# Magnetic Nanohybrid Materials for Water Purification

**Anindita Bhuyan and Md. Ahmaruzzaman**

**Abstract** In recent years, the constant population growth worldwide has put pressure on the need for clean and safe water for human consumption. Magnetic nanomaterials (MNMs) have emerged as a promising material in the past few decades due to their unique physiochemical properties. MNMs possess the desired characteristics for their application in water purification. However, it is important to develop well defined magnetic nanomaterials for efficient removal of water pollutants. This requires effective synthetic methods for the synthesis of shape, size and morphology-controlled nanomaterials. Various physical, chemical and biological methods including ball milling, gas phase deposition, thermal decomposition, hydrothermal, solvothermal, co-precipitation, sol–gel, etc. have been explored over the years for their synthesis. The presence of organic and inorganic pollutants in water even in trace concentration has extreme adverse effects on human and environmental health. Hence, it is the need of the hour to develop effective and economical methods for application in water remediation. In this chapter, the problem of water pollutants, their threatening effects and the use of MNMs in water purification have been addressed. These MNMs are characterized using methods such as UV, IR, XPS, XRD, SEM, TEM, VSM, AFM, RS, $^{1}$HNMR, etc. Finally, the application of these materials for water purification have been discussed in detail—highlighting the removal of pesticides, dyes, pharmaceutical drugs, inorganic anions, heavy metals and oil spill from water.

**Keywords** Magnetic nanomaterials · Water remediation · Morphology

## 1 Introduction

Nanotechnology is one of the most emerging and widely researched areas in modern times. It aims at developing new materials and devices of structures having nanoscale dimension. Nanoscience explores materials in the nanoscale range (1–100 nm) in atleast one dimension. Confined structure and dimensionality of nanomaterials

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makes them an attractive as well as a promising material for various applications in industries, water remediation and health care sector among others.

Today water pollution is one of the major concerning environmental issues. Water covers a vast majority of the earth surface, yet only a small amount is fit for human consumption. Modernization and industrialization have led to the widespread influx of pollutant into the water bodies leading to serious environmental consequences including adverse effect on human health. Some of the major contributors for water contaminants are industrial effluent, agricultural runoff, domestic sewage and hospital effluent. Contaminants are present as inorganic, organic and biological form and it is extremely crucial to implement methods to purify wastewater [63].

The removal of these pollutants is a quite challenging field and possesses serious issues. One of the major concerns is the removal of contaminants present in low concentrations which are often missed out in conventional water treatment methodologies. Nanotechnology is able to solve this problem to quite an appreciable level owing to their unique properties which include size selective adsorbent phenomenon and quantum confinement. Nanomaterials can be utilized for efficient uptake of organic (dyes pharmaceuticals, pesticides and insecticides), inorganic (heavy metals) and biological (microbes) pollutants. Another issue in the removal of toxic pollutants is to ensure that no degradation by-product and nanomaterial residue are left behind in the water source. Henceforth, it is necessary for the nanomaterials to not only have high affinity toward pollutants for their efficient removal but it is also important to develop materials that are easy to recover from the purified water. Magnetic nanomaterials can be a promising candidate for the water purification purpose [41].

## 2 Magnetic Nanomaterials

One class of nanomaterials that have attracted intensive research in recent times is magnetic nanomaterials (MNMs). These nanomaterials are utilised in variety of applications such as catalysis, chemical sensors, magnetic hyperthermia, drug delivery, magnetic resonance imagimg, etc. (Fig. 1). MNMs are generally composed of one magnetic element which can either be a monocomponent such as (i) metallic nanoparticles like Fe, Co or Ni (ii) metal alloys such as FePd or FePt (iii) metal oxides such as $Fe_3O_4$, $Fe_2O_3$ (iv) metal carbides like $Fe_2C$ or $Fe_3C_2$ or they can be bicomponent materials like $CoFe_2O_4$ and $NiFe_2O_4$ They can also be heterostructure materials such as $Fe_3O_4$@Ag, $Fe_3O_4$@$SiO_2$, $Fe_3O_4$@biochar, etc. [136].

Magnetic particles in nanoscale range display unique physical and chemical properties. Owing to their small size, large surface area and high porosity, these materials have gained interest in their widespread application in adsorption and catalysis. These materials can be easily synthesized and modified into various nanocomposites for targeted applications in different fields. Further, these nanomaterials can be easily controlled using an external magnetic field. Conventional magnetic materials generally lose their permanent magnetic properties when they are reduced to nanoscale range. Superparamagnetic nanoparticles (NPs) show magnetic properties only in

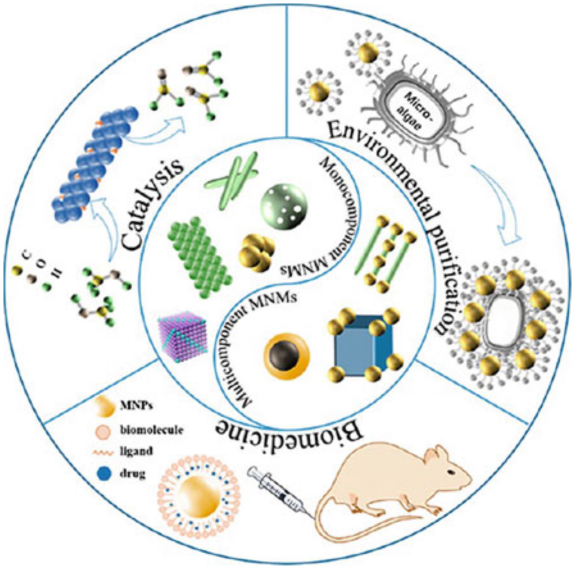


**Fig. 1** Application of magnetic nanomaterial in environmental purification, catalysis and biomedicine (Reprinted with permission from [136])

the presence of external magnetic field (EMF). Hence, MNMs can also be used in biological mediums for biomedical application.

Iron based nanomaterials constitute the most widely studied magnetic particles. They show excellent magnetization and biocompatibility. Further they show unique properties including superparamagnetism, high crystallinity, coercivity, high specific surface area and dispersity [4]. However, these materials are prone to agglomeration and corrosion. The MNMs are often capped with polymers like polyethylene glycol or incorporated with inorganic components like activated carbon, silica, alumina, Au, or Pt. This increases the stability of the MNMs and prevents the agglomeration, oxidation and provides protection against corrosion. The properties of these materials are highly dependent on the size, shape and morphology of the NPs. The synthesis methods and conditions thereby influence their properties and application.

## 3 Synthesis Techniques

It is quite necessary to obtain well-defined structure and morphology of MNMs to achieve its unique properties. Over the years, synthesis of MNM has been well developed. A Variety of methods such as hydrothermal, solvothermal, co-precipitation, sol–gel process, thermal decomposition, etc. have been utilized in the preparation of MNMs. The size, shape and morphology can be controlled by varying the reaction parameters. MNMs can be synthesized either by using physical methods (milling), chemical methods (hydrothermal, co-precipitation, sol–gel) [55, 72] or biological methods. Some of the commonly used chemical synthesis method includes.

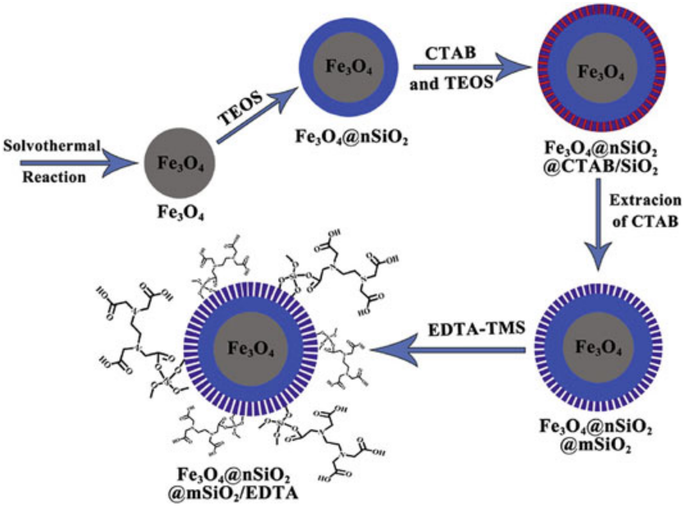


**Fig. 2** Schematic diagram of the preparation of $Fe_3O_4$@n$SiO_2$@m$SiO_2$/EDTA (Reprinted with permission from [119]

## *3.1 Hydrothermal/Solvothermal*

Hydrothermal method is one of the most commonly used synthetic approaches used in the synthesis of shape-controlled and stable nanoparticles [92]. Generally, hydrothermal method is carried out in a reaction chamber at high temperature and pressure. The solvothermal method [119] follows basically the same principle as hydrothermal with the only difference in the solvent used (Fig. 2). Elemental metals, metal oxides and alloys have all been synthesized using this route. A novel hydrothermal synthesis of $\gamma$-$Fe_2O_3$, $Fe_3O_4$ and $\alpha$-$Fe_2O_3$ [54] was reported by Jayanthi et al.

## *3.2 Sol–Gel Method*

Sol-gel method is based on hydroxylation and condensation of precursors. Gao et al. reported the synthesis of $Fe_3O_4$ using this method [42]. Small sized and monodispersed nanoparticlesof nearly 8 nm were obtained. This method has also been used in the synthesis of Co-Cr-Ferrite with excellent properties [71]. Some other synthesis of magnetic nanoparticles such as $Fe_3C/C$ [120], $NiFe_2O_4$ [112] $Mg_{0.5}Zn_{0.5}FeMnO_4$ [86] etc. have been reported using sol-gel method.

### 3.3 Co-Precipitation

It is a very common method used in the synthesis of $Fe_3O_4$ [25]. The process generally involves preparation of iron oxide nanoparticles from aqueous solution of ferrous and ferric salt by addition of alkaline substances [79]. Although this method is easy to use, it has poor control over size and shape of the nanoparticles. However, the addition of surfactant or polymer coating helps control the size of the nanoparticles [38, 81].

### 3.4 Thermal Decomposition

This method generally involves the decomposition of organometallic precursors using capping agents (surfactants) in the presence of organic solvents. The synthesis processes are carried out under anaerobic conditions. Manipulation of reaction conditions like reaction temperature, reaction time and annealing temperatures helps to control the morphology and size of the nanoparticles [31]. The advantage of this method is that it is useful to synthesize morphology controlled, high crystalline NPs in high yields [114]. However, the use of surfactants during synthesis process makes it difficult for further surface modification for improved properties [78].

### 3.5 Polyol Method

It is a liquid phase synthesis method, where polyols (example: ethylene glycol or N-methyldiethanolamine) simultaneously acts as solvent, surfactants and reducing agent. Here precursors are dispersed in polyol and reflexed at high temperature [23]. This method is especially useful to synthesize uniform magnetic nanoparticles as well as elemental NPs such as Fe, Co and Ni from their oxides [103]. The morphology and size of the NPs can also be modified by changing the reaction time or the solvent used. MNMs possessing interesting properties have been synthesized using this method.

### 3.6 Biological Induced Methods

Magnetic nanomaterials are also synthesized via biological systems. Simple, eco-friendly and cost-effective techniques make it an emerging area of research. Nanomaterials are either synthesized using plant extracts as precursors or the biological entities are used as stabilizing agents and modification for the magnetic nanomaterials. Siddiqui and Chaudhry used a seed mediated coprecipitation technique to grow $MnFe_2O_4$ on *Nigella sativa* plant seeds (Black cumin) [50]. The composite was applied for adsorption of organic pollutants (methylene blue) and also inhibits bacterial growth. Huang et al. carried out an enzyme immobilization on magnetic chitin nanofibre composite (MCNC) [124]. Enzymes being excellent biocatalyst, these nanomaterials can be applied onto various applications. Mixed fungal biomass coated with magnetic nanoparticles (MNP-FB) [100] were used for the removal of toxic Cr(VI) ions from aqueous solution. They isolated two fungal biomass, namely *Aspergillus fumigatus* and *Aspergillus niger* and used them as precursor for synthesis of an adsorbent. A maximum adsorption of 249.9 mg/g was achieved indicating it to be a proficient and environment friendly adsorbent material.

Several other physical, chemical and biological methods are also explored for the synthesis of magnetic nanomaterials especially super-paramagnetic iron oxide nanoparticles (SPIONS), as depicted in Fig. 3.

## 4 Characterization Techniques

The properties of MNMs are directly influenced by size, shape and structures of the nanoparticles. Several methods are adopted for analysis and characterization of the MNMs.

Fourier Transform Infrared Spectroscopy (FTIR) is a spectroscopic method used to determine the quality of material as well as used to confirm the identity and presence of functional groups present in the modified MNMs. The stretching vibration of Fe–O bond of iron oxide NPs changes on the formation of nanocomposites with other materials such as $SiO_2$ or ZnO [133], thereby confirming the formation of hybrid composites. $Fe_3O_4$ synthesized by Vojoudi *et al.* showed adsorption band around 457 and 573 $cm^{-1}$ which correspond to Fe–O stretching frequency [117]. However, after coating the iron oxide with mesoporous silica, new adsorption peaks around 805 and 1095 $cm^{-1}$ were obtained. Further, by analyzing the formation and nature of bonds formed between the adsorbent material and the adsorbate (substrate), the mechanism of adsorption can also be determined.

The size of the magnetic NPs can be determined by several methods such as X-ray diffraction (XRD), Transmission Electron Microscopy (TEM) and dynamic light scattering (DLS). DLS method determines the size of the MNMs where the nanoparticle suspension is exposed to an electromagnetic wave and scattering is recorded. Apart from size, the crystalline structure of the MNMs can be analyzed using XRD as the diffraction patterns correspond to the plane of the NPs [73].

TEM is one of the best methods to analyze the size of nanomaterials. TEM is based on the technique where an electron beam is transmitted through the specimen, and these electrons interact with the specimen as they pass through it. It is used for the determination of internal structure of nanomaterials. The core dimension and coating dimension directly influence the magnetic nature of the magnetic NPs. The major disadvantage of TEM is the tedious sample preparation process and selection of suitable volatile and inert solution for sample preparation. For core shell MNMs, TEM can be used to clearly observe and confirm the core–shell structures [57]. TEM images also successfully show the presence of any agglomeration that may occur in

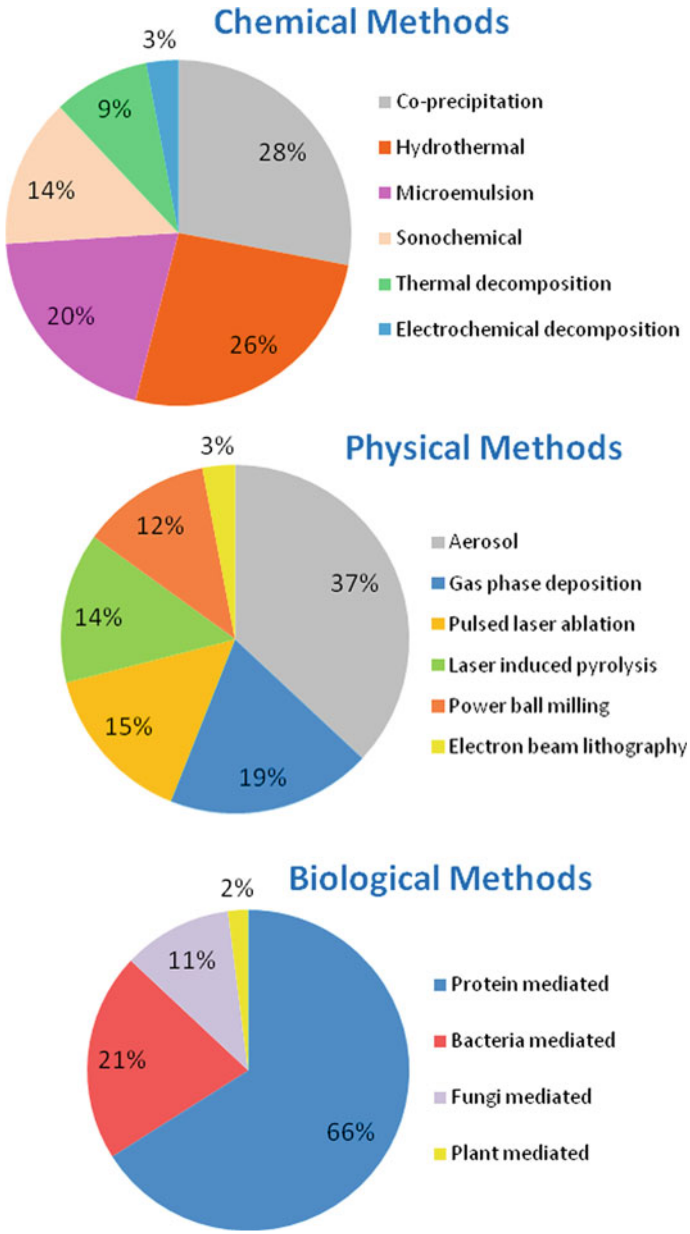


**Fig. 3** Synthesis of SPIONs by three different routes viz., Physical, chemical and biological (Preprinted with permission from [5])

the MNM dispersion. Moreover, it is also helpful to study the effect of core shell on the agglomeration of the MNMs.

Energy dispersive X-ray (EDX) analysis of TEM images confirms the presence of the various elements present in the magnetic nanocomposites. Moreover, the weight percentage of the components and nanomaterials as a whole can also be determined. It is also useful in the detection of any impurities that may be present in the MNMs. Scanning electron Microscopy (SEM) detects high energy electron emitted from the surface of the nanoparticles and creates surface images. The SEM image analysis determines the surface morphology of the MNMs. The layers of the NPs and the surface, whether planar, smooth or rough; regular or irregular can all be observed by studying the images.

Vibrating sample magnetometry (VSM) analysis is a very important technique to study the magnetic properties of magnetic nanomaterials. VSM plots the variation of magnetization (M) against the applied magnetic field. Here, the magnetic hysteresis loop is studied at room temperature. Magnetic characteristic of the MNM, whether ferromagnetic or superpaarmagnetic can be derived from the readings. Moreover, magnetic properties variation with core size can also be studied using VSM. It is also helpful to quantify the number of magnetic components in MNMs. This technique thus helps to not only understand the magnetic properties but also considers magnetic accountability, recovery and re-dispersible nature of the nanoparticles for their potential application as suitable magnetic nanomaterials in different fields [126].

Brunauer–Emmett–Teller (BET) analysis is a method used to determine the specific surface area (SSA), pore diameter and volume of the MNMs. The pore size and SSA is a major parameter which determines the adsorption capacities of the MNMs.

Photoluminescence (PL) spectroscopy helps to understand the transport, migration and recombination of electron–hole pairs. Understanding the recombination process and electron transfer helps to study the photocatalytic activity of the MNMs [46].

Zeta potential study is also carried out to study the surface charge of the nanocomposite which further helps to understand the stability of the NCs [48].

X-ray photoelectron spectroscopy (XPS) studies deals with surface characterization and help to determine the chemical composition and electronic state of the individual elements in the hybrid nanocomposites.

Raman spectroscopy (RS) [24], Thermogravimetric Analysis (TGA) [113], Elemental mapping, Atomic Force Microscopy (AFM) [43], Scanning Tunneling Microscopy (STM), $^1$HNMR [99] are some other methods used for analysis of magnetic nanomaterials and their interaction with the targeted substrate materials.

## 5 Application in Water Purification

Water contamination is one of the major environmental issues in recent era. Millions of deaths in human are caused every year due to inadequate or unsafe access to pure water. Consumption of polluted water leads to a variety of diseases and even deaths of living organisms. It also leads to deterioration of natural environment in various aspects. Hence, it is very important to seek removal of these toxic contaminants as a topic of critical concern.

Recently, magnetic nanomaterials have gained interest for their unique physiochemical properties. These nanomaterials show high adsorption and removal efficiency towards toxic contaminants. In addition, easy recovery and reusability of the MNMs makes them an efficient and cost-effective nanomaterial. Magnetic nanomaterials have been widely explored for their adsorption and catalytic action toward various types of pollutants (Fig. 4). Organic pollutants including pesticides, fertilizers, dyes pharmaceutical and personal care products; inorganic metals and anions, biological pathogens impose major threat to the environment as well as human health. MNMs through their adsorption and catalytic properties form a suitable candidate for waste water remediation.

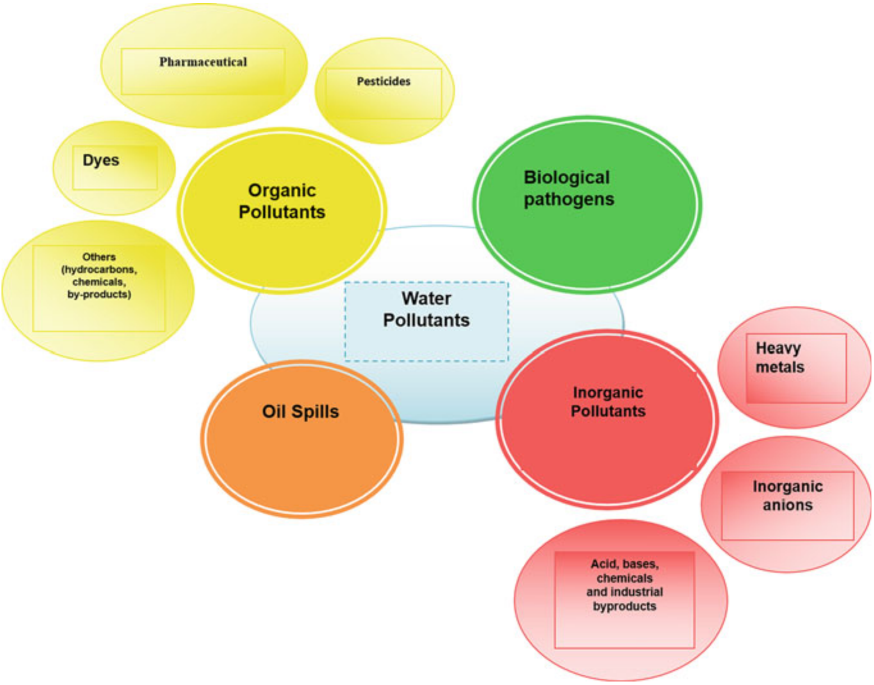


**Fig. 4** Overview of magnetic nanomaterial application in water purification

## 5.1 *Organic Pollutants*

Organic pollutants can be considered a critical environmental threat due to its persistent nature. Organic pollutants are hence often referred to as persistent organic pollutants (POPs). These POPs can bioaccumulate and undergo biomagnifications as it enters the food chain, thereby harming not only the aquatic life but also causing adverse effect on human. Unlike inorganic pollutants which mainly include heavy metals, cations and anions, organic pollutants comprise of a wide range of compounds and functional groups having different chemical and physical properties. Organic pollutants include hydrocarbons, dyes, pesticides, insecticides, herbicides, pharmaceuticals, health and personal care products, detergents and other toxic chemicals used in our everyday life.

### 5.1.1 Pesticides/Insecticides

These chemicals are used in agriculture to protect the crops from unwanted weeds, insects and mites. Increasing population leads to the increase in food demand which leads to extensive use of chemicals for increased production. However, this has led to some serious pollution issues of soil and water alike. The pesticides and insecticides enter the water bodies via rainwater and irrigation runoffs. They also make way into the groundwater through leaching. These chemicals are highly toxic and non-biodegradable. Once they enter the food chain they lead to biomagnifications and can adversely affect human health. Not only human, but marine life, birds and other animals can also be affected. Long term exposure can cause various health problems including imbalance in hormonal activity, birth defects, nervous and reproductive diseases and even cancer and death. The most commonly used class of pesticides is organophosphorus pesticides (OPPs). They include diazinon, chloropyrifos, parathion, ethion, malathion, profenofos, etc.

Nanomaterials have been used in the adsorption and degradation of these chemicals due to their high sensitivity as compared to conventional materials [9]. Over the years various magnetic nanomaterials are being developed and used in the detection, adsorption, oxidation and degradation of pesticides. Among various MNMs used, magnetite ($Fe_3O_4$) and maghemite ($Fe_3O_2$) have gained the most popularity.

A $Fe_3O_4$/CNT nanocomposite [76] was synthesized by hydrothermal method and was utilized as an adsorbent for the removal of organophosphorus pesticides, fenithrothion, profenofos and ethion. The adsorption process was carried out by solid phase extraction and liquid chromatography. Another $Fe_3O_4$/Red mud nanoparticle (NP) [13] was researched for the removal of organophosphorus pesticides (diazonin, parathion, malathion). During the same time a $Fe_3O_4$/C nanocomposite [97] was synthesized using a novel technique. They obtained the magnetic $Fe_3O_4$ using a typical wet chemical synthesis method and the carbon NPs were synthesized using walnut shells. The $Fe_3O_4$ was then added to the carbon solution under continuous stirring to form the nanocomposite (NCs). They applied the as-synthesized NCs for the removal of diazonin, a pesticide commonly used against sucking and chewing mites and insects. By using 0.155 g of the $Fe_3O_4$/C NCs at optimum pH 6 and 35 min contact time, 100% efficiency was obtained for solution containing 0.5 ppm diazonin.

An amino-functionalized $Fe_3O_4$-$WO_3$ NP [82] was synthesized by using two methods simultaneously, namely co-precipitation and hydrothermal. The tested the NPs for the photolytic degradation of diazonin from both synthetic and real water sample in the presence of UV-radiation. Although in the absence of any other organic compounds removal efficiency of 96.4% was obtained, however in the presence of citric acid, folic acid, EDTA, phenol and humic acid, it decreased to 70.3, 69.39, 67.37, 60.12 and 3.22%. Very recently an iron oxide decorated carbon fiber composite (Fe-ACF/CNF) [107] was synthesized by chemical vapour deposition (CVD) and was utilized for the extraction of 29 pesticides using gas chromatography. The adsorbent was reusable upto 4 cycles and had high accuracy of 70–120%.

Several other MNMs like silica supported $Fe_3O_4$ [85], $Fe_3O_4$/grapheme [17], $Fe_3O_4$/$SiO_2$ core shell [36], $Fe_3O_4$/biochar NCs [113], $Fe_3O_4$ decorated β-cyclodextrin polymer [99], g-$C_3N_4$/$Fe_3O_4$/Ag [46], $Fe_3O_4$@$SiO_2$-MWCNT [123] $Fe_3O_4$@ZIF-8@polymer core–shell shell [118], and $Fe_3O_4$@N-ZnO [6] have been utilized in the removal and degradation of pesticides.

### 5.1.2 Dyes

Dyes are coloured organic compounds that bonds chemically to the substances to which it is applied and imparts colour to it. They are different from pigments in the sense that pigments do not bind to the substances chemically. Dyes can be broadly classified into ionic dyes which include cationic and anionic dyes and non-ionic dyes comprising of vat dyes and disperse dyes.

Dyes have been used by mankind over several decades for various applications. Earlier natural dyes derived from plants and insects were used. However, this tends to fade away with time when exposed to sunlight and water. Synthetic dyes were first discovered in 1856 and since then it has been produced and utilized on large scales. Artificial dyes have complicated structure and are persistent in nature. When released into the waterbodies, they consume oxygen and increase the biological oxygen demand. Also due to their lower density they form a layer on the water surface cutting off sunlight and air from water. This imposes major threat to the aquatic life. Moreover, aromatic structures of dyes show mutagenic and carcinogenic effect towards human. Dye traces may also cause allergies and skin irritation to living organisms. Therefore, removal of dyes is a major environmental concern. Some of the major industries that contribute to the discharge of dye effluent into the waterbodies include textile, paper and pulp, food industry, tanning industry, cosmetic, plastic, paints and dye manufacturer and many others. The textile industry forms the highest contributor among all [62].

Several methods have been researched for the removal of dyes from water, which include chemical oxidation, degradation, adsorption, biological degradation, etc.

Adsorption has an advantage compared to other methods owing to its simple technique, cost effectiveness and most importantly the fact that it does not leave behind any by-product behind provided it is efficiently removed [35]. Most commonly used adsorbents include silica, activated carbon, zeolites, clays and other polymeric materials.

The importance towards the concept of easy and fast recovery and reusability of adsorbent materials, MNMs has gained interest towards the removal of dyes. Magnetite ($Fe_3O_4$) is one of the most widely explored magnetic materials for dye removal. These materials have affinity towards external magnetic field which allows their easy separation. A novel $Fe_3O_4$@GPTM@Gly NP [133] was synthesized and utilized for the adsorption of cationic and anionic dyes (MB, OR, AR, MEB, AZ). Another hybrid shell adsorbent, κ-carrageenan-Silica core shell encapsulated $Fe_3O_4$nanoparticles ($Fe_3O_4$@$SiO_2$/SiCRG) [110] were researched for the adsorption of MB. High adsorption capacity of 530 mg/g was observed at 25 ºC and pH 9, which was higher than many other magnetic materials. Magnetite-bioadsorbent nanocomposite (MLMC) [51] of Moringa oleifera leaves and cotton shell powder was explored for removal of brilliant green dye from water. Recently a novel chitosan-vanadium-titanium-magnetite (CS-VTM) nanocomposite [131] was synthesized through a one pot synthesis method to study its adsorption towards congo red. For CR dose of 100 mg/L, an adsorption capacity of 62.2 mg/g was achieved at optimum pH 6 and 65 ºC. Overall, the CS-VTM was found to be efficient and environment friendly nanoadsorbent. A porous magnetite (PMNs) [32] was also synthesized and used as a highly efficient adsorbent for removal of AR57 and RR dyes. The adsorption of the dyes was found to be endothermic in nature. The $q_m$ obtained was 888.8 mg/g for AR57 and 808.43 mg/g for RR dye. Magnetite-bioadsorbent NPs are also explore for removal of dyes from wastewater.

Panda et al. reviewed various other uses of magnetite nanomaterial composites as adsorbent for dye removal [91]. Several other works of magnetite incorporated composite used as dye adsorbents include $Fe_3O_4$/rGO [111], $Co_3O_4$@$SiO_2$ NC [45],$Fe_3O_4$/GPTMS@P-Lys [127], $Fe_3O_4$/chitosan [49], $NiFe_2O_4$/ZnO [135], $Fe_3O_4$/Pectin; $Fe_3O_4$/silica pectin/hybrid [11], Mn doped $Fe_3O_4$ [64], Au-$Fe_3O_4$-NCs-AC [29], rGO/$Fe_3O_4$ [59], Ggh-g-PAcM/ $Fe_3O_4$ [66], Humic Acid(HA)- $Fe_3O_4$ NP [1], etc.

### 5.1.3 Pharmaceuticals

Over the last few decades, urbanization and modernization has taken over human life. With increased human activity, pharmaceutical products have become a vital part in improving the quality of human life. The various classes of pharmaceutical compounds (PhCs) include antibiotics (tetracycline, amoxicillin, and sulfonamides), antihistamines, analgesics and antipyretics, antiseptics, non-steroidal anti-inflammatory drugs (NSAIDs) (diclofenac, ibuprofen), beta-blockers (propranolol, metoprolol), hormones, steroids, etc. Pharmaceuticals not only form a major part of human life, but are also widely used in several other sectors such as industries, veterinary, agriculture [69] livestock and animal husbandry, etc. Pharmaceutical products and byproducts, as emergent pollutants, have gained interest in recent times due to its persistent nature, low degradability and its potential effect on human health even when present in trace amounts.

These contaminants make way into the waterbodies mainly through drug manufacture industries, hospitals and municipal effluents. High amounts of pharmaceuticals have been detected in surface water, groundwater as well as drinking water. The adverse effects they may cause to humans include allergies, chronic diseases, antibiotic resistance, metabolic perturbations, and endocrine disruption among other. Prolonged presence of antibiotics in water is observed to produce harmful bacteria. Moreover, they result in bioaccumulation and eutrofication thereby adversely affecting marine life which includes disruption in fish spawning and reduced fertility. Pharmaceutical wastewater release is often unregulated. Moreover, due to its versatility and trace concentration makes it go undetected in conventional waste water treatment plants. Therefore, it is very important to develop high effluent and adequate removal techniques for pharmaceutical waste treatment. Several methods used to remove these contaminants from water include ultrafiltration, membranes, biodegradation, oxidation, adsorption, etc. Activated carbon, Grapheme oxides, zeolites, nanoclay, biochar, chitosan, etc. and their hybrid nanocomposites are some commonly used materials for the adsorption and degradation of this target molecules.

Magnetic nanomaterials and their composite counterparts are a major breakthrough for their removal process. Various MNMs like $Fe_3O_4$ [19], $Fe_3O_4$@C [60], $Fe_3O_4$-red mud NCs [14], $Fe_3O_4$-$MoO_3$-AC [77], $MnFe_2O_4$@$TiO_2$-rGO [22] have been used in the removal of common antibiotics such as ciprofloxacin, rimapicin, cephalexin, levofloxacin and amoxicillin. Raha and Ahmarruzaman synthesized g-$C_3N_4$@ $Fe_3O_4$/ZnO nanorods [95] and g-$C_3N_4$/NiO/ZnO/$Fe_3O_4$ [94] for the photolytic degradation of pantaprazole and esomeprazole. These are a class of drugs known as proton pump inhibitors and are commonly used against acidity in stomach. NSAIDs such as Ibuprofen, naproxen, diclofenac, etc., form a very important and commonly used class of drugs. Hence it forms a major part of the pharmaceutical contaminants in water [89]. Magnetic nanoparticles like $\gamma$-$Fe_3O_4$-Zeolite [12], $CoFe_2O_4$ [27], $Fe_3O_4$@decanoic acid [10], $Bi_2O_4$/ $Fe_3O_4$ [126], $NiFe_2O_4$/AC [39], $Fe_3O_4$@Ag [116], etc. have been explored for their removal.

### *5.2 Inorganic Pollutants*

Inorganic pollutants in wastewater mainly comprises of heavy metals, non-metallic salts, acids and bases, fluorides and plant nutrients such as phosphates, nitrates, ammonium ion, etc.

### 5.2.1 Heavy Metals

Metallic chemical elements having high density which are generally toxic and present low concentrations in the environment can be referred to as heavy metals. Although heavy metals such as Cd, Hg, As, Pd, Pb, Cr, Al, Co, Ni, Cu, Fe, Mn are mostly present in trace amounts, they are one of the most widely spread and toxic water pollutants. Heavy metals enter the waterbodies through industrial discharge (tanning, paints, pesticides, etc.), mining and through agricultural runoffs (fertilizers and pesticides). These pollutants accumulate in the environment and cause severe threat to human life. These are non-biodegradable components and some metals like Cd have infinite lifetime. They also accumulate in the food chain and result in biomagnifications and are highly toxic and carcinogenic. Heavy metals such as Ni, As and Cr can cause skin diseases, pulmonary fibrosis, nausea, dizziness, etc. Other metals such as Pb, Cd and Hg can affect lung and kidney functions, cause mental retardation, vision loss, anorexia, thyroid dysfunction and birth defects. Moreover, accumulation in the water bodies adversely affects the aquatic life damaging gills of fish and even deaths. Removal of these toxins, therefore, is a topic of critical concern.

Magnetic nanomaterials are being used for heavy metal removal from wastewater. Magnetic properties of the nanomaterials aid in the easy extraction of pollutants as well as the recovery of the adsorbent. $Fe_3O_4$ silica core shell MNM and modifications of the MNM with cetylmethylammonium bromide (CTAB) and bis(3-triethoxysilylpropyl)tetrasulphide (MSCMNP-$S_4$) was researched as a novel and efficient adsorbent for removal of heavy metals [117]. They could successfully remove Hg(II), Pd(II) and Pb(II) with 303.03, 256.41 and 270.27 mg/g maximum adsorption capacity ($Q_m$) and efficiency of 98.8, 96.4 and 95.7%. A novel magnetic nanoparticle (NP), iron oxide magnetic NP grafted hyperbranced polyglycerol (HPG-MNP) was synthesized and used for removal of Cu, Ni and Al ions from industrial wastewater. [7].

Surface modification of iron oxide NPs with materials like silica result in increased stability of the NP and also prevents aggregation. Magnetic iron oxide nanoparticles are considered a suitable material for removal of As due to the strong interaction between Fe and As. Table 1 summarizes the various MNMs used for heavy metal removal from water and wastewater.

### 5.2.2 Inorganic Anions (Phosphates/Nitrates/Fluorides)

Phosphates and nitrates are vital nutrients for plants. Phosphorus is commonly present as phosphates in nature. Phosphates are commonly used in fertilizers. It is also found in toothpaste, detergents, water softener, cured meat, pharmaceuticals and processed cheese. Nitrates are common nitrogenous compounds that are used as plant nutrients. Nowadays, fertilizers are used on large scale in agricultural farms to increase production. Excessive use of fertilizers and pesticides has resulted in release of nitrates into the waterbodies. These nutrients are detected in rivers, lakes, reservoirs, groundwater and even drinking water. High nitrate and phosphate concentration in water lead to

**Table 1** Heavy metal removal from water and wastewater using various MNMs

| MNM | Target Metal | $Q_m$ (mg/g) | References |
|---|---|---|---|
| $Fe_3O_4$@rGO | As (III)<br>As (V) | 13.10<br>5.83 | [21] |
| Thiol@magnetic $mSiO_2$ | Pb(II)<br>Hg(II) | 91.5<br>260 | [70] |
| $Fe_3O_4$@APS@AA-Co-CA MNPs | Pb(II)<br>Cd(II)<br>Cu(II) | 166.1<br>29.6<br>126.9 | [44] |
| Ascorbic acid@$Fe_3O_4$ | As(III) | 46.06 | [37] |
| $Fe_3O_4$ | As (III)<br>As(V) | 188.69<br>153.8 | [75] |
| $Fe_3O_4$@graphene | Cr(IV)<br>Pb(II)<br>Hg(II)<br>Cd(II)<br>Ni(II) | 17.29<br>27.95<br>23.03<br>27.83<br>22.07 | [47] |
| MDA-magMCM-48 | Pb(II)<br>Cu(II)<br>Cr(VI)<br>Cd(II) | 127.24<br>125.80<br>115.60<br>114.08 | [8] |
| $Fe_3O_4$@$SiO_2$-SH | Hg((II) | 132 | [121] |
| MSCMNP-$S_4$ | Hg(II)<br>Pd(II)<br>Pb(II) | 303.0<br>256.41<br>270.27 | [117] |
| $Fe_3O_4$<br>Si- $Fe_3O_4$ | As(III) | 88.19<br>67.92 | [52] |
| $Fe_3O_4$@$mSiO_2$ core shell | Cu(II) | 84.4 | [57] |
| $Fe_3O_4$ | As(III)<br>As(V) | – | [18] |
| FSP<br>FSBP | Pb(II) | 202.8<br>143.7 | [130] |
| $Fe_3O_4$@biochar | As(III) | 5.49 | [87] |
| MNPs35@$SiO_2$ | Pb(II) | 14.9 | [88] |
| $Fe_3O_4$@$nSiO_2$@$mSiO_2$/EDTA | Cr(III) | 30.59 | [119] |

eutrophication. The nutrients enter the waterbodies through irrigation and rainwater runoff. They also enter the ground water through leaching. They accumulate in the water and result in algal bloom excessive growth of aquatic plants. This results in the depletion of dissolved oxygen in water. This further leads to the death of aquatic organisms. In human, increase in amount of phosphorus in the body can lead to phosphorus retention. This led to chronic kidney diseases, cardiovascular morbidity and even deaths in people undergoing hemodialysis. Exposure to nitrates can cause blue baby syndrome or methemoglobinemia, a condition common in infants that occurs due to lack of enough oxygen in blood.

Various magnetic NPs such as $Fe_3O_4/ZrO_2$/chitosan [56], $Fe_3O_4$@$SO_2$@MPS@poly(4-vinylpyridine) core shell-shell [53], rectorite/$Fe_3O_4$-CTAB [121], AFMCS [68] $Fe_3O_4$@chitosan core–shell [40], $Fe_3O_4$@GO-Sr [102] and $Fe_3O_4$@GO-CMC [61] have been researched for the removal of phosphates and nitrates from water.

Fluorides are another important water pollutant which affects human in a major way. All water generally contains some fluoride. Natural causes of high fluoride concentration in water include weathering of minerals like fluorospars ($CaF_2$), flurapatite ($Ca_5(PO_4)_3F$) and cryolite ($Na_3AlF_6$) as well as through volcanic eruption. Fluorides are used in industries for production of glass, semiconductors, ceramics, rubber and fertilizers. Fluoride upto a concentration of 1.0 mg/L is an essential component of human health. Instances have shown that fluoride is even added to municipal water because it is believed to prevent tooth decay in local population. It is also present in toothpastes and mouthwashes. However, it become hazardous if the concentration exceeds 1.5 mg/L [125]. Consumption of fluoride contaminated water can cause ache and other skin problems, cardiovascular diseases, muscle spasm, abdominal pain, nausea and high blood pressure. Exposure to fluoride for longer time can also result in skeletal and dental fluorosis, early puberty and low fertility in women, thyroid dysfunction, neurological problems and bone cancer.

Some of the defluoridation technique used for drinking water is ion-exchange, reverse osmosis, adsorption and precipitation. Fluorides are hard base having small size and high electronegativity and has high affinity towards multivalent metallic ions like Fe(III) and Al(III). Hence magnetic nanoadsorbents are a good candidate for its removal. Aluminium when is combined with iron, very efficient adsorbent having advantages of both components is obtained. Chai *et al.* synthesized $Fe_3O_4$embedded $Al_2O_3$ for fluoride adsorption and obtained maximum adsorption of 88.48 mg/g at pH 6.5. Following similar route $Fe_3O_4/Al_2O_3$ NPs were developed for removal of fluoride from drinking water. Following similar route, a novel sulfate doped $Fe_3O_4/Al_2O_3$ was developed for fluoride removal from drinking water [20]. Similarly, batch adsorption of fluoride was carried out using magnetic corn stover biochar [84]. Magnetic Ce-Ti@$Fe_3O_4$ nano core shell [80] was also used for fluoride treatment of drinking water.

A biochar $Fe_2O_3/Fe_2O_4$nanoadsorbent [28] was also developed and used for the adsorption of nitrates and fluorides. Mohammadi et al. also synthesized carboxylated chitosan-iron complex [83] and studied its action for simultaneous removal of fluorides, phosphates and nitrates.

## 5.3 *Oil Spills*

Oil spills are generally accidental discharge for petroleum products into the water bodies. It has become a concerning environmental issue. Oil and petroleum forms one of the most important and primary source of energy and provides raw material for a number of chemical industries. With the depletion oil reserves in the land, men have moved their eyes to exploit the ocean. Majority of the oil spills occur during extraction and transport, through leakage in drilling rigs, pipelines and oil vessels, transfer of oil, oil spills from ships and tanker accidents, etc. The common components of oil spills include crude oil, gasoline, fuel oil diesel, petrol, kerosene, etc.

The oil spilled into the water bodies cut the oxygen supply from the water which adversely affects the biological oxygen demand (BOD). This hampers the respiration of aquatic organisms and photosynthesis of aquatic plants. Ultimately, this may lead to the devastating loss of aquatic life cycle. The oil spills also undergo weathering [98] which leads to the evaporation, emulsification, sedimentation and biodegradation and may result in the spread of oil components to other part of the environment, harming not only the marine life but also adversely affecting human. Hence it is an absolute necessity for the removal of oil spills to conserve the environment.

Nowadays, a variety of methods are employed for cleanup of oil spills. These can be categorized into chemical, mechanical and bioremediation methods [105]. The various techniques include use of booms, shimmers, surfactants, dispersants and adsorbents like silica, zeolite, polyethylene, grapheme, cellulose, etc. An efficient adsorbent material must be cost effective, must have high adsorption capacity and must be reusable. A number of researches have been conducted to develop novel and efficient adsorbents for oil spills. The major issue faced by the currently used adsorbents is the removal and recovery of the sorbent material along with the oil. Secondary pollution, low efficiency and long process time are some other problems faced by other removal methods.

This disadvantage may be overcome by the utilization of magnetic nanoparticles. Magnetic nanomaterials are considered as a promising candidate for oil spill remediation. These materials can either be employed alone or functionalized into nanocomposites. The nanocomposites are generally composed of an inorganic magnetic component dispersed/coupled with an organic polymer, surfactant, biomolecule, carbon, silica, ionic liquid among others. The composites do not lose their magnetic properties. Additionally, the addition of hydrophobic component further increases the potential and efficiency of the magnetic nanoparticle. Superparamagnetism, high magnetic susceptibility with high stability, non-sinking properties, high adsorption capacity and recyclability makes them a suitable material for oil remediation. The advantage of using magnetic nanomaterial is that the resultant aggregation of the oil with the MNMs exhibit magnetic properties which can then be easily extracted by external magnetic field. However, these materials must be biocompatible so that it does not lead to any secondary pollution. The nanoadsorbent can then be reused after washing them with solvents such as hexane and ethanol. Lu et al. demonstrated an excellent recyclability of upto 7 cycles was exhibited by a chitosan capped magnetic nanomaterial [74].

The most common materials researched are the maghemite ($Fe_2O_3$) and magnetite ($Fe_3O_4$) based nanomaterials. Acetylated curauafibres/$Fe_2O_3$ incorporated magnetic

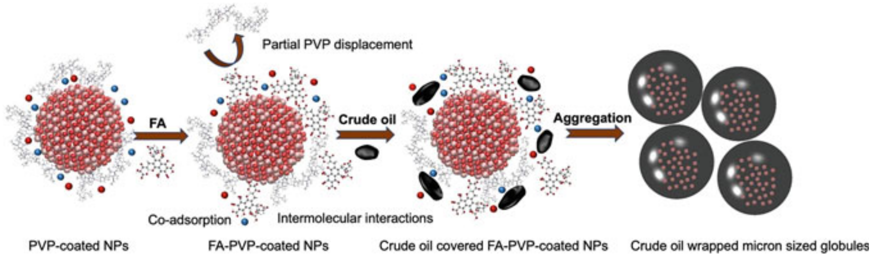


**Fig. 5** Schematic diagram of adsorbtion of crude oil on PVP coated $Fe_3O_4$ NPs (Reprinted with permission from [15])

polymer nanocomposite [33] was developed for removal of oil. Another MNM developed from coconut shell-based AC-iron oxide NC [96] was developed for magnetic separation of oil. A novel sulfonated asphalteen coated magnetic NP [3] was used for removal of crude oil. The asphaltene capping agent forms colloidal particles with the crude oil through strong hydrogen bond, electrostatic and $\pi$-$\pi^*$stacking forces. The as—synthesized nanomaterials were successful in removal of crude oil with adsorption capacity of 22.5 g/g. Flaked polyolefin-based adsorbents (PA) shows excellent properties for oil removal. However, these materials, due to their structure are not fully recoverable. To overcome this problem, very recently Kim et al. synthesized polyolefin based nanoadsorbent (PMA) [65]. This facilitated easy removal of the nanoadsorbent. Further, the oil-PMA composite can be fully paralyzed and converted to refined oil. Hence it forms a promising material for oil spill removal. Other magnetic nanocomposites used for these purpose includes SPION/β-cyclodextrin core shell [67], $Fe_3O_4$@OA (Saber et al., 2015), $Fe_3O_4$@$SiO_2$ [128], $Fe_3O_4$@PS [128], CS-grafted $Fe_3O_4$ [74], yeast-magnetite bionanocomposite (YB-MNP) [26], SPIONS-chitosan NC [106], magnetic ferrogels [101] and PVP- $Fe_3O_4$ nanoparticles [15] (Fig. 5).

## *5.4 Biological Pathogens*

Pathogenic microorganisms present in water for human consumption can cause adverse effect on human. Thus, elimination and disinfection of these water pollutants are a necessary step to ensure safety to human health. Bacteria, cyanobacteria, viruses, protozoa and fungi are various pathogens that can cause severe harm to human being as well as pose threat to aquatic life. Health issues including cholera, typhoid, jaundice, abdominal and intestinal infections, respiratory disorders, gastroenteritis, respiratory and digestive system inflammation, polio, hepatitis, encephalitis, giardiasis, etc. are all caused by microbes present in water.

Every year, a million people face death caused by these waterborne diseases (WBD). Hence WBD can be considered as a major global challenge. Various techniques such as filtration, flocculation, chlorination, disinfection, adsorption, etc.

have been used for elimination of biological contaminants. The inefficiency of these methods calls for the development of more efficient systems for pathogen treatment. Magnetic nanomaterials and their biologically compatible functionalized nanocomposites are a newly emerging material that shows high efficiency for the removal of a wide range of pathogens.

Abdolmaleki et al. synthesized β-cyclodextrin-poly(isophthalamide)-magnetic nanoparticles (CDPA-MNP) [2] that exhibited effective removal of both gram positive (*Baccilus cereus*) and gram negative (*Escherichia coli*) bacteria with high efficiency. The $MnFe_2O_4$/BC composite synthesized by Siddiqui and Chaudhry (Siddiqui and Chaudhry, 2018) showed bacterial growth inhibition against Straphylococcus aureus and Escherichia coli cells. This may be due to the phytogenic content on the black cumin (BC) seeds which interact with the lipid layer of bacteria cell membrane and inhibit their growth. NiO nanoparticles was synthesized by a sol-gel method using Salvia macrosiphon Boiss extract as limiting agent. The nanoadsorbent shows excellent photocatalytic properties and was used for degradation of methylene blue with 80% efficiency. Similarly, Pinto et al., synthesized a series of magnetic nanoparticles (FeO, FeO/AC, MnFeO, CuFeO, CoFeO) [93] and used them as low cost and simple materials for the elimination of both gram positive and gram-negative bacterial strains from the water. Mesoporous $Co_2O_3/Cu_2O_3:Al_2O_3:SiO_2$ magnetic NC was used as a novel material for water disinfection from 7 bacterial strains (*Escherichia coli, Salmonella enterica, Pseudomonas aeruginosa, Listeria monocytogenes, Staphylococcus aureus, Enterococcus faecalis, Bacillus subtilis*).

PQA-MNP [30], PEG-$Fe_3O_4$ [108], $Fe_3O_4$@Arg, $Fe_3O_4$@Lys, and $Fe_3O_4$@PLL [58], $Fe_3O_4$-$SiO_2$-$NH_2$ [129], chitosan-olligosaccharide/$Fe_3O_4$ [104], Graphene/$Fe_3O_4$ [137], AF-$CoFe_2O_4$ [16] and SWCNT-iron oxide [34] are other magnetic nanomaterials that have been used for the removal of pathogens from waste water.

The magnetic nanomaterials can be considered as a promising material with potent disinfection properties and their viable application in waste water treatment. These nanomaterials pose additional advantage such that they can be easily separated and reused.

## 6 Perspective and Conclusion

Over the last few decades, nanotechnology has emerged as a promising tool for their application in various fields. Magnetic nanomaterials have attracted much attraction recently. MNMs are highly stable and efficient nanoparticles owing to their high specific area, high selectivity and fast separation. Moreover, these materials can be synthesized and modified using simple techniques. These materials are widely explored for their interesting and unique physiochemical properties. The size, shape and morphology of these nanoparticles directly influence the properties of the MNMs. Hence, synthesis methods are primary requisite for development for materials with desired properties. Monodispersed magnetic nanomaterials can be successfully synthesized using different methods like hydrothermal, sol–gel, co-precipitation, etc. Further, surface modification of the MNMs with inorganic materials and organic coatings play a crucial role for the development of nanomaterials with improved fuctionalization. The properties of the nanomaterials also influence the interaction of the MNMs with the substrates. The application of these nanomaterials for adsorption, degradation and removal of different water pollutants is a topic of research. MNMs are being used for water decontamination and removal of pollutants like pesticides, pharmaceuticals, dyes, chemicals, heavy metals, inorganic anions, etc. However, the eco-friendly synthesis and environmental fate of these nanomaterials must also be considered for their efficient and widespread application for water purification. The MNMs reduces the use of chemicals to a certain level by reducing the number of extraction steps since they can be extracted using external magnetic field.

Despite the fact that magnetic nanomaterials are a promising material for their application in water purification, certain aspects need to be addressed. The excessive use of hazardous chemicals for the synthesis and fabrication of MNMs caused stress for the proper disposal of the harmful chemicals and solvents. If fabrication is done taking safety measures and the hazardous toxins are discharged without treatment, they may lead to secondary pollution and also transform into more toxic substances. Biological approaches are being explored to overcome this issue. Biological synthetic methods are often cheap, safe, non-toxic and eco-friendly in nature.

Although there is a rise in the interest and research of these nanomaterials, most work is based on batch experiments carried out in small scale. Therefore, sufficient effort must be taken to explore their potential in large scale industrial conditions. Efficient adsorption, regeneration, performance stability and economical availability must also be addressed during their large-scale application.

In conclusion, MNMs opened up new development in environment application and can be considered as a promising candidate for their application in water purification. Finally, the cost effectiveness, enhanced adsorption, regeneration and reusability of the MNMs are important criteria that require intense study and research for development of efficient materials for water remediation.

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