# **Chapter 16** Green Synthesis of Nanoparticles and Thei[r](http://crossmark.crossref.org/dialog/?doi=10.1007/978-981-13-1891-7_16&domain=pdf) **CHOCK GONERATER Applications in Water and Wastewater Treatment**



#### **Contents**



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**Abstract** Green synthesis of nanoparticles (NPs) is an emerging research trend in green nanotechnology as this method is nontoxic or less toxic, eco-friendly, efficient, and cost-effective as compared to other conventional physical and chemical methods. Green synthesis of NPs employs various biological agents such as plants, bacteria, algae, and fungi, but nowadays plant-based green synthesis of NPs is gaining more attention among researchers from around the world. A variety of green synthesized NPs are currently being used in water and wastewater treatment due to their high efficiency and biocompatible nature. Green synthesized NPs are highly proficient for recycling and removal of heavy metal from wastewaters without loss of their stability and degradation of a variety of organic pollutants from wastewaters and, thus, purify the wastewaters for reuse and recycling and could solve various water quality issues worldwide. However, regeneration and reusability are the main challenges to researchers and scientist yet in the green synthesis approach as a technology transfer from laboratory scale to commercial applications. In this chapter, we discussed the green synthesis approach for NPs and their applications in water and wastewater treatment and dye degradation from wastewaters. Further, challenges and issues related to the use of green synthesized NPs in water and wastewater treatment are also discussed.

**Keywords** Green nanotechnology · Green synthesis · Nanoparticles · Wastewater treatment · Environmental protection

## <span id="page-1-0"></span>**16.1 Introduction**

Water is the necessary element for all life forms and covers two-thirds of earth's surface. It has variable physical, chemical, or biological properties after the addition of different organic/inorganic materials, pathogens, heavy metals, or other pollutants which make it harmful to the whole ecosystem. (Bora and Dutta [2014\)](#page-25-1). Moreover, the water on earth is a precious and limited resource, which is boundlessly recycled in the water cycle. Hence, the sustainable management of water resources is immediately required due to their increasing contamination with various organic and inorganic pollutants discharged along with urban wastewaters including industrial wastewaters.

The global challenge of the twenty-first century is to overcome the lack of clean water and to provide safe and clean water for all living beings to survive. The scarcity of treated and clean water influenced the living standard of our society and deducted the equal opportunities of development in the society (Qadir et al. [2007\)](#page-29-0). According to a report of the World Health Organization (WHO) in 2012, 780 million people from all around the world still lack access to safe and clean drinking water supply (Qu et al. [2013](#page-29-1)). In most of the developing countries, 90% of all diseases arise due to the use of contaminated water for drinking purpose (WHO Report [2012](#page-30-0)). According to the United Nations World Water Development Report [\(2017](#page-30-1)), over the past 5 years, clean water scarcity has become a major risk to the surviving human population (UN Water Development Report [2017\)](#page-30-1). In a report of the World Economic Forum (WEF) in 2016, it is reported that water scarcity was evaluated as the universal risk of premier concern for individuals and economies for the next 10 years (<http://unesco.org/images/0024/002471/247153e.pdf>). In the countries including India, China, Australia, Western South America, Central America, and Western North America, around 500 million people live in areas where water consumption exceeds by a double factor than treated water resources (Mekonnen and Hoekstra [2016\)](#page-28-0). According to the reports of the United Nations International Children's Fund (UNICEF) and WHO in 2015, around two-thirds of the world's population has acquired developed sanitation, and developing countries are still facing a lack of sanitation or sewer connections (Kjellén et al. [2012\)](#page-27-0). In some of the developing countries, there is no central facility to provide the connections to a water supply; generally the people depend on the self-provided services or nongovernment organization (NGO) support ([http://unesdoc.unesco.](http://unesdoc.unesco.org/images/0024/002471/247153e.pdf) [org/images/0024/002471/247153e.pdf\)](http://unesdoc.unesco.org/images/0024/002471/247153e.pdf).

Wastewater is a major source of water pollution in the developing countries and is generally classified into two main categories (Bora and Dutta [2014\)](#page-25-1): (a) residential wastewater (municipal wastewater) and (b) nonresidential wastewater (urban or industrial wastewater). According to UN-Water Report in 2015, wastewater is roughly composed of 99% water and 1% suspended colloidal and dissolved solids [\(http://](http://unesdoc.unesco.org/images/0024/002471/247153e.pdf) [unesdoc.unesco.org/images/0024/002471/247153e.pdf](http://unesdoc.unesco.org/images/0024/002471/247153e.pdf)). Residential wastewater originates from the public residence and contains 99.90% water and 10% solids, organic and inorganic content, nutrients, microorganism, and biodegradable organic materials (Bora and Dutta [2014\)](#page-25-1). The organic and inorganic contents (metals) in wastewaters are considered to be highly toxic for humans as well as the environment (Goutam et al. [2018](#page-27-1); Bharagava et al. [2017c;](#page-25-2) Saxena and Bharagava [2015,](#page-29-2) [2017;](#page-29-3) Gautam et al. [2017;](#page-26-0) Saxena et al. [2016\)](#page-29-4). However, the organic content of wastewater can be measured in terms of biochemical oxygen demand (BOD) and chemical oxygen demand (COD), the main water quality parameters, and represent the degradability of wastewater (Henze and Comeau [2008\)](#page-27-2).The amount of residential wastewater is influenced by the lifestyle and living standard of people (Chandrappa and Das [2012\)](#page-25-3). Further, nonresidential or urban wastewater mainly originates from industries, agricultural fields, and commercial actions, and its composition depends on their sources.

To provide a safe and clean water supply to all, the sustainable management of water and wastewaters is an urgent need of the present time. Currently, many conventional and nonconventional wastewater treatment technologies are available to remove pollutants from various types of wastewaters, but these technologies are sometimes not effective and require high fund investment, specific operative conditions, and maintenance cost and high energy necessities (Amin et al. [2014](#page-25-4); Baruah et al. [2012](#page-25-5); Bora and Dutta [2014](#page-25-1)). On contrary, nanotechnology offers a great potential for the development of next-generation water and wastewater treatment technologies and could replace the conventional wastewater treatment technologies (Goutam et al. [2018](#page-27-1)). Nanoparticles (NPs) are increasingly applied for the wastewater treatment and purification due to their high reactivity and degree of functionalization, large surface area, size-dependent properties, etc. (Gawande et al.

[2011\)](#page-26-1). Recently, nanomaterials offered many advantages in wastewater treatment owing to the unique characteristics which elicited from nanoscale sizes, such as improved catalysis and adsorption properties as well as high reactivity (Gawande et al. [2011;](#page-26-1) Guo et al. [2014\)](#page-27-3).

Various nanoparticles have been successfully reported for water and wastewater treatment such as zerovalent metal nanoparticles (silver nanoparticles, iron nanoparticles, zinc nanoparticles), metal oxide nanoparticles (titanium dioxide nanoparticles, iron oxide nanoparticles, zinc oxide nanoparticles), carbon nanotubes, and nanocomposites (Mueller and Nowack [2009\)](#page-28-1). NPs, especially, the chemically synthesized titanium dioxide  $(TiO<sub>2</sub>)$ , have been extensively applied in the photocatalytic treatment of industrial wastewaters (Filipponi and Sutherland [2010\)](#page-26-2). Nano-sized  $TiO<sub>2</sub>$ -based photocatalytic treatment is a highly effective method for the degradation and detoxification of recalcitrant organic and inorganic pollutants from industrial wastewaters (Ray and Ray [2009;](#page-29-5) Lu et al. [2015\)](#page-28-2). The chemical synthesis of NPs under specific conditions requires highly expensive and toxic and hazardous chemicals, and their release in the environment creates a serious ecotoxicological concern (Bhattacharya et al. [2013](#page-25-6)). Nonetheless, the green synthesis is a simple, eco-friendly, and less toxic way of synthesizing NPs from biodegradable materials such as plant extracts, microbes, and enzymes (Bhattacharya et al. [2013;](#page-25-6) Tiwari et al. [2008\)](#page-30-2). However, the synthesis of NPs using plant extracts is the most useful strategy as it lessens the chance of associated contamination while reducing the reaction time and maintaining the cell structure (Tiwari et al. [2008\)](#page-30-2). Moreover, the application of green NPs for industrial wastewater treatment could be an excellent strategy to cope with environmental pollution.

To date, only very few studies reported the application of green synthesized NPs in wastewater treatment (Bhattacharya et al. [2013](#page-25-6); Savage and Diallo [2005\)](#page-29-6). Therefore, this chapter is mainly focused on the green synthesis of nanoparticles using plant extract and their applications in dye removal and water and wastewater treatment. In this chapter, we discussed the green synthesis approach for NPs and their applications in water and wastewater treatment and dye degradation from wastewaters. Further, challenges and issues related to the use of green synthesized NPs in water and wastewater treatment are also discussed.

### <span id="page-3-0"></span>**16.2 Nanotechnology: An Overview**

The science and technology associated with various nanostructures has become a wide area of research for the sustainable development of human society, and the technology is growing up incredibly day by day to have a great impact in the future for commercialization. The term "nanotechnology" was first introduced by Taniguchi in 1974. According to him, nanotechnology is a technology which comprises the processes of separation, consolidation, and deformation of materials by single atom or molecule (Iqbal et al. [2012\)](#page-27-4). In other words, nanotechnology is also used for the study of nanomaterials that exhibit amazing properties, functionalities,

and phenomena due to the influence of small size (nanoscale) (Khan et al. [2017](#page-27-5)). It is the ability to understand, fabricate, and manipulate the materials at the nanoscale (Roco [2004\)](#page-29-7). The word "nano" comes from the Greek word *Nanos* which refers to small animals or plants. The one-nanometer scale is equal to 10−<sup>9</sup> m. The aim of nanotechnology is to measure and manipulate the matters at nanoscale; the scale of its dimension lies in the range of 1–100 nm (Mansoori and Soelaiman [2005\)](#page-28-3). At nano-level, material shows unique chemical, physical, and biological properties due to higher surface to volume ratio where the surface area of the nanoparticle is inversely proportional to particle size, which probably occurs due to the increasing order of surface atoms with respect to the decreasing order of particle size (El Saliby et al. [2008](#page-26-3)). Thus, as a result of the extremely large surface area along with high surface energy, the nanoparticle reveals distinctive optical, electrical, and magnetic properties than the bulk one (Ichinose et al. [1992](#page-27-6)).

Nanoscience and nanotechnology have various promising applications due to the affectedly changed and excellent properties of materials at the nanoscale (https:// royalsociety.org/~/media/Royal\_Society\_Content/policy/publications/2004/9693. pdf). It covers a wide spectrum of various technologies, which are based on various types of physical, chemical, and biological processes relied on nano-level, not only to create nanomaterials but also to operate them or to influence them or to use them according to their projected motives ([https://www.eolss.net/sample-chapters/C05/](https://www.eolss.net/sample-chapters/C05/E6-152-01.pdf) [E6-152-01.pdf](https://www.eolss.net/sample-chapters/C05/E6-152-01.pdf)). As the size of materials rises from bulk to nanostructure, the properties and functionalities of the materials may be different. Conclusively, if two parameters of the materials, structural arrangement of atoms or molecules and the length scale of the materials, are molded appropriately, then variation in the properties of materials can easily be achieved (Heath [1995\)](#page-27-7). Nanotechnology provided some of the promising techniques for wastewater treatment such as (a) photocatalysis, (b) nanofiltration, and (c) nanosorbents (Bora and Dutta [2014](#page-25-1)).

#### <span id="page-4-0"></span>*16.2.1 Photocatalysis*

Photocatalysis is a process defined as "change in the rate of a chemical reaction or its initiation under the action of ultraviolet, visible, or infrared radiation in the presence of a photocatalyst, a substance that absorbs the light and involved in the chemical transformation of the reaction partners" (McNaught and Wilkinson [1997\)](#page-28-4). It is a surface phenomenon which commonly includes the following five mechanistic steps: (i) diffusion of reactants to the surface of the catalyst, (ii) adsorption of the reactants on the surface of the catalyst, (iii) reaction at the surface of the catalyst, (iv) desorption of the products from the surface of the catalyst, and (v) diffusion of the products from the surface of the catalyst (Pirkanniemi and Sillanpaa [2002](#page-29-8)).

Photocatalysis is currently being applied in wastewater treatment as two different types of applications: one is solar photocatalysis, and another is photocatalytic application system equipped with artificial UV-visible light ([https://www.sswm.](https://www.sswm.info/sites/default/files/reference_attachments/MULLER et al 2010 Photocatalysis for Water Treatment.pdf) [info/sites/default/files/reference\\_attachments/MULLER%20et%20al%202010%20](https://www.sswm.info/sites/default/files/reference_attachments/MULLER et al 2010 Photocatalysis for Water Treatment.pdf)

<span id="page-5-0"></span>

Fig. 16.1 Mechanism of TiO<sub>2</sub>-based photocatalysis. (Adapted from Goutam et al. [2018\)](#page-27-1)

[Photocatalysis%20for%20Water%20Treatment.pdf](https://www.sswm.info/sites/default/files/reference_attachments/MULLER et al 2010 Photocatalysis for Water Treatment.pdf)). Nanostructured semiconductor materials are the most suitable photocatalyst because they have the most of the photogenerated electrons and holes, which are available at the surface. Photocatalysis plays an important role in the removal of organic and inorganic contaminants such as heavy metal ions and microbes.

A recent study by Goutam et al. ([2018\)](#page-27-1) presents an excellent example of the photocatalytic treatment/degradation of tannery wastewater using green synthesized  $TiO<sub>2</sub>$  nanoparticles, although some other previously reported literatures by Devatha et al.  $(2016)$  $(2016)$ , Wang et al.  $(2014)$  $(2014)$ , Ghaly et al.  $(2011)$  $(2011)$ , and Bordes et al. [\(2015](#page-25-7)) have also shown the significance of photocatalysis in wastewater treatment. Nanoparticle (NP)-based photocatalytic treatment is a highly effective method for the degradation and detoxification of recalcitrant organic and inorganic pollutants from wastewaters (Mahlambi et al.  $2015$ ). A general mechanism of  $TiO<sub>2</sub>-NP-based$ photocatalysis is shown in Fig. [16.1](#page-5-0).

The photocatalytic reduction of inorganic contaminants  $(Cr^{+6}$  to  $Cr^{+3}$ ) from wastewater along with photooxidation of organic pollutants can be easily understood by the following reaction mechanisms (Goutam et al. [2018;](#page-27-1) Malakootian and Mansuri [2015\)](#page-28-6):

$$
TiO2 (catalyst) + hv \rightarrow h+ + e-
$$
 (16.1)

$$
Cr_2O_7^{2^-} + 14H^+ + 6e^- \rightarrow +2Cr^{3+} + 7H_2O
$$
 (16.2)

$$
2H_2O + 4h^+ \to O_2 + 4H^+ \tag{16.3}
$$

$$
H_2O + h^+ \rightarrow OH + H^+ \tag{16.4}
$$

$$
OH + \text{organics} \rightarrow \text{CO}_2 + \text{H}_2\text{O} \tag{16.5}
$$

<span id="page-6-0"></span>
$$
H^+ + \text{organics} \rightarrow CO_2 + H_2O \tag{16.6}
$$

## *16.2.2 Nanofiltration*

The second popular technique for wastewater treatment is nanofiltration. It is a type of membrane process and has emerged as a promising area for extended membrane applications. Membrane filtration is a pressure-driven process in which membrane acts as selective barriers to restrict the passage of pollutants such as organics, nutrients, turbidity, microorganisms, inorganic metal ions, and other oxygen-depleting pollutants and permit the pass-through of comparatively clear water (Drioli et al. [2015;](#page-26-6) Mulder [1997\)](#page-28-7). With technological advances and the ever-increasing stringency of water quality criteria, membrane processes are becoming a more attractive solution to the challenge of water quality and water reuse (Shannon et al. [2008](#page-29-9)).

Numerous studies have previously reported on the application of microfiltration and ultrafiltration for wastewater treatment and reuse (Seo et al. [1996](#page-29-10), [1997;](#page-29-11) Snoeyink et al. [2000;](#page-30-6) Shon et al. [2013;](#page-30-5) Visvanathan et al. 2000; Ben Aim and Semmens [2001](#page-25-8); Matsui et al. [2001aa](#page-28-8), [b](#page-28-9)). Generally, membrane process has been classified into four broad categories depending on their pore sizes: microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) membranes. As per previously reported in literature, NF membranes are considered to be the best alternative to water purification. NF membrane is generally polyamide membranes and has lower salt rejection facilitating a diverse cut-off based on the molecular weight of a species with more than 92% rejection (Shon et al. [2013](#page-30-5)).The NF membrane is the relatively newly introduced technology in wastewater treatment system. Due to the less size of NF membrane  $(-1)$  to 5 nm), very small uncharged solutes from wastewater are becoming highly rejected (Kim et al. [2001;](#page-27-8) Shon et al. [2013\)](#page-30-5). Over the last few years, NF technology is developing as a feasible approach for the removal of pollutants from pulp-bleaching effluents and textile effluents, separation of pharmaceuticals from fermentation broths, demineralization in the dairy industry, and metallic pollutants and viruses from wastewaters (Bowen et al. [2002](#page-25-9)). A general mechanism of nanofiltration for the removal of different pollutants from wastewater is shown in Fig. [16.2](#page-7-1).

## <span id="page-6-1"></span>*16.2.3 Nanosorbents*

Nanoparticles (NPs) as nanosorbents have been widely used for the removal of microbes, organic dyes, and heavy metal pollutants from water and wastewaters. Nanoparticles have specific properties like extremely small size, high surface area

<span id="page-7-1"></span>

**Fig. 16.2** Schematic mechanism of nanofiltration of contaminants from wastewaters

to volume ratio, specificity for pollutants, biocompatibility, and adaptability which attributed to the relevant technology for the enhanced and efficient removal of pollutants from wastewaters. Metal oxide at nanoscale shows the higher level of adsorption as compared to bulk size because of formation of metal-ligand precipitation (Stietiya and Wang [2014](#page-30-7)). As reported previously, pH value of the wastewater affects the efficiency of metal pollutant removal by adsorption mechanism. The removal efficiency of metallic pollutants (heavy metal ions) from the wastewater increased with a pH value of wastewater. Deprotonation at nanosorbent surface is enhanced with an increased pH value of wastewater and forces of attraction between positively charged metallic ions, and the negative sites on adsorbents also increased due to increased negatively charged sites. Nassar ([2012\)](#page-28-10) reported a brief review on iron oxide nano-adsorbent for the removal of various pollutants from wastewater (Nassar [2012\)](#page-28-10). Zhang et al. ([2014\)](#page-30-8) reported the synthesis of Fe-La composite oxide for the removal of As (III) from wastewater (Zhang et al. [2014\)](#page-30-8).

According to Khajeh et al. [\(2013](#page-27-9)), nanomaterials can be classified in different groups on the basis of their role or surface property in adsorption applications such as (a) carbon nanomaterials, (b) silicon nanomaterials, (c) nanomaterials, and (d) nanoparticles as adsorbents. Although,  $TiO<sub>2</sub> NPs$  and other metal oxide nanoparticles such as  $ZrO_2$ ,  $Al_2O_3$ , and  $SiO_2$ ,  $ZnO$  have been reported with very high adsorption capacity because of their specific surface chemistry (He et al. [2008;](#page-27-10) Lian et al. [2005;](#page-28-11) Hadjiivanov et al. [1991;](#page-27-11) Bolis et al. [1991;](#page-25-10) Cui et al. [2006](#page-26-7)). A general mechanism of nano-adsorbent for the removal of different pollutants from wastewater is shown in Fig. [16.3.](#page-8-0)

## <span id="page-7-0"></span>**16.3 Techniques for Nanoparticle Synthesis**

Recently, the development of several steadfast methods to synthesize nanoparticles by controlling their sizes, shapes, and chemical composition becomes a marvelous area of research interest in the field of nanotechnology. Nanoparticles in association with organic molecules facilitate collective nature and cause the formation of

<span id="page-8-0"></span>

**Fig. 16.3** Schematic mechanism of nanosorbents for pollutant removal from wastewaters

<span id="page-8-1"></span>

**Fig. 16.4** Basic approaches for nanoparticle fabrication

different dimensional (1-D, 2-D, 3-D) mesostructures, which also gave a great interest in research. Nanoparticle synthesis methods generally use precursors in the form of solid, liquid, or gas phase, following chemical and physical deposition processes to construct various nanostructured materials.

The nanoparticle synthesis methods are primarily categorized into two approaches: top-down and bottom-up approaches (Hu and Shaw [1999](#page-27-12)) as shown in Fig. [16.4.](#page-8-1) Top-down approach includes the physical processes such as milling,

<span id="page-9-1"></span>

**Fig. 16.5** Synthesis methods for nanoparticles

crushing, etc. where bulk particles break up into small to smaller particles; as a result, nanoparticles are formed. Though this approach has a negative impact on the surface structure due to crystallographic injury during the formation of the nanostructure, on the other hand, bottom-up approach includes chemical and biological processes such as sol-gel, laser pyrolysis, plasma spraying process, aerosolbased process, green synthesis method, etc. where the formation of nanoparticles starts from the basic atomic or molecular levels, i.e., the nanostructures are formed by the addition of atom to atom, molecule to molecule, or cluster to cluster. The bottom-up approach is very fabulous and best as compared to top-down approach to produce nanostructured materials with uniform structures and distributions (Saravanan et al. [2008](#page-29-12)). Various techniques that are used to synthesize nanomaterials are shown in Fig. [16.5.](#page-9-1)

## <span id="page-9-0"></span>*16.3.1 Mechanical Milling Method*

Mechanical milling method mainly associated with the production of powders where fine particles are created from the surfaces of small particles reduced from bulk materials during the rubbing with one another by the introduction of an

agitator medium or high-speed air jets to produce enormous pressure and friction together (Ichinose et al. [1992](#page-27-6)). It is very high energy consumption route where structural defects, chemical disorders, and elastic strain are introduced to parent bulk material during the whole process. As a result of which, nanostructures are produced (Saravanan et al. [2008](#page-29-12)). Ball milling is an appropriate example of mechanical milling.

## <span id="page-10-0"></span>*16.3.2 Physical Vapor Deposition*

Physical vapor deposition is a technique to produce nanostructures at gas phase using a focused beam of electrons that heat the parent bulk material. This method does not depend on any catalytic interaction during the synthesis process, and there occurs no chemical reaction from start to end of the process during the formation of final production from parent entity, namely, sputtering, laser ablation, laser pyrolysis, etc.

## <span id="page-10-1"></span>*16.3.3 Sputtering*

Sputtering is one of the most influential physical vapor deposition techniques and useful for nanostructured film production where surface atoms of evaporated material become separated to generate that occurrence [\(https://sci-hub.tw/10.1533/](https://sci-hub.tw/10.1533/9781845696689.58) [9781845696689.58](https://sci-hub.tw/10.1533/9781845696689.58)).

# <span id="page-10-2"></span>*16.3.4 Laser Ablation*

Laser ablation is one type of heating technique where materials like metal and compounds are melted and evaporated by using a laser beam to generate nanostructures under high vacuum system (Ichinose et al. [1992](#page-27-6)).

## <span id="page-10-3"></span>*16.3.5 Laser Pyrolysis*

Laser pyrolysis is a laser-supported deposition technique where the vaporized reactant material is decomposed by using a potent laser beam in some inert gas environment. In this method, the reactant vapor atoms interact with inert gas atoms under collision, then deposited on a substrate to grow nanostructured film (Kulkarni [2015\)](#page-27-13).

# <span id="page-11-0"></span>*16.3.6 Sol-Gel*

Sol-gel is a chemical process to synthesize nanoparticles by gelation followed by precipitation and calcination, respectively. Generally, the gel is configured from stabilized sols where sols are often present in the form of colloidal aggregates of small metal oxy-hydroxy particles in an aqueous solvent. However, it is possible to break up this aggregation in the presence of some capping agents. Hydrolysis and condensation in a controlled way play a pivotal role during gelation of the system. The relative rates of hydrolysis and condensation depend on various physical and chemical parameters such as temperature, pH, the concentration of metal ions precursor solution, etc. The change in pH value affects the surface properties of the synthesized material remarkably, which occur due to the change in porosity of gel. As usual for the preparation of nanostructures, metal alkoxides are taken as precursors in some organic solvent. Sometimes in place of alkoxide, chelate also can use to be a precursor which has the ability to stabilize the metal cations. Chelating facilitates mainly for the preparation of multicomponent gel, for example,  $TiO<sub>2</sub> - SiO<sub>2</sub> mixed gel, etc.$  (Kung and Ko [1996\)](#page-27-14).

## <span id="page-11-1"></span>*16.3.7 Chemical Vapor Deposition*

Chemical vapor deposition technique is used to generate a highly pure nanostructural thin film having high performance. In this method, the precursor is deposited on the surface of a substrate by heating followed by evaporation to generate vapor, and the deposition occurs through a chemical reaction under vacuum in such a way to create a difference in chemical properties between precursor material and product. After exposing the surface of a substrate to the vaporized precursor, primarily a template is formed on the surface; thereafter, nanostructures are formed on that template (Filipponi and Sutherland [2010\)](#page-26-2). The formation of the nanostructure is always influenced by the reaction temperature, the reaction rate, and also the precursor concentration during the deposition occurrence (Kim et al. [2004](#page-27-15)). Overall, this method facilitates for uniform coating of nanostructures on the substrate surface, though there are some restriction due to the higher temperature requirement in the process.

# <span id="page-11-2"></span>*16.3.8 Colloidal Method*

Colloidal method is a chemical precipitation method where different ionic solutions are mixed up to get precipitated by the adjustment of the reagent's concentration and capping agents and also by using the temperature and pressure in a controlled way. This method requires a stabilizer to prevent agglomeration that arises mainly

due to the presence of van der Waals forces between colloidal nanoparticles which can be stabilized electrically or sterically by Coulombic repulsions and adsorption of surfactants onto the nanoparticles surfaces, respectively (Fendler [2001](#page-26-8)). This method is very popular to produce metal and metal oxide nanoparticles and useful in various organic and pharmaceutical fields.

# <span id="page-12-0"></span>*16.3.9 Spray Pyrolysis*

Spray pyrolysis is a chemical deposition technique which is extensively used for the preparation of nanomaterial thin film. The formation of thin film depends on numerous parameters such as the spray rate, the temperature of the substrate, concentration of chemical solution, etc. The deposition efficiency can be improved by controlling the sizes of droplets and their distribution on the substrate during spraying. It is a very easy approach to prepare nanostructures on the substrate as a film by taking facilities for doping of several elements with any proportions in spraying solution. There are no limitations on quality, dimension, and surface properties of the substrate, and thus, the method has certain positive impacts compared with the other vapor deposition techniques to have opportunities for industrialization (Patil [1999](#page-28-12)).

# <span id="page-12-1"></span>**16.4 Green Synthesis Approach for Nanoparticles Fabrication**

Conventional methods are bound with various limitations such as being expensive, the generation of hazardous toxic chemicals, the requirement of high temperature and pressure, etc. (Sharma et al. [2015\)](#page-29-13). Due to these drawbacks of conventional methods, researchers are currently focusing on the biological systems and giving preference toward green synthesis.

Green synthesis is an environment-friendly, cost-effective process to synthesize nanostructural material to have adjustable structures, morphologies, and particle size distributions (Khan et al. [2017\)](#page-27-5). Recent progress displays an important role in biological synthesis for the production of nanomaterials. Green synthesis methods employ organisms like plant, bacteria, fungi, etc. for the synthesis of nanomaterials, and it has become a very quickly rising area of research due to their less toxic or nontoxic nature, environment-friendly behavior, and low cost of preparation (Das et al. [2016](#page-26-9); Goutam et al. [2017](#page-26-10), [2018](#page-27-1)). For plant-mediated synthesis method, the plants are used to synthesize nanoparticles that have excessive potential in heavy metal accumulation mechanism and detoxification (Iravani [2011](#page-27-16); Goutam et al. [2018\)](#page-27-1). Plant extract can be produced from leaves, stems, flower, and seeds of various plants. The extract contains biomolecules such as protein, amino acid, enzymes,

vitamins, terpenoids, flavonoids, alkaloids, phenolic acids, etc., which act as capping and reducing agents that can reduce metal ions during the bioreduction process to produce nanoparticles or nanostructures with different shapes and sizes (Goutam et al. [2018;](#page-27-1) Iravani [2011;](#page-27-16) Shiv Shankar et al. [2003,](#page-29-14) [2004](#page-30-9)). For example, *Pelargonium graveolens* extract reduces gold ions into nanoparticles of size 20–40 nm; *Trigonella foenum-graecum* extract can reduce gold ions into nanoparticles of size 15–25 nm (Shiv Shankar et al. [2003](#page-29-14); Aromal and Philip [2012](#page-25-11)). *Alternanthera dentata* leaf extract reduces silver ions into nanoparticles of size 50–100 nm (Kumar et al. [2014\)](#page-27-17), while leaf extracts of *Ziziphora tenuior*, *Ficus carica*, *Cymbopogan citratus*, and *Premna herbacea* reduce the silver nanoparticles to the sizes 8–40 nm, 13 nm, 32 nm, and 10–30 nm, respectively (Sadeghi and Gholamhoseinpoor [2015](#page-29-15); Ulug et al. [2015;](#page-30-10) Geetha et al. [2014;](#page-26-11) Masurkar et al. [2011;](#page-28-13) Kumar et al. [2013](#page-27-18)).

Terpenoid and phenolic are the organic polymers which exhibit strong antioxidant activities and act as stabilizing agents for synthesizing nanomaterials (Shiv Shankar et al. [2003](#page-29-14)). Phenolate ions were capable of transferring electrons to the metal ions during nanoparticle formation (Lukman et al. [2010](#page-28-14)). For example, eugenol is the leading phenolic of *clove* extract which plays the foremost role in biore-duction of AgNO<sub>3</sub>and HAuCl<sub>4</sub>to form nanoparticles (Singh et al. [2010](#page-30-11)). Flavonoids such as anthocyanin, flavonol, flavone, flavanone, isoflavonoid, chalcone, etc. are also composed of polyphenolic compounds that are capable of reducing the metal ions during the formation of nanoparticles. For example, *Ocimum basilicum* plant extract that contains flavonoids like luteolin, apigenin, etc. plays a significant role in the formation of silver nanoparticles by reducing silver ions (Ahmad et al. [2010\)](#page-25-12). Amino acids are also found to be able to bind metal ions and reduce them into nanoparticles. It is observed that amino acids such as cysteine, arginine, methionine, lysine help to bind silver ions for the formation of silver nanoparticles (Gruen [1975\)](#page-27-19).

Nanoparticle formation during bioreduction by plant extracts depends on various chemical and physical parameters such as pH, temperature, etc. (Iravani [2011](#page-27-16)). It is found that at low pH value, the rate of nucleation of metal ions becomes very low such that agglomeration occurs in metal nanoparticles, for which there may be a chance of formation of large-sized nanoparticles due to low pH value, while the higher pH value can help to the formation of small-sized nanoparticles (Thanh et al. [2014\)](#page-30-12). The shape of synthesized nanoparticles also depends on pH. At higher pH, spherical and decahedral types of nanoparticles are generated. It is observed that the case of synthesizing the silver nanoparticles using the plant extract of *Curcuma longa*, alkaline pH associates with large numbers of functional groups in comparison with acidic pH, which facilitates binding huge numbers of silver ions to form greater numbers of silver nanoparticles having a very small diameter (Sathishkumar et al. [2010\)](#page-29-16).

The preeminent activities of biomolecules that are present in plant extract are influenced by the pH of the surrounding medium which highly affects the reaction of biomolecules with the metal ions during nanoparticles production. As per the previous report on the formation of silver nanoparticles using *Medicago sativa* extract, at pH value 11, a high monodispersity is found during the reaction that causes the formation of nanoparticles with average size 11.5 nm, but at pH 2 no such reaction happens (Lukman et al. [2010](#page-28-14)).The temperature also affects the nucleation rate during nanoparticles synthesis process using plant extract. The sizes, shapes, and production rate of nanoparticles change with the variation of reaction temperature during synthesis (Sathishkumar et al. [2010\)](#page-29-16). Incubation temperature also affects the metal ion reduction process and indicates a variation of colors due to surface plasmon resonance. For example, during synthesis, with a variation of temperature, gold nanoparticles show different color effects such as yellow-brown, purplishpink, and pinkish-brown colors at the temperatures 60  $^{\circ}$ C, 80  $^{\circ}$ C, and 100  $^{\circ}$ C, respectively (Bankar et al. [2010\)](#page-25-13). According to a previous report, it is necessary to maintain the temperature greater or equal to 30 °C to synthesize silver nanoparticles using *M. Sativa* plant seed extract (Lukman et al. [2010\)](#page-28-14). The structural form of synthesized silver nanoparticles using *Cassia fistula* plant extract varies with temperature. At room temperature due to the accumulation of silver nanoparticles in a linear manner which causes recrystallization, as a result, formation of silver nanowires occurs. But, when calcined at  $400\degree C$ , then raising the temperature, the interaction between biomolecules and surfaces of silver nanoparticles also changes, which hampers the coalescence of nanoparticles; as a result, some irregular nanorods and spherical silver nanoparticles are perceived (Lin et al. [2010\)](#page-28-15). Nucleation depends on reaction temperature such that at high temperature, there action rate becomes high and maximum gold ions are used to form nuclei which prevent the secondary reduction process on the surfaces of that nuclei. As a result, spherical nanoparticles are made. Generally, the secondary nucleation occurs at low temperature (Das et al. [2011\)](#page-26-12). A schematic representation of green synthesis mechanism of nanoparticles using leaf extract of the plant is shown in Fig. [16.6.](#page-15-0)

# <span id="page-14-0"></span>**16.5 Application of Green Synthesized Nanoparticles in Wastewater Treatment**

Green nanotechnology has diverse applications in different fields (Fig. [16.7\)](#page-16-0). However, green synthesized metal or metal oxide nanoparticles have been extensively used in the treatment of different type of wastewaters (Table [16.1\)](#page-17-0). In a study, silver nanoparticles have been synthesized using *P. thonningii* leaf extract and used for the effective removal of heavy metals (magnesium, copper, lead, iron) from simulated wastewater (Shittu and Ihebunna [2017\)](#page-29-17). Green synthesis of silver nanocomposite using leaf extract of *Ocimum tenuiflorum* (Black Tulsi) has been reported by Banerjee et al. [\(2014](#page-25-14)) for the treatment of textile dye. Treated wastewater can be efficiently reused for industrial and domestic purposes.

Ehrampoush et al. [\(2015](#page-26-13)) reported the efficient cadmium removal from wastewater by green synthesized iron oxide nanoparticles using peel extract of tangerine. In this study, peel extract of tangerine was used as a stabilizing agent, which attributed to the inexpensive and eco-friendly synthesis of iron oxide nanoparticles as it also plays an important role to control the size and morphology of nanoparticles

<span id="page-15-0"></span>

Fig. 16.6 Workflow for green synthesis of nanoparticles using plant leaf extract (Source: authors own)

during synthesis. Ehrampoush et al. ([2015](#page-26-13)) showed that the concentration of peel extract of tangerine impact the size of nanoparticles. The synthesized iron oxide nanoparticles were applied as a good adsorbent for the removal of cadmium from wastewater. The average size of synthesized iron oxide nanoparticles observed decreased when the concentration of peel extract increases. In the study of Ehrampoush et al. [\(2015](#page-26-13)), a maximum of 90% removal of cadmium ions has been reported (Ehrampoush et al. [2015\)](#page-26-13).

In the study of Rosales et al. [\(2017](#page-29-18)), green synthesis of zerovalent iron nanoparticles using two different extracts, green tea (*Camellia sinensis*) and rooibos (*Aspalathus linearis*), has been performed. In this study, it was found that the reactivity of synthesized nanoparticles of zerovalent iron is higher when using rooibos (*Aspalathus linearis*) extract, although the antioxidant content was highest in green tea (*Camellia sinensis*). Further, green synthesized zerovalent iron nanoparticles were applied in the degradation of textile dye and demonstrated a better performance in the treatment of the wastewater.

In the study of Goutam et al. ([2018](#page-27-1)), anatase phase of green titanium dioxide (TiO2) NPs using *Jatropha curcas* L. leaf extract was successfully synthesized to evaluate their performance for the photocatalytic treatment of TWW after the secondary treatment process. The synthesized anatase phase of the spherical  $TiO<sub>2</sub>$ 

<span id="page-16-0"></span>

**Fig. 16.7** Applications of green nanotechnology

NPs unfolds the presence of phytochemicals in leaf extract, which might involve in the capping/stabilization of NPs. Moreover, the green synthesized  $TiO<sub>2</sub>$  NPs were applied for the first time to testify its potential for the simultaneous removal of chemical oxygen demand (COD) and chromium (Cr) from secondary treated TWW. In this study, treatment of tannery wastewater using green synthesized TiO2 NPs in a self-designed and fabricated parabolic trough reactor (PTR) resulted in the 82.26% removal of COD and 76.48% removal of Cr from TWW (Goutam et al. [2018](#page-27-1)).

Chaudhary et al. [\(2017](#page-25-15)) reported a rapid method of green chemistry approach for the synthesis of gold nanoparticles (AuNPs) using *Lagerstroemia speciosa* leaf extract. The green approach for the synthesis of AuNPs showed strong photocatalytic activity in the reduction of dyes, viz., methylene blue, methyl orange, bromophenol blue, and bromocresol green, and 4-nitrophenol under visible light in the presence of NaBH4 (Choudhary et al. [2017](#page-25-15)). This study proposed the nontoxic and cost-effective approach for the synthesis of AuNPs using plant extract.

<span id="page-17-0"></span>

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Table 16.1 (continued) **Table 16.1** (continued)



Devatha et al. ([2016\)](#page-26-4) reported the green synthesis of iron nanoparticles using plants. The motive of this study was to prepare leaf extracts, precursor, and synthesis of iron nanoparticles and to assess its efficacy in the treatment of domestic wastewater. The synthesis of iron nanoparticles was performed using leaf extracts of *Mangifera indica*, *Murraya koenigii*, *Azadirachta indica*, and *Magnolia champaca* to check its potential for the treatment of domestic wastewater. A significant removal in total phosphates, ammonia nitrogen, and chemical oxygen demand from domestic wastewater was achieved upon treatment with the synthesized nanoparticles. Among the different plant-mediated synthesized iron nanoparticles, *Azadirachta indica* showed the satisfactory results in the removal of 98.08% of phosphate, 84.32% of ammonia nitrogen, and 82.35% of chemical oxygen demand (Devatha et al. [2016\)](#page-26-4).

Al-Qahtani ([2017\)](#page-25-16) reported the synthesis of zerovalent silver nanoparticles by using an environmentally eco-friendly method without using hazardous compounds with the help of *Ficus benjamina* leaf extract. The synthesized silver nanoparticles were satisfactorily capable to remove the  $Cd^{2+}$  from contaminated solution, and various parameters like adsorbent dose, heavy metal concentration, pH, agitation speed, and contact time were studied (Al-Qahtani [2017](#page-25-16)).

Davar et al. ([2015\)](#page-26-16) synthesized ZnO NPs as a photocatalyst using lemon juice and zinc acetate as precursors and also investigated the effect of sucrose addition on the initial precursors. In this study, it has been showed that the photocatalytic activity of synthesized material was tested for the degradation of methyl orange, methyl red, and methylene blue solutions and resulted in the photocatalytic degradation of organic dyes. Moreover, synthesized ZnO NPs were used in decolorization processes and the treatment of textile dyes (Davar et al. [2015](#page-26-16)).

According to the study of El-Kassas et al.  $(2016)$  $(2016)$ , Fe<sub>3</sub>O<sub>4</sub>-NPs were successfully synthesized using two seaweeds *Padina pavonica* L. Thivy and *Sargassum acinarium* L. Setchell 1933 water extracts. Further, the algal extract was used as a reducing agent for FeCl<sub>3</sub> causing the photosynthesis of Fe<sub>3</sub>O4-NPs. The biosynthesized Fe3O4-NPs were entrapped in calcium alginates beads and used in Pb adsorption experiments. Green synthesized Fe<sub>3</sub>O<sub>4</sub>-NPs alginate beads via *P. pavonica* L. Thivy had a high capacity for 91% removal of Pb while that of *S. acinarium* L. Setchell 1933 had 78% removal capacity after 75 min (El-Kassas Hala et al. [2016](#page-26-15)).

Mystrioti et al. [\(2016\)](#page-28-17) successfully synthesized nano-iron suspensions for application in Cr(VI) reduction. In this study, five plant extracts and juices, i.e., extracts of *Camellia sinensis* (green tea, GT), *Syzygium aromaticum* (clove, CL), *Mentha spicata* (spearmint, SM), *Punica granatum* juice (pomegranate, PG), and red wine (RW), have been taken to study their effectiveness in the synthesis of nano-iron suspensions. In the extract of plant and juice, polyphenols contained in extracts act as reducing agents for iron ions in aqueous solutions and stabilize the nanoparticles produced from additional agglomeration and oxidation. In this study, the concentration of non-iron in suspensions was estimated and observed. The maximum concentration of nano-iron in suspensions was estimated using RW and PG at a mixing ratio of iron solution to extract equal to 2 was obtained

22 mM and lower concentrations, were obtained using GT and CL extracts up to 18 mM. Thus, PG juice and RW showed the effective nature for the synthesis of nano-iron. PG juice and RW with GT extracts effectively reduce the Cr(VI) with removal capacity as high as 500 mg Cr(VI) per g of iron in nanoparticles (Mystrioti et al. [2016\)](#page-28-17).

Wang et al. ([2014\)](#page-30-3) reported the first time synthesis of spheroidal iron nanoparticles through a one-step room-temperature biosynthetic route using eucalyptus leaf (EL) extracts. In this study with the help of X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectrometer, it has been showed that some polyphenols are bound to the surfaces of EL-Fe NPs as a capping/stabilizing agent. Reactivity of EL-Fe NPs was evaluated for the treatment of swine wastewater, and results indicated that 71.7% of total N, 30.4% of total P, and 84.5% of COD were removed, respectively (Wang et al. [2014](#page-30-3)).

Kuang et al. [\(2013](#page-30-17)) reported the successful synthesis of iron nanoparticles (Fe NPs) using tea extracts as a catalyst for the Fenton-like oxidation of monochlorobenzene (MCB), where 69%, 53%, and 39% of MCB were, respectively, degraded by Fe NPs synthesized using green tea extracts along with tea extracts and black tea extracts. Further, it has been observed that Fe NPs synthesized using green tea extracts (GT-Fe NPs) demonstrated the best degradation since green tea contains a high concentration of caffeine/polyphenols used as both reducing and capping agents in the synthesis of Fe NPs. In this study, results showed that the oxidation of MCB and the removal of chemical oxygen demand (COD) using GT-Fe NPs were 81% and 31%, respectively, at optimal conditions, where dosages were 0.6  $g/L$ GT-Fe NPs,  $0.045$  mol/L  $H_2O_2$ , and preliminary pH of 3.0. Matched to homogeneous Fenton oxidation of MCB, GT-Fe NPs as a heterogeneous catalyst indicated that  $Fe<sup>2+</sup>$  and  $Fe<sup>3+</sup>$  filtered from GT-Fe NPs nanoparticles and consequently reduced the formation of iron sludge. Conclusively, GT-Fe NPs have shown satisfactory removal of MCB and possible Fenton-like oxidative mechanism of MCB from wastewaters (Kuang et al. [2013](#page-30-17)).

# <span id="page-22-0"></span>**16.6 Stability and Reusability of Green Synthesized Nanoparticles**

From the literature survey, it has been clearly observed that the green synthesized NPs have greater potential in terms of efficient, safe, nontoxic, clean, and environment-friendly synthesis for higher pollutant removal performance. The application perspective of nanoparticles in pollutant removal/water and wastewater treatment cannot be ignored in terms of regeneration and reusability as these factors are important in the cost-benefit analysis of the nano-based water and wastewater treatment technology and its sustainability (Venkateswarlu et al. [2016](#page-30-18)). It has been reported in many studies that nanoparticles are applied as adsorbent and produce zero effluent (Wang et al. [2013;](#page-30-19) Dinesh and Pittman Jr [2006;](#page-26-19) Srivastava et al. [2017\)](#page-30-20).

Moreover, it is worth to consider that NPs have the ability to produce zero effluent owing to the presence of organic functional groups on the surface of adsorbent which could eventually degrade after a certain period of time and same characteristic causes to minimize the reusability of NPs (Lunge et al. [2014](#page-28-19)).

The reusability of NPs, repeated recycling, and adsorbent stability are essential key factors to economic concerns and commercial success. Srivastava et al. [\(2017](#page-30-20)) reported that the spherical magnetic NPs with an average diameter of 8.76 nm synthesized using *Lagerstroemia speciosa* bark (LB) extract by co-precipitation method showed significant Cr(VI) removal from aqueous solution. In this study, adsorption kinetics and equilibrium have been well described by Langmuir isotherm and pseudo-second-order model separately. After the maximum adsorption of Cr(VI), magnetic NPs were collected effortlessly from the aqueous solution by a magnet, and the adsorbed Cr(VI) by synthesized magnetic NPs was found to be more than 93.72% after 11 successive adsorption-desorption cycles (Srivastava et al. [2017\)](#page-30-20). Martinez et al. ([2016\)](#page-28-20) synthesized the iron oxide NPs using *Eucalyptus globulus* plant extract at different ratios of iron and plant extract. In this study, synthesized iron oxide NPs were found effective in arsenic adsorption. However, the desorption studies of As(V) clearly showed that synthesized iron oxide NPs can be easily regenerated using moderate concentration basic solutions (Martínez-Cabanas et al. [2016\)](#page-28-20). Further, Lingamdinne et al. ([2017\)](#page-28-21) synthesized magnetic inverse spinel iron oxide nanoparticles (MISFNPs) using seed extract of *Cnidium monnieri* L. Cuss (CLC). The synthesized MISFNPs was further used for the removal of Pb(II) and Cr(III) from aqueous solutions through batch studies. From this study, it has been concluded that green synthesized NPs synthesized by a green route are capable of recycling and removal of heavy metals without loss of its stability, and the synthesized MISFNPs were easily recoverable and reusable for the removal of heavy metals up to at least five times without a loss of stability (Lingamdinne et al. [2017\)](#page-28-21). Further research is required in this direction to use green synthesized nanoparticles effectively in water and wastewater treatment.

## <span id="page-23-0"></span>**16.7 Challenges and Future Prospects**

Green synthesized NPs are nontoxic in nature and eco-friendly and can be synthesized by simple green chemistry approach using plant extract that contains essential metabolites, which act as reducing/capping agents during NP synthesis (Ali et al. [2017\)](#page-25-18). Green synthesized NPs have been successfully applied in the pollutant removal from water and wastewater during treatment and, thus, can produce zerovalent effluent discharge. However, some challenges and future prospects should be considered (Ali et al. [2017](#page-25-18)):

(a) The toxicity of long-term exposure of the green synthesized NPs to human beings should be assessed before application from desorbed pollutants.

- (b) Despite the simple synthesis method, the use of solution extract volume, temperature, solvent type, pH, strength of precursor, and functional groups from plant metabolites should be optimized to avoid any change in the magnetic behavior and saturation magnetization value of the green synthesized NPs.
- (c) Efforts are required to devise the NP morphology and saturation magnetization value by optimizing the synthesis method to maintain the stability of NPs for efficient pollutant removal from water and wastewaters and NP magnetic separability.
- (d) There is a need to synthesize new NPs with a wide range of organic functional groups by manipulating the plant metabolites and synthesis method for the selective as well as multi-pollutant removal from water and wastewaters.
- (e) Cost-benefit analysis should be performed for commercial purposes as there is no cost data available to date.
- (f) Further, biocompatibility test should also be performed for biomedical applications.

# <span id="page-24-0"></span>**16.8 Conclusions**

Green nanotechnology is rapidly contributing to the removal of emerging pollutants, organic/inorganic contaminants such as dyes and heavy metal ions from water and wastewaters. Nanoparticles can greatly influence the domain of wastewater treatment in the coming future; however, focusing on the improvement in the existing treatment methods by increasing the efficiency of the processes and enhancing the reusability of nanomaterials can save the cost of operation of the plant or processes. Current wastewater treatment technologies can remove organic and inorganic pollutants from water and wastewater, but these methods are energy intensive and uneconomical because of their inability to completely purify water/wastewater as well as to reuse the retentates. However, the green synthesis of NPs is a very promising approach of green nanotechnology, and green synthesized NPs can easily be separated and reused because of their specific nature and high stability. Moreover, the use of green synthesized NPs in water and wastewater treatment is not only an environment-friendly option but also a promising technology for low-economy countries that can also fulfill the concept of zero effluent discharge after wastewater treatment using low cost and energy. Thus, green synthesized NPs could be an essential component of water and wastewater treatment systems in the future. Further, future research is warranted in this direction to commercialize the use of green synthesized NPs in pollutant removal from wastewater/wastewater treatment.

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