOSAL OF IRON OXIDE NANOMATERIALS

The Nanotechnology Safety and Health Program of the Office of Research Services Division of Occupational Health of the National Institutes of Health provides guidelines for mitigating occupational exposure risk and safeguarding oneself using personal protective equipment (PPE), gloves, respirators, dust masks, or surgical masks (2014). Guidelines and best practices for properly handling nanomaterials in research labs and industry are provided by nanoscience and technology. That study states that, in the hazard area designated by the relevant authorities, milligram ranges of nanomaterials should be disposed of in sealed containers that are adequately labeled and removed by following the usual method (Centre for Knowledge Management of Nanoscience and Technology, 2016). Different methods can be used for the disposal of the nanomaterials.

15. W&WT APPLICATIONS OF IONP BASED NANOMATERIAL

Various specialized applications of IONP-based nanomaterials for W&WT were discussed in previous sections i. e. section 7, section 8, section 10, and 12. In section 7 various synergistic applications of the materials such as synergistic oil removal/ recovery with microbial species, the synergistic photocatalytic effect of other metals doped IONPs, synergistic thermal degradation of dyes by α-Fe_2O_3-CuO NC and the synergistic effect of ferrate (VI) within the degradation of antibiotic pollutant discussed. In section 8, specific mechanism-based applications of water remediation by IONP-based nanomaterials such as adsorption/ desorption, photocatalysis, photo-Fenton processes, electrochemical processes, chemical oxidation, and alternative advanced oxidation processes and sulfate radical-based advanced oxidation processes (AOPs) were discussed. In section 10, IONPs-based methodologies for W&WT such as Nano, Ultra, Reverse Osmosis (RO) filtration systems, MXene Composite Material for Ultra and RO filtration, Iron Oxide Functionalized Membranes for Toxic Metal Removal from Power Plant Scrubber Water, 3D Printed Filtration System and micro/nanorobots for water remediation were discussed. A few applications based on methods for evaluation of water treatment efficiency of Nanomaterials were discussed in section 12. Nanotechnology allows for the modification of materials at the nanoscale to achieve specific properties and functions. Iron oxide nanoparticles are widely used in wastewater treatment due to their low cost, high surface area, strong adsorption capacity, and ability to be separated using external magnetic fields. They are effective in pollution control, sensing and detection, and treatment and cleanup. A few more applications of IONP-based nanomaterials are discussed.

15.1. IONP-based removal of ammonia from Fish Culture Tanks and microplastic

Since ammonia buildup in fish culture tanks can be harmful to aquatic ecosystems, it is a serious concern. Saharan et al. 2014 suggested the traditional techniques for removing ammonia, such as chemical treatment and biological filtration, can be resource-intensive and may not always work (Tang et al.; Hao et al. 2024). Using iron oxide nanoparticles, which have been demonstrated to successfully remove a variety of pollutants, including ammonia, from aqueous environments, is one of the promising substitutes (Saharan et al. 2014). Although research on the application of iron oxide nanoparticles for ammonia removal in fish culture tanks is still in its infancy, earlier investigations have produced encouraging findings (Mohamed et al. 2023).

Aquaculture is one of the emerging fields in food production. Maintaining the water's quality is crucial in this rapidly expanding field to ensure sustainable fish production. Barik et al. 2023 discussed the synthesis of Fe nanoparticles using Bacillus megaterium collected from the soil samples. The bacteria sample was isolated and identified and its 16S rRNA gene PCR-amplified sequences confirmed the identification of the bacteria. Further, it was subjected to resuspension in FeCl3 which resulted in the formation of Fe nanoparticles. The synthesized nanoparticles were characterized using UV-Vis, FTIR, XRD, DLS-zeta potential, and TEM analysis. Their efficacy to remove ammonia was evaluated under ex-situ and in-situ conditions from Common carp, and Cyprinus carpio culture tanks. The ammonia was removed from the tanks by the chemisorption mechanism. Despite recent developments in this area, it is still unknown how effectively nanoparticles work to remove ammonia from fish culture tanks.

Various industries have increased their use and demand for plastics because of the versatility, durability, resistance, and cost-effectiveness of manufacturing their products. Plastics are essential to packaging, automotive parts, electronics, and medical devices. Plastics are supported with advanced manufacturing techniques that allow them to be utilized according to desired properties as required in the industrial sector. E-commerce is growing, as well as consumer goods production has exacerbated consumption.

However, this rise also raised environmental concerns, encouraging research into more sustainable solutions (Heo et al. 2022a). Because of their microscopic size, microplastics can evade the majority of filtration systems, making their removal from water extremely challenging. Heo et al. 2022b assessed Magnetic iron oxide (Fe_3O_4) nanoparticles as viable options for the adsorptive removal of microplastics. Their study focused on the possibility of using magnetic Fe_3O_4 nanoparticles to filter the water of microplastics through adsorption. They used polystyrene (PS) microparticles to model the behavior of microplastics. The researchers investigated the isothermal adsorptive properties and process kinetics of polystyrene microparticles on Fe_3O_4 nanoparticles. They additionally conducted adsorptive tests for distinct groups of polystyrene microparticles with variable average diameters to look into the adsorbing efficacy of Fe_3O_4 nanoparticles. Fe_3O_4 and polystyrene adsorption involve both hydrophobic and electrostatic dynamics.

15.2. Oil field application

Enhanced Oil Recovery (EOR): IONPs have gained attention in enhanced oil recovery (EOR) due to their unique properties. IONP-based nanomaterials have very useful properties and their application in oil recovery was reviewed and their effectiveness was compared with other NPs. Role of IONPs in surface coatings, challenges in reservoir applications, and potential solutions. Surface treatment has shown its higher potential applications to enhance stability, transport, and minimize rock adsorption for increased oil recovery. Thus, IONP-based nanomaterials are economical, ecologically benign, and have potential for oil field applications. (Yakasai et al. 2022). Oil Droplet Removal: IONPs find applications for removing oil droplets from water in oilfields through coagulation and flocculation procedures (Jabbar et al. 2022).

15.3. Coagulation and Turbidity Removal

Almarasy et al. 2019 investigated and synthesized hematite iron NPs (α-Fe_2O_3) removal of turbidity. The efficacy of the NPs was compared with alum samples. The study revealed 93.8% turbidity removal by α-Fe_2O_3 NPs. In another reviewed study on the use of magnetic nanoparticles in water treatment, the findings highlighted the benefits of integrating magnetic nanoparticles in the coagulation/flocculation process. The finding revealed various benefits of IONP materials towards coagulant/flocculant-based W&WT. Better coagulant/flocculant by IONPs was achieved due to high charge density (Fe^{2+} and Fe^{3+}) in a shorter time. Recovery of magnetic NPs and coagulant/flocculant can be achieved using an external magnetic field providing a scale of economy (Mohamed Noor et al. 2022). The Iron-loaded zeolites and ozonation (O_3/Fe-ZA) is a new household-level treatment that removes arsenic and bacteria from tap water using a combination of iron-loaded zeolites and an ozonation process (Ikhlaq et al. 2021).

16. FUTURE PROSPECTS

Although there has been significant advancement, magnetic nanoparticles are still mostly used in laboratories, with only a few marketed technologies. Using magnetic nano-scavengers for wastewater treatment poses a significant challenge due to concerns about human health and environmental danger. There have been reports of toxicity associated with the usage of these nanoparticles, maybe that is the reason why they are not widely accepted as a treatment for wastewater. Practical uses of enhanced co-precipitation and hydrothermal procedures provide challenges for large-scale synthesis. Green synthesis is an essential goal for establishing the manufacture of sustainable and economical adsorbents. Furthermore, in order to facilitate their actual application in water treatment technologies, scalable and reasonably priced production procedures are being created. Fe3O4 adsorbents must be functionalized with surface agents to increase their adsorption capacity, stability, efficiency, and selectivity against pollutants.

Recent advancements in the application of iron oxide nanoparticles (IONPs) for wastewater treatment have illuminated potential challenges and avenues for future research in this field. The study also identified potential obstacles and solutions related to the long-term sustainability and environmental impact of such water purification systems. Research has demonstrated the great potential of ferrofluid, magnetic ionic liquids, magnetic microrobots, and nanorobots for water remediation. To meet commercial demands, however, the translation of this technology into practical applications would require the collaborative efforts of scientists from diverse fields, such as engineers, physicists, chemists, and biologists.

Future studies should focus on selectively removing certain pollutants from industrial effluents using low-cost and efficient technologies. To ensure industrial-scale application, a multidisciplinary approach involving relationships between enterprises, academic research centers, and governments is important. Also focus should be on developing magnetic nano adsorbents that are selective for specific industrial applications. Exploring these materials for also pollutant, as previous studies have removed multiple contaminants simultaneously.

17. CONCLUSIONS

The review emphasizes the great potential of iron oxide nanoparticles (IONPs) in a range of applications, especially biomedicine and environmental remediation. Understanding the composition, structure, and thermal stability of IONPs is essential for optimizing their functionality and effectiveness.

1. One of the primary issues with conventional water treatment procedures is the removal of a wide range of pollutants, which IONPs can effectively address because to their special magnetic properties.
2. Improved pollutant adsorption and recovery methods are now possible due to the considerable improvements in IONP performance and application brought about by synthesis methodology innovations such as green synthesis and surface functionalization.
3. The scalability and economic viability of IONP technologies must be the primary focus of ongoing research in order to enable their wider use in industrial and municipal wastewater treatment systems.

4. Ultimately, addressing the global water crisis and preserving a healthy planet for future generations need the implementation of iron oxide nanoparticles.

18. ABBREVIATIONS

AOPs, Advanced oxidation processes; CED, Cathodic Electrochemical deposition; CFU/ml, Colony-forming units per milliliter; EAOPs, Electricity-driven advanced oxidation processes; IONMs, Iron oxide nanomaterials; MION, Magnetic iron oxide; MMM, Mixed matrix membrane; MNMs, Micro/nanomotors; MOFs, Metal oxide frameworks; MONPs, Metal oxide nanoparticles; MXI, MXene Iron oxide; N-N, Nitrate or nitrite; PAA, Polyacrylic acid; PEG, Polyethylene glycol; PEI, Polyethyleneimine; PVDF, Polyvinylidene fluoride; RO, Reverse Osmosis; ROS, Reactive oxygen species; SPIONs, Superparamagnetic iron oxide nanoparticles; TKN, Total Kjeldahl nitrogen; TP, Total phosphorous; UFM, Ultra filtration membranes; W&WT, Water and wastewater treatment methods.

Author Contributions: Niraj S. Topare, and Vishnu P. Choudhari conceived the presented idea, and they wrote the manuscript with support from Saumya M. Mankad, Sakshi C. Chaudhari, Saloni P. Ghodmare, Rohan R. Mahajan, Siddhi H. Susladkar. Niraj S. Topare, Vishnu P. Choudhari, Satish Khedkar, Prashant Thorat have supervised the whole process. All authors read and approved the final manuscript.

Funding: Not applicable.

Institutional Review Board Statement: Not applicable

Conflicts of Interest: The authors declare no competing interests

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