Chapter 17
Nano-Adsorbents and Nano-Catalysts for Wastewater Treatment

Zeenat Sheerazi and Maqsood Ahmed

Contents

17.1 Introduction

Environmental conditions are very alarming all around us due to the anthropogenic activities disturbing the water bodies (Ray and Shipley 2015; Schwarzenbach et al. 2010). Even though anthropogenic processes are responsible for polluting water, natural processes also introduce some toxic metals into the water bodies due to weathering conditions, erosion of rock and soil, and rainwater (Wang and Mulligan

Z. Sheerazi
Department of Chemistry, Jamia Millia Islamia, New Delhi, India
e-mail: maqsood@csiriict.in

M. Ahmed (✉)
Indian Institute of Chemical Technology, Hyderabad, India

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2006). Water is the nectar for our life, but its quality is disturbed due to the contamination by industrial wastes, pesticides, harmful synthetic dyes and organic pollutants (pharmaceuticals, pesticide, phenols, fertilizer, plasticizer, detergent, oils, hydrocarbons etc.). Around the world more than 0.78 billion people face shortage of fresh and hygienic water. This causes serious infections and death of more than 200 million people every year, with 5000 to 6000 children deaths (Amin et al. 2014). Pollutant-removal techniques including biological treatment system, physicochemical, chlorination, ozonation, UV photolysis and ion exchange are not more effective for polluted water treatment (Amin et al. 2014). Due to enhancing nanotechnologies the role of nanomaterials in water purification attracted the attentions due to their nano size, large surface area, reusability, stability, electrical and optical properties (Lu et al. 2016; Prasad and Thirugnanasanbandham 2019). Various shapes of nanomaterials like nanowires, single and multi-walled carbon nanotubes (CNT), nano-colloids, carbon quantum dots (CQD), nano-membrane and films have been studied for diverse applications including wastewater treatment, due to their small size and large surface area (Fig. 17.1). These materials can be used in the form of absorbents for the capture of pollutants and catalytic degradation of larger organic molecules from polluted water (Khajeh et al. 2013). Metallic oxide nanoparticles including mono-metallic, bi-metallic and tri-metallic oxides are used in the form of absorbents and catalysts for wastewater remediation; the magnetic behaviour and presence of variable oxidation states make them efficient absorbents as well as catalysts (Ray and Shipley 2015; Khajeh et al. 2013). Nanostructural silica- and alumina-based materials were also studied in the form of nano-porous materials which provide accessible active sites. (Jadhav et al. 2019; Banerjee et al. 2019; Afkhami et al. 2011; Kalfa et al. 2009a). Polymer nanocomposite with hierarchical surface porosity is also an interesting material for the treatment of waste water (Zhao et al. 2018a). Moreover the nanomaterials can be functionalized with different types of

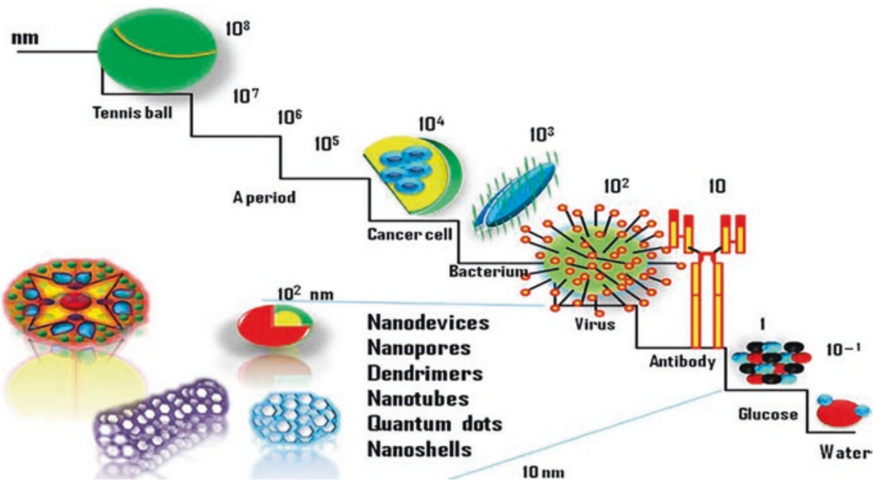

Fig. 17.1 Different morphologies of nanostructural materials

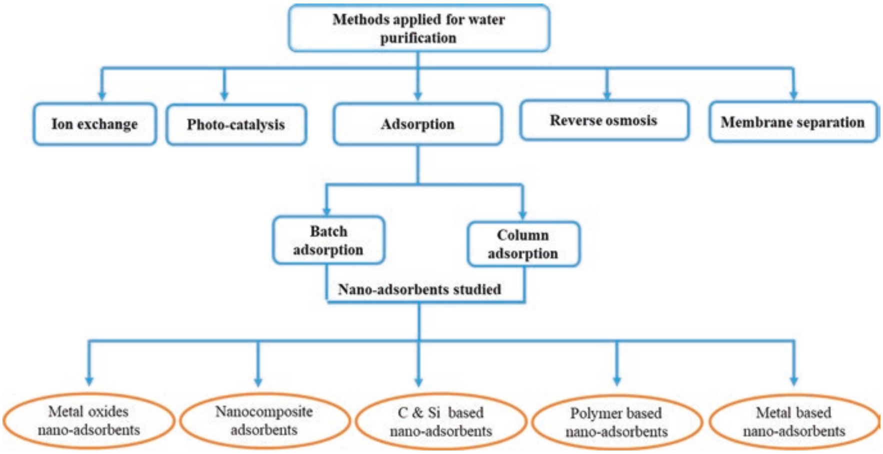

Fig. 17.2 Various chemical methods for waste water treatment and nano-adsorbents

organic functionalities possessing higher affinity for toxic metal ions (As, Cd, Hg, Cr etc.). Such materials include organo-modified nanostructural materials which can be functionalized via ex situ as well as in situ synthesis. The common methods used for the synthesis of metallic based nanoparticle involves co-precipitation, sol-gel and hydrothermal techniques. The nanoporous materials are commonly prepared through soft templating routes by using some surfactant and block copolymers as templates.

The most common methods applied for water purification are ion exchange (Akieh et al. 2008; Ismail et al. 2010; Qiu and Zheng 2009), reverse osmosis (Dialynas and Diamadopoulos 2009; Mohsen-Nia et al. 2007), electrochemical treatment (Hunsom et al. 2005; Deng and Englehardt 2007; Rana et al. 2004), membrane filtration (Qdais and Moussa 2004), photo catalysis and adsorption (Fu and Wang 2011). But the adsorption technique is the most recognised and convenient, as it does not require high amount of energy. The adsorption methods involve two different procedures viz. batch adsorption and column adsorption. The batch adsorption is carried out by adding the adsorbate into the sorbent solution under continuous agitation or stirring. The latter will be carried out through the continuous flow of contaminated water over fixed bed of adsorbent and is typically applied at industrial scale. Various chemical methods for waste water treatment and nano-adsorbents are depicted in Fig. 17.2.

17.2 Synthesis Approaches for Nano-Catalysts/Adsorbents

Most commonly the nanomaterials can be synthesised in two major categories viz. top to bottom and bottom to up approaches (Fig. 17.3). In top to down approach the bulk material is breakdown to nanoscale by mechanical process with uniform

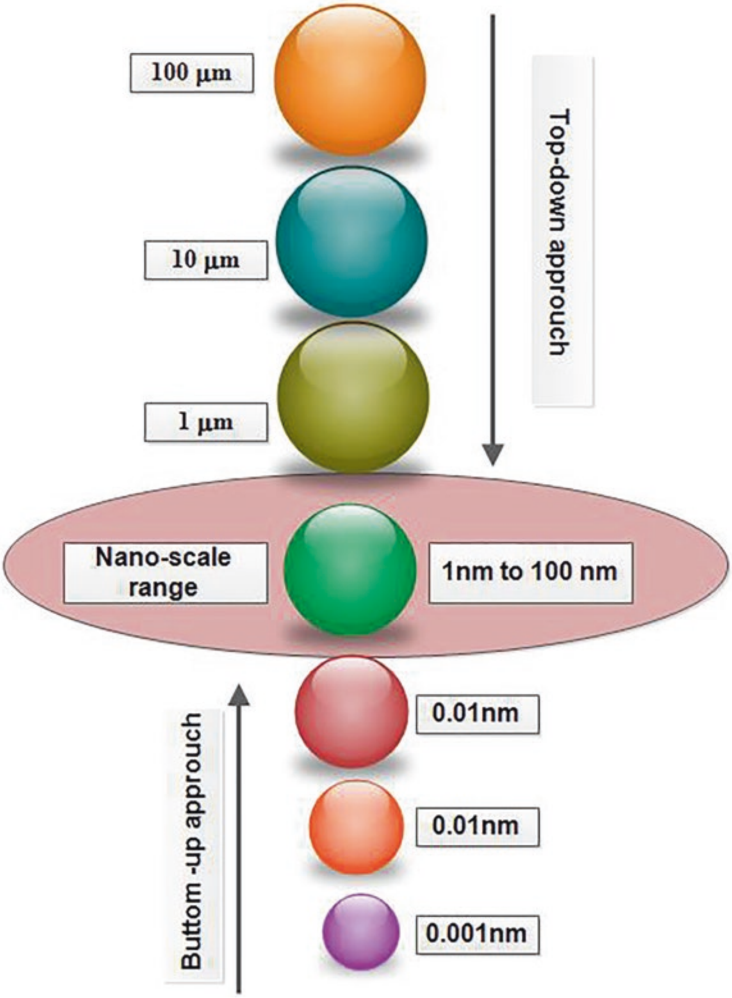

Fig. 17.3 Top-down bottom-up approaches for synthesis of nanomaterials

particle size and morphology. The latter involves the assembling of molecules and atoms to create various ranges of particles in nanoscales (Prasad et al. 2016). It is further divided into chemical, physical and biological methods. Among the three, the chemical method is more simplistic, low cost and quick in action. The chemical method is well established and more common, in which the nanomaterials are prepared through the interaction of molecules and atoms. It enables the easy functionalization of nanomaterials, which can be applied as adsorbents and catalysts in wastewater treatment. These chemical methods involve different synthetic chemistry (depicted in Fig. 17.5) viz. sol-gel, micro-emuslion, hydrothermal synthesis, Co-precipitation method, chemical vapour deposition (CVD) & Chemical vapour synthesis (CVC), microwave and ultrasound-assisted synthesis (Devatha and Thalla 2018; Rane et al. 2018).

17.2.1 Sol-Gel Method

In sol-gel technique the colloidal solution called sol of the metal precursor or any inorganic species is formed through hydrolysis, followed by the condensation or formation of gel-like diphasic system containing both liquid and solid phases. The morphologies of these materials range from discrete particles to the polymer network range. Nanomaterials of different inorganic metals and their oxides like TiO_2, ZnO, SnO_2, CdSe, Fe_2O_3, ZrB_2, $GdVO_4$, Ta_2O_5, CeO_2 and nanoalumina are synthesised through sol-gel techniques and some of them are studied in the field of waste water treatment (Xiao et al. 2009; Sharma et al. 2008; Bayal and Jeevanandam 2012; Nautiyal et al. 2015; Reda 2010; Zhang et al. 2011; Chumha et al. 2014; Sreethawong et al. 2013). Variety of mesoporous silica nanoparticles (MSN) are synthesised through sol gel methods and studied for adsorption processes (Chen et al. 2018; Qin et al. 2018). Jie Chen et al. demonstrated the study of different morphologies of MSN synthesised through sol-gel and micro-emulsion method (Chen et al. 2018). The synthesis of MSN involves the soft templating route, in which surfactants are used as templates (Fig. 17.4). Mostly the silica

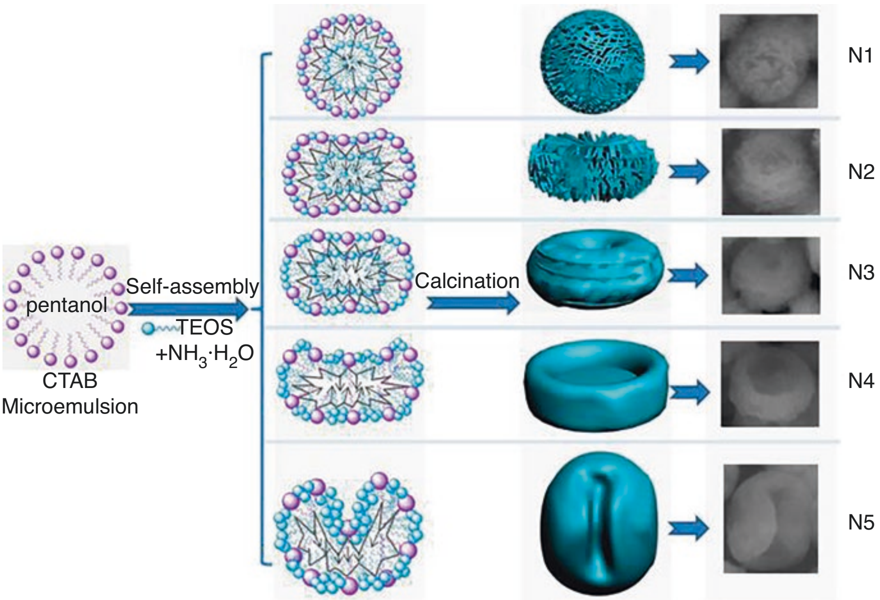

Fig. 17.4 Illustration of the synthetic procedure of MSNs with different morphologies. (Reprinted with permission from Ref. (Chen et al. 2018) Copyright (2020) American Chemical Society)

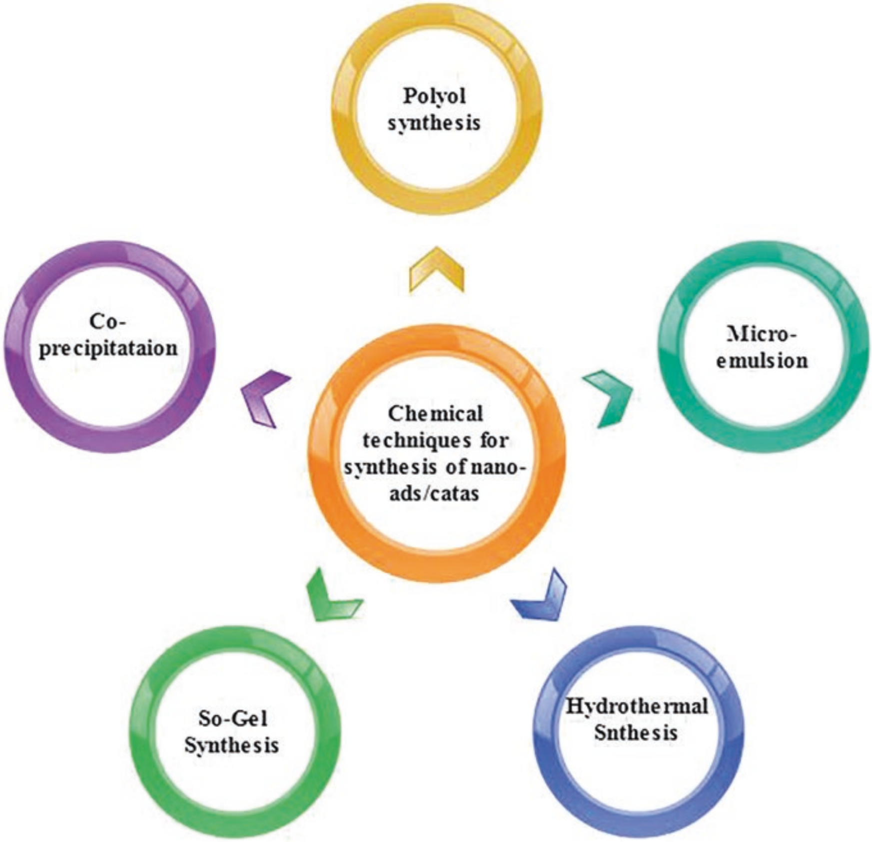

Fig. 17.5 Schematic representation of different techniques used for synthesis of nano-adsorbents and nano-catalysts through chemical method

precursors-tetraethylorthosilicate (TEOS) first hydrolysed and gets start condensing at different pH ranges. The surfactant/template is removed through calcination or solvent extraction methods. The same approach can be applied to other mesoporous materials by using their salt precursors with modified procedures.

17.2.2 Micro-Emulsion Method

Diameter range of 600 nm to 800 nm monodispersed spherical droplets of oil in water (o/w) or water in oil (w/o) depending upon the type of surfactant forming emulsion which is called as micro-emulsion system. Water in oil (w/o), also called as reverse micellar system, acts as a best reaction site for nanomaterial synthesis like Ag, Cu, CdS, Ni, Au, Rh, silica-CdS, Ag-Au, Pd-Au and Pd-Ag nanoparticles. Other non-metallic nanomaterials of polystyrene (20–30 nm), cholesterol, rhodiarome, retinol, polyaniline, etc. can be synthesized through micro-emulsion technique. (Joshi and Kumar 2018; Wang et al. 2010a; Dhand et al. 2010).

17.2.3 Hydrothermal Synthesis

The process of crystallization under autogenesis pressure produced at higher temperature in the presence of water vapours is called hydrothermal synthesis. The synthesis is usually carried out in a stain less steel autoclave hydrothermal rectors, or any closed autoclavable container. Hydrothermal synthesis is a fast and facile process for the synthesis of different nanostructural materials such as Fe_3O_4, NiO, CuO, $CoFe_2O_4$ and ZnO (Zhao et al. 2007; Yang and Pan 2012; Guo et al. 2012; Choi et al. 2013; Cao et al. 2013; Behbahani et al. 2012; Maryanti et al. 2014). Guohong Qiu et al. demonstrated the microwave-assisted hydrothermal synthesis of α-Fe_2O_3 and studied the applications for As (III) removal from water (Qiu et al. 2011). Synthesis of various nanomaterials like Cu nanoparticles, hydroxyapatite/biochar nanocomposite, hydroxyl sodalite zeolite nanoparticles, Mn_2O_3 and $CoFe_2O_4$ were carried out through hydrothermal synthesis (Kumar et al. 2014a; Kumar et al. 2014b; Nassar et al. 2016; Abdelrahman et al. 2019; Hermosilla et al. 2020). These materials were studied for the removal of different contaminating agents from water. Microwave-assisted hydrothermal synthesis is also important for nanomaterial synthesis, which can be used for adsorption process and in catalysis.

17.2.4 Co-precipitation

In co-precipitation, continuous occurrence of nucleation, growth coarsening and agglomeration will take place during synthesis. Nucleation is the key step to take place and the product formed is usually the insoluble species under the condition of super saturation. Super saturation condition is mandatory to start precipitation, which are usually the result of following chemical reaction (Rane et al. 2018)

$$XAy + (\text{aqueous}) + YBx - ((\text{aqueous}) \rightarrow \text{AxBy}(\text{solid})$$

17.2.5 Polyol Synthesis

Polyol methods is the synthesis route for the preparation of a wide range of metal-based nanoparticles (Ag, Pr, Pt, Pd, Cu), metal oxides nanoparticles (ZnO, indium-tin-oxide; ITO, Gd_2O_3), magnetic nanoparticles and mixed metal nanoparticles (Dhand et al. 2015). Polyethylene glycol is used as a solvent and a complexing and reducing agent at the same time, so the process is called as polyol process. Sharif Ahmad et al. (Ghosal et al. 2013) demonstrated the formation of nickel nanostructure using natural polyol and studied their dye adsorption properties. Nanoparticles of cuprous oxides were obtained in the forms of nanoboxes, nanocubes and nanospheres through polyol process by Lei Huang et al. (Huang et al. 2008).

17.3 Types of Nano-Adsorbents and Nano-Catalysts

A majority of inorganic adsorbents and catalysts belong to the transition metals and rare earth metals and their oxide forms. A few elements of main groups (like Si, Al, Mg) also play a role in the formation of active adsorbents and catalysts. The metallic oxides are well-studied nano-adsorbents in the form of mixed metal oxides (mono-metallic, bi-metallic, or tri-metallic oxides). In addition to metal oxides, pure metallic nano-adsorbents are also studied for their applications. Other adsorbents like surface-modified metal oxide, alumina, zeolites, and silica-based mesoporous materials are also good adsorbents for waste water treatment. Different forms of nano-adsorbents and nano-catalysts are described below.

17.3.1 Metal-Based Nano Adsorbents

Nanomaterials of transition metal are well studied in the various applications of catalysis, adsorption, and in the formation of semi-conductors and other devices. Due to the demands of efficient catalysts and adsorbents with paramagnetic behaviour for removal of harmful toxic metals and synthetic dyes from textile water, researchers focused on the transition-metal based nanomaterials for this application (Ge et al. 2012). Although number of transition metal ions are toxic in their ionic form like As(III), Cr (VI), Pb (II) and Hg (II) which contaminate water through their contamination but well design material is not soluble into water and work as adsorbents or catalyst. The introduction of organo-functionality onto the surface of nanomaterials also enhances the desirable activity of the material (Ge et al. 2012). Different types of metal-based nano-adsorbents with different structures and properties are discussed in the sections that follow.

17.3.1.1 Iron Oxide Nano-Adsorbents

Separation of adsorbents along with adsorbate from aqueous solution is a challenging task. The magnetically active forms of iron oxides nanomaterials (Fe_3O4 and γ-Fe_2O_3) act as excellent adsorbents for the capture of toxic elements, which can be easily separated from solution. These nanomaterials used for the removal of different toxic heavy metals (viz. Cr, Co, Pb, Cu, As and Ni from water (Badruddoza et al. 2013; Lei et al. 2014; Tan et al. 2014; Shipley et al. 2010). Such materials can be synthesised in different forms like nanorods, nanoparticles, and nanotubes. Nanoparticles of magnetite (Fe_3O_4) are ideal nano-adsorbents for the capture of As (III) and As (V) from water (Shipley et al. 2010). Arsenic adsorption studies along with the effect of common containments present in water were carried out by Shipley et al. in (Shipley et al. 2010) through batch adsorption study. About 83 mg L^{-1} of arsenates were adsorbed within 1 hour by using 0.5 g L^{-1} magnetite nanoparticles as adsorbents at optimized conditions. In 2013, Roy et al. studied maghemite nanotubes (MHNT) as effective nano-adsorbents for Cu(II), Pb(II) and Zn(II) ion capture from water (Roy and Bhattacharya 2012). The nanotubes of γ-Fe_2O_3 were synthesised with microwave and were studied for adsorption kinetics (Roy and Bhattacharya 2013). The adsorption isotherm model (Langmuir and Freundlich) is used to study the adsorption capacity of adsorbents, and the Langmuir model showed good agreement to the observed data of the adsorption of metal ion onto the MHNT then Freundlich model (Roy and Bhattacharya 2012). Favourable adsorption study was done for different metal ions (Cu (II), Zn (II), and Pb (II) studied which are depicted in Table 17.1**.** Magnetite (Fe_3O_4) nano-rods were also studied as nano adsorbents by Karami in 2013 for the capture of Fe (II), Pb(II), Zn(II), Ni (II), Cd(II) and Cu(II) ions at lab scale with best fitted adsorption data with the theoretical models as shown in Table 17.1 (Karami 2013). When magnetite nanorods are compared with

Table 17.1 Various iron oxide nano-adsorbents studied for removal of toxic metal ions

Iron oxide nano-adsorbents	Shape and size of material (nm)	BET surface area (m^2g^{-1})	Targeted metal ions	Isothermal models	Sorption capacity	Ref.
Fe_3O_4	Nano-sphere, 19.3	60	As(V) As(III)	Langmuir q_m (mgg^{-1})	1.19 1.13	(Shipley et al. 2010)
Fe_3O_4, nano-rods	Rods, 55–65 Length 900–1000	–	Fe(II) Pb(II) Zn(II) Ni(II) Cd(II) Cu(II)	Langmuir q_m (mgg^{-1})	127.01 112.86 107.27 95.42 88.39 79.1	(Karami 2013)
γ-Fe_2O_3	Tubes, 10–15 Length 150–250	321	Cu(II) Zn(II) Pb(II)	Langmuir q_m (mgg^{-1})	111.11 84.95 71.42	(Roy and Bhattacharya 2012)
α-Fe_2O_3 (3D flower like)	Flower shape, 5000–7000	–	As(V) Cr(VI)	Langmuir q_m (mgg^{-1})	41.46 33.82	(Liang et al. 2013)
α-Fe_2O_3 (3D sphere)	Spheres, 37	31	Pb(II) Cd(II) Cu(II)	Freundlich qe mgg^{-1}	3.11 0.51 0.051 0.31	(Shipley et al. 2013)

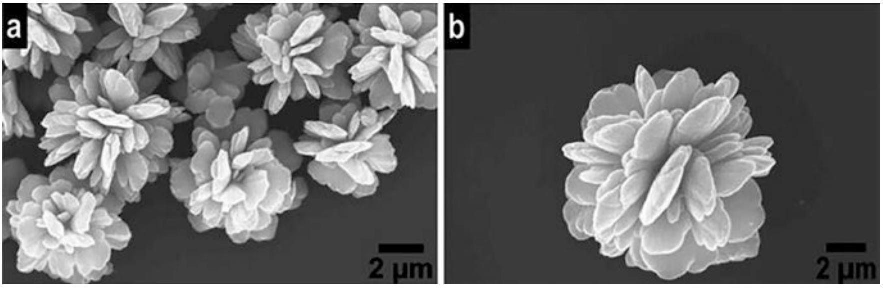

Fig. 17.6 SEM images of α-Fe_2O_3 micro-flowers after hydrothermal treatment at 150 °C for 12 h. (Reprinted from Ref. (Liang et al. 2013), Copyright 2020, with permission from Elsevier)

maghemite nanotubes, the former showed better adsorption capacity for Zn (II) and Pb (II) but exception for Cu, which might be due to the morphology of the nanorods (Karami 2013).

Nano-size hematite (α-Fe_2O_3) phase of iron oxide is a non-magnetic which is used in catalysis and environmental applications (Liang et al. 2013). Shipley et al. study the role of nono α-Fe_2O_3 for the removal of Pb (II), Cd ((II), Cu (II) and Zn(II) from aqueous solution (Shipley et al. 2013). The different adsorption parameters like pH, which affects the charge of adsorbent and temperature, were studied to elucidate the best absorption capacity. The surface chemistry of nano α-Fe_2O_3 depends upon optimized adsorption pH, which is attributed to the presence of –OH groups on the exterior surface of nano-adsorbents responsible for binding with toxic metals (Shipley et al. 2013). The isotherms models applied to the experimental data are in agreement with the observed conditions and are depicted in the Table 17.1. Hierarchical nano-structures of α-Fe_2O_3 with more surface area were studied by Liang et al. as adsorbents in order to achieve maximum adsorption capacity (Liang et al. 2013). The prepared structures are self-assembled and flower-like (SEM image shown Fig. 17.6), synthesised through hydrothermal treatment (Liang et al. 2013). Both the adsorption models, viz. Langmuir and Freundlich models, were studied in order to investigate the adsorption capacity of the flower-shaped nano α-Fe_2O_3 adsorbents.

17.3.1.2 Titanium Oxide Nano-Adsorbents

Titanium dioxide (TiO_2) has many applications of photo catalysis and photovoltaic, H_2 sensing, coatings and environmental applications for removal of pollutants (Bavykin et al. 2006a). Lao et at study the adsorption of arsenic by using TiO_2 at optimized parameters with 21 successive treatment adsorption cycles with regenerated TiO_2 (Luo et al. 2010). They study the adsorption kinetics through batch adsorption study which is fitted to pseudo second-order kinetic model (R value >0.999) and rate constant of 0.84 g mg^{-1} h^{-1}. Commercial TiO_2 nanoparticles were studied by Engates and Shipley et al. in 2011 for the adsorption of Pb(II), Cd(II) and

Table 17.2 Various iron oxide nano-adsorbents studied for removal of toxic metal ions

TiO_2 Nano-adsorbents	Shape and size of material (nm)	BET surface area (m^2g^{-1})	Targeted metal ions	Isothermal models	Sorption capacity	Ref.
TiO_2 nanoparticles	Nano-sphere, 8.3	185	Pb(II), Cd(II), Ni(II)	Langmuir q_m (mgg^{-1})	83.04 15.19 6.75	(Engates and Shipley 2011)
Layered protonated titanate sheets	Nano-sheets 2-15 nm thickness 0.78 nm interlayer distance	379	Pb(II)	Langmuir q_m (mgg^{-1})	366 mgg^{-1}	(Yang et al. 2008a)
$Na_2Ti_3O_7$ -T3	Nano fibres	321	Ba(II), Sr(II), Pb(II)	Sorption saturate capacity (mgg^{-1})	160.64 55.20 279.45	(Yang et al. 2008a)
$Na_{1.5}H_{0.5}$ Ti_3O_7 -T3(H)	Nano fibres	—	Ba(II), Sr(II), Pb(II)	Sorption saturate capacity (mgg^{-1})	130.44 49.94 244.26	(Yang et al. 2008a)
Titanate nano-flower	Flower shape, 600–100 nm	290	Pb(II), Cd(II), Ni(II), Zn(II)	Langmuir q_m (mgg^{-1})	304.3 168.6 88.05 98.1	(Huang et al. 2012)
Titanate nano-tubes	Tubes. 200 nm length, 7–10 nm outer dia.	230	Pb(II), Cd(II), Ni(II), Zn(II)	Langmuir q_m (mgg^{-1})	147.4 76.76 40.09 44.67	(Huang et al. 2012)
Titanate nano-wires	Wires shape 10 μm length, 40–240 nm dia	30	Pb(II), Cd(II), Ni(II), Zn(II)	Langmuir q_m (mgg^{-1})	106.09 47.55 24.83 27.66	(Huang et al. 2012)

Ni(II), and the results are depicted in Table 17.2 (Engates and Shipley 2011). The Langmuir adsorption model is well fitted to the process and indicates the monolayer adsorption on to the surface of TiO_2 nano-adsorbents, the adsorption efficiency of TiO_2 nanoparticles is more in comparison to bulk anantase TiO_2 (Engates and Shipley 2011). In addition to $TiO_{2,}$ titanates are also useful as adsorbents for the removal of heavy metals from water (Kasap et al. 2012). Titanates in different forms like nano-sheets, nano-fibers fibers were also reported for adsorption process (Bavykin et al. 2006b; Lin et al. 2014; Yang et al. 2008a; Yang et al. 2008b; Bancroft et al. 1982). Huang et al. studied the titanate nanoflower, titanate nanotubes and titanate nanowires for the removal of $Zn^{2+,}$ $Ni^{2+}$and Cd^{2+} using a ternary system (Huang et al. 2012). These three nanomaterials were synthesized through hydrothermal methods in alkaline condition which is followed by protonation in acidic media (Huang et al. 2012). The adsorption study suggested the strong adsorption capacity of titanate nanotubes.

17.3.1.3 Cobalt Oxide Nano-Adsorbents

Nanostructural cobalt oxide with various structural morphologies has been synthesised by hydrothermal and solvothermal techniques (Nassar and Ahmed 2012; Ribeiro et al. 2018). The Co-based nanomaterials are found to be good agents for waste water remediation. M.Y. Nassar et al. prepared the cobalt oxide nanomaterials with different morphologies and studied the application for removal of organic dye (methylene blue dye). The material showed maximum adsorption of 99.19% for MB in 24 h (Nassar and Ahmed 2012). Surface functionalized nano-adsorbents with various organo-functionalities like amine and thiol is more interesting due to the grafting of proper and desirable active sites on to the surface of nanomaterials. Qurrat-ul-Ain et al. synthesised magnetic Co-Fe oxide nanoparticles (CoFeNp) and functionalized their surface with two separate amine functionalities (hydrazine and dodecyl amine) (Khurshid et al. 2020). After complete characterization, the material was carried out for adsorption of six different negatively charged azoic dyes, which include acid Orange 7, reactive Red-P2B, naphthol Blue Black, Acid Orange 52, reactive Orange 16 and amaranth. The experimental data showed the pseudo-second order kinetics, in which film diffusion was the dominant phenomenon compared to intra-particle diffusion. The composite of $CoFe_2O_4$ modified with tragacanth gum was prepared and studied for methyl orange (MO) and methyl red (MR) from waste water (Moghaddam et al. 2020).

17.3.1.4 Zinc Oxide Nano-Adsorbents

Zn oxide is not a much-studied material for adsorption; however it has more applications in photocatalysis and gas sensing. Its nontoxic nature and availability of surface hydroxyl groups makes it as good adsorbent for the removal of Zn(II), Cd(II), and Hg(II) ions from aqueous solution (Sheela et al. 2012). Nanomaterials are prepared through the precipitation method and calcined at 400 °C and carried out for batch adsorption study. The adsorption study is well fitted with the theoretical isotherm models; it is suggested that due to small hydrated ionic radii of Hg (II) and more electronegativity adsorption efficiency for Hg is more as compare to Zn(II), and Cd(II) ions (Sheela et al. 2012). ZnO hollow microspheres were prepared by Wang et al., and their adsorption study was compared with ZnO nanopowder and nano-plates (Wang et al. 2010b). Hollow microspheres showed better adsorption performance over ZnO nanopowder and nano-plates.

17.3.1.5 Mixed Metal Oxides Nano-Adsorbents

Synthesis of nanoscale mixed metal oxides like spinel (Wang and Kang 2012; Giri et al. 2002; Khedr et al. 2006), Ti-based bimetallic and trimetallic oxides (Galindo et al. 2007), In (III)-Sn (IV) oxides have many applications in electrical, magnetic and conducting properties. These bi-metallic and tri-metallic oxide nano-adsorbents were also synthesised through similar methods and studied for adsorption of heavy metal ions and synthetic dyes. Gupta et al. studied Fe-Ti mixed oxides for arsenic removal from ground water in West Bengal (India) and Bangladesh. Iron doped titanium oxide adsorbent was prepared by Lin Chen et al. in 2012 through precipitation method using $Ti(SO_4)_2$ and $FeSO_4$ salts (Chen et al. 2012). Adsorption study of the materials was carried out for removal of fluoride from drinking water which showed the adsorption capacity of 53.22 mg/g, obtained by fitting experimental data to the Langmuir isotherm model. It is suggested that Fe doped into the titanium oxide increases the –OH groups on adsorbent surface, which enhance the adsorption efficiency for fluoride (Chen et al. 2012). Mesoporous Ce-Zr mixed oxides were synthesised through salvo-thermal synthesis by Qi Li et al. (Su et al. 2015). These nanomaterials were studied for the removal of phosphate ions from water through batch adsorption study. The phosphate adsorption capacity is ~112.23 mg/g. The material can be regenerated after desorption by NaOH solution. Yaswanth K. Penke and co-workers studied the tri-metallic oxides (Mn-Al-Fe and Cu-Al-Fe) as nano-adsorbent for the removal of arsenic. XPS studies showed the redox behaviour of adsorbents and showed 75-90% adsorption of As (III) (Penke et al. 2019). Co-Fe oxide and many other mixed ferrites like $MnFe_2O_4$, $ZnFe_2O_4$, $MgFe_2O_4$, $NiFe_2O_4$, $CuFe_2O_4$, and $CoFe_2O_4$ have been studied by Hu et al. (2007) for the removal of Cr((VI) (Hu et al. 2007). Different forms of bi-metallic, tri-metallic and mixed oxides nano-adsorbents are summarized in Table 17.3**.**

17.3.1.6 Aluminium Oxide Nano-Adsorbents

Aluminium trioxide nanoparticles, Al_2O_3, are well-studied and efficient nano-adsorbents due to their large surface area. Afkhami et al. studied the adsorption of Pb(II) and Cr(III) ions on the surface of modified alumina nanomaterials (Afkhami et al. 2011). Kalfa et al. used nanoscale Al_2O_3 on single-walled carbon nanotubes for adsorption study of Cd ions. The material was prepared through sol-gel technique and mentioned to be a better absorbent for Cd in caparison to the single-walled carbon nanotubes (Kalfa et al. 2009b).

17.3.1.7 Magnesium Oxide Nano-Adsorbents

Nanoparticles of magnesium oxide (MgO) are useful for the removal different heavy metal ions. Different morphologies like microsphere were studied by Gupta et al. in 2015 for removal of heavy metal. Various nanostructures of MgO like

Table 17.3 Mixed metal oxide nano-adsorbents and catalysts studied for water purification

Nano-catalyst/ nano-adsorbent	Targeted metal ion	Method of preparation	Sorption capacity (%) Qe(mg/g)	Process	Ref.
$Ag\text{-}Sc_{0.01}Ti_{0.99}O_{1.99}$	RhB	PPM	90%	Photo catalytic	(da Silva et al. 2014)
TiO_2-Flakes	RhB	Sol-gel (Dip-coating)	73.2%	Photo catalytic	(Li et al. 2015)
FAP-TiO_2	Pb^{2+},Cd^{2+} Cr^{3+},Fe^{3+}	Solvothermal	99%	Adsorption	(Wang et al. 2020)
Nano- TiO_2	Cr(VI), Phenol	–	~99%	Photo catalytic	(Chi et al. 2019)
$Ti_3C_2/SrTiO_3$	U(VI)	Hydrothermal	77%	Photo catalytic	(Deng et al. 2019)
V_2O_5	MB	Hydrothermal	437	Adsorption	(Avansi et al. 2015)
CS-VTM	CR	Hydrothermal	99.1%	Adsorption	(Zhang et al. 2020a)
Ni-V_2O_5	RhB	Sol-gel	100%	Photo catalytic	(Rafique et al. 2020)
$MnFe_2O_4$	RhB	Sol-gel	90%	Photo catalytic	(Zhang et al. 2020b)
MnO_2@PmPD	Pb^{2+}	Oxidation	104.88	Adsorption	(Xiong et al. 2020)
OMS-2	U(VI) Eu(III)	Hydrothermal	348 106	Adsorption	(Yin et al. 2020)
FPL	M_O	Hydrothermal	833.33	Adsorption	(Natarajan et al. 2020)
Fe_2O_4/COP	AO, RhB	Solvothermal	107.11, 131.23	Adsorption	(Shakeri et al. 2020)
HFOR	p-ASA As(V)	Co-ppt	22 60	Adsorption	(Liu et al. 2020)
$CoFe_2O_4$@ɣ-Fe_2O_3	Cr(VI)	Hydrothermal coprecipitation	50	Adsorption	(Campos et al. 2019)

nanorods, nanotubes, and nanocubes are reported as nano-adsorbents for heavy metals. MgO nanomaterials were prepared and used for the removal of azo and anthraquinone reactive dyes from water by Gholamreza Moussavi et al. (Moussavi and Mahmoudi 2009).

17.3.2 Polymer-Based Nano-Adsorbents

In addition to the inorganic adsorbents, polymer-based nanocomposites are also well-studied nano-adsorbents due to their macromolecular structure and the variety of functional groups. These materials possess good physical properties, large surface area, mechanical rigidity, and can be regenerated. These materials usually include, polyaniline, polystyrene and polyacrylic ester matrix. Compared to single polymers nano-adsorbents, dual polymers are more efficient for adsorption, due to their abundant surface functional groups (Wu et al. 2016). The combination of Polypyrrole (PPy) with PANI and polyacrylonitrile is found to be a suitable adsorbent for Co (II) (Javadian 2014a; Javadian 2014b; Wang et al. 2013). The adsorption study Co (II) on PANI/PPy polymer nanaofiber showed efficiency of 99.68% in 11 mins. The data are well fitted with Freundlich model and followed the pseudo-second-order kinetic model. Checkol et al. studied an efficient material consisting of poly (3,4 ethylenedioxythiophene)/polystyrene sulfonate (PEDOT/PSS) and the lignin (LG) for adsorption Pb(II) (Checkol et al. 2018). PPy/polyacrylonitrile core-shell nanostructures were also prepared and studied for Cr(VI) removal from aqueous solution (Wang et al. 2013). A variety of polymer-based nano-adsorbents including dual polymers, polymer-carbon composites, polymer silica composites, and polymer-metal composites are well studied and described in detail by Xiangke Wang et al. (Zhao et al. 2018b).

17.3.3 Silica- and Carbon-Based Nano-Adsorbents

Carbon nanotube in combination with other metals as support enhances the adsorption behaviour of adsorbents. Di et al. reported supported Ce in the form of CeO_2 on to the carbon nanotube and study its adsorptive behaviour for wastewater treatment (Di et al. 2006). The hydrated forms of these rare earth metals have high affinity for anions like fluoride, arsenate and phosphate (Zhang et al. 2003; Tokunaga et al. 1995). Carbon nanotubes were also used for supporting magnetic iron oxide by Gupta et al. (Gupta et al. 2011). The combined adsorptive behaviour of both the components in nanocomposite enhances the adsorption behaviour for Cr removal.

Silica nanoparticles are well-studied adsorbents for waste water treatment. They are also used for coating of metal oxides nanoparticles before their functionalization, with some organic functionality to improve adsorption. Bulk mesoporous silica like SBA-15, MCM-41, MCM-48, and HMS are found to be more superior adsorbents due to their large surface area, uniform pore size distribution and abundant hydroxyl groups. Their surface can be modified with various functional groups (like amines, thiol) to adsorb metal ions.

17.3.4 Nano-Catalysts for Wastewater Treatment

In addition to the adsorption of the toxic pollutants from water by nano-adsorbents, nano-catalysts are also play a role in polluted water remediation. Both the materials (nano-adsorbents & nano-catalysts) mostly have similar structure, composition, stability, surface area, and particle size, but differ only in their mode of action for purification. Nano-absorbents capture the whole polluting agents (adsorbate) on to their surface through physiosorption or chemisorption. On the other hand nano-catalysts simply act as catalysts to make more toxic ions into less toxic ones, like reduction of nitrite and catalytic degradation of dye molecules into smaller fractions. Recently nano-sized Pd-Ag alloy was studied as nano-catalyst by J. P. Troutman et al. in 2020 and used for the reduction of nitrite in drinking water (Troutman et al. 2020). Nitrate is one of the most common pollutants present in ground water. It is harmful after ingestion and causes methemoglobinemia due to the formation of nitrite after the reduction of nitrate (blue-baby syndrome). Catalytic reduction of nitrite to N_2 gas or NH_3 is a promising route for the detoxification of nitrite ions. Titanium-based nano-catalysts are well used photocatalysts for the degradation of dyes. Polycarpos Falaras et al. in 2013 studied anion-doped mesoporous titania materials for the degradation of a hazardous material microcystin-LR (MC-LR) cyanotoxin pollutant in the presence of visible light (Likodimos et al. 2013). The materials were developed through sol-gel technique and co-doping of N and F anions (Likodimos et al. 2013). Rajender S. Varma and his co-workers studied and compiled the literature about green synthesis of nano-catalysts and their application in wastewater treatment (Nasrollahzadeh et al. 2020). There are many routes for the fabrication of bio-based chemicals to the nanotubes, nanowires etc. which are found to be excellent catalysts for reduction and degradation of water pollutant (Nasrollahzadeh et al. 2019a; Nasrollahzadeh et al. 2019b; Singh et al. 2016). Usually the metal salts precursors and plants or microorganisms are mixed and fabricated to develop nanostructures. The terpenoids, phenolic acid, carbohydrates, proteins, vitamins and alkaloids will work as capping reducing agents for the development of sustainable nanostructure (Prasad 2014; Bhuyan et al. 2015; Prasad et al. 2016, 2018; Srivastava et al. 2021). Metal oxide-based photocatalysts like mesoporous TiO_2 are also well-studied catalysts for the degradation of dyes present in water (Ahirwar et al. 2016). Most of the 3D metal-based materials are used as adsorbents and catalysts for wastewater treatment through adsorption and photo-degradation respectively. TiO_2 is a well-known photocatalyst used in the degradation of dyes present in water, due to their strong oxidizing properties (Lian et al. 2020). Most of the functionalized nonporous zeolites type materials studied for other catalytic activities can also be studied for adsorption as well as catalytic activities for wastewater treatment (Ahmed and Sakthivel 2017; Yadav et al. 2016; Ahmed et al. 2016). Bimetallic oxide-based nano-catalysts are summarized in Table 17.3.

17.4 Conclusion

The whole chapter presents the detailed study of nanomaterials' applications in waste water treatment. Varity of nanomaterials in different morphology, shape and size we studied as nano-adsorbent and nano-catalyst for the adsorption of various effluents from waste water are summarized in the current chapter. The different chemical techniques (viz. hydrothermal, co-precipitation and sol-gel) used for the synthesis of nano-adsorbents and catalysts are stated with examples. Metal-based nano-adsorbents and nano-catalysts in the form of oxides, and bimetallic, and tri-metallic oxides are discussed in details. Considerable work has been done to develop magnetic metallic oxides which were studied for the removal of toxic metal ions and are easy to separate from the water. The nano-types of such materials are very interesting due to the small size and large surface area. Moreover the porous silica, alumina and TiO_2 nanomaterials are also included in the chapter due to their potential application in metal ion capture and photocatalytic degradation.

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