REVIEW

Nanoadsorbents for wastewater treatment: next generation biotechnological solution

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Abstract

Day by day, the water sources are increasingly being adulterated due to various reasons including the uncontrolled discharge of pollutants from the point and nonpoint sources. Therefore, it is a timely need to develop suitable, inexpensive and efficient treatment techniques for water purification. This review aims at evaluating different water treatment technologies, their basic principles, cost and suitability for pollutants' removal from wastewater. Among various water treatment technologies, adsorption technique appears to be techno-economically more attractive due to its inexpensiveness, universality and environment friendliness. Here, wide varieties of adsorbents (silica gel, activated alumina, clays, limestone, chitosan, activated carbon, zeolite, etc.) and their capacities for pollutant removal are described. The limitations of conventional adsorbent applications for water treatment are also discussed. Recently, nanotechnology has introduced nanoadsorbents, which have drawn additional attention due to their unique properties and are considered to be the viable alternative to conventional adsorbents. The potential applications, separation and regeneration of nanoadsorbents for wastewater treatment are also included in this review. Furthermore, prospects including commercial and health aspects of nanoadsorbents are also added.

Keywords Nanoadsorbent · Wastewater treatment · Regeneration · Adsorption · Nanotechnology · Biotechnological solution

Introduction

The first United Nations World Water Development Report addressed water's importance as "Water for People Water for Life." Water is the prime need for sustaining all lives on the earth and plays a key role in maintaining ecosystems (Nemerrow 1978; Lehr et al. 1980; Franklin 1991; Helmer and Hespanhol 1997). But in the twenty-first-century reliable access to clean water remains a major global challenge (Qu et al. 2013a, b). Rapid population growth, enormous industrialization, enhancement of agricultural activities to meet food demand, other geological, environmental and global changes have contributed to both the increased variety and volume of pollutants continuously contaminating the water sources (Nemerow and Dasgupta 1991; Tchobanoglous and Franklin 1991; Yang 2011; Ali and Aboul-Enein 2004; da Silva and Gouveia 2020; Karimi-Maleh et al. 2019, 2020; Shamsadin-Azad et al. 2019). As a result, the quality of water sources for industrial, agricultural and human consumption is worsening globally. Therefore, water quality preservation and improvement become a serious concern for scientists, water regulatory authorities and governmental agencies (Ali et al. 2012). Generally, the water pollutants are classified as inorganic, organic and biological pollutants (Gupta et al. 2012; Ali et al. 2019; Elsayed et al. 2019; Karimi-Maleh et al. 2020).

In the last couple of decades, various technologies for water purification and recycling were reported in the literature. These technologies differ in their physical, chemical, biological, thermal and electrical principles. The most

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important water purification technologies include screening, coagulation–flocculation, filtration, biological treatment, oxidation, reverse osmosis, ion exchange process, distillation, electrochemical dialysis, adsorption, etc. (Barakat 2011; Wasewar 2010; Gupta et al. 2012; Hoque et al. 2018; Tlili and Alkanhal 2019; Blanco et al. 2019). Among these purification techniques, adsorption processes are the most popular treatment method due to its universality, low construction and maintenance costs and ease of operation (Gupta et al. 2012). The removal capacity of inorganic, organic and biological pollutants from wastewater via adsorption can be as high as 99.9% (Ali et al. 2012). The attributes which determine adsorption efficiency are porosity and pore size of adsorbents and their surface area (Singh and Kaushal 2013). Currently, common adsorbents used for wastewater purification process include clays (El-Guendi et al. 1995), activated alumina (Singh and Pant 2004), silica gel (Do 1998), limestone (Aziz et al. 2008), activated carbon (Aggarwal et al. 1999), zeolites (Ming and Dixon 1987), chitosan (Crini 2006), etc. Among all of them, activated carbon is the most extensively used adsorbent (Babel and Kurniawan 2003). Nevertheless, despite its versatility, activated carbon suffers from several shortcomings. Activated carbon shows limited porosity, surface area and pore volume and loses its adsorption capacity quickly. Recovery of adsorption capacity upon regeneration is also poor (Freeman 1998; Taiwo and Adesina 2005). Generally, surface functionalization of the conventional adsorbents is an attractive method to improve the adsorbent performance. However, these functionalized materials require complex, multistep synthesis process which is not amenable for their large-scale production and therefore are expensive (Bhatnagar et al. 2013). In addition, convenient separation of the adsorbent from treated water and removal of pollutants at ppb levels are major challenges for conventional adsorbent (Li et al. 2011; Mohan and Pittman 2006).

Recent advances in nanotechnology showed the potential routes to synthesize nanomaterials are mechanical, solgel, combustion, sonochemical, chemical, microwave and so on from numerous sources such as biomass residues, agriwastes and residues (Biswas et al. 2017, 2019; Rangari et a. 2017). To overcome the aforementioned limitations of the conventional adsorbents, researchers introduced next-generation adsorbents (nanoadsorbents) for the water treatment system (Förstner and Wittmann 1985; Ali et al. 2012). Nanoadsorbent possesses an array of exceptional physical and chemical properties like high surface area, high chemical reactivity, conductivity, catalytic, selectivity, magnetic and optical properties. The high surface area offers a higher number of active sites for various pollutants to react with nanoadsorbent. These properties make them better adsorbent than their corresponding bulk materials (Kalfa et al. 2009; Liu et al. 2005; Hurt et al. 2006; Ilisz et al. 2004). Nanoadsorbents open a potential market in environmental industries at both the domestic and international levels. It is expected that the market value of the nanoadsorbents will reach €1.6 trillion by 2020 and create over a million jobs (Donlan et al. 2009).

Nature of water pollutants

Before addressing water treatment technologies, it is important to have a good idea of the nature of the major water pollutants both qualitatively and quantitatively. The nature and amount of the pollutants found in wastewater vary based on sources such as agricultural, industrial and municipal discharge effluents. The toxicity of these pollutants is only observed when they exceed the permissible limit. Generally, wastewater pollutants are classified into three categories such as organic, inorganic and biological pollutants. These water pollutants may be present in water either in colloidal, solvated or in the suspended form (Gupta et al. 2012).

Inorganic pollutants

Inorganic pollutants include heavy metals found in various waste streams that include industrial wastewater, river sediments, mine drainage, ashes and electronic scraps wastewater (Hoque and Philip 2011). Heavy metal comprises cations of lead, zinc, chromium, cadmium, iron, copper, arsenic and mercury, and anions of nitrates, sulfates, phosphates, fluorides, chlorides and oxalates are common inorganic water pollutants (Gaston 1979; Hutson and Roberts 1990; John 1990). The maximum contaminant level (MCL) established by USEPA (US Environmental Protection Agency) for some heavy metals is mentioned in Table 1 (Babel and Kurniawan 2003).

Table 1 List of heavy metals and their maximum concentration limit

Heavy metals	(MCL) Maximum concentration limit (mg/L)
Mercury	0.00003
Arsenic	0.05
Lead	0.006
Cadmium	0.01
Chromium	0.05
Zinc	0.8

Organic pollutants

The organic pollutants are very stable in the environment due to one or more cyclic rings in chemical structure, absence of polar functional group and a few halogen substitutions (Ali et al. 2012). The toxic organic pollutants are comprised of pesticides (insecticides, fungicides, herbicides), phenols, fertilizers, plasticizers, biphenyls, polybrominated biphenyls, formaldehyde, greases, detergents, hydrocarbons, oils, pharmaceuticals, etc. (Gaston 1979; Hutson and Roberts 1990; John 1990). Agricultural and chemical industries are the most important sources of organic pollutants.

Biological pollutants

Various biological agents like viruses, bacteria, algae, fungi, amoeba and other worms also pollute water and induce different types of diseases (Gaston 1979; Hutson and Roberts 1990; John 1990). The water pollutants cause serious health problems like kidney damage, nausea, rheumatoid arthritis, chronic asthma, diseases of the nervous system, circulatory system and, in extreme cases, death (Barakat 2011).

Wastewater treatment technology

The wastewater treatment technologies have been classified into three stages: (1) primary, (2) secondary methods and (3) tertiary (Fig. 1). The primary stage includes screening, filtration, coagulation–flocculation, centrifugation, gravity and sedimentation methods. Generally, screening is the first operation in wastewater treatment. These techniques can protect the equipment and/or water plant from potential damage or blockages by removing large non-biodegradable floating solids or sediments present in the wastewater, such as cloth debris, paper, fiber, fecal solids, wood, hair and kitchen refuse. Sometimes, activated silica, alum or iron materials are used to remove non-settable solids by coagulation process (Franklin 1991; Nemerow and Dasgupta 1991; Latifossglu et al. 1997).

In secondary water treatment systems, different biological microbes (usually bacterial and fungal strains) are employed for the removal of different pollutants (Franklin 1991; Kato et al. 1997; Zinkus et al. 1998). These microbes produce different by-products like water, carbon dioxide and ammonia gas after reacting with organic pollutants (Pendashteh et al. 2010; Joss et al. 2006). Tertiary water treatment technologies are needed to achieve a water quality that meets the existing standards for human consumption. This is the final treatment strategy for water purification. The technique includes oxidation, precipitation, ion exchange, adsorption, reverse osmosis, etc. (Gupta et al. 2012).

These water treatment technologies are applied for groundwater, surface water and wastewater treatment purposes. Due to uncontrolled industrial discharge, groundwater is adulterated by toxic metal cations and anions continuously. Tertiary treatment is necessary to remove these pollutants. On the other hand, both secondary and tertiary treatment methods are required for surface water, where contamination is initiated by biological, organic and inorganic pollutants. Generally, utilization of tertiary water treatment methods varies depending on wastewater nature, economic feasibility and efficiency of pollutant removal. For better water treatment a good hybridization of these three treatment technologies is always required. (Gupta et al. 2012).

Current state of the art for wastewater treatment

Generally, water treatment processes are employed for three purposes, i.e., wastewater treatment, water source reduction and recycling. Researchers all over the world are looking for low-cost and effective methods for wastewater treatment and reuse (Gupta et al. 2012). Over the last few decades, numerous processes have been applied to remove metal ions from industrial wastewater. The commonly used techniques for removing water pollutants are filtration, biological treatment, ion exchange process, chemical precipitation, oxidation, electrochemical dialysis and adsorption (Barakat 2011). Here, these water treatment techniques are shortly discussed mentioning their mechanism of action, advantages and shortcomings.
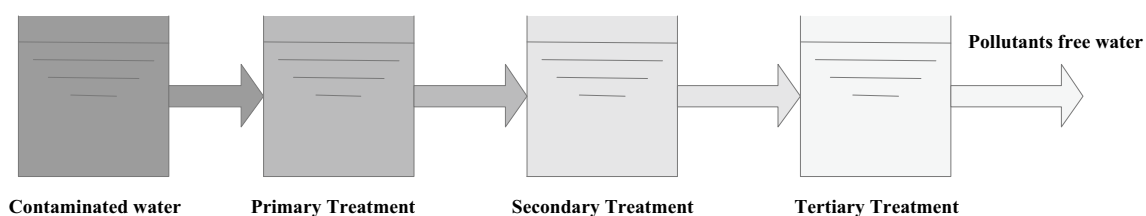

Fig. 1 Different wastewater treatment stages

Chemical precipitation

In most of the countries, chemical precipitation is a commonly applied method for the elimination of heavy metal ions from wastewater due to availability of cheap precipitant like lime and limestone (Mirbagherp and Hosseini 2004; Aziz et al. 2008). Besides these, alum, sodium bicarbonates, ferric chloride and ferrous sulfate are also commonly applied precipitants for the chemical precipitation purposes (Gupta et al. 2012). Inorganic effluent with a metal concentration of higher than 1000 mg/L can be treated by lime precipitation as reported in the literature (Aziz et al. 2008). Limestone plays the prime role to remove heavy metals via chemisorption reaction because of its rough surface which provides higher contact areas to react even at very low concentrations. Besides, the pH of the resultant solution goes up above the solubility value due to calcium carbonate of limestones, thus allowing metal impurities to precipitate via forming metal oxides and carbonates (Sturchio et al. 2003; Xu et al. 1996; Stipp et al. 1992). The theoretical mechanism of heavy metal elimination via chemical precipitation technique is shown by the following reaction (Wang et al. 2004):

$$M^{2+} + 2(OH)^- \leftrightarrow M(OH)_2 \downarrow$$
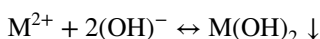
where M^{2+}, OH^- and $M(OH)_2$ imply dissolved metal ions, precipitant and insoluble metal hydroxide, respectively. Here, the removal efficiency can be improved by adjusting the temperature and alkalinity (pH 9–11) (Wang et al. 2004). Aziz et al. (2008) reported that limestone significantly removed (more than 90%) Cd^{2+} and Cu^{2+} from aqueous solution at pH 8.5 (Aziz et al. 2008). Though it can remove heavy metals from industrial effluent, the requirement of a large amount of chemicals, excessive sludge formation, poor metal precipitation, slow settling, presence of oil and grease as well as the aggregation of metal precipitates during treatment make its application limited for a broad range of applications (Aziz et al. 2008).

Biological treatment

In this system, organic compounds are degraded in modified sludge tanks through the application of aerobic or anaerobic conditions, under the strict control of temperature and the system's chemical oxygen demand (COD) (Britto and Rangel 2008).

Aerobic treatment In aerobic treatment, bacteria and fungi are the most widely used because of their ability to destroy pollutants in the presence of oxygen in biological treatment processes. The prime benefit of the aerobic approach is the production of the non-toxic end product as well as significant influence to remove nitrates, phosphates, BOD, COD,

dissolved and suspended organics, etc. (Gupta et al. 2012). Different enzyme-secreting bacteria in the wastewater are selected for their pollutants breakdown capacity (Rai et al. 2005). For example, *Kurthia sp* strain can efficiently decolorize (92–100%) a wide variety of dyes (organic pollutants) like magenta, pararosaniline, brilliant green, ethyl violet, crystal violet and malachite green (Sani and Banerjee 1999). The successful decolorization of pollutants depends on several factors like temperature and initial pH of the effluent, the concentration of pollutants and the concentrations of dyes (Christie 2007). The aerobic treatment has some shortcomings (Husain 2006; Kulla 1981; Gupta et al. 2012):

(i) development of aerobic bacterial strains is costly and challenging
(ii) no uniform decomposition of synthetic dyes and
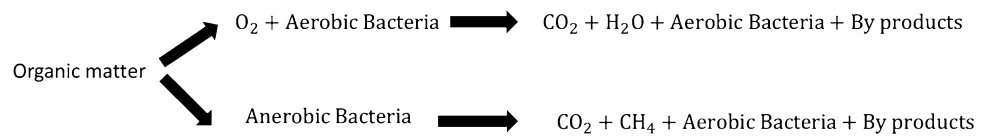(iii) costly due to high production of biosolids and its management

Anaerobic treatment The anaerobic decomposition or putrefaction occurs when dissolved oxygen is not available in the wastewater (Venkata Mohan et al. 2007; Van Der zee and Villaverde 2005). Delee et al. (1998) demonstrated the potential applications of anaerobic approach for a wide range of artificial dyes. The anaerobic treatment process offers the following advantages over an aerobic treatment process (Delee et al. 1998).

- cheap and alternative of aerobic process where costly aeration and bulk sludge are a major concern,
- dye decolorization can be done via the reduction process with low cost but efficient removal of BOD levels,
- heavy metals can be removed via sulfate reduction,
- high effluent temperatures are favorable and
- high pH of effluent can be reduced and thus initiates neutralization of organics.

Zee van der et al. (2001) showed the treatment and decolorization of 20 azo dyes using anaerobic granular sludge and investigated their viability of the process (Van der Zee et al. 2001). Despite these advantages, still, the anaerobic process faces some challenges like removal of BOD is not sufficient, dyes and other organics are not neutralized, nutrients (N, P) are not eliminated and sulfates form sulfide (Delee et al. 1998).

This treatment process requires 20 to 200 US$ per million liters depending on the material used for treatment purposes (Franklin 1991; Zinkus et al. 1998). A simplified representation of the biological treatment is shown in Fig. 2.

$$\text{Organic matter} \begin{cases} O_2 + \text{Aerobic Bacteria} \longrightarrow CO_2 + H_2O + \text{Aerobic Bacteria} + \text{By products} \\ \text{Anaerobic Bacteria} \longrightarrow CO_2 + CH_4 + \text{Aerobic Bacteria} + \text{By products} \end{cases}$$

Filtration technology

This technology is considered to be the primary technique that received huge attention for drinking water and wastewater treatment applications. This approach showed excellent performance not only for removing suspended solids and organic matters but also for heavy metals and other inorganics (Barakat 2011). Numerous membrane filtration techniques are available that include microfiltration, nanofiltration, ultrafiltration, reverse osmosis and so on which are applicable depending on particle size to be removed (Fig. 3) (Barakat 2011; Gupta and Suhas 2009). To evaluate the potential of ultrafiltration technique, Saffaj et al. (2004) used cheap $ZnAl_2O_4$–TiO_2 ultrafiltration membranes and achieved 93% Cd (II) and 86% Cr(III) rejection, respectively. This high removal efficiency might be because of higher interactions between the divalent cations (pollutant) and the filter membrane (Saffaj et al. 2004). Based on membrane properties, this ultrafiltration method can attain more than 90% removal capacity at a wide pH range and 2–5 bar of pressure for metal concentration with a range of 10 to 112 mg/L (Kurniawan et al. 2006a, b). Recently, a polymer-supported ultrafiltration method has been introduced for the removal of heavy metal ions from industrial effluent. This method uses soluble polymeric ligand that binds to metal or ions of interest and form macromolecules complexes. These complexes are rejected by the membrane and produce an effluent without targeted metal ions (Rether and Schuster 2003).

In addition to the ultrafiltration method, sometimes polymers have been used as binders to remove metal ions reported in the literature. Those polymers include carboxyl methylcellulose (Barakat 2008), polyethyleneimine (Aroua et al. 2007), diethylaminoethyl cellulose (Trivunac and Stevanovic 2006), etc. The selection of appropriate membranes for filtration technique depends on several factors including characteristics of the wastewater, heavy metals concentrations, pH, temperature, compatibility with the polluted water and cleaning agents to abate surface fouling (Madaeni and Mansourpanah 2003). However, surface fouling exhibits prime difficulty in the filtration approach, which reduces productivity and wide application (Zularisama et al. 2006). Researchers use both physical and chemical procedures for recovering membrane permeability. The membranes have been reported to regain the initial membrane efficiency by the treatment with different chemical agents like sodium hypochlorite (NaOCl), sodium hydroxide (NaOH), hydrochloric acid (HCl) and nitric acid (HNO_3). But these regeneration processes are very expensive and may cause unavoidable membrane damage and generate toxic by-products (Park et al. 2002). The cost of this water treatment process ranges from 25 to 450 US$ per million liters of treated water (Franklin 1991; Nemerow and Dasgupta 1991).

Oxidation

Oxidation is one of the most widely used approaches for decolorization of organic matters like dyes. Generally, two forms of oxidation processes are used for industrial wastewater treatment such as chemical oxidation and UV-assisted oxidation. Various kinds of oxidizing agents such as chlorine
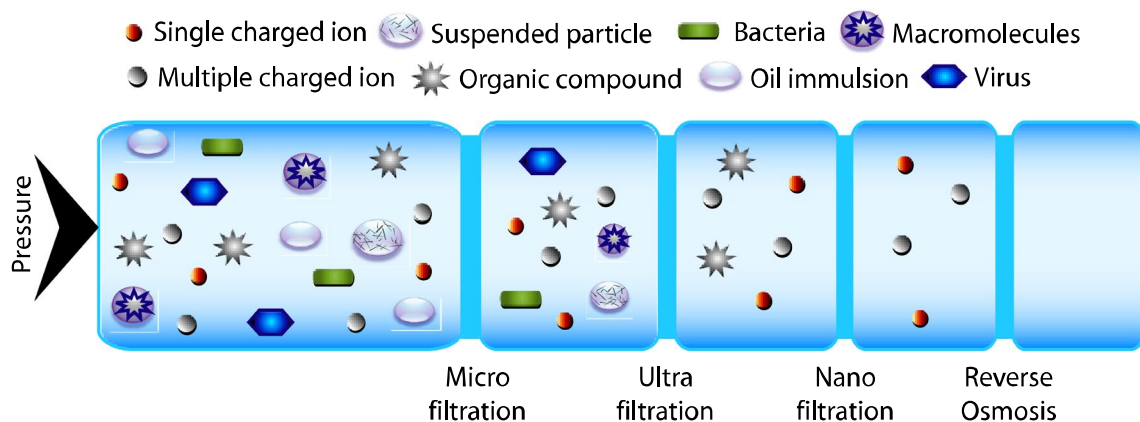

Fig. 3 Schematic representation of filtration technology

(Namboodri et al. 1994), hydrogen peroxide (Hage and Lienke 2006), Fenton's reagent (H_2O_2 + Fe catalyst) (Wang 2008) and ozone (Wu et al. 2008) are applied for the effluent treatment after the primary treatment (sedimentation) process. Oxidation processes completely degrade the pollutants into low molar mass substances such as nitrogen, sulfates, aldehydes and carboxylates (Gupta and Suhas 2009). It is worth mentioning that the pH of the solution and applied catalysts greatly influence the efficiency of the oxidation process. For example, in the decomposition of the metal–dye complex like chromium, copper, iron and nickel are generated and exhibit a catalytic effect that enhances decolorization. Chlorine is used as a strong oxidizing agent in low-cost approach for decolorizing dye water, but unfortunately, it shows unavoidable side reactions, generates toxic compounds such as trihalomethane, increasing adsorbable organic halogen content in the treated water, and liberates metals in metal–dye complex degradation which encounters corrosion in metallic containers (Gupta and Suhas 2009). The cost of wastewater treatment by oxidation ranges from 100 to 2000 US$ per million liters of clean water (Gupta et al. 2012).

Advanced oxidation processes (AOPs) Sometimes, a single oxidation process fails to degrade the organic pollutants completely. The process where more than one oxidation processes are applied simultaneously to produce powerful, non-selective hydroxyl radicals in an aqueous medium for pollutant degradation is termed as advanced oxidation processes (Garciamontano et al. 2006). These hydroxyl radicals are highly reactive but unstable due to their high oxidation potential (Mahamuni and Adewuyi 2010). In 2009, Klavarioti and his co-worker discussed and compared different types of advanced oxidation processes, such as ozone-assisted chemical oxidation processes, combined ozone and peroxide, ultraviolet-enhanced oxidation such as UV/H_2O_2, UV/ozone, UV/air, wet air oxidation, electrochemical oxidation and catalytic wet air oxidation (Klavarioti et al. 2009).

The reaction between hydroxyl (.OH) radical and natural organic matter proceeds in three steps: (i) addition of .OH to double bonds, (ii) abstraction of H-atom resulting carbon-centered radicals and (iii) organic substituents donate an electron to .OH, radical resulting carbon-contained radicals, which react very quickly with oxygen and form peroxyl radicals. At the end of the reaction, ketones or aldehydes and/or carbon dioxide are formed (Kleiser and Frimmel 2000). The rate of the reaction depends on several factors like oxygen, the presence of free radicals and also pollutant concentrations (Parsons 2004). Techniques included in the advanced oxidation processes are photo-Fenton's oxidation process (Moncayo-Lasso et al. 2009), ultraviolet (UV) photolysis (Gjessing and Kallovist 1991) and ultrasonic irradiation (Nagata et al. 1996). At ambient temperature

and pressure, advanced oxidation processes are capable of complete degradation of dyes and also show benefit over biological approach for wastewater treatment comprising toxic or bio-contaminants (Matilainen and Sillanpaa 2010).

Namboodri and Walsh (1996) performed a comparative study between oxidation and advanced oxidation for color removal. They reported that the oxidation alone can only remove 10–20% color, but after the addition of peroxide the removal efficiency reached 90% (Namboodri and Walsh 1996). De Witte et al. (2009) investigated the advanced oxidation process with a 120 mL/min O_3 flow rate for removal of ciprofloxacin (pharmaceutical activated product). The highest degradation was found with the highest concentration of ozone (660–3680 ppm) and the lowest concentration of ciprofloxacin (22.64–135.81 μM). The degradation was pH-dependent. At pH 10, the highest concentration (99%) of the degraded product (diethylene ciprofloxacin) was identified using HPLC–MS technique (De Witte et al. 2009). Common pharmaceuticals (diclofenac) were completely removed within 100 min from water by solar-driven photocatalysis. The stock solution concentration was 50 mg/L (Pérez-Estrada et al. 2005). Though advanced oxidation processes have proven potential for dye removal, it has some shortcomings like the formation of undesired by-products, incomplete mineralization, dependent on pH, a high degree of pretreatment obligation and operational challenges. Moreover, the processes are found to be pretty expensive in the small-scale sector, especially in developing countries (Gupta and Suhas 2009; Comninellis et al. 2008).

Ion exchange treatment

Generally, ion exchange involves adsorption of dissolved and colloidal matters from industrial effluent (ionic matter) (Kurniawan et al. 2006a, b). A solid sorbent (ion exchanger) can accomplish exchanging cations or anions with the surrounding substances (Choi et al. 2007). This treatment can remove pollutants at lower concentrations (up to 250 mg L^{-1}) (Gupta et al. 2012). There are two types of ion exchange membranes like anion or cation exchangers. In anion exchanger, the membrane infused with the positively charged ions (NH_3^+, NH_2^+, NR_3^+) which permit anions to pass through, but discard cations. On the other hand, the cationic membranes infused with negatively charged ions (SO_3^{2-}, $RCOO^-$, PO_3^{3-}) permit cations to pass through rejecting anions. Based on the preparation method, the membranes are categorized into heterogeneous and homogeneous (Xu 2005). Polymeric membrane (styrene and acrylic resins) is mostly used because of its structural integrity, chemical inertness and great selectivity (Dickert 2007). The basic principle of the ion exchange process is described in Fig. 4.

Synthetic organic ion exchange resins (Barakat 2011), zeolites (Vassilis 2010) are found to remove pollutants such

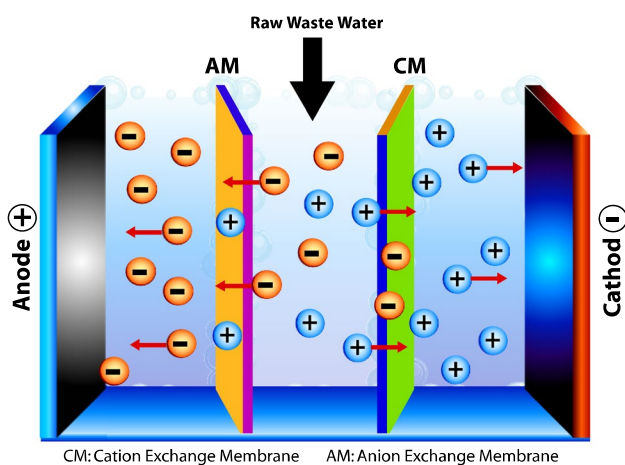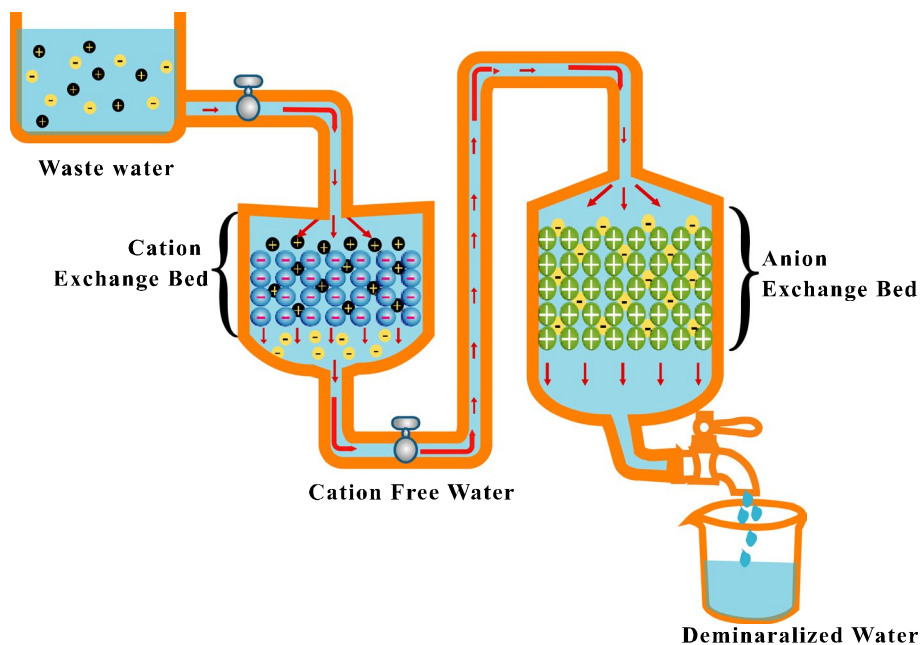**Fig. 4** Schematic representation of ion exchange treatment

Fig. 5 Schematic representation of the electrodialysis technique for water treatment

as Cr^{3+}, Fe^{3+}, Cu^{2+}, Pb^{2+} and NH_3. However, this kind of technique has some disadvantages like expensive membranes, poor electrochemical properties and high porosity (Xu 2005; Nagarale et al. 2006). Moreover, it needs backwashing and regeneration after long use. Sometimes, the organic materials fix the resin and cause the appearance of fouling in the membrane (Üstün et al. 2007). Ion exchange is a low-cost reversible process because the adsorbent resin can be used for a long period before replacement is mandatory (Homem and Santos 2011). The cost varies from 50 to 200 US\$ for the treatment of one million liters of wastewater (Gupta et al. 2012).

Electrodialysis

Electrodialysis technique for water pollution management was developed based on electrochemistry principles, where ionized species are separated by applying a high voltage on both sides of an ion exchange membrane (Barakat 2011) (Fig. 5). The basic difference between electrodialysis and ion exchange membrane treatment process is that in the ion exchange process the concentration difference (diffusion and dialysis) is the driving forces, whereas, in electrodialysis, the externally applied electric potential is the driving force (Koter and Warszawski 2000). Generally, electrodialysis cell is made of thin films of polymeric products having either anionic or cationic in nature (anion exchange membrane and cation exchange membrane) (Chen 2004). The ionic functional groups of the membrane originate from the chemical nature of the polymer (Blackburn 1999). Such anionic exchange membranes have positive fixed charges due to the presence of quaternary amine groups in the polymer; in contrary, cation exchange membranes have negative charges resulted due to the presence of sulfonic acid groups in the polymer. A solution containing ionic species is added to an electrodialysis cell. Then, potential is applied by the electrodes on both sides of the dialysis cell. The anionic exchange membranes with fixed positive charges attract anions and allow them to pass through and reject cations. The reverse scenario works in a cationic exchange membrane (Blackburn 1999).

Electrodialysis can provide high selectivity for successful separation and recovery of the desired compounds and metals (Scott 1995). The electrodialysis cell can be used as a reactor for obtaining desirable products from both

electrolytes and chemicals. The successful operation of the cell depends on selecting the cell structure and the right operating conditions. Electrodialysis ensures high water recovery after treatment. It also reduces water hardness. The flexibility to operate when needed and no requirement of any toxic chemical make this treatment process attractive compared to other processes (Koter and Warszawski 2000).

Moreover, electrodialysis not only removes undesirable impurities from water but also produces highly concentrated liquor for recovery for reuse, such as valuable metals Cr and Cu (Blackburn 1999). The success of the electrodialysis depends on membrane characteristics, feedstock nature, composition and process parameters such as initial flow rate, operation conditions and cell geometry (Mohammadi et al. 2004). Due to long use and higher current, membrane fouling occurs and consequently decreases the efficiency of the membrane. The presence of other ions also decreases the efficacy of the membrane separation process (Korngold et al. 1998; Messalem et al. 1998; Hansen et al. 1997). Pretreatment is necessary to avoid membrane fouling. For better performance, electrodialysis process needs a continuous supply of new feedstock, continuous monitoring on the operation and regular maintenance to avoid damages. The treatment cost ranges from 15 to 400 USD (Gupta et al. 2012) (Table 2).

Sometimes, combined techniques are applied for water pollutants removal; for example, in India, Kenya, Senegal and Tanzania, the most commonly used technique for defluoridation is the Nalgonda technique. In this method, the required amounts of alum, bleaching powder and lime are applied to treat water. After that, the water proceeded with disinfection, flocculation, filtration and sedimentation. The whole process takes 2–3 h in a batch experiment. But this treated water contains 2–7 mg/L residual aluminum concentration which is higher than the WHO standard (0.2 mg/L) (Ayoob et al. 2008; WHO 2004; Meenakshi and Maheshwari 2006).

Adsorption

Reverse osmosis, ion exchange and oxidation are commonly used technologies for wastewater treatment. These methods introduce toxic secondary pollutants into the ecosystem which make their use limited in wastewater treatment at potable and industrial levels (Crini 2005; Gaya and Abdullah 2008). Therefore, there is a need to explore other alternative techniques, which are effective in removing pollutants and are economical at the same time. The increasing environmental awareness and concern among scientists led to developing the adsorption technique, which is effective for purification and separation of water and wastewater

pollutants. Among all the water purification treatment methods, adsorption is one of the popular techniques which is meant to be an efficient, economical and eco-friendly technique for wastewater treatment (Crini 2006). Adsorption is the mass transfer process where pollutants are concentrated or adsorbed on a solid substrate from the liquid phase or gaseous surroundings in contact with the substrate. Here, pollutants are referred to as adsorbate and substrate as an adsorbent (Singh and Kaushal 2013).

Kayser in 1881 first introduced the term adsorption in the literature and postulated the adsorption process as a surface accumulation of materials (Dabrowski 2001). It is a surface phenomenon if the adsorbate or the species are attached to the adsorbent surface physically without any chemical bonding; the process is denoted as physisorption. In physisorption, the bonding forces are van der Waal forces, weak H-bonds, hydrophobicity, interaction due to polarity and steric effect, dipole–dipole interaction, π–π interaction, etc. (Ali et al. 2012). On the other hand, a chemical bonding also participates in the adsorption process, referred to as chemisorption (Ali et al. 2012). It is hard to remove chemisorbed compounds from the substrate (Yadla et al. 2012). The pollutants are adsorbed onto solid sorbents surface in three stages: 1. transport of pollutants from balk solutions to the adsorbent; 2. adsorption on the surface of any particles; and 3. transport within the absorbent particle (Barakat 2011). The mechanism of adsorption is shown in Fig. 6.

Different factors affect the adsorption process, such as pH, temperature, types of adsorbates and adsorbents, the concentration of pollutants, the presence of other pollutants, contact time, surface functional group and other atmospheric and experimental conditions. Sometimes, prefiltration is required to avoid the unwanted effect of suspended particles, oils and greases in water during adsorption (Ali 2010). The adsorption process is especially suited for wastewater treatment and more so in the case of high concentration but a low volume of wastewater. Watonabe and Ogawa (1929) first demonstrated the application of activated carbon heavy metals adsorption. The adsorption process is effective to remove soluble as well as insoluble organic pollutants. The removal efficiency can be up to 99.9% as reported in the literature (Ali et al. 2012). As a result, researchers considered that adsorption is the greatest universal method as it can remove various types of inorganic as well as organic pollutants from wastewater. It is economically viable for the treatment of a large volume of wastewater due to low construction, maintenance and operation cost of the adsorption equipment (Gupta et al. 2012). The adsorption method has the benefit to remove analytes instead of creating potentially more hazardous metabolites (Rivera-Utrilla et al. 2009; Putra et al. 2009). Many industries like dyes, textiles, paper and plastic

Table 2 Technical advantages and drawbacks of existing treatment technologies

Treatments	Advantages	Drawbacks	References
Chemical Precipitation	Process simplicity Cheap and convenient safe operations	Too much sludge production Poor settling Slow metal precipitation Aggregation of metal precipitates	Aziz et al. (2008)
Biological treatment	Cost-competitive Suitable for a variety of dyes Removal of BOD, COD, dissolved and suspended organics	Production of large quantities of biosolids Limited design and operation flexibility Low biodegradability of the pollutants Requires larger space and longer times for decolorization–fermentation processes	Franklin (1991), Zinkus et al. (1998), Robinson et al. (2001), Bhattacharyya and Sarma (2003), Crini (2006), Gupta et al. (2012)
Filtration technology	Nanofiltration and ultrafiltration techniques: Virtually all types of dyestuffs can be removed Reverse osmosis: Capable of decoloring and desalting process Produced water by reverse osmosis close to pure H_2O.	Pretreatment require for primary solid removal High working pressures High cost of membrane Relatively short membrane life Higher level of technical expertise required Significant energy consumption	Marmagne and Coste (1996), Al-Bastaki (2004), Marcucci et al. (2001), Sostar-Turk et al. (2005), Gupta (2009), Franklin (1991), Nemerow and Dasgupta (1991)
Oxidation	Require low quantities of chemical and short reaction times Complete oxidation can reduce the complex molecules to CO_2 and water	Water-insoluble disperse and vat dyes resistant to decolorization by oxidation Chlorine causes inevitable side reactions that result in the generation of organochlorine compounds (e.g., toxic trihalomethane, etc.) May lead to corrosion in metallic vessels due to release of metals in metal complex dyes Treatment process is pH-dependent and produces sludge Too much oxidant and/or catalyst used for oxidation Ozone and hydrogen peroxide, metals, metal oxides and metal salts normally toxic to microorganisms	Gupta (2009), Namboodri et al. (1994), Oller et al. (2011), Gupta et al. (2012), Gupta (2009), Namboodri et al. (1994), Oller et al. (2011), Gupta et al. (2012)
Advanced oxidation treatment	Organic pollutants oxidized to CO_2 Accelerating pollutants removal via solar, UV–Vis rays or ultrasonic radiation No sludge production Considerable reduction in chemical oxygen demand Non-selective pathway allows the treatment of multiple pollutants in a single step	No possibility of complete mineralization Process pH-dependent Involvement of extra energy sources (solar, UV–Vis rays or ultrasonic) Limitation of fine catalyst separation from the treated liquid, fouling of catalysts and light penetration Deteriorated potentiality by chlorine oxidation, causing the formation of hypochlorite or chlorine High cost, particularly for the small-scale sector	Gupta (2009), Foo and Hameed (2010), Gupta et al. (2012)
Ion exchange treatment	Reversible process Low operational cost Low energy consumption	Membranes—frequently contain large pores, bad electrochemical properties and too expensive Appearance of fouling Cannot handle concentrated metal solution Ion exchange non-selective High sensitivity to the pH of a solution Improper for treating chlorinated pesticides Microorganisms can reduce the exchange capacity	Foo and Hameed (2010), Xu (2005), Nagarale et al. (2006), Üstün et al. (2007), Barakat (2011), Homem and Santos (2011), Gupta et al. (2012)

Table 2 (continued)

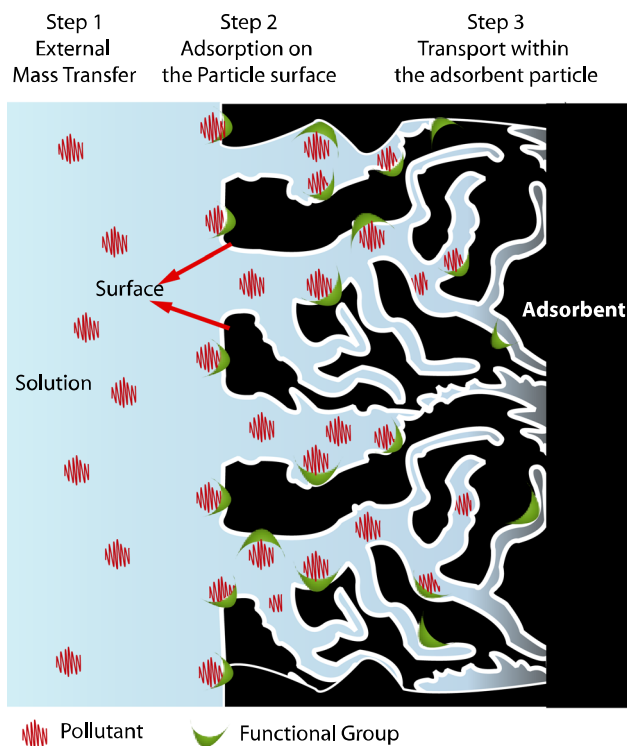| Treatments	Advantages	Drawbacks	References
Electrodialysis	Provide high selectivity for separation Act as a reactor Reduce water hardness	Membrane fouling High energy consumption Presence of other ions reduces the efficiency Require clean feed, careful operation, periodic maintenance	Blackburn (1999), Gupta et al. (2012), Hansen et al. (1997)

–, not available

Fig. 6 Schematic representation of the adsorption process and its mechanism

discharge different types of dyes in the water. These dyes are detrimental to aquatic life (O'Neill et al. 1999). The removal of these color substances is very difficult because the dyes are intractable organic molecules, inert to aerobic digestion, stable under light, heat and oxidizing agents (Ravi Kumar et al. 1998). Adsorption shows the best results for the removal of various kinds of dyes (Ho and McKay 2003; Derbyshire et al. 2001; Jain et al. 2003).

Different conventional water treatment techniques like pre-/post-chlorination, coagulation/flocculation/filtration, cannot efficiently remove cyanobacterial toxins from wastewater (Lambert et al. 1996; Huang et al. 2007; Momani et al. 2008). For example, the flocculation treatment can eliminate cyanobacterial cells from water by an appropriate concentration of flocculent. But the treatment increases the concentration of cyanobacterial toxins by cell lysis, which cannot be removed by this treatment (Lepistö et al. 1994). Huang et al. (2007) reported successful removal of cyanobacterial toxins by the adsorption process (Huang et al. 2007).

Adsorption has advantages over conventional techniques, which are shown below (Volesky 1999):

- *Cheap* Adsorbent is often made from abundant agricultural or waste materials, which makes adsorbents cheap.

- *Metal selective* The metal sorbing capability of different kinds of biomass adsorbents can be dependent on metal ions as well as the nature of biomass, composition in the solution, preparation and physicochemical treatment of biomass.
- *No sludge generation* Unlike other techniques, no secondary sludge generation occurs with adsorption.
- *Regenerative* The adsorbent can be regenerated and used for several cycles of adsorption.
- *Metal recovery* It is possible to recover metal (pollutants) ions after being sorbed.

Major operating variables of adsorption

Based on the review of the latest published research on the application of adsorption process for water treatment, major operating variables are discussed below.

Effect of initial concentration and contact times on adsorption of pollutants

The initial concentration and contact time influence the metal ion (Cu^{2+}) adsorption shown in Fig. 7. At pH 6.0 and adsorbent dose 2 g/L, the adsorption experiment was carried out at initial concentration varying from 25 to 100 mg/L. The figure shows that the metal ions adsorption increased simultaneously with the increase in contact time at all concentration, and after 3 h, the adsorption became constant. It indicates that the contact time was not dependent on metal ion concentration. The optimum uptake period of Cu^{2+} adsorption was about 3 h. The
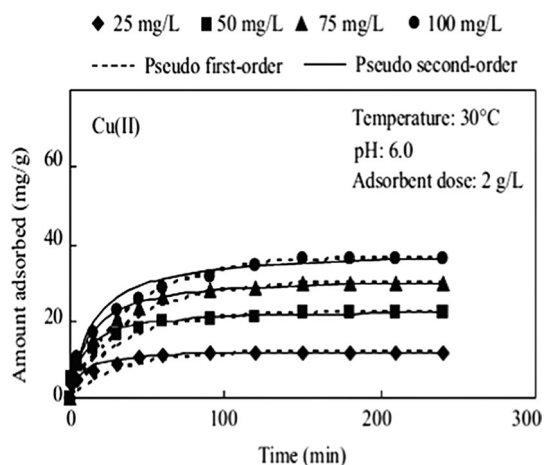

Fig. 7 Comparison of experimental contact time data and the fittings to pseudo-first-order and pseudo-second-order kinetic models for the adsorption of Cu (II) onto AC at different concentrations (Anirudhan, and Sreekumari 2011)
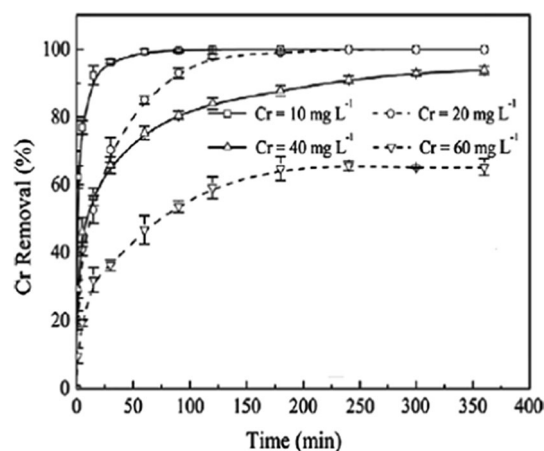

Fig. 8 Effect of initial Cr(VI) concentration on Cr(VI) removal (pH, 7.0; nZVI-to-MWCNTs mass ratio, 1:2; ZVI amount 0.1 g/L) (Lv et al. 2011)

adsorption of Cu^{2+} increased from 11.92 to 36.40 mg/g as the initial concentration changing from 25 to 100 mg/g (Anirudhan and Sreekumari 2011).

But the different scenario was found, when Lv et al. (2011) studied different initial concentrations (10–60 mg/L) of Cr(VI) for removal by nanocomposite. The complete removal was found at concentrations 10, 20 and 40 mg/L. However, at a concentration of 60 mg/L, the removal capacity decreased to 40% as shown in Fig. 8. The reason might be the lack of available sites for adsorption in the constant amount of nano-zerovalent particles for high Cr(VI) concentration (Lv et al. 2011).

Effect of pH on adsorption

pH has a direct effect on pollutant removal. The adsorbent should be stable under working pH conditions. For example, the efficiency of Cr(VI) removal reduced from 100% to 91% with increasing pH from 5.0 to 9.0. The reason is electrostatic repulsion between nano-zerovalent iron multiwalled carbon nanotube (composites) and dichromate ions. The positive zeta potential of nano-zerovalent iron multiwalled carbon nanotube (nanocomposites) at pH < 7.5 accelerated attraction of negatively charged Cr contaminant and resulted in the highest removal efficiency and kinetics. When pH raised above the pH (point of zero charges), negatively surface charge accelerated electrostatic repulsion (Lv et al. 2011; Pradhan et al. 1999; Das et al. 2000).

The influence of pH was further investigated with activated carbon at pH range (2–9) to remove Cu (II) and Hg(II) as shown in Fig. 9. The increase in the pH value of the solution increased the pollutant removal percentage. The point of zero charges of activated carbon (adsorbent) was 5.2. Below pH 5.2, the surface of the activated carbon remains positive
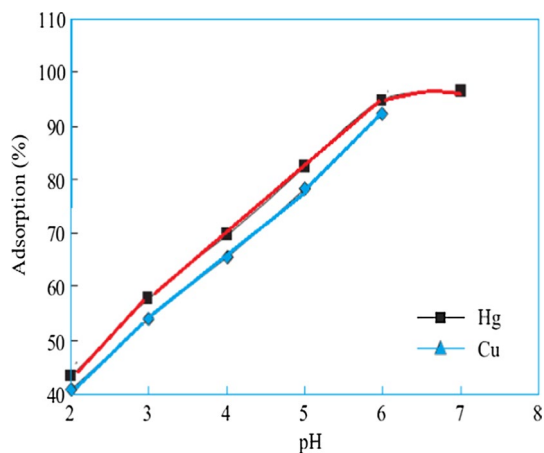
Fig. 9 Influence of pH on the adsorption of pollutants (Hg and Cu) by activated carbon. [condition: adsorbent dose 2 g/L; temperature 30 °C; equilibrium time 3 h; concentration 25 mg/L (Anirudhan and Sreekumari 2011)

due to the presence of H^+; as a result, electrostatic repulsion occurs between adsorbent and metal cations. On the other hand, above pH 5.2 the surface charge of the adsorbent becomes negative and electrostatic attraction occurs between adsorbent and cationic pollutants. The optimum value of pH for the removal of Cu (II) and Hg(II) was observed to be 6 and 7, respectively (Anirudhan and Sreekumari 2011).

Effect of ionic strength on adsorption

The ionic strength (i.e., electrolyte concentration) of a solution influences the binding of adsorbate and adsorbent. It influences the interface potential and width of the double layer around the adsorbent. Generally, the outer sphere complexes are more susceptible to ionic strength disparities than inner-sphere complexes of the adsorption process. For example, an experiment was performed in three different ionic strength solutions (0 M, 0.05 M and 0.1 M) containing Cr(VI). At strength 0 M, the removal efficiency reached above 95% by 2 h; when it raised to 0.05 M, the rate of Cr(VI) removal was enhanced and within 1 h it showed above 95% removal efficiency. However, the removal efficiency decreased to 81% within 2 h, due to the increase in ionic strength (0.1 M). The moderate ionic strength (0.5 M) might strengthen the adsorbent capacity for the high removal of dichromate ions. On the other hand, high ionic strength would have the opposite effect (Lv et al. 2011). Hayes and Leckie (1987) postulated that the effect of electrolyte concentrations on adsorption can replicate the adsorption type (Hayes and Leckie 1987).

Adsorption isotherms are used to investigate the solute–solvent interaction, adsorption capability of adsorbent and accumulation degree of adsorbate on the adsorbent surface (Anirudhan and Sreekumari 2011). The two most common isotherms Langmuir and Freundlich are used extensively in the literature to elucidate the application of various adsorbents (Mor et al. 2007; Arslan and Pehlivan 2007). Langmuir isotherm describes the monolayer formation of adsorbent on a surface which contains a finite number of binding sites, without any lateral interaction between adsorbed molecules (Arslan and Pehlivan 2007). On the other side, Freundlich isotherm describes the multilayer adsorption having interaction among adsorbed molecules (Anirudhan and Sreekumari 2011).

Effect of adsorbent

The efficacy of adsorption depends on the porosity, surface area and pore size of the adsorbent (Singh and Kaushal 2013; Estevinho et al. 2007). To be an effective adsorbent, the pore size of the adsorbent and the diameter of the adsorbate molecule should be comparable to each other (Tinge et al. 1987). The characteristics of good adsorbents are high adsorption volume and quick separation of a large amount of solutions, selectivity, renewability, high porosity resulting large surface area, chemical and physical stability, low diffusion resistance, very low solubility in the respective contacting fluid, sorbent preservation and its properties, high hardness and compressive strength to prevent crushing and erosion, high confrontation to biofouling to extend long life, no side reactions, suitable for both batch and continuous processes, compatibility and cost-effective (Singh and Kaushal 2013; Ali et al. 2012; Mayadevi 1996; Mohanty et al. 2006; Mei et al. 2010). Ali et al. (2012) also gave importance to the presence of a high volume of carbon or oxygen in the adsorbent moiety for better adsorption.

The most commonly used adsorbents for wastewater treatment are clay (El-Guendi et al. 1995), activated alumina (Singh and Pant 2004), silica gel (Do 1998), limestone (Aziz et al. 2008), activated carbon from different raw materials (Aggarwal et al. 1999; Rao et al. 2009; Kadirvelu et al. 2001; Mohan et al. 2000), zeolite (Ming and Dixon 1987), and chitosan (Crini 2006), etc. The adsorbents are discussed in the following section.

Clays

Clay materials got attention and used as adsorbent due to their abundance, low cost, high adsorption and ion exchange properties (Crini 2006). In recent years, clay materials have been applied for the removal of inorganic and organic pollutants. Various researchers studied clay particles for dyes removal from wastewater (El-Guendi

et al. 1995; Alkan et al. 2004; Ozdemir et al. 2004; Gürses et al. 2004; Alkan et al. 2005). Different types of clay materials like smectites (saponite, montmorillonite, etc.), kaolinite, mica (illite), serpentine, pyrophyllite (talc), vermiculite and sepiolite are used for pollutant removal purposes. Among them, montmorillonite clay exhibits the highest surface area and cation exchange efficiency (Shichi and Takagi 2000). The adsorption abilities of clay materials are due to the net negative charge which came from the chemical structure of minerals. Clay materials can adsorb positively charged species due to this negativity. Besides, their high sorption behavior is due to the high surface area which can go up to 800 m²/g and high porosity (Alkan et al. 2004; Cadena et al. 1990). In 1996, El-Guendi investigated the removal performance of natural clays and activated clays for cationic dye such as Basic Blue 69 and Basic Red 22 from waste aqueous solution. Natural clays showed 390 mg/g and 365 mg/g adsorption capacities (maximum, q_{max}) for Basic Blue 69 and Basic Red 22, respectively. The adsorption efficiency increased by 23% for Basic Blue 69 and 13% for Basic Red 22 after activation of natural clays with H_2O_2 (El-Guendi 1996). Like other adsorbent materials, clay materials can be modified to improve their ability to remove pollutants. Ozdemir and his group (2004) modified sepiolite with quaternary amines and applied to adsorb different types of azo-reactive dyes. This experiment exhibited substantially improved adsorption of the pollutants (Ozdemir et al. 2004). Though clay materials are considered as low-cost adsorbent, the removal efficiency of clays for heavy metals is not adequate (Babel and Kurniawan 2003).

Zeolites

Zeolites are microporous in structure and can be found both naturally as silicate minerals and prepared synthetically like magnetically modified zeolite, bio-zeolite, etc. (Ming and Dixon 1987; Adebajo et al. 2003; Nah et al. 2006; Bai et al. 2010). Clinoptilolite is the most abundant zeolite in nature (Ming and Dixon 1987). They are also used as an adsorbent due to high ion exchange capability (Adebajo et al. 2003). Babel and Kurniawan (2003) and Bose et al. (2002) reported high selectivity of clinoptilolite (natural zeolites) for some specific heavy metal ions like Pb^{2+}(2.4 mg/g), Cd^{2+}(1.6 mg/g), Zn^{2+}(0.5 mg/g) and Cu^{2+}(1.64 mg/g). Magnetically modified zeolite prepared by Nah et al. (2006), exhibited high adsorption capabilities for the Pb^{2+} ion and good chemical inertness in the pH range 5–11. In 2010, Bai et al. applied bio-zeolite for wastewater treatment containing pyridine and/or quinoline. This adsorbent is comprised of mixed bacteria and reformed zeolites. Bacteria degraded the organic pollutant, and the modified zeolite removed the ammonium ion derived from pyridine and quinoline degradation (Bai et al. 2010). Although zeolites originate from low-cost natural resources, their selectivity and competitive adsorption to various ions make their use limited. Moreover, these materials do not show the good capability to adsorb anionic ions and organics. Further modification of zeolite is needed to improve its efficiency to adsorb organics and anions and can be done via acid wash, ion exchange and/or surfactant functionalization (Wang and Peng 2010). Zeolites show low permeability and require external support during column operations (Calzaferri et al. 2000).

Activated alumina

A highly porous, granular crystalline gel used as an adsorbent (Singh and Pant 2004) due to having a high surface area (Do 1998) varies from 200 to 300 m² g⁻¹. Researchers have used alumina due to its amphoteric properties (Naiya et al. 2009) for the removal of dyes (Huang et al. 2007) as well as other ions such as cadmium, led, arsenate and arsenite (Naiya et al. 2009). Activated alumina was also investigated for defluoridation of water. At neutral pH (pH = 7), adsorption efficiency was found to be 1450 mg/kg (Ghorai and Pant 2004). This defluoridation occurred by nonspecific adsorption. But alumina fluoro complexes were formed due to the presence of aluminum ions in the treated water at pH < 7. However, the regeneration of activated alumina can lower its removal efficiency of pollutants.

Silica gel

Various workers also used silica gel as an adsorbent for its comparatively higher surface area (250 to 900 m² g⁻¹) than alumina (Do 1998). It also has some other advantages like local availability and high thermal resistance (Jal et al. 2004; Sharma et al. 2003). Some researchers also modified this material by salinization techniques to enhance pollutant removal efficiency (Jal et al. 2004; Sharma et al. 2003). Preliminary researches reported that various elements from wastewater, such as Pb, Cd, Zn, Cu, Fe and Mn, could be effectively removed by functionalized silica gels (Jal et al. 2004; Sharma et al. 2003). Though the adsorption of basic dyes onto silica was significant, the high price of silica restricts its wide applications as adsorbent (McKay et al. 1999; Woolard et al. 2002); moreover, the regeneration of adsorbent is a complicated process (Seshadri and Kettrupt 1982). Silica shows high sensitivity toward alkaline

solutions that limit the usage in the media of pH higher than 8 (Ahmed and Ram 1992).
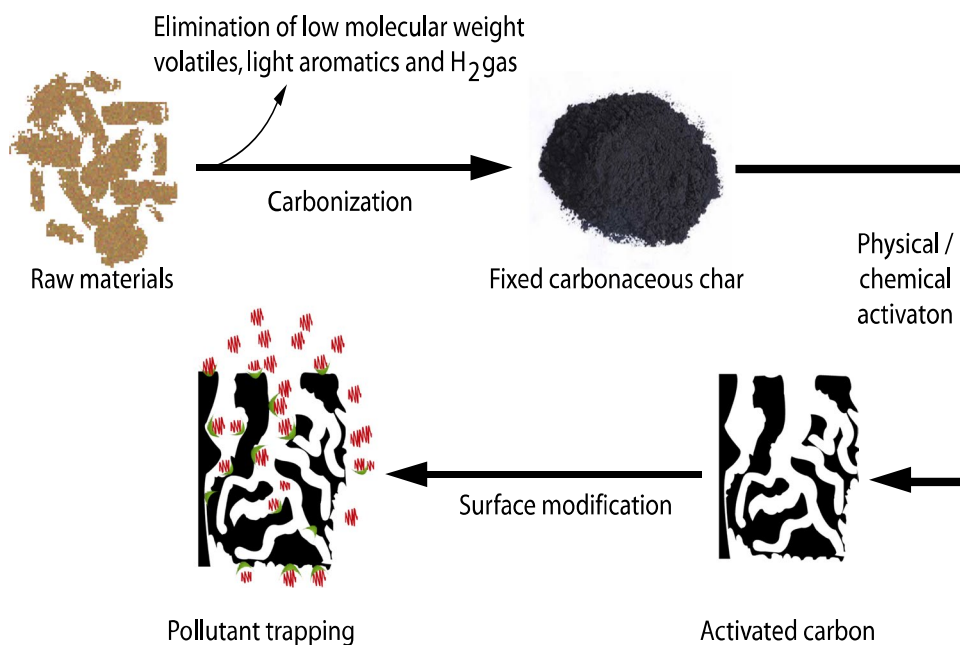
Chitosan

Chitosan, a cationic amino polysaccharide biopolymer, gets extra attention from researchers due to its sorption capacity of dyes and heavy metals at very low concentrations (ppm or ppb levels) (Crini 2006). Among other biomaterials, chitosan provides high reactivity, better chemical inertness and selectivity toward pollutants (Ravi Kumar 2000; Varma et al. 2004; Guibal 2004). The structural flexibility, presence of hydroxyl group and primary amino groups in the chemical structure favor the formation of a stable configuration for adsorption of pollutants (metal ions) (Babel and Kurniawan 2003). Natural chitosan has been modified to improve the adsorption capability for different types of pollutants (Ravi Kumar 2000). Zhu et al. (2012) synthesized xanthate-modified magnetic chitosan and used it for adsorption of Pb(II), Cu (II) and Zn (II). The highest adsorption efficiency was found to be 76.9, 34.5 and 20.8 mg/g for Pb(II), Cu (II) and Zn (II), respectively. Chitosan also has some disadvantages in wastewater treatment. Chao et al. (2004) stated that chitosan has a low attraction for cationic (basic) dyes. The powder/flake-form chitosan is responsible for pressure drop in the sorption column which eventually lowers the adsorption efficiency. Also, chitosan is acid sensitive which triggers the necessary physical and chemical modification of chitosan powder before using in acid condition (Crini 2006).

Activated carbon

Activated carbon, among the conventional adsorbents, is the most prevalent and extensively used adsorbent for wastewater treatment all over the world (Babel and Kurniawan 2003). The US Environmental Protection Agency (EPA) has referred to the activated carbon-based adsorption as one of the best technologically feasible methods (Derbyshire et al. 2001). The credit goes to Raphael von Ostrejko, who first developed commercial activated carbon and got patent in 1900 and 1901. The most common organic precursors are wood, olive stones, anthracite coal, bituminous coal, lignite, peat shell, and almond and coconut shell. The carbon contents of those materials vary from 40 to 90% (w/w) (Bansal et al. 1988; Holden 1982).

Preparation and surface modification The activated carbons are prepared by controlled pyrolytic decomposition of the precursors (Fig. 10) (Fitzer et al. 1971). During carbonization, different low molar mass volatiles, light aromatics and hydrogen gas are released (Hucknall 1985). The resulted fixed carbonaceous char contains tarry pyrolysis residue, which blocks the pores of the char. The resulted char can be activated by physical or chemical treatment (Ali et al. 2012). The physical treatment requires high temperature and steam, CO_2 and air as an activator (Lizzio et al. 1990). In chemical treatment, different activators such as $ZnCl_2$, H_3PO_4, H_2SO and KOH are impregnated into the precursors. The final material is carbonized followed by an activation step in a single action by two different temperatures (Smíšek and Černý 1970). Generally, the required temperature for chemical activation is 400–800 °C (Ali et al. 2012). This cata-

Fig. 10 Schematic representation of activated carbons preparation and pollutant removal

Elimination of low molecular weight volatiles, light aromatics and H_2 gas

Raw materials —Carbonization→ Fixed carbonaceous char —Physical / chemical activation→ Activated carbon —Surface modification→ Pollutant trapping

lytically activated sample further needs a post-activation treatment to remove the residual catalyst. The advantages of chemical treatment over physical treatment are (1) it needs low temperature and (2) the yield is high because the burning of char can be avoided.

The heating rate for carbonization and activation is the prime process parameter, which influences pore structure consisting of macro-, meso- and micropore, surface area and surface functional group (Rodriguez-Reinoso 1986; Wigmans 1986; Bhatnagar et al. 2013). The key functional groups of the activated carbon are carboxyl, carbonyl, phenols, lactones and quinines. These functional groups arise by thermal treatment, activation process and post-chemical treatment on the activated carbon surface. The properties and concentration of functional groups are modified by appropriate chemical or thermal treatment to enhance the adsorption efficiency of activated carbon (Bhatnagar et al. 2013). Different researchers reported that activated carbon comprises both cationic and anionic ions (Lukens 2007). During basic treatment, the increasing trend of net cationic ions is due to a decrease in the number of anionic ions on the activated carbon surface (Lukens 2007). Generally, surface modification is done after the activation process. The alteration can be performed by various approaches that include base treatment, acid treatment, microwave treatment, impregnation treatment, plasma treatment, ozone treatment and surfactant treatment (Bhatnagar et al. 2013). Some of the surface modification processes are discussed below:

Acid treatment It oxidizes the surface of the porous activated carbon as well as improves its hydrophilic properties (Shen et al. 2008). During the acid treatment, huge percentages of oxygen-containing functional groups appear on the carbon surface by replacing hydroxide groups and thus increase cation exchange properties (Ahn et al. 2009). The acidic treatment removes mineral elements from activated carbon (Shen et al. 2008). Generally, nitric acid and sulfuric acid are most widely used for acid treatment (Bhatnagar et al. 2013). Here, metal ions form complexes due to strong interaction with the negatively charged acid groups (electrostatic adsorbate–adsorbent interaction) by replacing (H^+) ions from the oxidized carbon surface (Bhatnagar et al. 2013). The adsorption method depends on pH competing for metals. The capacity of activated carbon to adsorbed metallic compounds is closely related to the number of surface functional groups (Ahn et al. 2009). Jia and Thomas (2000) oxidized activated carbon by nitric acid treatment and reported the incorporation of acidic oxygen functional group (carboxylic acid group, phenol and quinine groups). This surface-modified activated carbon shows cation exchange behavior over long range of pH values as well as exhibited multifunctional nature (Jia and Thomas 2000). However, this oxidation treatment sometimes decreases the

surface area and reduces carbon porosity (Alvarez-Merino et al. 2005).

Base treatment This treatment creates a positive surface charge of the activated carbon which adsorbs negative pollutants in higher amounts (Menendez et al. 1996; Faria et al. 2004; Shaarani and Hameed 2011). Here, activated carbon is treated at an elevated temperature in inert, hydrogen or ammonia atmosphere (Faria et al. 2004; Shaarani and Hameed 2011). The basic surface properties are due to the development of basic nitrogen functionalities like amines, amides, protonated amides, pyridine-type structures, etc. (Mangun et al. 2001; Jansen and Vanbekkum 1995; Raymundo-Pinero et al. 2003). This surface-functionalized activated carbon adsorbs organic dye molecule by electrostatic forces or dispersive interaction. The dispersive forces arise due to the delocalization of π electrons, which are present at Lewis basic sites in the basal planes of a carbon atom and free electrons of the dye molecules present in the aromatic rings (Bhatnagar et al. 2013). Przepiorski (2006) modified the activated carbon by gaseous ammonia at 400 to 800 °C. The resulted adsorbent shows 29% more adsorption capacity for phenol than untreated one (Przepiorski 2006). Shaarani and Hameed (2011) also modified the surface through ammonia and revealed that the adsorption capacity for 2,4-dichlorophenols increased from 232.56 to 285, 71 mg/g.

Impregnation The impregnation technique involves the fine distribution of chemical/metal particles in activated carbon pores. Iron (Vaughan Jr and Reed 2005), copper (Yeddou et al. 2011), silver (Miyanaga et al. 2002) and aluminum (Tchomgui-Kamga et al. 2010) are deposited on the activated carbon using the impregnation process. Huang and Vane in 1989 synthesized iron impregnated activated carbon for arsenic removal. They reported a tenfold increase in arsenic removal by treated activated carbon than untreated activated carbon. The enhancement of adsorption was due to the adsorption of ferrous ion and the formation of arsenate complexes (Huang and Vane 1989). The main advantages of impregnated carbon include catalytic properties optimization of activated carbon by enhancing its inherent catalytic oxidation properties and promote synergism between activated carbon and the impregnated material.

The adsorbents produced by these aforementioned methods possess highly porous morphology with a high surface area of 500 to 2000 $m^2 g^{-1}$ (Carrott et al. 1991). The adsorption on activated carbon usually happens through van der Waals forces. Different types of activated carbon are commercially available in the market, such as powdered activated carbon (PAC), granular activated carbon (GAC), activated carbon pellet and activated carbon fiber (ACF). Among them, granular activated carbon is mostly used due to its adaptation to continuous interaction with pollutants

and no need of additional step to remove the carbon from the bulk (Najm et al. 1991). Activated carbon is extensively used as adsorbents to remove various types of dyes (Al-Degs et al. 2001; Pelekani and Snoeyink 2000), organic and inorganic pollutants such as metal ions (Gabaldón et al. 2000) phenols (Carrott et al. 2005), pesticides (Hu et al. 1998), humic substances (Lee et al. 1983), chlorinated hydrocarbons (Urano et al. 1991), *polychlorinated biphenyls* (Pirbazari et al. 1992), detergents (Malhas et al. 2002), organic compounds which cause taste and odor (Lalezary et al. 1986) and many other chemicals and organisms (Carrott et al. 2000).

Removal of inorganic pollutants by activated carbon

Rao et al. synthesized activated carbon from *Ceiba pentandra* hulls and used it for Hg^{2+} removal. At pH 6, this activated carbon showed higher adsorption capacity (25.88 mg/g). The increased adsorption capacity is because of the presence of sulfur groups on the surface of activated carbon. Since Hg^{2+} is a soft Lewis acid, according to Pearson theory, it will interact with surface sulfur groups (soft bases). The adsorption isotherm is well fitted to the Freundlich model and adsorption kinetics followed pseudo-second-order models. Here, the adsorption equilibrium state is achieved by the hydrophilic character (Rao et al. 2009) and also by the affinity of activated carbon to mercury species. Kadirvelu et al. 2004 prepared activated carbon from waste materials of the sago industry by treating with H_2SO_4 and $(NH_4)_2S_2O_8$. The adsorption capacity of Hg^{2+} was observed to be 55.6 mg/g at pH 5.0. The adsorption process obeyed Langmuir equilibrium isotherm model (Kadirvelu et al. 2004).

Arsenic (As) adulteration in pure drinking water leads to serious health problems. EPA recommended the arsenic level for drinking water 0.01 mg/L (Bohlen 2002). During the last few decades, activated carbon is widely applied to remove arsenic from contaminated water (Budinova et al. 2009; Mondal et al. 2008a; b; Navarro and Alguacil 2002; Mohan and Pittman 2007; Kalderis et al. 2008, Daus et al. 2004). Asadullah et al. (2014) prepared activated carbon from jute stick and loaded with iron by impregnation method. They used it for separation from polluted water. The presence of iron oxide on activated carbon initiates the oxidation of As (III) into As (V) which can more strongly be adsorbed on the surface (Mondal et al. 2007, 2008a; Fierro et al. 2009). The iron-loaded chemically activated carbon reduced as concentration to 3 µg/L when the initial concentration was 100 µg/L and pH 7 (Asadullah et al. 2014). Iron oxide-loaded activated carbon removed 99.90% As (V) within 5 min of the batch adsorption process. This highest adsorption capacity was observed 27.78 mg/g at pH 7. The adsorption process

well fitted both Langmuir and pseudo-second-order kinetic models (Yurum et al. 2014). In 2011, Kocabas and Yurum used iron-loaded red mud for arsenic removal. At pH 2.0, the adsorbent showed a maximum capacity of 11.64 mg/g (Kocabas and Yurum 2011).

Alslaibi et al. (2013) synthesized microwave-irradiated activated carbon from olive stone and investigated its performance for Cd^{2+} removal for aqueous solution. Microwave treatment provides lesser holding time for heating than the conventional heating method. This olive stone-activated carbon showed an adsorption capacity of 11.72 mg/g for Cd^{2+} removal. The adsorption followed Langmuir isotherm, i.e., monolayer adsorption occurred on the activated carbon surface. The value well fitted with pseudo-second-order kinetics and directs chemisorption which is the rate-controlling reaction step during Cd^{2+} removal (Alslaibi et al. 2013). Activated carbon synthesized from coconut buttons was investigated through batch adsorption process to remove heavy metal ions like Pb^{2+}, Hg^{2+} and Cu^{2+}. The activated carbon was synthesized by steam activation at 400 °C. The adsorbent shows good adsorption at pH 6.0 for Pb^{2+}, Cu^{2+} and Hg^{2+} at pH 7.0. The monolayer adsorption capacity for Pb^{2+}, Hg^{2+} and Cu^{2+} was found to be 92.72, 78.84 and 73.60 mg/gm, respectively, at 30 °C. The adsorption isotherm best fitted with the Freundlich model and indicates a heterogeneous surface. The adsorption obeys the pseudo-second-order kinetics model. The adsorption process was influenced by the concentration of metal ions, contact time, solution pH, ionic strength and adsorbent concentrations (Anirudhan and Sreekumari 2011).

Removal of organic pollutants by activated carbon

Activated carbon has also been extensively used to eliminate different classes of industrial dyes from water. McKay used activated carbon filtrasorb type (1.4–2.8 mm) to remove dyestuffs from water and observed outstanding adsorption for acidic, basic and dispersed dyes compared to direct dyes (Mckay 1982). Mendez-Diaz et al. (2010) carried out adsorption of imidazole, sulfonamides and trimethoprim on activated carbon and found almost 90% removal of pollutants. Similar research was studied by Kim et al. (2010) where they investigated trimethoprim adsorption as a batch or continuous process and found above 90% removal efficiency.

Nowadays, pharmaceutically activated compounds and endocrine-disrupting compounds have emerged as novel contaminants in environmental water (Cabrita et al. 2010). Most of them are not biodegradable and cannot be eliminated completely from water by conventional water treatment processes (Villaescusa et al. 2011; Domínguez et al.

2011). Various natural materials such as hydrous oxides (Gu and Karthikeyan 2005), soils (Figueroa and MacKay 2005), clays (Pils and Laird 2007) and silica (Bui and Choi 2010) have been used for adsorption of pharmaceutical pollutants from wastewater. Activated carbon shows promising roles for the efficient removal of these pollutants. It can remove the pharmaceuticals without generating any toxic by-products (Dutta et al. 1999). Baccar et al. (2012) synthesized activated carbon from agricultural waste (olive-waste cakes) at a laboratory scale and used it for adsorption study of four common pharmaceutically activated compounds (diclofenac, naproxen, ketoprofen and ibuprofen) (Baccar et al. 2012). At 25 °C, the adsorbent shows maximum adsorption volumes of 56.17, 39.52, 24.69 and 10.83 mg/g for diclofenac, naproxen, ketoprofen and ibuprofen, respectively.

The adsorption process follows Langmuir and pseudo-second-order adsorption kinetics. The authors also investigated pH and temperature influence on the adsorption of these pollutants. Increasing pH gradually decreases the adsorption of these four pharmaceutically activated compounds (Baccar et al. 2012). Trimethoprim is a common pharmaceutical product used extensively as human and veterinary medicine. It is poorly metabolized by the animal body, and approximately 60% of trimethoprim is discharged in the environmental water in the original form (Lindberg et al. 2004; Hirsch et al. 1999; Molu and Yurdakoc 2010). In 2012, Liu and his co-workers investigated the removal efficiency of four kinds of oxyacids of phosphorus-activated lotus stalk for trimethoprim (Liu et al. 2012). These oxyacids were H_3PO_4, $H_4P_2O_7$, HPO_3 and H_3PO_3. All the prepared activated carbon contains more acidic oxygen functionalities. The order of sorption capacity of trimethoprim by activated carbon was activated carbon–$H_4P_2O_7$ > activated carbon–H_3PO_4 > activated carbon–H_3PO_3 > activated carbon-HPO_3. The adsorption process followed both the Freundlich and Langmuir models, which indicates that both chemisorption and physisorption mechanisms took place simultaneously on the surface of the activated carbon (Liu et al. 2012) (Table 3).

Table 3 List of different adsorbents for water pollutants removal

Pollutants	Adsorbent—conventional	Removing capacities	pH	References
Cd	Zeolite	2.4 mg/g	–	Babel and Kurniawan (2003)
	Modified zeolite	123 mg/g	5-11	Nah et al. (2006)
	Limestone	0.0184 mg/g	8.5	Aziz et al. (2008)
	Activated carbon (olive stone)	11.72 mg/g	–	Alslaibi et al. (2013)
Pb	Zeolite	1.6 mg/g	–	Babel and Kurniawan (2003)
	Limestone	0.0167 mg/g	8.5	Aziz et al. (2008)
	Activated carbon (coconut buttons)	92.72 mg/g	–	Anirudhan and Sreekumari (2011)
Zn	Zeolite	0.5 mg/g	–	Babel and Kurniawan (2003)
	Limestone	0.0115 mg/g	8.5	Aziz et al. (2008)
Ni	Zeolite	0.4 mg/g	5-11	Babel and Kurniawan (2003)
	Modified zeolite	8 mg/g	–	Nah et al. (2006)
	Limestone	0.0380 mg/g	8.5	Aziz et al. (2008)
Cu	Zeolite	1.64 mg/g	–	Babel and Kurniawan (2003)
	Limestone	0.0145 mg/g	8.5	Aziz et al. (2008)
	Activated carbon (coconut buttons)	73.60 mg/g	–	Anirudhan and Sreekumari (2011)
Cr(III)	Limestone	0.0161 mg/g	8.5	Aziz et al. (2008)
Hg(II)	Activated carbon (*Ceiba pentandra* hulls)	25.88 mg/g	6	Rao et al. (2009)
	Activated carbon (sago industry waste)	55.6 mg/g	5	Kadirvelu et al. (2004)
	Activated carbon (coconut buttons)	78.84 mg/g		Anirudhan and Sreekumari (2011)
As (V)	Iron-loaded activated carbon (jute stick)	3.0 µg/L	7	Asadullah et al. (2014)
	Iron-loaded activated carbon (Charcoal)	27.78 mg/g	7	Yurum et al. (2014)
	Iron-loaded red mud	11.64 mg/g	2	Kocabas and Yurum (2011)
Basic Blue 69	Natural clays	390 mg/g	–	El-Guendi (1996)
Basic Red 22	Natural clays	365 mg/g	–	El-Guendi (1996)
Diclofenac	Olive-waste cakes (activated carbon)	56.17 mg/g	–	Baccar et al. (2012)
Naproxen	Olive-waste cakes (activated carbon)	39.52 mg/g	–	Baccar et al. (2012)

–, not available

Challenges of conventional adsorbent

Some major disadvantages of conventional adsorbents are given below.

Cost

The activated carbons are commonly used to remove various kinds of pollutants, but sometimes their applications are restricted because of high-quality activated carbon and its higher cost (Babel and Kurniawan 2003). On the other hand, low-quality adsorbent has generally shown low adsorption capabilities, so large volumes of materials will be required for water purification (Aksu and Kabasakal 2004). For improvement in removal performance, activated carbon is treated with different complexing agents. This additional cost makes the use of activated carbon less attractive in small-scale industries (Babel and Kurniawan 2003). Both physical and chemical activations have some shortcomings, such as physical activation needs a longer time as well as high temperature for activation. Chemical activation requires long washing for the removal of chemical agents.

Exhaustion

During the treatment of wastewater, the adsorbent (activated carbon) becomes exhausted and loses its competence to further adsorb the pollutants. Different techniques like thermal, chemical, oxidation and electrochemical are used for the regeneration of exhausted adsorbent for further use in wastewater. This regeneration process also adds extra cost as well as lowers the adsorption efficiency compared to activated carbon (Taiwo and Adesina 2005; Zhou and Lei 2006).

Surface modification

The surface modification also introduces some shortcomings like the cost involved in the surface modification process, leaching of impregnated metals, regeneration of surface-modified activated carbon and limitation in column, pilot or full-scale study of real wastewaters, surface water and groundwater treatment (Bhatnagar et al. 2013). The presence of other metal ions reduces the adsorption capacity of activated carbon for specific pollutants (Makeswari and Santhi 2013). This process only transfers pollutants from liquid to solid phase, where further treatment is required for complete removal (Homem and Santos 2011).
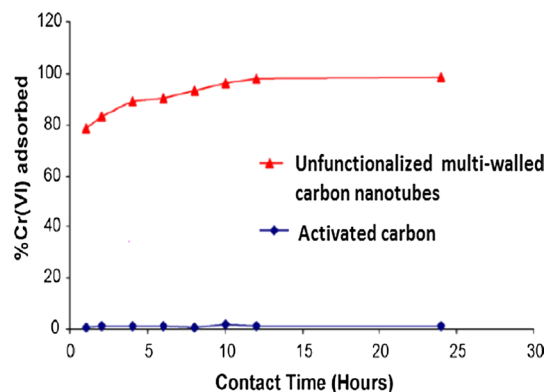

Fig. 11 The influence of contact time on the amount of Cr(VI) adsorbed by each adsorbent (Nxumalo 2006)

Separation of adsorbent

Convenient removal of adsorbent from treated wastewater is another concern for promising adsorbent. The traditional adsorbents are hard to remove after treatment; moreover, the conventional separation processes like centrifugation, precipitation, filtration and chromatography induce malformation like deformation and inactivation of an adsorbent. This operation adds extra cost and time to water purification. Therefore, economically feasible and effective separation techniques are in urgent demand (Li et al. 2011; Liu et al. 2011).

Removal efficiency

Generally, activated carbon cannot reduce the concentration of the pollutants at ppb levels. It adsorbs only a few milligrams of pollutant per gram of absorbent. Moreover, activated carbon shows poor adsorption for inorganic pollutants due to its nonpolar surface (Mohan and Pittman 2006). In the case of activated carbon adsorption, the adsorption equilibrium achieved very slowly due to slow pore diffusion (Lu and Chiu 2006; Deng and Bai 2004). In Fig. 11, activated carbon shows less than 1% adsorption, whereas non-functionalized multiwall carbon nanotubes show a greater ability to adsorb Cr(VI) like 98% adsorption. The lower adsorption efficiency of activated carbon might be due to the filling of pores by water. As a result, Cr(VI) cannot retain the solid surface of activated carbon (Nxumalo 2006).

The above points indicate that the application of activated carbon in wastewater treatment also encounters various challenges. Carbon fibers were developed as second-generation carbonaceous adsorbents to solve the above-mentioned problems. These adsorbents show higher adsorption kinetics than activated carbon due to the highly porous structure, resulting in a shortening of the diffusion time of pollutants to adsorption sites. Carbon nanotube, an excellent third-generation

carbonaceous adsorbent, was synthesized in 1991 with hollow and layered structures. The adsorption sites are located both on the outer and on the inner layer surface of this nanostructure (Liu et al. 2013).

Nanoadsorbents

Nanotechnology has successfully introduced nanoadsorbents for wastewater treatment, which has emerged as an enormous growing and enthralling area of interest (Förstner and Wittmann 1985). Generally, nanotechnology is related to the creation, processing, characterization and application of materials at the nanoscale (1–100 nm) in diverse areas (Biswas et al. 2017, 2019; Mamalis 2007; Stander and Theodore 2011; Padmanabhan et al. 2019; Golieskardi et al. 2019; Ragurajan et al. 2018; Rangari et al. 2017, 2019; Nuge et al. 2020). Nanomaterials own exceptional properties, which are absent in their corresponding bulk materials. These properties are conductivity, selectivity, and catalytic, magnetic and optical properties and high surface area per mass ratio (Lu et al. 2019). Nanomaterials are chemically active due to large surface areas with a large number of active surface sites. Carbon nanotubes, ordered mesoporous carbon (OMC), graphene, carbon nanofibers, titania nanotubes and their modifications are some of the popular nanomaterials that have been used in wastewater treatment. The higher concentration of surface defects arises from large surface area and facilitates interaction with pollutants. Sometimes, nanomaterials are coated with other coating agents to enhance their adsorption properties. For example, when TiO_2 was coated with carbon nanotubes (CNTs), the composite showed larger surface area as well as high removal efficiency than their precursor in the as-received form (Hurt et al. 2006; Ilisz et al. 2004). Photocatalytic degradation of pollutants carried out by using TiO_2-based nanomaterials results in non-toxic end products (Mahmoodi et al. 2007). These nanomaterials possess outstanding absorbing capabilities and chemical stability (Chen et al. 2005).

Nanomaterials as adsorbent

The structural properties (porosity, surface area, pore size and pore volume), chemical composition and thermal stability of nanoparticles depend on the synthesis procedure and operating conditions. Solgel, ion sputtering, impregnation, co-precipitation, spray pyrolysis, thermal spraying, arc discharge, chemical vapor deposition (CVD), mechanical alloying/milling, laser ablation, thermal plasma synthesis, catalytic growth and electrodeposition are the common methods applied for nanoparticle synthesis (Liu and Zhang 2007; Sharma et al. 2009). The solgel method gets extra attention for the synthesis of nanoparticle due to its low

cost, homogenous product formation, high purity and environmental friendliness (Zeng et al. 1998). Different types of nanomaterials are synthesized and used as an adsorbent for pollutant removal, for example, alumina, TiO_2, Al_2O_3, Fe_3O_4, carbon nanotube (CNT), graphene, mesoporous carbon, $MnFe_2O_4$, nano-zerovalent iron, magnetite, etc. (Sharma et al. 2009). The role of nanoadsorbent to remove inorganic, organic and biological pollutants is discussed below with recently reported data.

Removal of inorganic pollutants by nanoadsorbents

Zerovalent iron nanoparticle, iron oxide nanoparticle, titanium oxide, aluminum oxide, etc., are commonly used for metal pollutants removal from wastewater. Zerovalent iron has been reported for adsorption of different metal ions like arsenic, chromium, cadmium, lead, selenium, silver and zinc (Kanel et al. 2005; Ponder et al. 2000a; Sturchio et al. 1997).

Arsenic (As)

Arsenic adulteration of groundwater is a major problem in whole around the world. The prevalence is the highest in Bangladesh, where about 35 million people are affected by arsenic contamination. A study carried on arsenic affected area/villages in Bangladesh reported that almost 90% of groundwater contained a higher concentration of arsenic than the permissible limit. Various water treatment techniques have been used for arsenic removal, but none of them is effective in community level due to high cost and handling problems (Khan et al. 1997; Dhar et al. 1998; Chatterjee et al. 1995; Das et al. 1995). Nanoadsorbents like akaganeite, magnetite, maghemite and zerovalent iron nanoparticle were used for the removal of arsenic pollutant from drinking water (Deliyanni et al. 2006; Chowdhury and Yanful 2010; Machado et al. 2006; Kanel et al. 2006; Li and Zhang 2007). At pH 7.5, Deliyanni et al. (2003) reported removal of 120 mg/gm as (v) from the solution by akaganeite nanoparticle (2.6 nm). After adsorption, nanoadsorbents can be separated by membrane filtration (0.45 μm) (Deliyanni et al. 2006). Hristovski et al. (2007) also studied the As (v) removal by 16 metal oxide nanoparticles in fixed bed columns, including TiO2, NiO, Fe_2O_3 and ZrO_2. Except for ZrO_2, all the nanoparticles showed ~98% removal capacity. The adsorption data fitted well with Freundlich adsorption isotherm (Hristovski et al. 2007). Zhu et al. (2009) impregnated non-zerovalent iron on activated carbon and used it to adsorb arsenic pollutant As (III). At pH 6.5, the composite removed approximately 1.997 mg/g in 2.0 mg/L arsenic solution. The authors proposed this adsorbent as an ideal candidate to treat arsenic-polluted drinking water (Zhu et al. 2007). Velickovic et al. (2012) and Peng et al. (2005) used iron (III) oxide comprised of ethylene

diamine-functionalized multiwall carbon nanotubes and ceria nanoparticles supported on carbon nanotube, respectively, for As (V) removal. Tang et al. (2013) successfully produced superparamagnetic ultrafine magnesium ferrite ($Mg_{0.27}Fe_{2.50}O_4$) nanoadsorbent which shows superior arsenic adsorption performance on both As (III) and As (V). Its adsorption efficiency of As (III) and As (V) was found to be 127.4 mg/g and 83.2 mg/g, respectively, at pH 7.0. The hydroxyl group played a major role in arsenic adsorption. They found that the arsenic adsorption capacity of the as-synthesized nanoadsorbent was higher than Fe_2O_3 nanoadsorbent. Moreover, this nanoadsorbent ($Mg_{0.27}Fe_{2.50}O_4$) could be easily removed by the external magnetic field and could be regenerated and reused for arsenic removal (Tang et al. 2013). Zhang et al. produced a series of iron oxide–graphene oxide composites to remove arsenate from water. They found that water with arsenate concentration at 51.14 ppm, the composites adsorb more than 95% arsenate, results in an absorption capacity of 23.78 mg arsenate/g of composite in the pH range of 4–9 (Zhang et al. 2010). Yu et al. synthesized magnetic iron oxide/graphene oxide with high iron oxide loading (51% by wt.). They reported significantly high adsorption capacities 54.18 and 26.76 mg g^{-1} for As (III) and As (V), respectively. In general, graphene-based adsorbent materials show improved adsorption capacities because they offer a large surface area and inhibit the agglomeration of the deposited nanoparticles (Yu et al. 2015).

Chromium (Cr)

Chromium contamination is generally initiated by steel mill waste, erosion of natural waste, electroplating wastewater and dye industries. The U.S. EPA fixed the discharge limit of chromium to 0.1 mg/L in surface water. Among various valence states of chromium, only trivalent, Cr(III) and hexavalent Cr(VI) forms are important for the environmental point of view. The hexavalent form exhibits more toxicity than its trivalent form (Browning 1969; Kowalski 1994; Singh and Singh 2002). Ponder et al. (2001) removed Cr(VI) by applying nano-size zerovalent iron (Ferragels) which is 10 − 30 nm in diameter. They fabricated the nanoadsorbent using a borohydride reducing agent to reduce aqueous iron salt. The authors explained pseudo-first-order reaction kinetics of the hexavalent chromium adsorption process. Here, Cr(VI) is reduced to Cr(III) by nano-size zerovalent iron. The author showed that the reduction rate of chromium was 7–12 times faster by prepared nano-size zerovalent iron than the equivalent weight of iron powder (Ponder et al. 2000a, b).

In 2005, Hu et al. synthesized and used maghemite (γ-Fe_2O_3) nanoparticles (diameter around 10 nm) to remove and recover Cr(VI) from wastewater. The highest adsorption performance of maghemite for Cr(VI) removal was
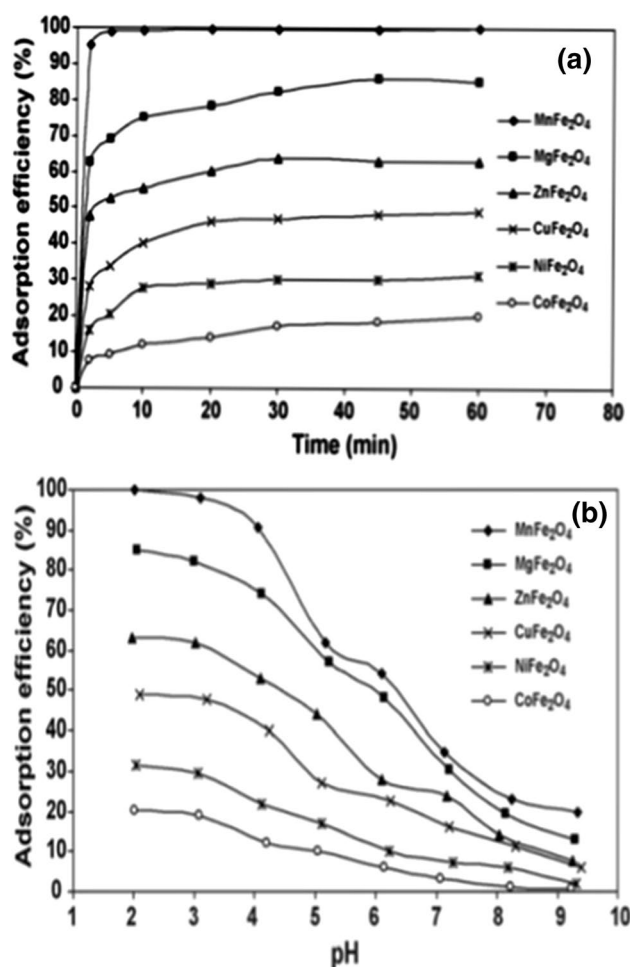

Fig. 12 The effect of contact time and pH on chromium removal by various ferrites; **a** absorption versus time and **b** absorption versus pH (Hu et al. 2007)

19.2 mg/g of maghemite at pH 2.5, and the saturation time for this method was only 15 min. The adsorption of Cr(VI) on the maghemite was because of electrostatic interactions and ion exchange (Hu et al. 2005). Hu et al. (2007) synthesized a group of magnetic nanoparticles of about 20 nm by chemical co-precipitation method. The synthesized magnetic nanoadsorbents are $CoFe_2O_4$, $CuFe_2O_4$, $MgFe_2O_4$, $MnFe_2O_4$, $NiFe_2O_4$ and $ZnFe_2O_4$. The major advantages in adsorption trailed by magnetic parting are rapidness, efficacy and easiness of the process. The author found adsorption equilibrium within 1 h for Cr(VI) by all kinds of nano-ferrites. At a similar primary Cr concentration (1000 mg/L), among all the ferrite nanoparticles, $MnFe_2O_4$ nanoparticles showed the highest Cr adsorption efficacy (99.5%) within the shortest adsorption time. This is because of the high surface area of $MnFe_2O_4$ (180 m^2/g). The removal efficacy was extremely pH-dependent, and the optimum adsorption occurred at pH 2. The effects of contact time and pH on chromium removal by various ferrites are shown in Fig. 12.

Fig. 13 **a** SEM and TEM images of nZVI supported on MWCNTs before adsorption, **b** SEM images of nZVI supported on MWCNTs after reaction (adsorption) with Cr(VI) for 2 h. The polygon shape appeared on the composite (Lv et al. 2011)
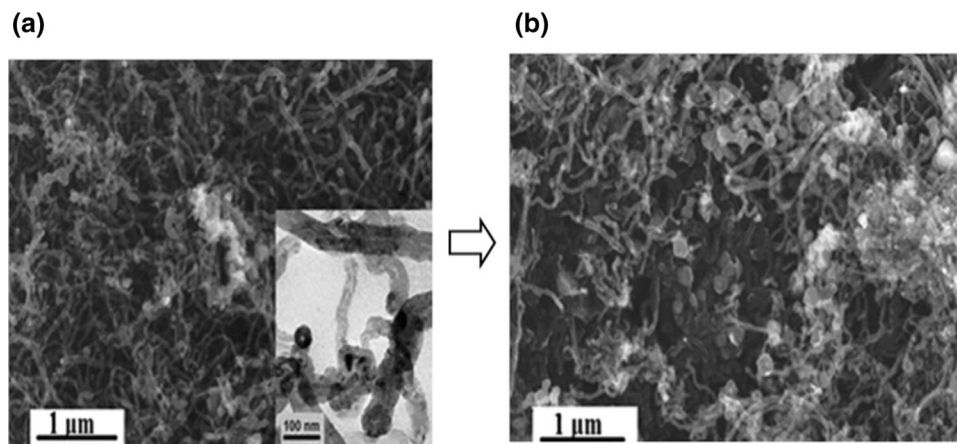
The figure shows that the adsorption capacities of magnetic nanoadsorbent followed the order: $CoFe_2O_4 < NiFe_2O_4 < CuFe_2O_4 < ZnFe_2O_4 < MgFe_2O_4 < MnFe_2O_4$ (Hu et al. 2007).

For the first time, Lv et al. (2011) used nanoscale zerovalent iron multiwalled carbon nanotube nanocomposite for Cr(VI) removal. In this composite, nano-zerovalent iron nanoparticles (20–80 nm) remain dispersed on the surface or into the network of multiwalled carbon nanotube (Fig. 13a). The study reported that this composite exhibited a 36% higher efficacy for Cr(VI) removal than bare nano-zerovalent iron or nano-zerovalent iron-activated carbon composite. The reason might be bare nano-zerovalent iron formed inert oxide film as it exposed to air or water. This oxidation layer prevents further reactions with contaminants. Coupling with multiwalled carbon nanotube prevents this oxide film formation on the surface and allowed more adsorption. This composite also completely removed Cr(III) from aqueous solution. At concentration 1.09 mg/L, the composite removed completely Cr(III) at 15 min. The removal process depends on ionic strength, initial concentration of pollutants, pH and presence of other anions. The experiment was allowed to run for 4 h and found complete removal of dichromate ions and other anions at their high concentrations (Lv et al. 2011). Figure 13b shows that after 2 h of reaction some polygon shape appeared on the nano-zerovalent iron multiwalled carbon nanotube. This is possible because of the co-precipitation of chromate and iron ions (Lv et al. 2011).

Chandra et al. (2010) synthesized magnetite (Fe_3O_4)–graphene composite. The composite showed adsorption capability of 5.5 mg/g of composite that is higher than the unsupported Fe_3O_4. Goharshadi et al. used functionalized graphene oxide to remove Cr(VI) ions. The functionalized graphene oxide has a high concentration of epoxy and hydroxyl groups, which assist in adsorption. The adsorption capability was found to be 1.66 mg/g of graphene.

Cadmium (Cd)

Karimi (2013) reported the synthesis of magnetite nanorods (average diameter 60 nm) by the electrochemical method in pulsed conditions. He used this nanoparticle as an adsorbent for various metal pollutants removals such as Cd^{2+}, Cu^{2+}, Fe^{2+}, Ni^{2+}, Pb^{2+}, Zn^{2+}, etc. The maximum adsorption capacities of this nanoadsorbent for Cd^{2+}, Fe^{2+}, Pb^{2+}, Zn^{2+}, Ni^{2+} and Cu^{2+} were 88.39, 127.01, 112.86, 107.27, 95.42 and 79.10 mg/g, respectively (Karami 2013). Like the removal of As(V) and Cr(VI), synthetic akaganeite nanocrystals (average crystallite size 2.6 nm) also efficiently removed Cd (II) from aqueous waste solutions. This nanoadsorbent showed 17.1 mg/g adsorption ability for Cd (II) ions. The sorbent capacity can be improved by increasing the temperature and pH, whereas it decreased by increasing the electrolyte ions in solution. Here, the adsorption of Cd (II) took place by a weak chemisorption process. The adsorption data fitted both Freundlich and Langmuir isotherms and obeyed those two models (Deliyanni and Matis 2005).

Recently, biodegradable and renewable organic polymers are used for coating nanoadsorbents which have been broadly used for the separation of heavy metal pollutants. These organic polymers originate from industrial wastes, agricultural wastes and biomass (Abdel-Halim and Al-Deyab 2011). Gong et al. (2012) used shellac, a biorenewable and biodegradable resin with ample hydroxyl and carboxylic groups that provided the possibility of chelating heavy metal ions. The authors coated the iron oxide nanoadsorbent with this shellac polymer and examined its capacity for Cd^{2+} removal from waste solution. The adsorption capacity of the coated nanoadsorbent increased with increasing pH. At pH 8.0, it showed the highest adsorption capability of (18.80 mg/g). After adsorption, the coated nanoadsorbent can be easily regenerated by organic acid. Though the adsorption capability was not high enough, there is still much room for development such as optimizing the synthesis parameters to coat a thicker shellac layer. Another

advantage of this coated nanoadsorbent was that it can be used in a high saline solution for Cd^{2+} removal. At 3.0% sodium chloride solution, this adsorbent showed 10.10 mg/g adsorption capacity. Adsorption mechanisms followed electronic attraction as well as chemical adsorption (Gong et al. 2012).

Pacheco et al. (2006) synthesized aluminum–silica nanoparticles by the solgel process. They used this nanoadsorbent for Cd^{2+} removal from wastewater and stated 96.4% adsorption on Si–Al nanoparticles. This nanoadsorbent has various kinds of functional groups on their surfaces, such as alkoxy, hydroxyl and oxy groups, and metallic ions (Cd^{2+}) that interact with one group by electrostatic attraction as well as by ionic exchange to another group (Pacheco et al. 2006).

Mercury (Hg^{2+})

Mercury is harmful to the human body. It can cross the blood–brain barrier. Mercury poisoning can damage the cardiovascular system, kidney and bones as well as cause neuronal disorder (Miretzky and Cirelli 2009; Clarkson 1993). Pan et al. (2012) fabricated mercapto-functionalized nanoscale Fe_3O_4 and carefully investigated the adsorptive characteristics of this nanoadsorbent for removal of Hg^{2+}. At the optimized condition, i.e., 308 K, and at pH 3.0, this functionalized nanoadsorbent showed the highest adsorption capacity toward Hg^{2+} (522.9 mg/g) with an initial concentration of 500gm/L. The adsorption of Hg(II) reached saturation within 60 min. The results fitted well to Freundlich isotherm and pseudo-second-order kinetic isotherm, and the process was endothermic (Pan et al. 2012). In 2013, Zhang et al. synthesized SiO_2-coated Fe_3O_4 nanoparticle. The coatings protected Fe_3O_4 nanoparticles from oxidation at low pH conditions, and Fe_3O_4 core is comprised of superparamagnetic and separate wastes which are magnetic

in nature. Further, they used 3-mercaptopropyltrimethoxysilane, for modifying coated Fe_3O_4 nanoparticle to prepare a unique mercaptopropyl-modified sorbents to remove mercury from solution. The highest adsorption capacity obtained from Langmuir fitting was 148.8 mg/g at pH 6.5. The researchers also found significant mercury removal efficiency (110 mg/g) from natural wastewater samples by this adsorbent (Zhang et al. 2013).

Recently, carbon-based nanomaterials like graphite oxide attracted researchers for its potential application as adsorbent (Seredych et al. 2011). Kyzas et al. (2014) synthesized two composites: (1) graphite oxide nano-filled with chitosan and (2) graphite oxide–magnetic chitosan. The groups evaluated the performance of untreated graphite oxide and two as-prepared composites to remove Hg^{2+} from aqueous solution. At pH 6 and 25 °C, the maximum adsorption capability for graphite oxide, graphite oxide nano filled with chitosan and graphite oxide–magnetic nanocomposite was 187, 381and 397 mg/g, respectively. The results supported the Langmuir isotherm well, and the adsorption mechanism followed chemisorption type. The authors evaluated the study by varying the parameters, i.e., pH, contact time and temperature (Kyzas et al. 2014).

Copper (Cu^{2+})

Amino-functionalized magnetite nanoparticles were investigated for Cu^{2+} removal from aqueous solution. The adsorption equilibrium achieved within 5 min of the adsorption process. The equilibrium data satisfactorily fitted by Langmuir isotherm, and the maximum adsorption was found to be 25.77 mg/g at pH 6 and 298 K (Mei et al. 2010). Here, the fast adsorption indicates that adsorption happened primarily on the adsorbent surface (Liu et al. 2008). The sorption mechanism between Cu^{2+} and NH_2 groups on the

Fig. 14 TEM images of **a** nano-geothite and **b** nanohematite (Chen and Li 2010)
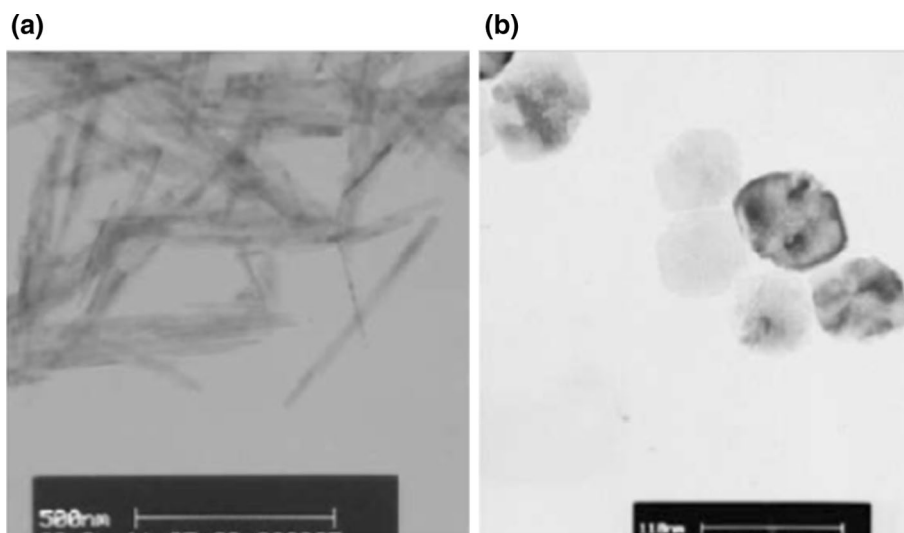
amino-functionalized magnetite nanoparticles was the chemisorption process. By 0.2 mol/L HCl solution, the amino-functionalized magnetite nanoparticles can be regenerated within one minute and retained the original metal removal capacity (Mei et al. 2010). Nanogeothite (α-FeOOH, average particle size 10–15 nm) and nanohematite (α-Fe$_2$O$_3$, average particle size 75 nm) were synthesized by the co-precipitation method (Fig. 14). The surface area of both the particles was 71.49 m^2/g and 24.82 m^2/g, respectively. It was found that both nanogeothite and nanohematite showed good adsorption capability of 149.25 and 84.46 mg/g, respectively for Cu^{2+}. Both the adsorption processes were befitting to Langmuir isotherm, and the adsorption onto nanomaterials was a spontaneous process (Chen and Li 2010).

Gum aerobic modified magnetic (Fe$_3$O$_4$) nanoadsorbent was developed for the removal of Cu^{2+} ions from various solutions. The interaction between gum aerobic and magnetic nanoadsorbent took place via the carboxylic groups present in the structure of gum aerobic and the hydroxyl groups present in Fe$_3$O$_4$. This surface modification led to the formation of the particle with a diameter range of 13–64 nm. The author compared the adsorption capacity of both gum aerobic modified magnetic nanoadsorbent and naked magnetic nanoadsorbent. The maximum adsorption capacities were 38.5 and 17.6 mg/g, for modified magnetic nanoadsorbent and naked magnetic nanoadsorbent, respectively. The modified magnetic nanoadsorbent adsorbed copper ions via complexation with the amine group of gum aerobic, whereas naked magnetic nanoadsorbent used surface hydroxyl groups for complexation with copper ions. Both adsorption data fitted to Langmuir isotherm (Banerjee and Chen 2007).

Zhao et al. prepared few-layered graphene oxides via the modified Hummers method. They studied the effect of operating conditions like pH and ionic strength to remove Cd^{2+} and Co^{2+} ions from water. They found that the presence of an oxygen-containing group on the graphene surface was the key to the adsorption of the ions. The adsorption capacity at near-neutral pH for Cd^{2+} removal was 106.3 mg/g. They also calculated the adsorption of Cd^{2+} on graphene which is endothermic, and the process is spontaneous (Zhao et al. 2011). In a separate study, Xu et al. also prepared graphene oxide layers for the removal of Cd^{2+} ions from water. The maximum adsorption capacity was found to be 44.64 mg/g at pH 4.00 with a loading of 2 g/L.

Mubarak et al. showed the comparison of the removal capacity of acid-functionalized multiwall carbon nanotubes (MWCNTs) and biochar for the Cd^{2+} from water. The introduction of functional groups like hydroxyl and carbonyl and the carboxylic acid act as anchor points for the cations. The highest Cd^{2+} adsorption of f-MWCNTs was found to be 83.33 mg/g, and it was 62.5 mg/g for the magnetic biochar at pH near 7 (Mubarak et al. 2015). Ramana et al. synthesized silver-coated MWCNT using the reduction method. The maximum adsorption capacity for Cd^{2+} removal was found to be 54.92 mg/g at the neutral pH (Ramana et al. 2013). Although graphene oxide and CNTs have shown good adsorption capacities, still there remains a great concern over their elution to the system. It has been shown that biochars can stabilize them by acting as host in the nanocomposite. Biochars have large pore volumes and surface areas. Therefore, biochar–CNT and biochar–graphene composite were tested for Cd^{2+} removal. Liu et al. fabricated carbon nanomaterial–biochar nanocomposites (SG–PySA–CNT and SG–PySA–GO) via pyrolysis of sweetgum biomass pretreated with carbon nanotubes (CNTs) and graphene oxide (GO). The adsorption capacity for Cd^{2+} removal was found to be 10.2 mg/g, which is low compared to the previously reported values. Several modifications of graphene oxide have been reported. Deng and Bai prepared iron oxide–graphene oxide nanocomposite for the simulation's removal dyes and Cd^{2+}. In a mono-component system, the maximum sorption capacity in ultrapure water for Cd (II) was 91.29 mg/g (Deng and Bai 2004).

Lead (Pb)

Corrosion of domestic plumbing and natural erosion are the prime effects of Pb contamination. Nanoscale zerovalent iron was successfully used by Ponder et al. (2001) for Pb(II) removal. Here, Pb(II) is reduced to Pb (0). The iron nanoadsorbent showed 30 times higher removal efficiency than from iron powder on a (Fe) molar basis (Ponder et al. 2000a, b). Wang et al. (2007) treated MWCNTs by nitric acid and used these functionalized MWCNTs for Pb(II) adsorption. After treatment, the acid-functionalized MWCNTs become more negatively charged because of the formation of –OH and –COOH functional groups and showed adsorption capacity up to 91 mg/g at pH 3.5. The adsorption is due to electrostatic interactions (Wang et al. 2007). The hexagonal arrangements of carbon atoms in its graphite structure and its large pore size enable the high adsorption of Pb(II) by carbon nanotubes. This adsorption might happen in various places such as interstitial pore space between the grove edge of its boundary bundles, tube handle or outer layer surfaces (Stafiej and Pyrzynska 2007).

Some researchers used single adsorbent for multiple metal ion adsorption. Polymer-based hybrid nanoparticle sorbents (ZrPS-001) were synthesized by Zhang et al. (2008) and applied for adsorption of different metal ions (cadmium, lead and zinc) from aqueous solution. After the treatment, water met the WHO requirements as drinking water standards (Zhang et al. 2008). Another research group developed nanoadsorbent (2,4-dinitrophenyl hydrazine immobilized on sodium dodecyl sulfate-coated nanoalumina) and studied its feasibility for removal of metal cations like lead, cadmium,

chromium, cobalt, nickel and manganese from water. The adsorbent showed the highest removal for lead, chromium and cadmium ions. The adsorption data matched well with Freundlich and Langmuir models (Afkhami et al. 2010).

Nano-hydroxyapatite (NHA), which is a major inorganic segment of human bone (Michael et al. 2016a, b), could also play a significant role in wastewater treatment. Nano-hydroxyapatite (Sairam et al. 2008), polypyrrole (PPy)/Fe$_3$O$_4$ magnetic nanocomposites (Bhaumika et al. 2011) and Fe–Al–Ce nanoadsorbent (Chen et al. 2009) were used for defluoridation of water. Nano-hydroxyapatite showed a defluoridation capacity of 1845 mg/kg. The ion exchange and adsorption processes were the main mechanisms (Sairam et al. 2008). Kassaee et al. (2011) investigated the removal efficiency of iron nanoparticle for nitrate ions. The author found that each experiment showed almost 76% reduction of nitrate ions after 12 h, above 90% after 24 h, and approximately 100% after 72 h (Kassaee et al. 2011).

Huang et al. fabricated graphene nanosheets (GNSs) via vacuum-assisted low-temperature exfoliation and applied to adsorb Pb^{2+} ions from an aqueous solution. They observed that Pb^{2+} ion adsorption depended on the solution pH and increased with it. They concluded that the Lewis basicity of GNSs is enhanced due to heat treatment under vacuum condition and favors simultaneous adsorption of Pb^{2+} ions and protons by GNSs. GNSs showed a desorption capacity of up to 35.7 mg/g at pH 3.5 (Huang et al. 2011). Fang et al. prepared magnetic chitosan–graphene composite and observed that the presence of Fe$_3$O$_4$ in the composites enables quick extraction after saturated adsorption completed. The unique characteristics of MCGO such as high surface area, abundant functional groups and the high volume of sp^2 carbon not only enhance its adsorption behavior but also help to immobilize and disperse the GO. The magnetic adsorbent exhibited high stability and eco-friendly having high Pb^{2+} ions adsorption and desorption capacity of 76.94 mg/g and 90.3%, respectively. This behavior makes them an excellent candidate to remove Pb^{2+} ions from agricultural and industrial wastewater (Fan et al. 2013).

Vilela et al. reported very innovative graphene-based microdots made from graphene sheets. Those microdots behaved like self-propelled structures to capture, transfer and remove heavy metals (i.e., lead) and its subsequent recovery for recycling purposes. Their structure is comprised of nanoscale graphene oxide, nickel and platinum in multilayers fashion, offering various functionalities. For example, the graphene oxide outer layer surface attracts lead, the inner layer of platinum decomposes hydrogen peroxide fuel for self-propulsion, whereas the middle layer of nickel permits external magnetic control of the microdots. Mobile GOx-microbots showed excellent efficiency to remove lead compared to non-mobile GOx-microbots. They exhibited excellent removal capability of lead and water pollutants

from 1000 ppb down to 50 ppb in 60 min. Furthermore, the saturated microdots can be regenerated and reused by removing lead from the surface (Vilela et al. 2016).

Nanoadsorbents for wastewater treatment

Removal of organic pollutants using nanoadsorbents

Naoadsorbents are also used for organic pollutants removal like different types of dyes, pesticides and hydrocarbons. Belessi et al. (2009) prepared mesoporous TiO$_2$ and used it for commercial azo dye Reactive Red 195 removal in the liquid phase at room temperature under dark condition. The mean synthesized particle size was ~ 8.8 nm. The nanoadsorbent exhibited the highest adsorption percentage (87.0 mg/g) at pH 3.0 and a temperature of 30 °C. The authors considered the influence of pH, dye concentration and adsorbent dose to remove dye molecules. The equilibrium data showed an excellent fit to Langmuir and pseudo-second-order kinetic models. The adsorption process showed high electrostatic attractions between the positively charged surface of the adsorbent and anionic dye (Belessi et al. 2009). Surfactant-coated magnetic nanoparticles have been used for 2-hydroxyphenyl extraction from wastewater (Bahaj et al. 2002). The perchloroethylene undergoes reduction by chemisorption on zerovalent iron nanoparticle in anaerobic conditions (Joo and Cheng 2006; Tratnyek et al. 2003).

Du et al. (2008) prepared chitosan nanoparticles by the ionic gelation method. They used a batch adsorption process to remove eosin Y (a model anionic dye) by this nanoadsorbent. Experimental results followed Langmuir isotherm, and the adsorption capability was found 3.333 g/g. The process was influenced by contact time, dye concentration, pH and temperature (Du et al. 2008). The Fe$_3$O$_4$ nanoparticles–carboxymethylated chitosan conjugate (anionic magnetic nanoadsorbent) was synthesized by the carbodiimide activation process. This conjugate was studied to remove acid dyes, such as orange G and acid green. In both cases, it is found that the adsorption capability reduced with increasing solution pH. The maximum adsorption capability for Orange G and acid green was 1883 and 1471 mg/g, respectively. Here, the adsorption route followed the pseudo-second-order kinetic model and the Langmuir model (Chang and Chen 2005). Due to hydrophobic nature, high surface area, hollow and layered structure, carbon nanotubes show a strong attraction to organic materials, especially to nonpolar organic materials, such as polycyclic aromatic hydrocarbons, benzene derivatives, phenolic compounds, organic dyes (Nas et al. 2019a, b), naphthalene (Gotovac et al. 2007), phenanthrene and pyrene (Yang et al. 2006). More interested readers are referred to a recent review by Apul et al. (2016).

Recently, degradation of methyl parathion (MP), methyl orange (MO), methylene blue (MB), sodium dodecylbenzene sulfonate (SDBS) and bisphenol A (BPA) in aqueous solution has been investigated using three different grades of acid-functionalized CNTs of different tube diameters in the presence of microwave radiation. The effect of irradiation time, initial concentration of the organic compound, CNT doses, microwave power and initial pH was studied. CNTs having diameter ranges from 10 to 20 nm showed the highest catalytic behavior under microwave treatment. Further, complete degradation was obtained using 10–20 nm CNTs within 7.0-min irradiation when 25 mL solution (25 mg/L), 1.2 g/L catalyst dose, 450 W, 2450 MHz and pH = 6.0 were applied. The concept of "hot spot" was believed to be behind the remarkable degradation by CNTs in the presence of microwave.

Graphene-based materials have been extensively used to remove organic dyes and oil from water. The high aspect ratio of π electronic surface favors the adsorption of organic contaminants on the graphene surface and their eventual removal from water (Nas et al. 2019a, b; Wang et al. 2016). Methyl orange, methylene blue, phenol, aromatic compounds, hydroquinone and phenol have been adsorbed on graphene. Graphene was decorated with flowerlike TiO_2 microspheres using a simple surfactant-assisted hydrothermal process. Poly(vinyl pyrrolidone) was used as a surfactant. The composite was used to degrade rhodamine B, and its adsorption capacity was compared with the TiO_2 microsphere and Degussa P-25. Excellent removal was achieved using the composite.

Removal of biological pollutants

Cyanobacteria are common habitants of water. These bacteria produce microsystems (nonribosomal peptides) which also contaminate water. So far, conventional technology faces difficulties in removing this pollutant completely from the water. Carbon nanotubes showed 98% removal capacity of this pollutant from water, which is four times higher than did granular activated carbon (GGC) (Yan et al. 2006).

Nanoadsorbent is reported to remove viruses from infected water. A comparative study was carried out on four commercial nanoparticles of iron oxide to remove bacteriophage phiX174. Among them, α-Fe_2O_3 showed the most efficiency (100% adsorption) at low initial virus concentration. The adsorption method followed both Langmuir and Freundlich adsorption isotherms. The authors also studied the effect of ionic strength, presence of cations and anions on the adsorption process. The presence of anion reduced the adsorption, whereas the reverse scenario was found in the presence of cation. The mechanism for adsorption of the virus on iron oxide nanoparticle was due to electrostatic interaction (Shen et al. 2010).

Singh et al. (2011) synthesized surface-modified magnetic nanoparticles (Fe_3O_4) by a soft chemical method. They prepared three types of surface-functionalized nanoparticles—functionalized Fe_3O_4 nanoparticles with (1) carboxyl group (succinic acid), (2) amine group (ethylenediamine) and (3) thiol group (2,3-dimercaptosuccinic acid). These nanoparticles successfully removed both toxic heavy metal ions (As^{3+}, Co^{2+}, Cr^{3+}, Cd^{2+}, Cu^{2+}, Ni^{2+}, Pb^{2+}, etc.) and bacterial pathogens (*Escherichia coli*) from water. In the case of a pathogen, the hydrophilic magnetic particles damaged the cell wall partially and/or completely, infiltrate into the lipid bilayer component of the membrane and disrupt its structural integrity (Fig. 15) (Singh et al. 2011).

In comparison with other low-cost adsorbents, nanoparticles show low contact time (1.0–15.0 min) and low adsorbent dose (μg/L) and can work in various conditions of pH (3–9) during the adsorption process. These parameters ensure the fast adsorption capacity of nanoadsorbent (Ali et al. 2012) (Table 4).

Regeneration and separation of nanoadsorbents

Regeneration of nanoparticle offers two aspects in nanoadsorbent application, i.e., reusability of nanoadsorbent and recovery of valuable metal species. Various research groups investigated the reusability of nanoadsorbents for pollutant

Fig. 15 TEM images of **a** *E. coli* (control) and **b** *E. coli* obtained after incubating these bacteria with carboxyl magnetic nanoparticle (MNP) (Singh et al. 2011)
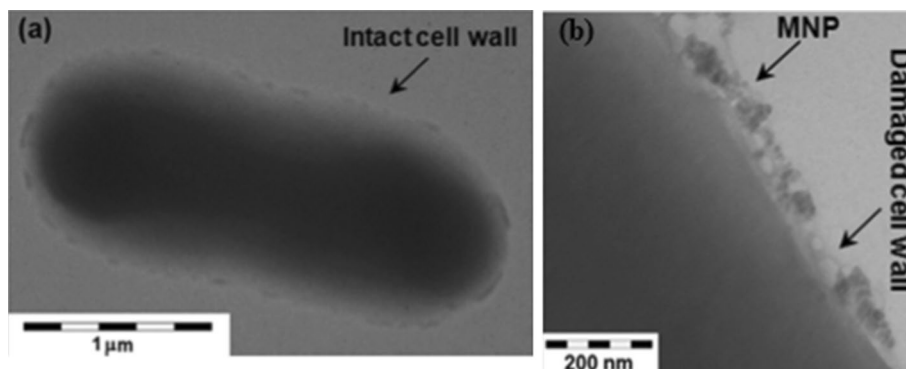
Table 4 Nanoparticles' applications to remove pollutants

Pollutants	Adsorbents	Removing capacities (mg/g)	pH	Separation of nanoadsorbent	References
As (v)	Akaganeite nanoparticle (2.6 nm)	120	7.5	Membrane filtration (0.45 μm)	Deliyanni et al. (2006)
	Magnesium ferrite (~ 3.7 nm)	83.2	7.0	External magnetic field	Tang et al. (2013)
As (III)	Zerovalent iron onto activated carbon	1.997	6.5	–	Zhu et al. (2007)
	Magnesium ferrite (~ 3.7 nm)	127.4	7.0	External magnetic field	Tang et al. (2013)
Cr(VI)	Maghemite (γ-Fe_2O_3) nanoparticles (~ 10 nm)	19.2	2.5	Electrostatic interactions and ion exchange	Hu et al. (2005)
Pb(II)	HNO_3 treated -MWCNT	91	3.5	Electrostatic interactions	Wang et al. (2007)
Cd^{2+}	Magnetite nano-rods (average diameter 60 nm)	88.39	5.5	Electrostatic force	Karami (2013)
	Akaganeite nanocrystal (average crystallite size 2.6 nm	17.1	–	Weak chemisorption process	Deliyanni and Matis (2005)
	Shellac-coated iron oxide nanoparticle	18.8	8.0	Electronic attraction and chemical adsorption	Gong et al. (2012)
$Hg2+$	Mercapto-functionalized nano-Fe_3O_4	522.9	3.0	Surface complexation	Pan et al. (2012)
	Graphite oxide nanofiller with chitosan	381	6.0	Chemisorption	Kyzas et al. (2014)
	Thiol-modified SiO_2 coated Fe_3O_4 nanoparticle	148.8	6.5	–	Zhang et al. (2013)
	Graphite oxide–magnetic chitosan composite	397	6.0	Chime sorption	Kyzas et al. (2014)
Cu^{2+}	Amino-functionalized magnetite nanoparticles	25.77	6	Chemisorption	Mei et al. (2010)
	Nanogeothite (average particle size 10–15 nm)	149.25	–	–	Chen and Li (2010)
	Nanohematite (α-Fe_2O_3, average particle size 75 nm)	84.46	–	–	Chen and Li (2010)
	Gum aerobic modified magnetic (Fe_3O_4) nanoparticle (diameter range 13–67 nm)	38.5	–	Surface complexation	Banerjee and Chen (2007)
Azo dye Reactive Red 195	TiO_2 (~ 8.8 nm)	87.0	3.0	Electrostatic interaction	Belessi et al. (2009)
Orange G	Fe_3O_4 nanoparticles—carboxymethylated chitosan conjugate (anionic magnetic nanoadsorbent)	1883	–	Surface complexation	Chang and Chen (2005)
Acid green 25	Fe_3O_4 nanoparticles—carboxymethylated chitosan conjugate(anionic magnetic nanoadsorbent)	1471	–	Surface complexation	Chang and Chen (2005)

–, not available

removal. Some of the findings are briefly discussed here. Peng et al. (2005) reported ceria nanoparticle-coated carbon nanotube and used it for arsenate removal. After being exhausted, the nanoadsorbent was regenerated by 0.1 M NaOH with a desorption efficiency of 94% (Peng et al. 2005). Through regeneration, carbon nanotubes become economically attractive for wastewater treatment. Zhou et al. (2009) modified the Fe_3O_4 by utilizing chitosan and α-keto glutaric acid to remove Cu (II). After the complete removal of metal pollutants, this nanoadsorbent was regenerated by using Na_2EDTA. This regenerated nanoadsorbent reused without varying its removal efficiency (Zhou et al. 2009).

Maghemite nanoparticles (diameter around 10 nm) were successfully applied for adsorption of different metal pollutants, namely Cr(VI), Cu (II) and Ni (II). This nanoadsorbent got saturated within 10 min. After successive adsorption, the exhausted nanoadsorbent was treated with 0.01 M of NaOH and 0.05 M of HCl for Cr(VI), Cu (II) and Ni (II) desorption, respectively. The removal efficiency was highly dependent on solution pH. After five consecutive

adsorption–desorption processes, the nanoadsorbents (maghemite) adsorption capacity remains almost constant (Hu et al. 2006).

In 2003, Deliyanni et al. used akaganeite-type nanocrystals for sorption of As (V). After each regeneration of nanoadsorbent, the capacity decay was 25–30% of its initial capacity. Through this method, only 75% of metals were recovered (Deliyanni et al. 2003). Through the regeneration of nanoadsorbent, the adsorption process for water treatment becomes more economical. Various separation processes are applied for nanoadsorbent separation, such as centrifugation (Novak et al. 2001), ultracentrifugation (Tsao et al. 2009), gel electrophoresis (Hanauer et al. 2007), diafiltration (Sweeney et al. 2006) and fractional crystallization. Due to the small particle size, the centrifugation process needs higher centrifugal force and time for sedimentation (Tsao et al. 2009). Among them, membrane filtration is a promising one, which provides continuous operation with small chemical use (Qu et al. 2013a, b). Diafiltration, a membrane-based method, provides a convenient way for the separation of water-soluble nanoparticles by a single step (Sweeney et al. 2006). It is a continuous flow process. As a result, materials cannot build up on the membrane surface like traditional ultrafiltration, thus preventing membrane fouling. Due to simple equipment and reusability of the membrane, diafiltration is cost-effective, suitable and versatile (Sweeney et al. 2006).

Recently, nanomaterials are immobilized on different support like resin, membranes to avoid the separation. But this immobilization reduces their efficiency (Qu et al. 2013a, b). Generally, magnetic nanomaterials provide extra benefit over nonmagnetic nanoparticles, because these magnetic nanoadsorbents provide a magnetic field for effective and efficient separation of inorganic pollutants. After being exhausted, this magnetic nanoadsorbent can be removed from the matrix by applying an external magnetic field. After separation, the harmful components can be desorbed by acidic or basic treatment, while these nanoadsorbents can be recovered and recycled without varying their initial efficacy (Kurniawan et al. 2006a; Ngomsik et al. 2005).

Future prospects of nanoadsorbents

Generally, the adoption efficiency and feasibility of any innovative technique depend on two factors: cost minimization and involvement of potential toxicity.

Cost and market opportunities Though nanoadsorbents offer excellent efficiency in water treatment in some cases, its production and application are costly (Qu et al. 2013a, b). They proposed some approaches to overcome the cost issue. These are (1) using low-purity nanomaterials without compromising adsorption efficiency. For example, Lee et al. (2010) prepared amino fullerene photocatalysts from fullerene soot rather than ultrapure C60 and saved average 90% production cost with minimal ($< 10\%$) loss of effectiveness, (2) long-term use of nanoadsorbent may compromise their cost-effectiveness and (3) the treatment cost can be minimized by regeneration and reusing nanomaterials (Qu et al. 2013a, b).

Recently, nanoadsorbent's synthesis increases, due to high demands for clean water treatment. In 2003, approximately € 2.28 million was utilized for environmental remediation (Li et al. 2006). According to a recent technical market report, the value of nanomaterials for environmental remediation in 2004 and 2005 was €3.9 and €9.11 million, respectively. In 2009, the market predicted to reach almost double (€20 billion). Day by day, the world population is increasing. By the next 50 years, the world population growth is expected to double with the existing population. To meet the demand of this huge population, water purification needs more investment. In 2015, the market value for nanomaterials related to environmental remediation would increase 250% (€ 1 trillion), and by 2020 it is expected to reach €1.6 trillion (Li et al. 2006; German Federal Ministry of Education and Research 2009). The increasing trend of nanotechnology products may open a potential market in environmental industries both domestic and international levels and can provide job opportunities among the increasing population. For example, at present European Union (EU) alone made €227 billion from nanotechnology related to environmental applications, which is accounted for 2.2% of its total domestic materials, and created 3.4 million employment (Roco 2003). This increasing trend of nanomaterials in the environmental application in positive not only in water treatment but also in economic competitiveness.

Nanotoxicity Nanoparticles attracted researchers for their efficient capacity to remove water pollutants. But due to its new identities in the water system, it might be a new concern soon. It may release in the environment from the synthesis process, application or disposal management. The special attributes such as small size and shape and the high surface area of nanomaterials are responsible for their toxicities also (Ali et al. 2012). For example, titanium dioxide (TiO_2) is a biologically inert chemical and has no toxicity effect in rat's

Table 5 Precautions for minimizing nanotoxicity

Before synthesis	During synthesis	After synthesis
(i) Ensure employees are properly informed, trained and supervised	(i) Always wear appropriate gloves, aprons and highly efficient respirators to minimize the health effects from non-toxic exposure	(i) Performing cytotoxicity and genotoxicity in vivo models before the commercialization of the nanoparticles. Because data are needed to access the environmental impact and risk assessment studies
(ii) Considering sustainability and biodegradability of the nanoparticle	(ii) Optimizing synthesis and sterilization steps	(ii) Careful investigation during water pollution treatment, because pollutant might transform more toxic products
(iii) Require in-depth knowledge on water treatment by nanoadsorbent	(iii) Do not disperse liquid droplets in the laboratory air	(iii) Transportation of nanomaterials in a closed system
(iv) The laboratory should be well set up and restricted for others (visitors). The working place with nanomaterials should be closed and always work with free nanomaterial powder in a closed fume hood	(iv) Synthesis of more efficient, selective and eco-friendly nanoparticle for the water treatment system	(iv) Proper management of exhausted nanoparticle. Do not spread nano-wastes in open
(v) Measure the concentration of airborne nanomaterial in the laboratory air shortly before, during and after the handling of nanomaterial		

lung at a single instilled dose (5 mg/rat) (Ferin and Oberdorster 1985). On the other hand, nano-size TiO_2 particles could be responsible to produce more pulmonary toxicity compared to their bulk counterparts (Ali et al. 2012).

The enhancement of the surface area provides a potential number of reactive groups on the particle surface. These properties make them chemically and biologically reactive, which consequences more reactive oxygen species (ROS) production. This reactive oxygen species is the primary mechanism of nanotoxicity. Due to its small size, it gets easy excess in the living systems. It is reported that nanoparticles may initiate oxidative stress, inflammation, DNA mutation, major structural injury to mitochondria and cell death (Savić et al. 2003; Li et al. 2003; Geiser et al. 2005; Ali et al. 2012).

So far, there is a lack of specific tests or systems developed for ascertaining non-toxicity. So, the safe and careful strategy should be undertaken during handling and deposal. In 2003, the Environmental Protection Agency (EPA, USA) launched a research plan to study the harmful effect of nanoparticles on the environment. At this moment, there is no specific guideline for dealing with nanoparticles. In 2010, Schmidt-Ott et al. mention some facts regarding nano-handling and disposal (Schmidt-Ott et al. 2010). For eco-friendly synthesis and application, the following precautions can be taken for minimizing the nanotoxicity (Table 5) (Schmidt-Ott et al. 2010; Freeland et al. 2012).

Conclusion

With the expansion of industrialization and agricultural activities meeting the demand for continued population growth in the world, water pollution has become a major global concern for the last few decades. Techno-economic assessment of various water treatment methods suggests that the adsorption process can be an efficient tool for water purification. However, the conventional adsorbents inherit some limitations such as high cost of adsorbent, exhaustion, poor separation efficiency, costly regeneration and so on. Recently, nanoadsorbents have received tremendous momentum in the water treatment process because of their unique properties like high surface/volume ratio with active sites, selectivity, catalytic and magnetic properties, etc. The exotic properties of nanoadsorbents and their incorporation with current technologies have added a new dimension in the revolutionization of the water treatment process around the world. So far, a wide range of nanoadsorbents has already been used successfully to remove inorganic, organic and biological pollutants. The regeneration and separation of magnetic nanoadsorbents after treatment make the process more user-friendly (i.e., convenient and cost-effective). The adsorbents show excellent results in the batch adsorption process. However, more pilot and industrial-scale studies

are essentially important to evaluate the overall efficiency of large-volume water treatment. Though the economic value of nanoadsorbents for environmental remediation is well recognized, still it requires appropriately safe and suitable strategies to avoid harmful effects (e.g., toxicity, etc.) of nanoadsorbents in the water treatment process.

Compliance with ethical standards

Conflict of interest The authors declare no conflict of interest.

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