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Preparation of Nano-Particles and Their Applications in Adsorption

Tooba Saeed, Abdul Naeem, Tahira Mahmood and Nazish Huma Khan

Abstract

The nano-technologies and nano-materials draw incredible consideration in recent years. Nano-particles are the particles having size ranging from 1 to 100 nm. The nano-particles are usually categorized into different classes, and their classification is based on size, shape, material production, and dimension. They show superior properties, i.e., enhanced reactivity, high BET surface area, sensitiveness, and steadiness as compared to their bulk materials. In this chapter, different approaches of synthesizing nano-particles, including sol gel, chemical vapor deposition, and biosynthesis are talked over. In the treatment of wastewater, nano-particles offer a possibility for effective adsorption of contaminants organic as well as inorganic. This chapter presents an overview on nano-particles, their types, characteristics, synthetic approaches, and applications in the field of surface chemistry.

Keywords: nano-particles, adsorption, sol–gel, chemical vapor deposition, biosynthesis, carbon nano-tubes, mechanical milling, nano-lithography, laser ablation, iron nano-particles, manganese oxide nano-particles, zinc oxide and magnesium oxide

1. Introduction

This chapter consists of three main sections. The first section gives an overview about the introduction of nano-particles. The next section is about the synthesis of nanoparticles and the last section describes the use of nanoparticles as adsorbents.

The preface "nano" is known for nineteenth century for its ever-increasing applications in various fields of science. A few nano-containing terms that are found in the record (usually in scientific reports and books) are nano-materials, nano-chemistry, nano-science or nanotechnology. The preface nano comes from a Latin nanos meaning dwarf that means extremely small. According to units system working internationally, it is used to represent a reduction factor of 109 times. Consequently, the nano-materials are usually dignified in nano-meters (1 nm is equivalent to 10^{-9} m) and it comprises systems having size less than macroscopic measurements and greater than molecular ones (mostly >1 nm and <100 nm) at least in one spatial dimension. This characteristic scale might be used for a particle size, diameter and layer thickness [1–5].

1.1 Classification of nano-materials

The nano-materials are different in structure, size and shape. They can be of various shapes like rod, globular, conical, hollow, coiled, plane, cylindrical and asymmetrical, while some are crystalline or amorphous.

Nano-materials are generally classified into nano-emulsions, nano-clays and nano-particles. Nano-particles are present as nano-composites or nano-structures. These nano-structures are made from basic units or blocks having small dimensionality i.e. zero, one, two and three dimensions. In zero dimensional nano-particles, the moment of electrons is cramped in all three dimensions, e.g. quantum dots. If electrons can move freely in x-direction only, they are one dimensional nanoparticles e.g. quantum wires. Whereas, in two dimensional thin films and three dimensional nano-structured materials, free electrons can move freely in x, y and x, y, z directions respectively.

Based on material production and role in sorption process, nano-particles can also be categorized into organic, mixed oxide nano-structures, magnetic, inorganic (metallic) and carbon based nano-particles. Organic nano-particles are self-assembled, three dimensional fabricated by synthetic and natural organic molecules, i.e. protein masses, milk suspension and lipid bulks etc. Commonly known organic nanoparticles are micelles, dendrimers, ferritin and liposomes. Inorganic nano-particles usually are manufactured from inorganic salt precipitations. They are non-carbon containing particles and their most common examples are metal and metal oxide particles. Carbon based nano-particles are manufactured entirely from carbon e.g. graphene, fullerenes, carbon nano-tubes, carbon nano-fibers, carbon black and sometimes activated carbon (**Figure 1**) [6–10].

In nano-materials (especially nano-particles) molecules and atoms act differently and reveal inimitable physical, chemical and electronic properties. These properties are different from their bulk counterparts and sometimes the same kind of nano-particles can show diverse characters. Physical properties of nano-particles
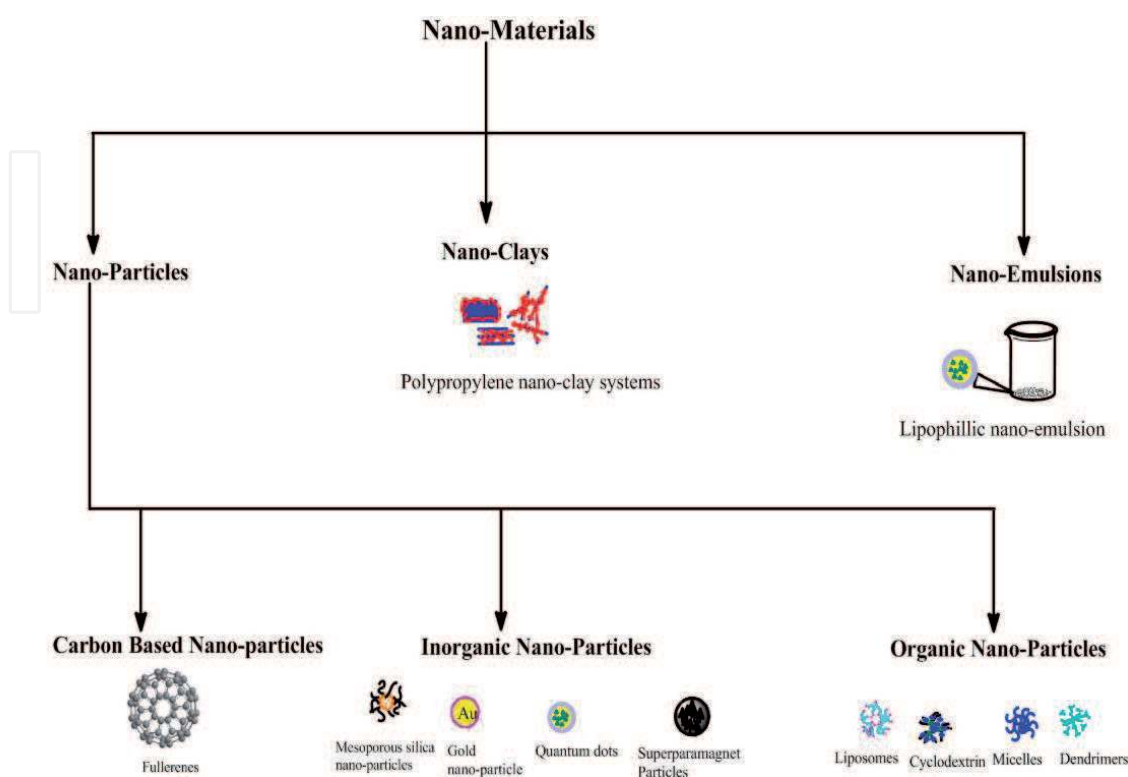

Figure 1.
Classification of nano-materials.

include absorption, reflection, light dispersion, color of nano-particles, hydro-phobicity, hydro-philicity, suspension and dispersion. When layered onto a surface or in the form of solution their absorption and reflection properties make them a perfect choice for different fields. They also show outstanding chemical properties, i.e. anti-destructive, oxidation, reduction, flammability, sensitivity and stability towards humidity, atmosphere, heat, light and dis-infection, non-toxicity, biode-gradability, anti-bacterial and fungal properties. These properties also enable them ideal materials for environmental and biomedical applications. Nano-particles also exhibit mechanical properties like elasticity, ductility, flexibility, tensile strength and electrical properties including semi-conductivity, conductivity and resistivity which have directed a route for them to be used in renewable energy applications.

These distinctive and inimitable properties of nano-particles make them perfect and formidable for amazing and interesting applications in physical science, material science, agriculture, food, engineering, industrial and biomedical sciences (**Figure 2**).

These applications encompass them to be used in electronic, drug delivery, optical, mechanics, catalysis, bio-encapsulation and wastewater treatment especially adsorption [4–15]. Besides all these properties, nano-particles have some toxic effects for aquatic and human health. As they have small size, they can easily enter through the skin of organisms and consequently enter into the body fluid. Furthermore, nano-particles used in sun screens can absorb deep inside and become toxic to the skin, bones and liver cells. Their greater surface area often makes them more sensitive, explosive and reactive. Inhaling directly in the environment of nano-particles can adversely affect the function of lungs especially in human being. However, highlighting the health issues caused by nano-particles, does not mean to ignore their extraordinary importance in technology, industries and environment [16–18].

It is important to mention that water is one among the basic necessities of every organism. From total water present on earth, only 0.01% portion is available for human [19, 20]. Shortage of drinking water is increasing day by day due to demolition of water means [21].
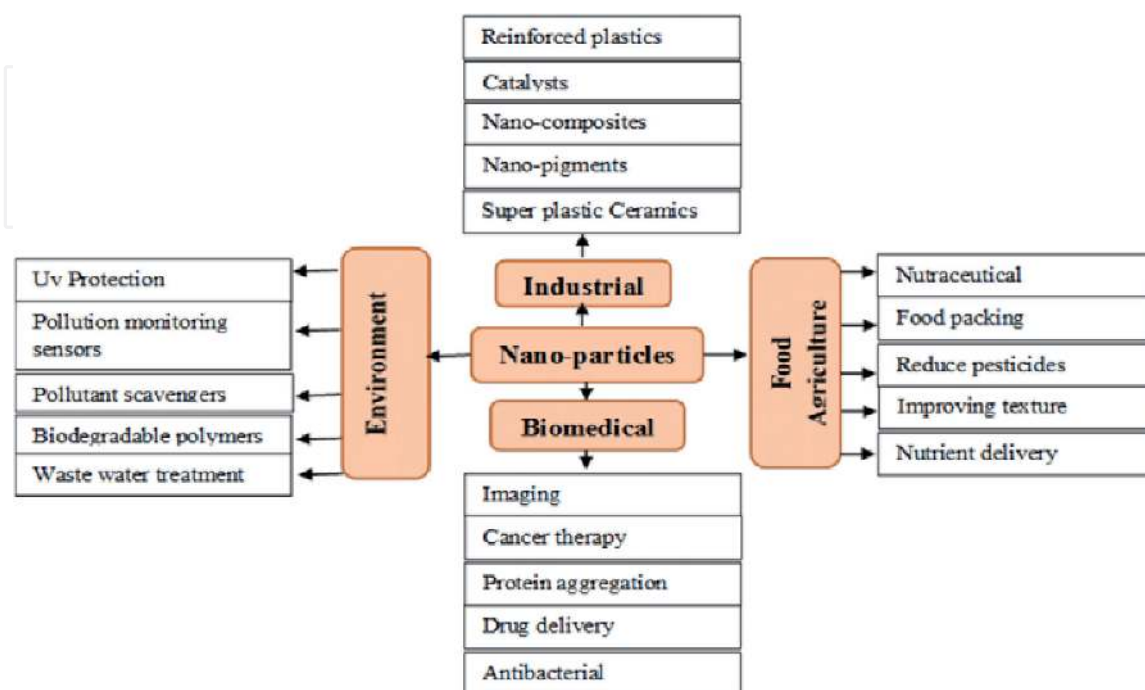

Figure 2.
Applications of nano-particles in different fields [20].

The main sources of water contamination are agricultural, industrial and domestic effluents. Industries can help on the one hand in the development of economy, whereas, on the other hand, they are mainly responsible for various environmental issues i.e. water, air and soil pollution. Drinking water containing agricultural and industrial effluents cause different diseases such as, cancer, eye irritation, dermatitis, cell damage and dysfunction of kidney, respiratory and reproductive system even in a very insignificant quantity. Hence, treatment methods for drinking as well as wastewater are one of the most important requirements for emerging and growing health and economy. Various techniques have been used for decontamination of pollutants from industrial wastewater, including reverse-osmosis, ion-exchange, chemical oxidation, flocculation or coagulation and precipitation. Each technique has its individual disadvantages as they are energy dependent, economically as well as technically not sound and achievable. Literature exhibited that from all these treatment techniques, adsorption is one of the most effective technique for water decontamination. Adsorption is simple, adaptable, highly potential, efficient and recyclable technique [22, 23]. A range of effective, low-cost and environment friendly nano-materials with outstanding properties have been developed for prospective applications in decontamination of industrial effluents, surface, ground and drinking water. Literature also revealed that nano-particles behave as an ideal adsorbent as they are environmentally benign, selective, efficient, recyclable, high surface area and maximum adsorption capacity even at a very low concentration [24–26].

The recent progress related to the different aspects of adsorption using nano-particles have described in several reviews and book chapters. This chapter focuses on the various techniques used for preparation of nano-particles and their applications in the field of adsorption.

2. Preparation of nano-particles

The nano-particles can be prepared by various processes divided into i.e. bottom up and top down techniques. Bottom up methods include the reduction of material components up to the atomic level and then with further self-assembly lead to the formation of nano-particles. However, during self-assembly, the physical forces functioning at nano-scale are used to connect basic units into macro structures. Pyrolysis, bio-synthesis, sole gel, spinning and chemical vapor deposition are most extensively used methods fall in this approach [6, 27]. Whereas, top down techniques including sputtering, laser ablation, nano-lithography, mechanical milling and thermal decomposition, starting with a pattern produced on a higher scale, then compacted to nanoscale. Both of these techniques are contradictory and schematically represented in **Figure 3**.

2.1 Bottom up technique

2.1.1 Sol gel

The sol is a colloid where the aggregates of fine particle are distributed in liquid phase. They are larger in size ranging from 1 nm to 1 µm than nano-particles. Whereas, solid macromolecules immersed in a solvent, called as gel. Sol gel is one of the simplest and, most commonly used method for the synthesis of nano-particles. It is a chemical method which comprises of a solution working as a precursor for an assimilated system of distinct particles. In this method, metal oxides, metal chlorides and alkoxysilanes (typically tetramethoxy and ethoxysilanes) are most
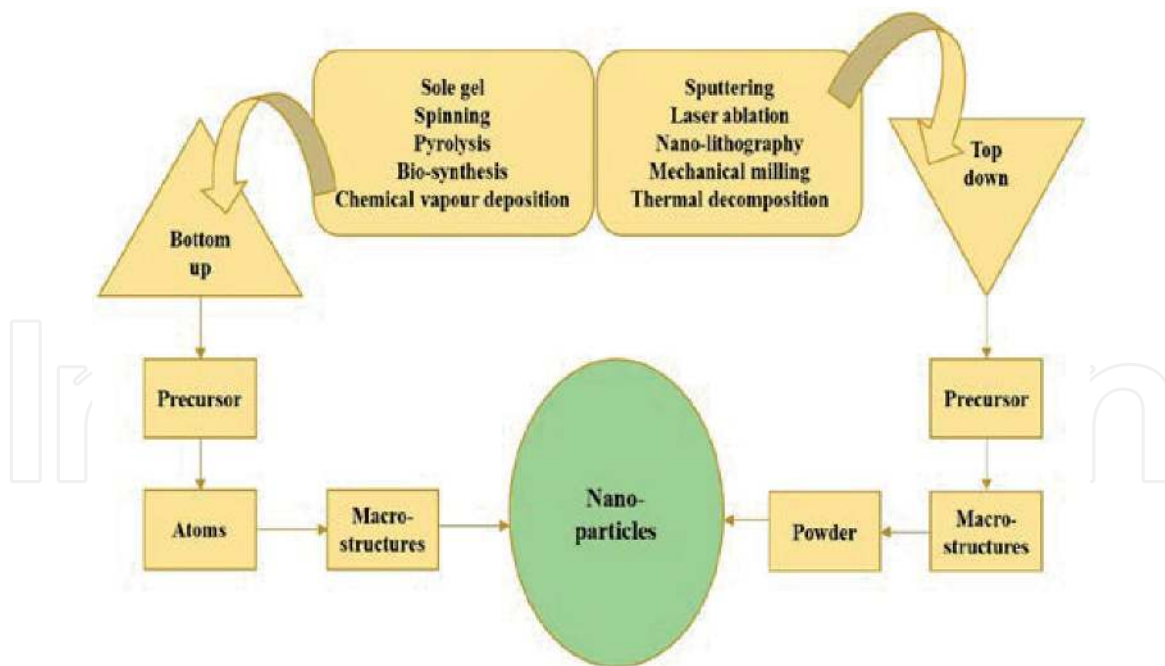
Figure 3.
Schematic representation of bottom up and top down techniques.

commonly used as precursors. The precursor is mixed by means of mixing, quivering sonicating or stirring and is then spread in second liquid which form a solid–liquid phase. Catalyst is commonly used to start the reaction and to control the pH of the system. Sedimentation, filtration and centrifugation are the typical methods used for phase separation to get nano-particles and then the sample is dried to remove moistness. The main advantages of this process are to attain uniform nano-structures even at a very low temperature, having controlled chemical composition and purity [6, 27–30]. This process is not easily scalable having different drying steps involved as well as it is difficult to control synthesis during this process.

2.1.2 Chemical-vapor deposition (CVD)

In this method of preparation, substrate is coated with a thin film of gaseous reactants. The gas molecules are combined at ambient temperature in a reaction chamber to carry out deposition. Upon heating substrate comes in vicinity of combined gas where a chemical reaction occurs and a thin film is formed on the surface of substrate. This thin film can be recovered and reused for different applications. The basic influencing factor in this method is the temperature of the substrate. The nanoparticles achieved through this method are highly pure, uniform in size, strong and have high mechanical stability. The disadvantages of CVD include the use of special equipment as well as the high toxicity of the gaseous by-products [8, 31, 32].

2.1.3 Biosynthesis

Biosynthesis is one of the inexpensive, green, safe, decomposable and environment friendly methods used for the synthesis of nano-particles. In this method bacterium, fungi and plant extracts are used in conjunction with precursor for bio-reduction and capping functions rather than conventional chemicals. This method has its distinctive and enriched properties that find its approaches in medical applications [33, 34].

2.1.4 Pyrolysis

Pyrolysis is the method used in industries to prepare nan-particles on large scale. In this method, precursor used can be liquid or vapor. A furnace is used in order to burn the precursor. Precursor is added to the furnace through a small opening where flame is applied to burn it. [13]. Nano-particles are collected by the gases produced as by product. Pyrolysis is an effective method for nano-particle preparation due to its simplicity, high product yield and sensitiveness [35, 36].

2.2 Top down techniques

2.2.1 Mechanical milling

One of the most widely used top down techniques to produce nano-particles is mechanical milling. In this method various elements are milled under an inert atmosphere and during this process particles are milled and post annealed. The influencing factor in this method is plastic distortion which end up with particle size, breakage that ends up in particle size, and cold-soldering that ends up to increased particle size [37–39].

2.2.2 Nano-lithography

Nanolithography is the investigation of manufacturing nano-scale structures of one dimension at least, with size ranging from 1 to 100 nm. There are different nano-lithographic forms, for example optical, electron-pillar, multiphoton, nanoimprint and filtering test lithography. Mostly lithography is the way towards printing a required shape or structure of a light sensitive material, which specifically evacuates a bit of material to make the ideal shape and structure. The primary advantage of nanolithography is to create a bunch from a solitary nano-particle with desired shape and size [40–42]. A sophisticated equipment is required in this method which is cost effective.

2.2.3 Laser-ablation

Laser-ablation is a typical method for the preparation of nano-particles from various solvents in solution. A metal immersed in a liquid solution is irradiated by the laser beam, resulting in the formation of plasma crest that yields into nano-particles. In this process, a chemical reduction of metals occurs to produce inorganic (metal based) nano-particles. As laser ablation gives a steady synthesis of nano-particles in natural solvents and water that does not require any balancing agent or synthetic substance. It is a 'green' process and its setup is shown in **Figure 4** [43–45].

2.2.4 Thermal decomposition

In this method heat is applied to decompose the chemical bonds of the compound. It is an endothermic chemical process where the nano-particles are synthesized by rotting a metal at a precise temperature called as decomposition temperature. As a result of this decomposition secondary products are also produced. This method is useful for the preparation of metal oxide and carbon based nano-particles [46, 47].
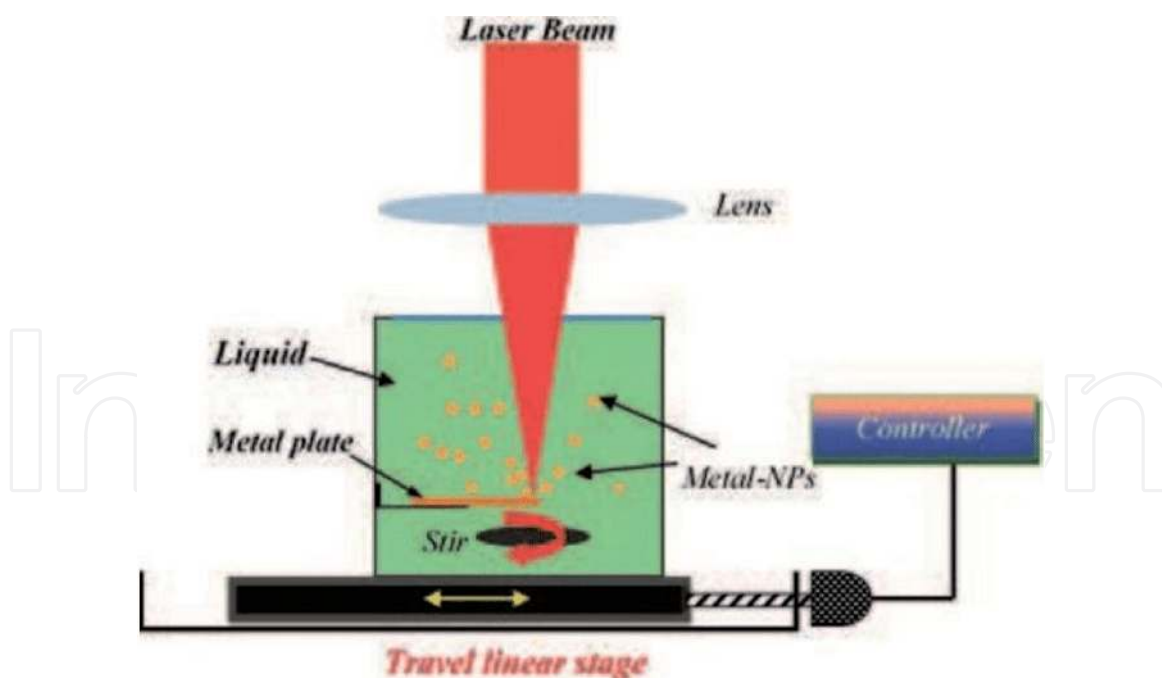
Figure 4.
Laser ablation setup [43].

3. Applications of nano-particles as adsorbent

Now a days, one of the foremost problems that is facing by the world is accessibility of clean drinking water. Demand for fresh and clean water is increasing day by day due to increasing population. In developing and industrialized countries, clean water deficiency is intensified by human as well as by the industrial effluents (metals and dyes). These effluents are directly discharged into water bodies and contaminate them. As described in introduction part of this chapter, sorption is declared to be one of the best and suitable methods for wastewater treatment [48–50].

The sorption method is a surface phenomenon during which sorbate is gathered on the sorbent surface. When adsorbate molecules from solution come to the vicinity of adsorbent surface, then some of the molecules adsorb onto the sorbent surface by intermolecular forces of attraction between surfaces of adsorbent and adsorbate molecules. The particular nature of interaction can be determined by the type of species concerned. However, the sorption method is usually classified as physi-sorption where the sorbate bound on the sorbent surface through valence or electrostatic bonding and chemi-sorption where molecule attached through chemical bonding [51–54].

Nano-particles have a high specific surface area, sorption active sites, solubility, efficiency and fractal dimension, short intra-particle diffusion distance, well defined chemical composition, small particle size and tunable pore size as compare to the their bulk counterparts that are responsible for their valuable features for effective sorption especially chemical activity and fine grain size. The high surface area and high sorption active site in nano-particles are due to high surface-energy and size dependent surface structure at nano-scale. The nano-particles have the highest efficiency towards sorption of organic and inorganic pollutants and their selectivity towards contaminants can be increased by functionalizing the surface of nano-particles. Iron oxide, titanium dioxide, manganese dioxide, silica nanoparticles, alumina, zinc oxide, dicalcium phosphate, copper, silver, maghemite, gold nano-particles, etc. are discovered as cheap, efficient, easy to synthesize and

environment friendly sorbents for the removal of pollutants. Among nano-particles (metal oxides), the magnetic nano-particles have acquired a substantial importance due to their interesting magnetic properties i.e. super para-magnetism, strong reaction even at minor applied magnetic field [6, 12, 55, 56].

Moreover, a recent improvement on carboniferous and siliceous nano-materials enclosed nano-sheets, nano-tubes and nano-particles of carbon and silicon declared as efficient adsorbents for sorption of metals and dyes from wastewater. Some oxides and carbon based nanomaterials are discussed below [55–57].

3.1 Iron nano-particles

Iron based nano-particles are most commonly used adsorbents for the removal of toxic materials from aqueous solutions. These nano-particles are declared as most efficient, cost effective and ecofriendly sorbent with less chance for the production of secondary contaminants. The adsorption process by iron oxide nano-particles is affected by pH, temperature, adsorbent dosage and equilibrium time. Modification of these materials increased their surface properties for the removal of metals, i.e. cadmium(II), lead(II), copper(II), chromium(II), nickel(II), arsenic(III) and anionic and cationic dyes [58–60].

3.2 Manganese oxide nano-particles

Manganese oxide nano-particles have a high specific surface area which makes them effective adsorbent for the removal of heavy metals i.e. arsenic(III), lead(II), cadmium(II) and ionic dyes. Manganese oxide nano-particles can also be modified into hydrous manganese oxide, nano-porous and nano-tunnel manganese oxide to improve their surface area and porosity for excellent adsorption [61, 62].

3.3 Zinc oxide

These are porous micro nano-structure with high Brunauer-Emmett-Teller (BET) surface area. Most widely used nano-sorbents of zinc oxide are nano-assembled, nano-sheets, nano-rods, nano-plates and micro-spheres for competent removal of dyes and inorganic pollutants from aqueous phase. Whereas, micro-porous nano assemblies of zinc oxide display maximum potential for the removal of lead(II), arsenic(III) and mercury(II) because of their electro-positive nature [63–65].

3.4 Magnesium oxide

The sorption capacity of the magnesium oxide nano-particles is much greater than its bulk counterpart. Their micro-spheres are innovative structure, with increased sorption capacity for the sorption of heavy metals. Various modification of magnesium oxide nano-particles i.e. nano-rods, nano-tubes, nano-wires has been reported as improved sorption affinity towards metals and organic effluents [66–68].

3.5 Carbon nano-tubes

They are the most widely used material for the sorption of heavy metals as well as organic dyes from aqueous media. Though, they have meager dispersal capacity, very small size of particles and separation complications are some difficulties for using carbon nano-tubes as sorbents. Whereas, these difficulties can be overawed

by modifying carbon nano-tubes into multi walled carbon nano-tubes. Literature revealed that the multi walled nano tubes and alumina supported carbon nano-tubes, more competently removed metals such as Mn(II), Cu(II) and Pb(II) more efficiently as compare to unmodified material [22, 69, 70].

4. Conclusion

Nano-technology is refining our everyday life by increasing the proficiency and purity of many substances. As described in this chapter, there are different techniques for the synthesis of nano-particles, but laser ablation chemical vapor deposition, nano-lithography, biosynthesis, mechanical milling, and sol–gel are the most suitable techniques because they are less time consuming methods. Nano-particles with inimitable chemical and physical characteristics, have a remarkable prospective for the adsorption of contaminant but still their applications for wastewater treatment are inadequate. However, nanoparticles have pronounced future due to their proficiency and environmentally benign property.

Acknowledgements

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