Chapter 11
Phytoremediation Technology: Sustainable Solution for Cleaning Up of Recalcitrant Pollutants from Disturbed Environs

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11.1 Introduction

Water is a fundamental substance for the sustenance and reproduction of life since it is indispensable for the development of biological processes (Mongue 2004). However, we currently find that various water sources such as wells and rivers are contaminated with large amounts of recalcitrant's pollutants (Wong Argüelles 2009). The term recalcitrant applies to compounds whose presence in the environment is prolonged due to their difficult degradation. These compounds can be from simple halogenated hydrocarbons to complex polymers. The recalcitrants can persist even in effluents that have received a previous treatment, which causes the presence of these compounds in the receiving environments causing cause health and

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R. A. Bhat et al. (eds.), *Bioremediation and Biotechnology, Vol 3*,
https://doi.org/10.1007/978-3-030-46075-4_11

environmental problems (Gupta and Thakur 2017). The low concentrations of recalcitrant in the environment make its treatment and elimination difficult using conventional treatments such as activated carbon, chemical precipitation, exchange resins, and filtration (Sandhya et al. 2005).

This chapter summarizes the information obtained, by scientific sources, about plants used in the phytoremediation of organic and inorganic recalcitrant contaminants. In addition, a study we conducted with *Typha latifolia* in the removal of heavy metals is presented.

11.2 Main Recalcitrants Pollutants

The recalcitrant pollutants are a variety of compounds such as heavy metals, chlorinated phenolic compounds (CPCs), complex dyes, explosive compounds, polyaromatic hydrocarbons (PAH), polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), pharmaceutical active compounds (PhACs) (Ahmadi et al. 2017) (Fig. 11.1).

Heavy Metals Heavy metals are natural components of the Earth's crust, and some are indispensable for cell growth and biological mechanisms, however, when released into the environment, and exceed permitted limits are considered toxic. Most heavy metals come from industrial sources and are considered hazardous soil contaminants because they have chronic and acute toxicity (Abdel-Shafy and

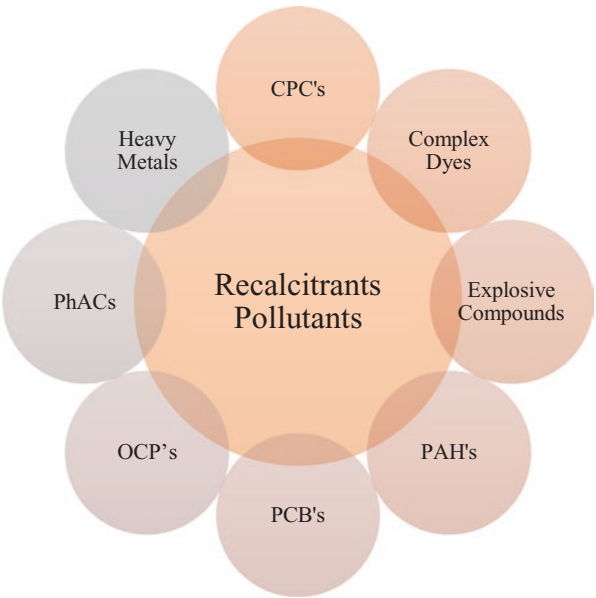

Fig. 11.1 Mains groups of recalcitrants pollutants

Mansour 2018). Heavy metals have a prolonged persistence because they do not degrade, which causes them to accumulate in living organisms causing toxic and potential adverse effects on humans and ecosystems (Khatoon et al. 2017). The major toxic heavy metals are Hg (I), Cd (II), Pb (II), Cr (IV), Fe (III), and Cu (II) (Sud et al. 2008).

Complex Dyes Most synthetic dyes are toxic and highly resistant to degradation due to their complex chemical structures. The textile industry is one of the largest generators of liquid effluent contaminants due to the high amounts of water used in dyeing processes. In recent years, interest in environmental control of dyes has increased due to their toxicity and carcinogenicity, as it contains carcinogenic compounds such as benzidine and other aromatic compounds (Ali 2010).

Explosive Compounds Explosives are highly stable compounds and have the tendency to bind with soil organic matter and contaminate it. Explosive materials are energy substances that, when released, present toxic hazards to the environment and biota. Around the world, soils are contaminated by such pollutants, whether due to manufacturing operations, military activities, conflicts of different levels, open burning/open detonation, ammunition discharge, etc. (Chatterjee et al. 2017).

Poly-Aromatic Hydrocarbons (PAHs) They are considered as one of the most dangerous chemical pollutants in the environment. These chemicals have carcinogenic, toxic, and mutagenic effects on the living body. In addition to their carcinogenic activity, these PAHs are recalcitrant, ubiquitous, and have bioaccumulative potential. Diesel and all hydrocarbon fuels are composed of an excessive amount of PAHs. Therefore, PAHs cause abundant pollution to ecosystems (Abdel-Shafy and Mansour 2018).

Polychlorinated Biphenyls (PCBs) They are among one of these groups of notorious contaminants. Polychlorinated biphenyls (PCBs) are members of chlorinated organic chemicals that theoretically can contain 209 different congeners. Highly chlorinated congeners are more stable and tend to have lower solubility in aqueous solution and also have high octanol water partition coefficients (K_{OW}) than low molecular weight PCBs. The high K_{OW} is partly responsible for their persistence and allows them to absorb strongly in the soil. Due to their environmental and ecological impacts, PBCs have been included in the Stockholm Convention as priority persistent organic pollutants (POPs), which must be removed from the environment by 2025.

Pharmaceutically Active Compounds (PhACs) In aquatic environments, PhACs have become an important issue that attracts the attention of the public due to their pseudo-persistence, possible toxic effects on microorganisms, and the widespread occurrence in surface waters (Yan et al. 2016). The aromatic side-chain compounds used in the pharmaceutical industry are difficult to biodegrade due to their high persistence in the environment and their resistance to degradation. These com-

pounds reach water systems from different sources, such as human excretion (wastewater), improper disposal, landfill leaching, water drainage, or industries. It has been found that PhACs have the potential to cause aquatic toxicity, development of resistance in pathogenic microbes, genotoxicity, and endocrine alteration (Couto et al. 2019).

Chlorinated Phenolic Compounds (CPCs) The contamination of the environment by chlorinated aromatic compounds is a serious problem due to its large-scale applications such as biocides, lubricants, solvents, etc. and its high stability to biodegradation (Colomban et al. 2014). CPCs are the precursors or intermediates in many process industries, such as the dye, resin, and plastic, pharmaceutical, and pulp and paper industries. CPCs are irritating at low levels and have a very negative impact on the central respiratory and nervous systems at higher doses. They also have adverse effects of acute toxicity and carcinogenic properties (Miran et al. 2017).

Organochlorine Pesticides (OCPs) These compounds are of environmental concern due to their recalcitrance in soils and sediments, global distribution, and toxicity. The OCPs had been applied to the soil to protect the crops until they were banned. Due to their properties, they could bind strongly to organic matter and become trapped within the soil matrix, which would lead to persistence several years after their last use. The presence of pesticides released into the soil represents a risk to the environment since they can migrate through the profile; be absorbed by biota and transported to aquatic systems by erosion (Mitton et al. 2012).

11.3 Phytoremediation Technology

Phytoremediation is a term that applies to a group of technologies that use plants to reduce, eliminate, degrade, or immobilize toxic compounds in the environment through biological processes that develop the same plants or through synergistic plant-microorganism processes that develop in the rhizosphere, with the objective of restoring the areas to a condition in which they are usable (Manahan 2007; Peer et al. 2006). This technology is used to remove heavy metals, radionuclides, nutrients (nitrate, phosphate, etc.), solvents, explosives, crude oil and organic pollutants such as persistent organic pollutants (POPs), polycyclic aromatic hydrocarbons (PAHs), and pesticides from wastewater and soils (Anand et al. 2019).

Phytoremediation of contaminated sites has gained popularity as a non-invasive, relatively inexpensive, and esthetically pleasing technology to the public compared to alternative remediation strategies that involve excavation, removal or stabilization, and chemical conversion in situ (Peer et al. 2006). The phytoremediation is a sustainable technology for cleaning up of recalcitrant pollutants from disturbed environs. The phytoremediation strategies have a number of promising advantages in comparison with other remediation techniques, in particular (Fig. 11.2).

There are factors that influence plant phytoremediation, which include the ability of the plant to absorb pollutants through its roots and transfer it to the shoots; the level at which plants tolerate contaminants and the available concentration of contaminants so that the plant can absorb them; and the rate of transforming toxic substances into less toxic by plants. Other factors that also influence phytoremediation are the root length of the plants, the types and concentration of the target metals, the environmental condition of the soil, and the interaction of the plants and associated soil microorganisms (Naila et al. 2019).

11.4 Plants Used in Phytoremediation Technology to Cleaning Up of Recalcitrant Pollutants

This chapter provides information of plants considered hyperaccumulants of recalcitrant contaminants mainly heavy metals in Table 11.1 and other recalcitrant pollutants are presented in Table 11.2. The information was searched by consulting the following electronic sources: ScienceDirect, SCOPUS, Web of Science, SpringerLink, SCIELO, PubMed, and Google Scholar finding 77 studies with different heavy metal accumulator plants such as Cd, Pb, Hg, Mn, Ni, B, Zn, Tl, Co, and Cu. Table 11.1 shows the plant species and the recalcitrant pollutant that removes the part where it accumulates.

Table 11.2 presents the 13 studies with plants removing other recalcitrant pollutants such as polycyclic aromatic hydrocarbons, oil hydrocarbons, phenol, nitrogen compounds.

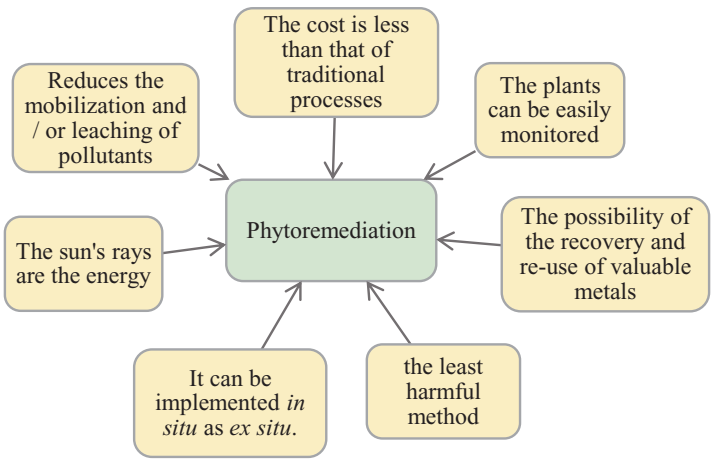

Fig. 11.2 Main advantages of phytoremediation (Dubchak and Bondar 2018; Abdel-Shafy and Mansour 2018)

11.5 *Typha latifolia* Plant Used in Phytoremediation Technology

Typha latifolia (cattail) is an emergent macrophyte; it is a perennial plant belonging to the Thyphaceae family, which grows in swamps, lakes, and lagoons. In addition, it has the advantage of growing in diverse climatic conditions and in environments contaminated with heavy metals (Alonso-Castro et al. 2009). The aquatic macrophyte presents rapid growth, easy spreading, high-pollutant uptake capacity, and easy harvesting, properties required for aquatic plants used in treatment system (Boyd 1970).

The accumulation of toxic elements by *T. latifolia* is a gradual process and depends on the concentration of elements and time of exposure. Additionally, this plant presents characteristics desirable for the remediation of aquatic sites such as a well-developed root–rhizome system, easy harvesting, and high biomass production. Further analyses are necessary to characterize the cellular mechanisms involved in the internalization of these elements in this plant. Previous research conducted by our research group indicated that *T. latifolia* presents the ability to remove and accumulate As, Cd, Pb, Ni, Cr, Mn, and Fe from *in situ* (an artificial lagoon) and controlled conditions (Carranza Álvarez 2005; Alonso-Castro et al. 2009; Carranza Álvarez et al. 2008; Leura Vicencio et al. 2013). In addition, promoter bacteria were isolated from the root of *T. latifolia*, finding that these exert a protective role in the phytoextraction of heavy metals. Nevertheless, the response to the toxic elements stress depends on the plant species.

11.5.1 Typha latifolia *as Biosorbent*

The heavy metals present in waters come mainly from industrial processes such as mineral extraction, wastewater treatment, metal casting and coating, nuclear and nuclear generation industries, among others. Heavy metals are introduced into food chains resulting in accumulation in the human body. Even at low concentrations they have important health effects such as stunted growth, spread of cancer, damage to organs, deterioration of the nervous system, and, in more severe cases, death (Aghababai and Beni 2020).

Lead is a non-essential element in the human body and its accumulation can cause some damage to the brain, leading to decreased learning and memory. It also affects bone metabolism, causing genetic mutations (Chu et al. 2019). There are several processes that can be applied in water treatment to reduce the concentration of heavy metals and other pollutants such as ion exchange, membrane separation, electrodialysis, reverse osmosis, solvent extraction, evaporation, etc. These methods are expensive and ineffective. Adsorption has become an economical and effective method for treating heavy metal wastewater due to its easy availability of raw materials and simple operation (Elhafez et al. 2017). Biosorption, considered as a type of

Table 11.1 Plants considered hyperaccumulants of recalcitrant contaminants

No.	Plant species	Recalcitrant pollutants that remove the part where it accumulates	References
1	*Abelmoschus manihot*	$Cd^{R, L, St}$	Wu et al. (2018)
2	*Achyranthes* sp.	$Fe^{R, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
3	*Actephila alanbakeri*	Ni^{L}	Van Der Ent et al. (2016)
4	*Ageratina sp*[a]	Zn^{Sh}	Bech et al. (2016)
5	*Alcea aucheri*	$Pb^{Sh, R}$ and Cd^{L}	Ravanbakhsh et al. (2016)
6	*Amaranthus spinosus* L.	Fe^{R}, $Zn^{R, Sh}$, and $Mn^{R, Sh}$	Chandra and Kumar (2017)
7	*Amaranthus viridis*	Pb	Eid and Shaltout (2016)
8	*Anisopappus chinensis*	Cu^{L} and Co^{L}	Lange et al. (2016)
9	*Antidesma tomentosum Blume*	Mn^{L}	Nkrumah et al. (2018)
10	*Antidesma coriaceum Tul.*	Mn^{L}	Nkrumah et al. (2018)
12	*Antidesma leucopodum* airy shaw	Mn^{L}	Nkrumah et al. (2018)
13	*Antidesma montanum* blume	Mn^{L}	Nkrumah et al. (2018)
14	*Antidesma Montis-silam*	Mn^{L} and $Ni^{L, B, St}$	Nkrumah et al. (2018)
15	*Antidesma neurocarpum Miq.*	Mn^{L} and Ni^{L}	Nkrumah et al. (2018)
16	*Antidesma neurocarpum* var. *Linearifolium* (Pax and K. Hoffm.) Petra Hoffm	Mn^{L}	Nkrumah et al. (2018)
17	*Antidesma puncticulatum Miq.*	Mn^{L} and Ni^{L}	Nkrumah et al. (2018)
18	*Antidesma stipulare Blume*	Mn^{L}	Nkrumah et al. (2018)
19	*Basella alba*	$Fe^{R, Sh, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
20	*Bassia indica*	Fe and Pb	Eid and Shaltout (2016)
21	*Blumea lacera*	$Fe^{R, Sh, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
22	*Brassica oleracea* var. *capitata*	Tl	Jia et al. (2013)
23	*Brassica rapa* var. *rapa*	$Cd^{St, L}$	Li et al. (2016)
24	*Calendula officinalis*	Pb^{R} and Cu^{R}	Shao et al. (2019)
25	*Camellia oleifera*	$Mn^{L, St}$	Yu et al. (2019)
26	*Cannabis sativa*	$Fe^{R, Sh, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
27	*Carduus nutans*	$Cd^{R, Sh}$	Palutoglu et al. (2018)
28	*Celosia argentea* Linn.	$Mn^{L, St, R}$	Liu et al. (2014)
29	*Centella asiática*	$Fe^{R, L, St}$	Bhat et al. (2016)
30	*Chenopodium album*	$Fe^{R, Sh, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
31	*Chlorophytum comosum*	Pb^{R} and Cu^{R}	Shao et al. (2019)
32	*Clethra barbinervis*	Co^{L}	Yamaguchi et al. (2017)
33	*Coronopus didymus* (L.)	$Cd^{R, Sh}$	Sidhu et al. (2017)
34	*Cortaderia hapalotricha*[a]	Pb^{Sh}	Bech et al. (2016)

(continued)

Table 11.1 (continued)

No.	Plant species	Recalcitrant pollutants that remove the part where it accumulates	References
35	*Croton bonplandianum*	$Fe^{R, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
36	*Datura stramonium*	$Fe^{R, Sh, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
37	*Eichhornia crassipes*	Cr $(VI)^{R, L, St}$, Cu, Pb, Hg, and Zn	Saha et al. (2017), Romero-Hernández et al. (2017)
38	*Epilobium denticulatum*[a]	Zn^{Sh}	Bech et al. (2016)
39	*Erato polymnioides*	Pb, Zn, Cu, and Cd	Chamba et al. (2016)
40	*Gevuina avellana*	Al^{L}	Delgado et al. (2019)
41	*Glochidion* cf. *sericeum*	Ni^{L}, Co^{L}	Van Der Ent et al. (2018)
42	*Helianthus annuus*	B	Barbafieri et al. (2018)
43	*Isatis cappadocica* subsp. *Cappadocica*	Ni^{AG}	Çelik et al. (2018)
44	*Kalanchoe pinnata*	$Fe^{Sh, L}$	Chandra and Kumar (2017)
45	*Lomatia dentata*	Al^{L}	Delgado et al. (2019)
46	*Macleaya cordata*[a]	$Cd^{R, St, L}$	Nie et al. (2016)
47	*Microthlaspi perfoliatum*	Ni^{AG}	Çelik et al. (2018)
48	*Myriophyllum aquaticum*	Cu, Pb, Hg, and Zn	Romero-Hernández et al. (2017)
49	*Noccaea caerulescens*	Zn^{Ec}	Kozhevnikova et al. (2017)
50	*Noccaea ochroleuca (Boiss and Heldr.) FKMey*	Ni	Salihaj et al. (2018)
51	*Odontarrhena oxycarpa*	Ni^{AG}	Çelik et al. (2018)
52	*Odontarrhena muralis*	Ni^{AG}	Çelik et al. (2018)
53	*Orites myrtoidea*	Al^{L}	Delgado et al. (2019)
54	*Panax notoginseng*	Pb^{R} and Cu^{R}	Shao et al. (2019)
55	*Parthenium hysterophorus*	$Fe^{R, Sh, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
56	*Phlomis* sp.	$Cd^{R, Sh}$	Palutoglu et al. (2018)
57	*Phyllanthus balgooyi*	$Ni^{L, St, P}$	Mesjasz-Przybylowicz et al. (2016)
58	*Phyllanthus rufuschaneyi*	Ni	Bouman et al. (2018)
59	*Phyllanthus securinegioides*	$Ni^{pR, pSt, L}$	Van Der Ent et al. (2017)
60	*Pluchea dioscoridis*	Fe	Eid and Shaltout (2016)
61	*Polygonum lapathifolium* L.	$Mn^{L, St, R}$	Liu et al. (2016)
62	*Portulaca oleracea*	Mn	Eid and Shaltout (2016)
63	*Potamogeton pectinatus* L.	Cd	Lu et al. (2018)
64	*Pseudosempervivum sempervivum*	Ni^{AG}	Çelik et al. (2018)

(continued)

Table 11.1 (continued)

No.	Plant species	Recalcitrant pollutants that remove the part where it accumulates	References
65	*Pterocypsela laciniata* (Houtt.) C. Shih	$Cd^{Sh, R}$	Zhong et al. (2019)
66	*Rinorea niccolifera*	Ni	Fernando et al. (2014)
67	*Saccharum munja*	$Fe^{R, Sh, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
68	*Salvinia cucullata*	$Cu^{Sh, R}$	Das and Goswami (2017)
69	*Sedum plumbizincicola*	Cd, Zn	Fan et al. (2019)
70	*Senecio conrathii* NEBr.	Ni^{L}	Siebert et al. (2018)
71	*Setaria viridis*	$Fe^{R, L}$, $Zn^{R, L}$, and $Mn^{R, L}$	Chandra and Kumar (2017)
72	*Silene latifolia*	Tl^{Sh}	Escarré et al. (2010)
73	*Solanum nigrum*	$Zn^{R, L}$, $Pb^{L, St, R}$, Co^{R}, Ni Cd, and Fe	Saad-Allah and Elhaak (2017), Eid and Shaltout (2016)
74	*Tagetes erecta* L.	Cd^{Tb}	Liu et al. (2018)
75	*Tagetes patula* L.	Cd^{Tb}	Liu et al. (2018)
76	*Taraxacum ohwianum* Kitam.	$Cd^{R, L}$	Cheng et al. (2019)
77	*Thlaspi rosulare*	Ni^{AG}	Çelik et al. (2018)
78	*Thlaspi triangulare*	Ni^{AG}	Çelik et al. (2018)
79	*Trichosanthes dioica*	$Fe^{R, Sh, H}$, $Zn^{R, H}$, and $Mn^{R, H}$	Chandra and Kumar (2017)
80	*Typha domingensis*	$Ni^{R,Sh}$ and $Cd^{R, Sh}$	Mojiri et al. (2016)

Part where the recalcitrant accumulates: *R* hyperaccumulation in root, *Sh* shoot, *L* leaves, *St* stem, *P* petioles, *Tb* total biomass, *pR* phloem root, *pSt* phloem stem, *B* bark, *Ec* epidermal cells, *AG* above ground parts

[a]Almost considered hyperaccumulator

adsorption using biomass, is a promising method for the treatment of Pb (II) contaminated water due to its low cost and minimization of toxic sludge (Liu et al. 2014). The removal of heavy metals using biomass is attributed to a stable and insoluble matrix with active groups on the surface mainly carboxyl, amino, and hydroxyl groups.

There are a large number of reported studies on metal removal using various biosorbents, but few have focused on the use of cattails as the basis for adsorbent materials. Cattails are perennial, herbage plants with high adaptability and large biomass that are usually used in the construction of wetlands for removing nitrogen, phosphorous, and organics from wastewater (Tang et al. 2017). These aquatic plants have taken an interest in the remediation of aquatic ecosystems contaminated by heavy metals. *Typha latifolia, Typha angustifolia* and *Salix matsudana* are low-cost materials that have been used directly or as precursors of activated carbons for the removal of heavy metals such as Pb(II), Cd(II), Cr(VI), Cu(II), Mn(II), Zn(II),

Table 11.2 Plants used in bioremediation of recalcitrant contaminants

No.	Plant species	Recalcitrant pollutants that remove the part where it accumulates	References
1	*alfalfa Crioula*	Polycyclic aromatic hydrocarbons	Alves et al. (2018)
2	*Aspilia africana*	Oil hydrocarbons	Anyasi and Atagana (2018)
3	*Carex praegracilis*	Benzotriazole	Pritchard et al. (2018)
4	*Chromolaena odorata*	Oil hydrocarbons	Anyasi and Atagana (2018)
5	*Cucurbita pepo* L	Polychlorinated dibenzo-*p*-dioxins (PCDD) and polychlorinated dibenzo-furans (PCDF)	Urbaniak et al. (2016)
6	*Cyperus alternifolius*	Carbamazepine, sulfamethoxazole, ofloxacin, roxithromycin	Yan et al. (2016)
7	*Lolium perenne*	Anthracene	Yarahmadi et al. (2017)
8	*Nasturtium officinale*	Azo dye C.I. acid blue 92	Torbati et al. (2015)
9	*Pistia stratiotes*	Naphth-1-yl acetic acid, nitrate nitrogen	Pavithra and Hina (2018)
10	*Rhizophora mangle* L.	Oil hydrocarbons	Moreira et al. (2011)
11	*Salix* sp.	*P,p′*-DDT, *p,p′*-DDE, *p,p′*-DDD	Mitton et al. (2012)
12	*Uvaria chamae*	Oil hydrocarbons	Anyasi and Atagana (2018)
13	*Vetiveria zizanioides* L. Nash	Phenol, oil hydrocarbons	Singh et al. (2008), Dudai et al. (2018)

among others. However, few studies evaluate the physicochemical properties of biosorbents and their relationship with the removal of the metal of interest (Bukhari et al. 2013; Kumari and Tripathi 2015; Liu et al. 2014; Rajaei et al. 2013; Song et al. 2013; Tang et al. 2017).

Therefore, the main objective of the present work was to use the root of *Typha latifolia* as an adsorbent material for the removal of Pb(II) from water as a low-cost alternative, as well as to relate its physicochemical properties and adsorption capacity.

11.5.2 Preparation of Typha latifolia *Root as Biosorbent*

T. latifolia samples were collected from a temporary lagoon located in the municipality of Ciudad Valles, San Luis Potosí, Mexico, by means of a meticulous collection of plants with similar characteristics such as height between 30 and 50 cm and degree of robustness of their root and rhizome between 10 and 20 cm long and 5 cm wide. Once collected, they were treated using a process of acclimatization at room temperature in a plant nursery for 30 days. During this acclimatization process, the

plants were provided with nutrients (K, P, N) and enough water to promote root growth. Next, the plants with the largest amount of root were selected and washed with abundant water to remove impurities, the roots were cut and washed again using distilled water, then introduced into the stove for drying at 70 °C for 72 h. The roots were then crushed and pulverized using an analytical mill. Once the root was pulverized, it was thoroughly washed with deionized water and sieved to an average particle size of 0.16 mm. Finally, the root was dried at 80 °C for 24 h. The biosorbent obtained was packaged and stored in a plastic container for later use.

11.5.3 Characterization of T. latifolia *Root*

The morphology of the surface of the natural root of *T. latifolia* was examined by means of a scanning electron microscope (SEM), Philips, model XL-30-SFEG, equipped with a Link/ISIS-OXFORD microanalysis system of dispersed energy (EDS) which allows to identify the elements present on the surface of the material used as biosorbent. The functional groups present in the natural root of T. latifolia were identified by infrared spectroscopy (IR). The IR spectra of the root were obtained using an infrared spectrophotometer, Thermo-Scientific, model Nicolet iS10 that has the technique of attenuated total reflectance (ATR). The active sites were determined by the acid–base titration method proposed by Boehm (1994). The total acid and basic sites were neutralized with 0.01N NaOH and HNO_3 solutions, respectively. Surface charge and zero point charge (pH_{PZC}) were determined by the procedure proposed by Kuzin and Loskutov (1996).

11.5.4 Determination of the Concentration of Pb(II) in Aqueous Solution

The concentration of Pb(II) in aqueous solution was determined using an atomic absorption spectrophotometer, Varian model SpectrAA-20 and the concentration in aqueous solution was estimated using a calibration curve with standard Pb(II) solutions.

11.5.5 Adsorption Isotherms of Pb(II) on T. latifolia *Root*

Experimental data on the adsorption equilibrium of Pb(II) on the root of *T. latifolia* were obtained in batch adsorbers as follows. In a volumetric flask of 50 mL a solution of a known concentration of Pb (II) was prepared from a 1000 mg L^{-1} standard solution and a buffer solution prepared by mixing NaOH and HNO_3 solutions was

gauged. Next, an initial sample of 10 mL was taken and subsequently analyzed to corroborate the initial concentration. To the batch adsorber, a certain root mass and 40 mL of the initial concentration solution were added. The adsorber was placed in a constant temperature bath, and the root and solution were left in contact until equilibrium was reached. In previous experiments, it was found that 5 days was enough time to achieve equilibrium. Once a day, the adsorbers were mixed in an orbital agitator at 200 rpm for 30 min.

The pH of the solution was measured periodically with a potentiometer and kept constant by adding drops of solutions 0.01, 0.1, and 1N of HNO_3 and NaOH, as necessary. The volumes of these solutions were recorded to calculate the final total volume of the solution. Once equilibrium was reached, a 10 mL sample was taken and analyzed to determine the final concentration of the solution. The mass of Pb (II) adsorbed on the root was calculated by means of a mass balance which is mathematically represented as follows:

$$q = \frac{V}{m}\left(C_0 - C_e\right) \tag{11.1}$$

where q (mg g^{-1}) is the mass of Pb(II) adsorbed per unit root mass, V (L) is the volume of the Pb(II) solution, m (g) is the mass of the root, C_0 (mg L^{-1}) is the initial concentration of Pb(II), and C_e (mg L^{-1}) is the concentration of Pb(II) in the equilibrium.

11.5.6 Results and Discussion About the Study of Typha latifolia *as a Biosorbent*

11.5.6.1 SEM, EDX, and IR Analyses of the *T. latifolia*

The surface, morphology, and particle distribution of *T. latifolia* root were observed by scanning electron microscope (SEM) and the elemental chemical composition of the surface was determined by microanalysis (EDX). Figure 11.3a, b shows the photomicrographs of fragments of *T. latifolia* root. Figure 11.3a shows a micrograph of the root increased to 36×, in which the irregular and rough surface is observed. Figure 11.3b is at 3500× and it is shown in great detail that the surface of the root does not have a uniform topography, also can be seen in it some irregular particles which are attributed to Pb, these in size range between ±5 μm.

Figure 11.4 presents the X-ray fluorescence (EDX) microanalysis of the *T. latifolia* exposed to Pb. It can be seen that C and O are the main elements, since it is a lignocellulosic material. The presence of Al, Si, and S was also detected in smaller quantities, as well as the characteristic peak of Pb.

The results of the IR spectra (not shown) revealed that the root of *T. latifolia* exposed to Pb(II) presents a slight change in the band of the functional groups O–H, C–H, C–O, and C–N. The results of the IR spectra revealed that the root of *T. latifolia*

exposed to Pb(II) presents a slight change in the band of the functional groups O–H, C–H, C–O, and C–N. However, the increase in the intensity for the groups C=O and C=C was more noticeable and is a clear indication of the interaction between the organic compounds of *T. latifolia* and Pb(II) in solution.

It is very important to mention that the influence of the surface chemistry of adsorbent materials confers a certain degree of selectivity, for example, the presence of carboxylic, lactonic, and hydroxylic phenolic groups is responsible for the acidic surface properties of activated carbon (Böehm 2002). These specific functional groups are essential in the adsorption of heavy metals due to their chelating quality forming complexes (Monser and Adhoum 2002; Kikuchi et al. 2006).

11.5.6.2 Adsorption Isotherms

The experimental Pb(II) adsorption equilibrium data were adjusted to the Freundlich and Langmuir isothermal models. These models are mathematically represented with the following equations:

$$q = kC_e^{1/n} \tag{11.2}$$

$$q = \frac{q_m KC_e}{1 + KC_e} \tag{11.3}$$

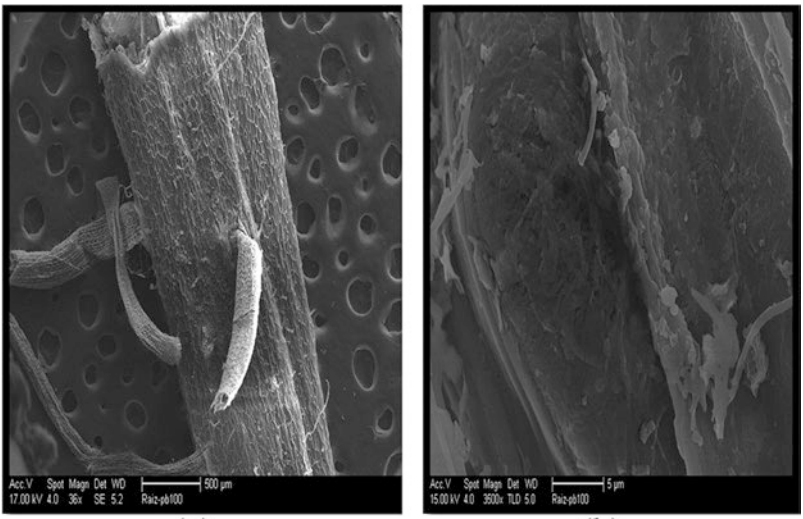

Fig. 11.3 SEM micrographs of *T. latifolia* saturated with Pb(II) (**a**) 36× and (**b**) 3500×

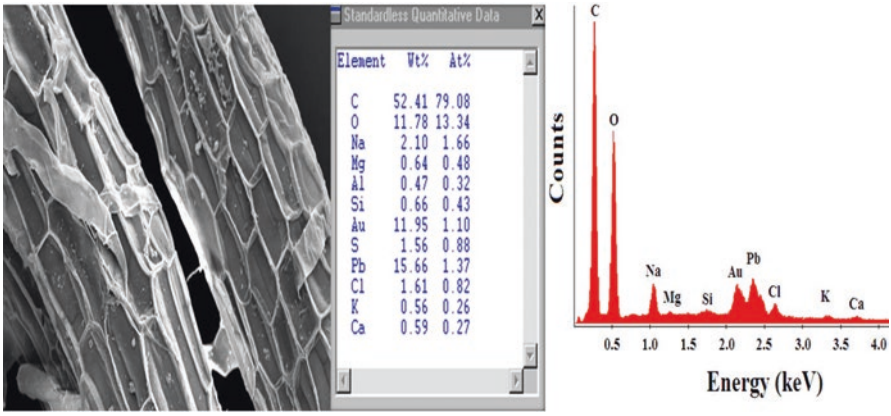

Fig. 11.4 SEM micrograph and EDX spectra of *T. latifolia* saturated with Pb(II)

The parameters K (L mg^{-1}) and q_m (mg g^{-1}) are the Langmuir constants for Pb(II) related to adsorption energy and maximum adsorption capacity, respectively. The parameters k (L$^{1/n}$ mg$^{1-1/n}$ g^{-1}) and n are the Freundlich constants for Pb(II) related to adsorption capacity and intensity, respectively. The parameters of these isotherms were estimated by a method of least squares based on the Rosenbrock–Newton optimization algorithm which uses the following objective function:

$$R = \sum \left(q_{\text{exp}} - q_{\text{cal}} \right)^2 = \text{minimum} \tag{11.4}$$

On the other hand, the average absolute percentage deviation %Dev was estimated using the following equation and the results are reported in Table 11.3.

$$\%\text{Dev} = \left(\frac{1}{N} \sum_{i=1}^{N} \left| \frac{q_{\text{ex}} - q_{\text{cal}}}{q_{\text{ex}}} \right| \right) \times 100\% \tag{11.5}$$

Table 11.3 shows that the isotherm that best adjusted the adsorption equilibrium data was the Freundlich isotherm since the %Dev was lower than the Langmuir isotherm for all cases. The %Dev was lower than 17.2 and 11.5% for the Langmuir and Freundlich isotherms, respectively. Therefore, it was considered that Freundlich isotherm better interpreted the experimental data. It should be noted that the model of the Freundlich isotherm suggests that the surface of the adsorbent is heterogeneous. However, it is important to clarify that the fit of the isotherm model to the adsorption equilibrium data is a statistical mathematical method and not a corroboration that the adsorption mechanism occurs according to the fundamentals that support the model that best adjusted the data.

11.5.6.3 Effect of Solution pH on the Adsorption Capacity

The solution pH is probably the most important factor in the adsorption of ions in aqueous solution onto porous solids. The pH has a considerable influence on the adsorption equilibrium since the surface charge of the adsorbent and the species or ionic complexes formed by the adsorbate are a function of the pH. The effect of solution pH on the adsorption capacity of Pb(II) in aqueous solution on the root of *T. latifolia* was investigated by determining adsorption isotherms at pH 2, 3, and 5 and T = 25 °C. The effect of pH on the adsorption capacity of Pb(II) in aqueous solution on the root of *T. latifolia* was investigated by determining adsorption isotherms at pH 2, 3, and 5 and T = 25 °C. Figure 11.5 shows the effect of pH on the adsorption capacity of *T. latifolia* root and it can be seen that pH significantly affects adsorption capacity.

The results revealed that maximum adsorption capacity occurs at pH 5 and decreases as it decreases from 5 to 3 and from 3 to 2. On the other hand, the averages of Typha root adsorption capacities were 0.96, 1.72, and 7.74 mg g^{-1} at pH 2, 3, and 5, respectively. This is a clear indication that the adsorption capacity shown by the *T. latifolia* root increases considerably with increasing pH. The effect of the pH of the solution on the adsorption capacity can be explained considering that the pH_{PZC} of the biosorbent determined in this study was 5.6 which indicates that its character is acid and that the surface charge can be positive, neutral, or negative when the pH < pH_{PZC}, pH = pHPZC, or pH > pHPZC, respectively.

It is important to mention that during the adsorption of Pb(II), the pH of the solution always remained variable during the adsorption progress and to keep it constant it was necessary to add solutions 0.1 and 0.01N of NaOH or HNO_3. In the experiments carried out at pH 5.0, the solution varied considerably, increasing with the days until reaching equilibrium. On the other hand, in the experiments performed at pH 2 and 3, the pH of the solution hardly changed. This could indicate that the H^+ ions from the root surface were transferred to the solution and the Pb(II) cations in solution were diffused to the *T. latifolia* root surface. In other words, Pb(II) was adsorbed by ion exchange.

Table 11.3 Parameters for the Langmuir and Freundlich adsorption isotherms

		Langmuir			Freundlich		
T (°C)	pH	q_m (mg g^{-1})	K (L mg^{-1})	%Dev	k ($L^{1/n}$ $mg^{1-1/n}$ g^{-1})	n	%Dev
15	5.0	17.3	0.156	17.2	3.39	2.45	8.22
25	2.0	28.8	0.003	10.4	0.10	1.13	7.17
	3.0	7.79	0.062	9.03	0.53	1.34	8.64
	5.0	19.5	0.205	16.3	4.68	2.63	7.23
35	5.0	28.8	0.058	16.6	3.62	2.14	11.5

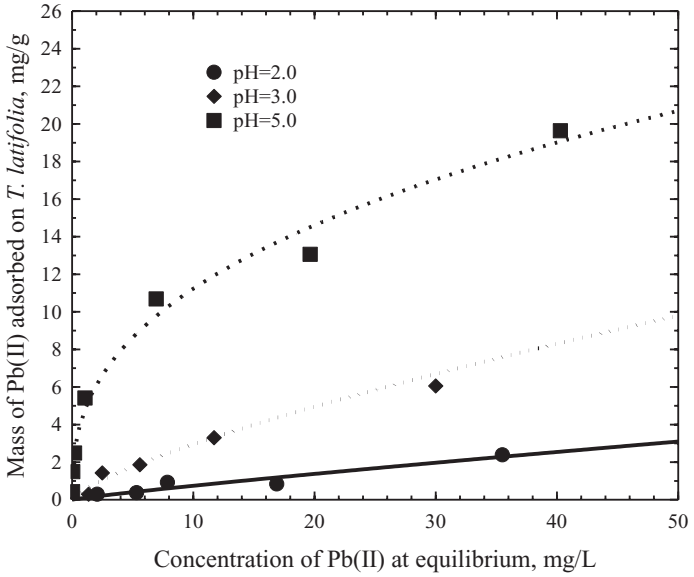

Fig. 11.5 Effect of the solution pH on the adsorption isotherm of *T. latifolia* at T = 25 °C. The lines represent the Freundlich isotherm

11.5.6.4 Effect of Solution Temperature on the Adsorption Capacity

The effect of the temperature of the solution on the adsorption of Pb(II) in the root of *T. latifolia* was analyzed by isotherms of Pb(II) adsorption at temperatures of 15, 25, and 35 °C, at pH of 5. In Fig. 11.6 it is observed that the capacity of the root to adsorb Pb(II) increased as the temperature increased. These results indicate that the adsorption of Pb(II) was endothermic. The effect of temperature can be checked by calculating the heat of adsorption (ΔHads), which when occurring at the same mass of adsorbed Pb(II), but at different temperatures is known as isosteric heat of adsorption. This calculation is made by means of the following equation (Medellin-Castillo et al. 2017):

$$\left(\Delta H_{\text{ads}}\right)_q = \frac{R \ln \dfrac{C_2}{C_1}}{\dfrac{1}{T_2} - \dfrac{1}{T_1}} \tag{11.6}$$

where $(\Delta H_{\text{ads}})_q$ (J mol^{-1}) is the isosteric heat of adsorption, R (8314 J mol^{-1} K^{-1}) is the universal constant of the ideal gases, C_1 (mg L^{-1}) is the concentration of Pb(II) to T_1 (K) at the same value of q in equilibrium, and C_2 (mg L^{-1}) is the concentration of Pb(II) to T_2 (K) at the same value of q in equilibrium. Thus, at a mass of Pb(II) adsorbed on the root of *T. latifolia* of q = 14.0 mg g^{-1}, the concentrations of Pb(II)

in equilibrium were $C_1 = 32.0$ mg L^{-1} and $C_2 = 14.8$ mg L^{-1}, at temperatures of $T_1 = 288.15$ K and $T_2 = 308.15$ K, respectively. The estimated isosteric heat was 28.5 KJ mol^{-1} indicating that the biosorption process of Pb(II) on the root of *T. latifolia* is endothermic ($\Delta H > 0$).

Experimental data from Pb(II) adsorption isotherms on *T. latifolia* root were correlated by Freundlich and Langmuir isotherms. The isotherm of Freundlich better adjusted the data under the criterion of the lowest percentage deviation. The study of the effect of pH and temperature in the Pb(II) adsorption isotherm on the root of *T. latifolia* revealed that the adsorption capacity of this biosorbent to remove lead is considerably dependent on pH and the temperature of the solution, increasing with increasing pH and temperature. The biosorption of Pb(II) on the root of *T. latifolia* is an endothermic process, with an isosteric heat of adsorption of 28.5 KJ mol^{-1}. Finally, it was concluded that Pb(II) can be efficiently removed from aqueous solutions by means of Typha latifolia root, which is widely distributed in the world and can be easily found in the Huasteca region in San Luis Potosi, Mexico and therefore can be considered a viable and low-cost option for the treatment of water intended for human consumption.

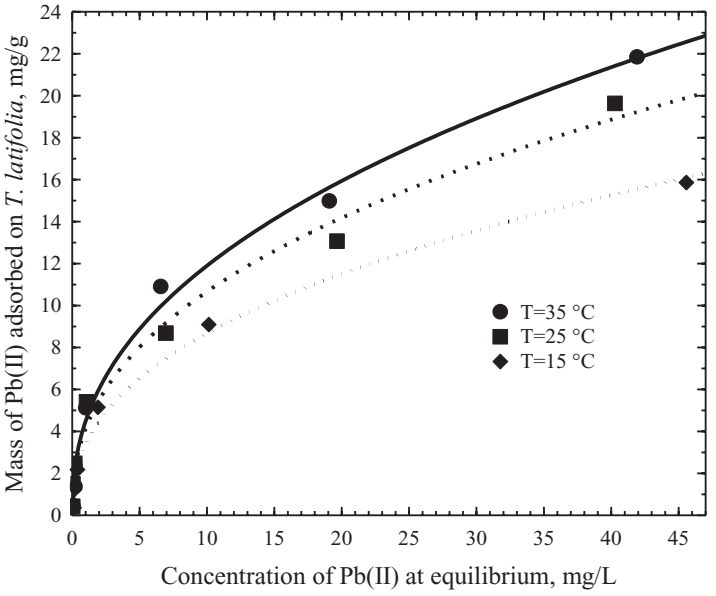

Fig. 11.6 Effect of the solution temperature on the adsorption isotherm of *T. latifolia* at pH = 5.0. The lines represent the Freundlich isotherm

11.6 Conclusions About the Study of *Typha latifolia* as a Biosorbent

Experimental data from Pb(II) adsorption isotherms on *T. latifolia* root were correlated by Freundlich and Langmuir isotherms. The isotherm of Freundlich better adjusted the data under the criterion of the lowest percentage deviation. The study of the effect of pH and temperature in the Pb(II) adsorption isotherm on the root of *T. latifolia* revealed that the adsorption capacity of this biosorbent to remove lead is considerably dependent on pH and the temperature of the solution, increasing with increasing pH and temperature. The biosorption of Pb(II) on the root of *T. latifolia* is an endothermic process, with an isosteric heat of adsorption of 28.5 KJ mol^{-1}. Finally, it was concluded that Pb(II) can be efficiently removed from aqueous solutions by means of Typha latifolia root, which is widely distributed in the world and can be easily found in the Huasteca region in San Luis Potosi, Mexico and therefore can be considered a viable and low-cost option for the treatment of water intended for human consumption.

11.7 Conclusion

Fitorremediation is a sustainable, low-cost technology because it uses the natural capacity of plants to remove contaminants. Plants are able to remove and accumulate in their plant tissues a wide variety of recalcitrant contaminants (organic and inorganic). To facilitate phytoremediation, native plants with a large amount of biomass and rapid growth such as *Typha latifolia* should be used. *T. latifolia* is a suitable plant for cleaning contaminated environments. *T. latifolia* is solution for cleaning up of recalcitrant pollutants from disturbed and can be used in situ or as a biosorbent material.

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