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CHAPTER

4

Methods for the synthesis of nano-biosorbents for the contaminant removal

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4.1 Introduction

The essential thing to all living things on earth's surface is water, also called a universal solvent due to its versatility used in every field. Water serves a crucial role in the human body which involve in many metabolism processes which are taking place in our body (Sharma and Bhattacharya, 2016). Though about 70% of the area on earth is covered by water, but drinking water is not enough for living things, another problem is nothing but the contamination of drinking water sources. Human as well as natural activity responsible for the discharge of unlike things called as contaminants, there are several types of contaminant reported by researchers like a pathogenic organism, plant nutrients, oxygen demanding waste, sediments, synthetic organic chemicals, micro-plastics, oil, radioactive substances, inorganic chemicals, and heavy metals. Several methods were used for the purification of water by removing the contaminants present in water mainly classified into chemical and physical approaches, such

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as chemical oxidation, filtration, precipitation, biological treatments, and coagulations (Tran et al., 2019). The degree of purification of water and operational cost is the main factor of discussion, some methods are cost-effective and can be used at high concentration but always question mark about the accuracy, precipitation method is much effective related to the high concentration in which contaminants allow to settle down at the bottom in the form of precipitate and filter off it. The biological contaminants are biodegradable, by using this principle biological waste can be removed from water sources (Kanamarlapudi et al., 2018). Nowadays, nanotechnology is the best tool to obtain multifunctional nano-material having fantastic applications in biomedicine, pharmaceuticals, industries, electronics, electrical devices, and environment, etc. The nan-omaterial's prepared using Top-Down and Bottom-up concepts, several methods were reported for the synthesis of nan-omaterial such as physical, chemical, and biological methods. Recently, nano-material prepared via biological or green methods is the first choice of the researcher because of their merits over known physical and chemical approaches, as the biological method is cost-effective, eco-friendly, quick, and one pot. Hence, biologically synthesized nano-range material having different morphology like nano-wire, nano-tube, nano-flower, nano-spheres, and nano-clusters, used as nano-biosorbent in the treatment of wastewater (Dabhane et al., 2020).

In present days, nano-biosorbent plays a key role in the purification of contaminated water with high efficiency and at low cost, nano-biosorbent can be synthesized using agriculture waste, biomass, biological waste, bacteria, fungus, algae, metal nanoparticles, and characterization was done with help of spectroscopic and microscopic techniques such as UV-DRS, FT-IR, Photo Luminescence, BET, FE-SEM, HR-TEM, EDX, XRD, DLS, AFM which gives information about bandgap, functional group, porosity, size, morphology, elemental composition, crystal plane, etc. Numbers of methods were reported by several researchers by which nano-biosorbent material was synthesized, Chemical method, Physical method, Microwave-assisted, Ball mill, Precipitation method, Sol-Gel method, Green Synthesis, Hammer mill, biological synthesis and used for the efficient removal of pollutants like synthetic dyes, hazardous chemicals, heavy metals, biological waste from water sources (Dabhane et al., 2021; Crini et al., 2019; El-Sayed and El-Sayed, 2014; Mahamadi, 2019; Huang et al., 2020; Tara et al., 2019; Srivastava et al., 2020; Beni and Esmaeili, 2019; Ali, 2012; Amin et al., 2014).

4.2 Types of nano-biosorbents

The synthesis or preparation of nano-biosorbent design is based on the concept of green chemistry, the classification of nano-biosorbent depends on the precursor from which it obtains. The different types of precursors were used for the synthesis of nano-biosorbent as shown below.

1. Agriculture waste.
2. Forest waste.
3. Extract of different parts of plant.
4. Biological precursor.
5. Chemical precursor.
6. Other

The various agriculture waste such as sawdust, sugarcane, DSP, peels of different fruits was used as reducing, and stabilizing agents for the fabrication of nano-biosorbent, similarly forest waste like Chestnut bur used for the same purpose. In most of the research articles, the synthesis of nano-biosorbent was reported using extract and waste of several parts of plants like flowers, roots, latex, leaves, etc. Along with that, biological precursors such as algae, fungus, bacteria were also reported for the biological synthesis of biosorbent which effectively remove the contaminants from water sources. The effective chemical synthesis of nano-biosorbent using a variety of chemical precursors such as chitosan, dextrin, etc., reported by some researchers, the nano-biosorbent synthesized by another precursor also like oil obtained from different natural sources.

4.3 Methods for the synthesis of nano-biosorbents and their applications

Several attempts were made for the synthesis of nano-biosorbent using different precursors from nature, the activity of nano-biosorbent depends on their morphology as well active sites. Table 4.1 summarized the reported synthesis of nano-biosorbent from a variety of sources.

Agriculture waste is the chief and best precursor which is easily available in nature for the synthesis of nano-biosorbent. It shows excellent adsorption activity towards contaminates present in water sources. Garima Mahajan and Dhiraj Sud reported the synthesis of nano carbonized biomass from *Dalbergia sissoo* pods (DSP) which contain lignocellulosic, nitrogenous agricultural waste biomass by calcination method. Initially, the biomass was collected and wash well to remove organic impurity from it, afterword it was dry at 120°C in the oven for 24 h, dried biomass was crush into the nano-size powder and further calcinated at 800° C in furness to obtained DSPC. The morphology of synthesized DSPC was investigated using spectroscopic and microscopic techniques like FTIR, XRD, and SEM. FTIR spectra confirm the presence of different anionic and cationic functional groups which coming from lignin, XRD techniques show the porous spacing and porous size of synthesized DSPC, and finally, SEM image confirms the nano-size of DSPC with regular morphology.

The author, study the application of DSPC for the remediation of Cd (II), Ni (II), and Pb (II), metal ions from water solutions. The batch experiment was conducted to carry out the removal of the above metal ions in aqua media, due to the presence of anionic sites in DSPC it effectively interacted with metal ions and self-undergo oxidation by doing the reduction of the metal. The interaction and reaction between metal and DSPC in aqueous media were confirmed by FTIR techniques which shows the conversion of anionic sites of DSPC into their oxidized form. The author also studied the effects of several parameters on the extraction of metal ions such as pH, adsorption dose, initial metal ion concentration, contact time, and stirring speed. pH is the main factor that influences the rate of adsorption or removal of metal, the present experiment was carried out at pH 1–7 and it was found that maximum adsorptions of Ni (II), Cd (II), and Pb (II) was 95%, 95%, and 98%, respectively. Another main factor is the adsorbent dose which is directly proportional to the rate of adsorption, the rate of adsorption also relies upon the initial concentration of a metal ion in an aqueous solution, when it is presented in higher concentration, the adsorbent dose must be increased for better result,

TABLE 4.1 Summarized the reported synthesis of nano-biosorbent from a variety of sources.

Sr. No.	Nano-biosorbent	Method	Precursor	Condition	Characterization	Product description	Contaminant removal	References
1	Carbonized waste biomass	Calcination	Delbergia sisso pods (DSP)	Calcination at 800 oC	FT-IR, XRD, SEM	Nano-Flaks	Cd (II), Pb (II) and Ni (II) metal ions	Mahajan and Sud (2014)
2	Chitosan-sunflower (CS), chitosan-sunflower-nanoiron (CSN)	Described procedure by Nadaroğlu et al., and Turgut.	chitosan and sunflower waste, an agro-industrial waste and modified using iron nanoparticles	–	FTIR, SEM-EDAX, TEM, XRD, and	Nano-sheet	Methylene Blue dye	Turgut et al. (2020)
3	Sawdust	–	Sawdust and sodium dodecyl sulfate	Dried at of about 60C for 5 h temperature	SEM	Heterogeneous and porous structure	Removal of methylene blue	Ansari et al. (2012)
4	Nanomodified sugarcane bagasse biosorbent	–	sugarcane bagasse (SB) and ferromagnetic nanoparticles (Fe3O4)	Dried at 50°C for 6 h where it was later milled in a rotor mill and stored	FTIR, XRD, and SEM/EDS	Flaks	Cu(II) removal	Carvalho et al. (2020)
5	FNPSA and FNPSOPR	Precipitation	Fe3O4, sodium alginate matrix and saponified orange peel residue	Ultrasound	FTIR, XRD, SEM-EDS DLS and VSM	Spherical	Fluoride from water	Christina and Viswanathan (2015)
6	Sugar beet pulp	Hammer mill	Sugar beet pulp	Air-dried at 40°C	–	sizes of 0.25–0.50 mm	Removal of Pb2+, Cu2+, Zn2+, Cd2+, and Ni2+ cations	Reddad et al. (2002)
7	GQDOs-Ba nano-biosorbent	A bottom-up method	Rice husk	Treated with N2 atmosphere at 700oC for 2 h	FT-IR, SEM, TEM,TGA, and XRD.	Regular aglomerzation	Removal of lead (II) and lanthanum (III)	Sharma et al. (2018)

8	Algal	–	*Ulva fasciata* and Sargassum dentifolium	Dried at 60 oC for 24H.	FT-IR, TEM, ZETA SIZER	0.220 and 0.309 µm	Cu^{2+}	El-Wakeel et al. (2019)
9	Brown algae and chitosan/ PVA nano-fiber membrane	planetary ball mi;;	Sargassum glaucescens	Planetary ball mill (Fara Pajouhesh Isfahan FP2) at 600 rpm for 1 h	SEM	Nanowire and spherical 30–300 nm	Removal of Nickel	Ozdemir et al. (2020)
10	Amberlite XAD-4 loaded with Anoxybacillus kestanboliensis	Biological Method	Anoxybacillus kestanboliensis, XAD-4.	120 rpm and 55°C for 1 day on a rotary shaker	SEM and FT-IR.	Aglomerization	Preconcentrations of Co(II) and Hg(II) in	Leung et al. (2016)
11	Amyloid fibrils	Biological Method	Hen lysozyme	Stirred for 4 h at 50°C and then centrifuged at 14,000 rpm for 4 min	TEM, PL and ZP	Irregular	Removal of dye pollutants	Arshadi et al. (2015)
12	Ostrich bone waste (OBW)	–	Ostrich bone waste (OBW)	Dried oven for 24 h at 70C,	FTIR, XRD, TEM	Aggregation	Phosphate removal	Jain et al. (2017)
13	Nano-cellulose	Precipitation	Aniline and persulfate	Stirred for 26H.	FT-IR, XRD, SEM	rod-like in shape, ∼60 nm in size	Chromium	Pipíška et al. (2020)
14	Magnetically functionalized moss biomass	Microwave	*Rhytidiadelphus squarrosus*	Dried at 60°C for 24 h	(FTIR), XRD, SEM, and EDX	moss leaves	Co^{2+} Ions and thioflavin T	Zare et al. (2018)

the analysis of adsorption studies done based on Freundlich isotherm and Langmuir isotherm models (Mahajan and Sud, 2014).

Synthetic dye is the major constituent of water pollution, different types of dyes were used for several purposes in industrial sectors. The unused dye was present in wastewater dump into water bodies causes many side effects on living things, such type of problem-solving using the nano-biosorbent. The author Esra Turgut and co-workers reported the synthesis of two nano-biosorbent chitosan-sunflower (CS) and chitosan-sunflower-nanoiron (CSN) from chitosan, sunflower waste and modified using iron nanoparticles. The author used reported methods (Gungor et al., 2015; Karaduman et al., 2017; Nadaroglu et al., 2015) for the green synthesis of iron nanoparticles using plant extract of *ficus carica* and $FeCl_2$-$FeCl_3$, the chitosan-sunflower (CS) and chitosan-sunflower-nanoiron (CSN) was prepared using chitosan, pre-dried powder of sunflower waste and green synthesized iron nanoparticles via the reported procedure of Turgut et al. (2020). The synthesized nano-biosorbents was a characterized using FT-IR, UV–Vis, SEM, and TEM technique which shows the low crystal structure and a highly porous surface. The FT-IR spectra of CS and CSN explain the interaction between chitosan-sunflower waste and chitosan-sunflower-nanoiron, respectively, XRD peaks of chitosan-sunflower (CS) at 19.20°, 27.49°, and 32.43° gives the CS structure peaks while the structure of the NPs connected to the peaks of the CS structure in addition to 32.28°, 32.17°, and 37.82° in the presence of peaks of Fe_3O_4 NPs were determined and confirm. The SEM, and TEM images of CS and CSN explain the nearly equal distribution of Fe NPs over the surface of CS which appears as cluster and aggregation due to the magnetic nature of Fe_3O_4 NPs. The author successively used synthesized CS and CSN for the removal of synthetic dyes; he chose Methylene Blue as a reference dye, and analysis was explored according to Langmuir and Freundlich's adsorption isotherm model. The various parameters which affect the rate of adsorption like biosorbent dosage, treating agent, contact time, initial dye concentration, solution pH, and temperature, the interaction during the reaction of methylene blue dye and biosorbent was also studied by UV–Vis, FT-IR, whereas the change in morphology of biosorbent after the adsorption of methylene blue was tested using SEM, and TEM image. It was found that the following optimum condition (pH 5, 30°C, and 60 min) give 80% of CS and 88% of CSN removed MB from aqueous media (Turgut et al., 2020).

Similarly, the author R. Ansari et al. also reported the synthesis of nano-biosorbent utilizing sawdust assisted with sodium dodecyl sulfate (SDS/SD) for the degradation of methylene blue dye. Sawdust is an agricultural waste that is easily available used as the precursor for the synthesis of SDS/SD biosorbent, initially sawdust was washed well and dried at 60°C for 5 h in the oven, then dried sawdust 35 g treat with 500 mL SDS and stirred at ambient temperature for 12 h then dried at 50°C to obtain biosorbent. The morphology of nanostructured SDS/SD was confirmed by SEM technique, SEM image showed the porous and heterogeneous structure of sawdust. Adsorption studies were carried out via two different methods like batch and fixed-bed column systems, and Thomas and the bed-depth service time model (BDST) were employed for study of sorption data, and determining of sorption ability. The analysis and result of biosorption of methylene blue were obtained using Langmuir and Freundlich isotherm equations, the effect of several factors such as initial dye concentration, pH, flow rate, and bed depth on the sorption of MB dye have been reported. The result of biosorption of methylene blue over surfactant-modified sawdust is much efficient than the untreated SD biosorbent (Ansari et al., 2012).

Sugarcane is the main agricultural product in Maharashtra state which is used for the production of sugar whereas sugarcane bagasse is used for the production of alcohol. The sugarcane bagasse was found to be a good biosorbent for the extraction of the pollutants in water, the author Juliana Tosta Theodoro Carvalho and co-workers synthesized nano-modified sugarcane bagasse biosorbent for the extraction of Cu (II) ions in the aqueous medium. The nano-modified sugarcane bagasse was prepared using sugarcane bagasse and ferromagnetic Fe_3O_4 nanoparticles by coprecipitation technique (Labuto et al., 2018; Mascolo et al., 2013; Panneerselvam et al., 2011) in which synthesized Fe3O4 nanoparticles mixed with SB at 80°C for 30 min. The synthesized SB/ Fe_3O_4 and MSB/ Fe_3O_4 were explored by FT-IR, XRD, SEM/EDS techniques, FT-IR spectra explain the presence of different functional groups like hydroxyl (O-H), hydrocarbons (C-H), carbonyl (C=O), C-O, ether (C-O-C), and Fe-O, the above functional group also enlighten the binding sites present in biosorbent. The structural information about the SB/Fe_3O_4 and MSB/Fe_3O_4 obtained from the XRD spectrum confirms the cubic structure, the SEM image also confirms the unequal morphology of SB/Fe_3O_4 and MSB/Fe_3O_4 biosorbent. To determine the charge on the biosorbent surface, it was treated with anionic and cationic such as amaranth red (AM) and methylene blue (MB) dyes. The cationic dye like methylene blue (MB) shows excellent interaction with the anionic surface of biosorbent and vice versa, then after kinetic study for the removal of Cu (II) was investigated at 25°C, pH 6.2 condition and different concentration of Cu (II) ion (Carvalho et al., 2020).

The synthesis of Fe_3O_4 nanoparticles (NPs) and saponified orange peel residue immobilized in sodium alginate matrix (FNPSOPR) as well as Fe_3O_4 NPs immobilized in sodium alginate matrix (FNPSA) as sorbents for fluoride extraction from polluted water reported by author Evangeline Christina, and Pragasam Viswanathan. Initially, Fe_3O_4 NPs were prepared by co-precipitation technique adapted by Thapa et al., further, the synthesized Fe_3O_4 NPs were ultrasonicated with 1.5% sodium alginate solution to obtained magnetic alginate nanoparticles, and to enhance the activity of the catalyst, the surface modification was done in three steps by cross-linking with glutaraldehyde for 20 h, carboxylation with chloroacetic acid for 20 h, and final step is loading with $FeCl_3$ for 24 h. The synthesized surface-modified nanoparticles were analyzed with help of FTIR, SEM-EDX, XRD, DLS, and VSM techniques, the size of Fe_3O_4 NPs was confirmed by SEM, EDX, and DLS techniques, the SEM image confirmed the agglomeration with a mean size of 21 nm was as the hydrodynamic size of the particles was found to be approximately 140 nm by DLS techniques. The XRD data confirmed the cubic morphology with high purity of Fe_3O_4 nanoparticles, also the VSM study shows that synthesized Fe3O4 nanoparticles are superparamagnetic with a coercivity of 70.541 Oe.

The adsorption capacity of synthesized nan-omaterial was found by batch methods with the help of sorption isotherm and reaction kinetics study, for this purpose the uniform bed of FNPSA and FNPSOPR was prepared and sorption study of chlorine ions was studied. The maximum sorption ability of FNPSA and FNPSOPR were found to be 58.24 and 80.33 $mg \cdot g^{-1}$ respectively. Five sorption-desorption cycles exhibited 100%, 97.56%, 94.53%, 83.21%, and 76.53% of regeneration of FNPSOPR (Christina and Viswanathan, 2015). Agriculture waste also has a marvelous capacity to adsorbed heavy metal present in water sources, sugar beet pulp is one of the most popular examples, which is used as bioadsorbent, the author Zacaria Reddad et al. reported the adsorption of several metal ions using cost-effective bioadsorbent. In the present study, the commercially available sugar beet pulp was crushed by hammer mill and get milled particle having size 0.25–0.50 mm which was used for further process. The obtained particles were washed well with help of distilled water to remove the impurity as well different ions, further, they are dried at 40°C and used for adsorption study of metals. The author tested the kinetic study of beet pulp particles by adsorption test, 2 g of beet pulp was dissolved in 800 mL of distilled water stir on a magnetic stirrer and initial concentration of metal chloride 0.0004 M allow to adsorbed on it, the extent of adsorption was recorded by atomic absorption spectrometer. Similarly, the sorption study of nano-biosorbent was carried out, in the same manner, using Langmuir and Freundlich adsorption isotherm (Reddad et al., 2002).

Methylene blue is an important dye used in different industries for several purposes, they also have some side effects on aquatic and human life, hence the need to remove it from water bodies. Several methods were reported and adapted for the removal of methylene blue dye, but one of the best and effective ways is the adsorption of methylene blue dye on the matter. The author Swati Sharma and co-workers reported the synthesis of nano-bioadsorbent using iron oxide nanoparticles and biomass. In the present paper, the author prepares nano-bioadsorbents by the combination of previously synthesized iron oxide NPs and Agrobacterium fabrum biomass in sodium alginate solution over calcium chloride. The morphological study of synthesized nano-bioadsorbent was done with help of the FESEM technique, the elemental composition was determined by EDX technique, and the involvement of functional group was understood by FTIR technique. The batch-wise adsorption study was carried out by determining the optical density of the resultant solution at various parameters like initial dye concentration, temperature, contact time, and pH. In the present study, 1 g of synthesized nano-bioadsorbent was added to different methylene blue concentrations of 25, 50, 100, and 200 mg/L with constant stirring at 160 rpm and 50°C. The amount of adsorption was studied by calculating the optical density of the resultant solution (Sharma et al., 2018).

The biological microorganism also plays the role of nano-bioadsorbent, the author Shaimaa T. El-Wakeel and et al. reported the algal bioadsorbent for the extraction of heavy metal from water medium. In the present study, the author collected the two macro-algal biomasses *Ulva fasciata* and Sargassum dentifolium, washed well, dried, and ground them in a ball mill to micro-size powder. The micro-size powder was characterized using FTIR, TEM, and DLS techniques, after the morphological analysis of nanobioadsorbent was used for the removal of Cu (II) ion from aqueous medium. The adsorption efficiency of nano-bioadsorbent was determined by batch experiment, where the 1 g adsorbent was added in 1lit solution of Cu (II) ion, the efficiency of adsorption was investigated by considering the vital parameter like pH, initial due concentration, temperature, adsorbent dose, and contact time. Finally, the resultant concentration of a metal ion in solution was investigated by inductively coupled plasma-Optical Emission spectroscopy (ICP-OES), and analysis were done with help of Langmuir, Freundlich, and Dubinin–Radush kevich models (Mahamadi, 2019). Similarly, the author A. Esmaeili and A. Aghababai Beni reported the use of brown algae and chitosan/PVA nano-fiber for the removal of nickel.

The preparation of chitosan/VPA nano-fiber and biomass using brown algae was made by the previous procedure given in Esmaeili and Aghababai Beni (2015a, b), the synthesized nano-bioadsorbent was subjected to the characterization using different spectroscopic, and microscopic techniques, FTIR technique confirms the presence functional groups imparted by chitosan/PVA nano-fiber and *S. glaucescens*. The morphology and surface area of nano-bioadsorbent was investigated with help of SEM and BET techniques. The continuous system was formed by the combination of two reactors which contain three parts, two membranes, and a reactor. In the reactor, the nano-bioadsorbent containing ligand sides bind with the nickel-metal ions and adsorb on its surface, where the biosorption was nickel ion on the surface of adsorbent explained by the mechanisms of electrostatics, ion exchange, complexation, covalent force adsorption, and micro-sized deposition (El-Wakeel et al., 2019).

A novel nano-bioadsorbent composed of Amberlite XAD-4 and Anoxybacillus kestanboliensis was reported by author Sadin Ozdemir and et al., the Anoxybacillus kestanboliensis was isolated from Turkey, were as the morphological and biological analysis was done. The fabrication of dried dead Anoxybacillus kestanboliensis and loaded biomass were prepared according to Ozademir et al. with some modifications. After the preparation of the nano-biosorbent, characterization was done with help of techniques like FTIR, SEM, and EDX mapping. The author also studied the effect of flow rate, amounts of nano-bioadsorbent and resin, influence of eluent type, concentration and volume, the influence of matrix ions on retention of Co(II) and Hg(II), the effect of sample volume and determination of enrichment factor on the efficiency adsorption. Finally, the author reported the maximum biosorption capacity of novel biosorbent as 24.3 and 27.8 mg g^{-1} respectively (Ozdemir et al., 2020). The author reported the synthesis and use of Amyloid Fibrils as a rapid and efficient nano-biosorbent for the removal of dye. In the present study, the Amyloid fibril of hen lysozyme, which is an excellent source of protein nano-fiber considered as nano-biosorbent, this protein nano-fiber was synthesized in one step using the green route. The zeta-potential analysis confirms the positive/negative charges on nano-biosorbent and the hydrophobic region as well, the presence of active charge sides in nano-biosorbent make them more reactive and can form interaction with organic dye and it explains the process of biosorption of organic dye present in aqueous media. The author reported the maximum sorption capacity of biosorbent is 60% in 15 min (Leung and Lo, 2016).

A new nano-biosorbent was reported for the removal of phosphate in synthetic and real water sample synthesized by the combination of Ostrich bone waste and nanoscale zero-valent iron, the author M. Arshadi et al. collected the Ostrich bone waste from the local store, washed well several times using distilled water to remove the unwanted fat and flash, then after put in the oven for 24 h at 70°C, the selected size of particles 305 μ were collected and reflux with the 0.1 M HNO_3 for 2 h to enhance the sorption efficiency towards phosphate. The nano zero-valent iron was synthesized using $FeSO_4.4H_2O$ dissolved in 4/1 (v/v) ethanol/water, then OBW-HNO3 solution was added dropwise and ultrasonicated for 25 min to result from homogeneous solution. Finally, the resultant black color nano-biosorbent was obtained by adding dropwise NaBH4 solution. The morphology of synthesized nano-biosorbent was investigated using TEM, and EDX techniques, where the surface area was known with help of BET techniques, and chemical composition was understood using chemical analysis and ICP-AES techniques. The adsorption study was carried out using batch experiments, were the 0.5 g of synthesized nano-biosorbent shaken with 50 mL solution having a concentration between 0.75 and 1000 mg/L (Arshadi et al., 2015). The author Priyanka Jain and co-workers reported the synthesis of difunctionalized-polyaniline-modified nanocellulose composite sorbent (PANINCC) has been used for the removal of trivalent and hexavalent chromium from water bodies. The polyaniline was synthesized by adding

15 mL ammonium persulfate solution into aniline-HCl solution, into that 10 g of nanocellulose was mixed at ambient temperature with constant stirring for 10 h result in nano-biosorbent. The morphology of the resultant nano-biosorbent was investigated using SEM, TEM, XRD, and confirm the rod-like structure with size 60 nm, where the elemental analysis was done with the help of EDX techniques. The sorption study of chromium chloride over synthesized nano-sorbent was done through batch experiment, in which known concentration of chromium chloride with the variable dosage of nano-sorbent, at different pH, different volume, and at different temperature stirred at 40 min, was the maximum capacity of sorbent was found to be 96% (Jain et al., 2017).

The author Martin Pipíška and co-workers reported the microwave-assisted synthesis of iron oxide nanoparticles and microparticles and they are used in the preparation of Moss biomass from *Rhytidiadelphus squarrosus* moss. The magnetical moss biomass was prepared according to the procedure of Safarik and Safarikova, in which 1 g of $FeSO_4.7H_2O$ was dissolved in 200 mL distilled water and 1 mol/L of NaOH was slowly added with constant stirring to reach the 12 pH of the solution, the resultant suspension was treated in the domestic microwave for 10 min. Finally, suspension mix with the 1 g of *R. squarrosus* biomass in water to get magnetically moss biomass after 60°C for 24 h. The surface characterization of magnetical biomass was done with the help of SEM-EDX, and XRD before and after sorption of Co^{2+} and thioflavin. The adsorption mechanism involves the active sides of nano-biosorbent, the oxygen functionality present in biomass form electrostatic interaction with Co^{2+} and thioflavin (Pipíška et al., 2020). The author reported the development of an effective nano-biosorbent based on poly-*m*-phenylenediammine grafted dextrin for the removal of Pb(II) and methylene blue dye in aqueous media. The synthesis of the present nano-biosorbent is carried out in three steps, first step involves the chemical synthesis of dextrin-g-poly-*m*-phenylenediamine (DgPmPDA), in which 1.25 g Dex was taken in 50 mL of distilled water in 100 three-naked flasks with constant stirring for 30 min at 50°C, after cooling 3.75 mL of HCl (1 M) was added to the same solution and again stir for 30 min at 0–5°C in an inert atmosphere. Then successive addition was taken place of APS solution in 30 min and mPDA solution in 2 min dropwise in an inert atmosphere, finally the resulting precipitate was with water, ammonia, and acetone to remove unreacted chemicals from solution. In the second step, the synthesis of graphene oxide was carried out according to modified hummer's procedure (Marcano et al., 2010). In this procedure the mixture of concentrated H2SO4/H3PO4 was added to a mixture of graphite flask and $KMnO_4$, the mixture was stirred for 12 h at 50°C. Finally, the reaction mixture was poured into an H_2O_2 solution, and obtained black powder was dried at 90°C for 24 h. Finally, the preparation of Dex-g-PmPDA@GO (DgPmPDA@GO) nano-biosorbent was done using the solution blending technique (Lago et al., 2016). In this procedure, both components dispersed in chloroform separately and then were mixed in an ultrasonic bath for 2 h at 25°C.

The characterization of each part of nano-biosorbent was done with help of FTIR, CHN, XRD, FESEM, AFM, and TGA techniques, which confirm the slight agglomeration present in synthesized nano-biosorbent. The adsorption study was carried out considering the effect of pH of the solution, adsorbent dosage, contact time, initial concentration of Pb(II) and methylene blue dye, and experimental data put forth in terms of Langmuir isotherm which is found to good (Zare et al., 2018).

4.4 Conclusion

The numerous techniques of synthesis of nano-biosorbents are discussed in this chapter, as well as their significant application in environmental remediation. The goal is to classify diverse raw materials and assess their uses in diverse environmental uses. The research presented here reveals minute data about the size, shape, characteristics, and uses of nano-biosorbents. For the preparation of nano-biosorbents, several biocompatible reagents and sources, as well as energy-efficient procedures, have been discussed in detail. This chapter explored many roles of nano-biosorbents as catalysts, adsorbents, and their toxicological effects. Furthermore, the synthesis of nano-biosorbents using a green approach, which is currently being developed, has been highlighted. Furthermore, the several green synthesis pathways were assessed critically in terms of their stability, size distribution, and various other aspects. In conclusion, diverse nanotechnology protocols have been investigated in this chapter, which will be useful in future research as a guide to study and use for further improvements in developing better nano-biosorbents to deal with real-world sewage and polluted solutions with the least risk of toxicological effects on the ecosystem. This chapter may be interesting to readers who want to learn more about the synthesis of nano-biosorbents and their remarkable effectiveness in diverse environmental applications.

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