Chapter 12
Adsorption of Pharmaceutical Pollutants Using Lignocellulosic Materials

Atul Sharma and Saif Ali Chaudhry

Contents

Abstract Water contamination through drug disposal is a prominent problem as it has harsh consequences on food chains. Over 100,000 tonnes of pharmaceutical products are consumed globally every year, and during their manufacture, use and disposal, active pharmaceutical ingredients (APIs) as well as other chemical ingredients are released into the environment. Dry plant matter is called lignocellulosic biomass which is easily available in abundance on the Earth's surface and is composed of carbohydrate polymers (hemicellulose, cellulose) and aromatic polymer (lignin). These polymeric carbohydrates contain different sugar monomers bounded tightly to lignin. Recently, great attention has been paid to remove pharmaceutical pollutants for which various treatment methods are known including both advanced (e.g. membrane, microfiltration, ozonation) and conventional (e.g. adsorption, biodegradation, activated sludge) processes. The aim of this chapter is to discuss the removal of pharmaceuticals using adsorption from wastewater using lignocellulosic materials. Adsorption capacity of various adsorbents from various sources have been reviewed for their capacity to remove pharmaceuticals from water. There are numerous adsorbents including most commonly used carbonaceous materials, clays and polymeric and siliceous materials. The adsorption capacity of various lignocel-

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lulosic materials for pharmaceutical removal from water is discussed in this chapter. The mechanism for adsorption of pharmaceuticals onto lignocellulosic adsorbents is also discussed herein.

Keywords Adsorption capacity · Adsorbents · Water treatment · Pharmaceuticals

12.1 Introduction

Pharmaceuticals are considered, and recently recognized, as major class of environmental pollutants. The presence of pharmaceuticals, either in surface water or groundwater, near to industrial and residential localities, is seriously a great problem (Fig. 12.1 (Licence Number: 4500651074774)). In the year 1960, the USA and Europe marked the first case of the presence of pharmaceuticals and personal care products (PPCPs), and the concerns about their potential risk were raised in 1999 due to the lowering of feminization of fish living downstream of wastewater treatment plants (WWTPs) after the presence of pharmaceuticals in river was found (Kyzas et al. 2015). Hundreds of different pharmaceuticals have been detected in the environment globally. Entry of pharmaceuticals in the environment can take place in a number of ways:

1. Emissions from manufacturers
2. Human consumption and excretion of pharmaceutical products
3. Improper disposal of pharmaceuticals down toilets and sinks

Designing of active ingredients of pharmaceuticals is done in order to stimulate a response in humans and animals, and some are modified so that they remain unchanged during their passage through the body. Unfortunately, its high stability makes it persist outside the body and, as a result, can have therapeutic effects on un-targeted organisms, and it starts accumulating up. Pharmaceuticals that enter the environment can have unexpected harmful effects on wildlife. One of the worst cases of wildlife poisoning by a chemical has been attributed to a pharmaceutical product, diclofenac. This non-steroidal anti-inflammatory drug (NSAID) caused a

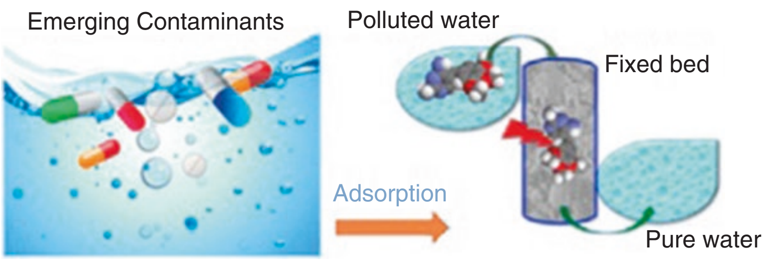

Fig. 12.1 Pictorial representation of contamination of water and its remediation by adsorption technique. (Reprinted with permission from M.J. Ahmed, B.H. Hameed 2018 copyright (2018) Elsevier (Licence Number: 4500651074774))

97% decline in three species of Old World vultures (genus *Gyps*) in Asia, with risk of extinction. Vultures feeding on carcasses of cattle treated with diclofenac suffered acute kidney failure and died within days (https://noharm-europe.org/content/europe/pharmaceutical-pollution-faq). Pharmaceuticals in the environment may also pose a threat to human health (Nabi et al. 2006; Pavithra et al. 2017). Although the concentration of pharmaceuticals may be low, exposure to mixtures of pharmaceuticals with other chemicals could pose a risk to human health. Synergistic effects can intensify the therapeutic properties, and even low concentrations can be dangerous to people for whom a medicine has been given.

There are many remedial methods available for pharmaceutical removal, but one of the advantageous methods with low cost is adsorption (Chaudhry et al. 2016; Siddiqui and Chaudhry 2017a–d). A phenomenon of accumulation of molecules of a substance on the surface of a liquid or solid leading to a higher concentration of those molecules onto the surface is called adsorption (Fig. 12.2) (Chaudhry et al. 2017; Siddiqui et al. 2018a–c). The substance thus adsorbed on the surface is called adsorbate (Siddiqui et al. 2019a–d; Tara et al. 2019), and the substance on which it is adsorbed is called adsorbent (Nilchi et al. 2012; Siddiqui and Chaudhry 2019).

After understanding the pharmaceuticals as pollutant and adsorption phenomenon, now we need to know about the use of adsorption technique in pharmaceutical removal using lignocellulosic material (which is the main aim of this chapter).

12.1.1 Effect of Parameters on Adsorption

12.1.1.1 pH Effect

The pH of solution plays a very significant role while dealing with the interactive sorption (Siddiqui and Chaudhry, 2018a, b). Studies revealed that without considering the nature of adsorbent, it is difficult to have a constant adsorption capacity over the entire pH range. So, it becomes very necessary to determine the optimum pH required for a specified adsorption process (Akhtar et al. 2015).

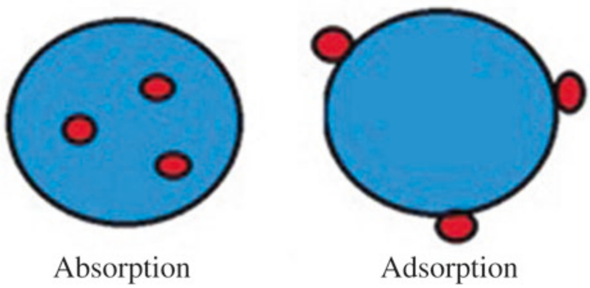

Fig. 12.2 Showing principle of absorption and adsorption

12.1.1.2 Adsorbent Dose

Studies revealed that with the increase in adsorbent dose, there is an increase in percentage removal of pharmaceuticals (Vergili and Barlas 2009; Rossner et al. 2009). This was explained on the basis of increase in availability of vacant sites at higher dosages. Studies reported that saturation value for pharmaceutical adsorption is achieved rarely [96]; therefore, it can be said that further increase in adsorbent dosage is not of measureable significance. Extra dosage leads to removal of extra pharmaceuticals (Akhtar et al. 2015).

12.1.1.3 Concentration of Pharmaceuticals

Initial concentration of pharmaceuticals is a very important factor as adsorption capacity and adsorption rate depend on it. Generally, it was found that adsorption of pharmaceuticals gets boosted by initial concentration. It was also observed that accessibility of pores for adsorbate molecules and interactions at solid–liquid interface increases due to concentration (Akhtar et al. 2015).

12.1.1.4 Temperature

For adsorption process, temperature is an important parameter. It was found that molecular activity at boundary layer interface increases at high temperature, which in turn increases the rate of diffusion of solute molecules. However, literature shows that adsorption behaviour of solute onto a specific adsorbent might also be exothermic in nature (Zawani et al. 2009).

12.2 Lignocellulosic Materials

Three polymers (cellulose, hemicellulose and lignin) constitute lignocellulosic materials (Fig. 12.3). The association of these polymers with each other depends on type, species and even source of the biomass. The relative abundance of cellulose, hemicellulose and lignin are inter alia key factors which determine the optimum energy (Bajpai 2016). The application of these lignocellulosic materials in the removal of pharmaceuticals is majorly studied in this chapter. The composition of monomers of lignocellulosic material is shown in Fig. 12.4. Plant is a major source of lignocellulosic materials (Fig. 12.5).

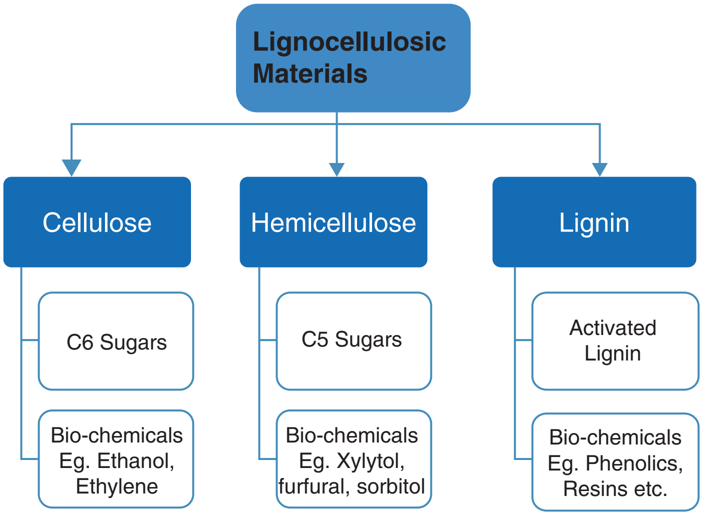

Fig. 12.3 Classification of lignocellulosic material

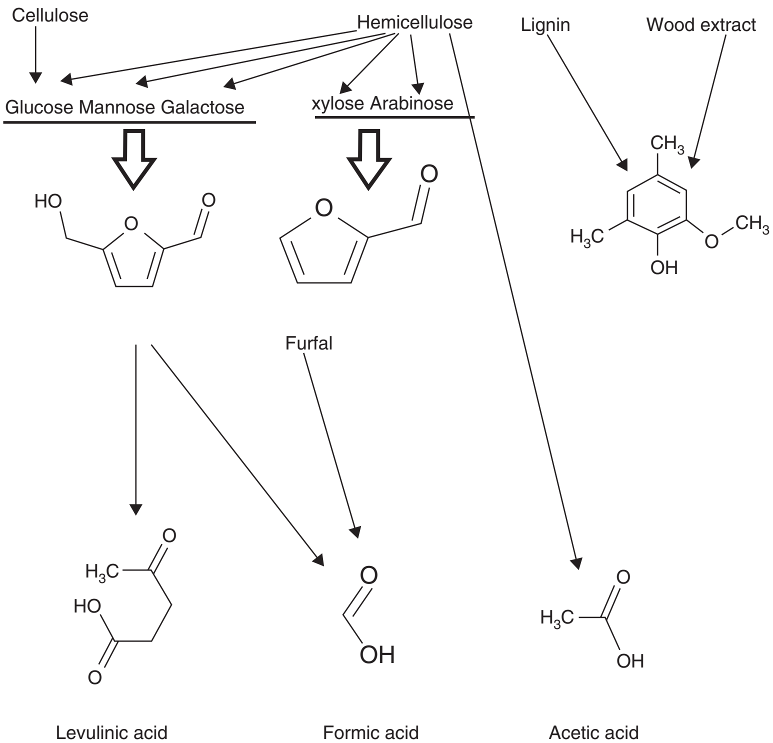

Fig. 12.4 Composition of monomers of lignocellulosic material

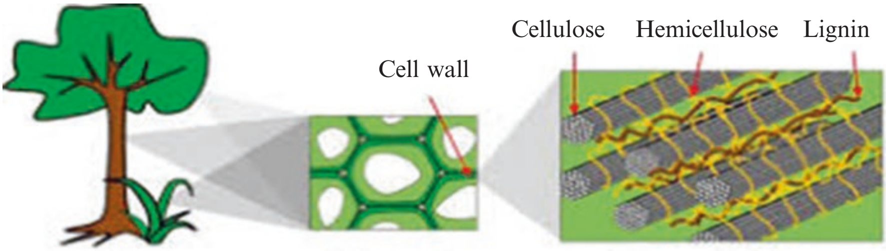

Fig. 12.5 Plants as an important source of lignocellulosic material. (Reprinted with permission from J. Vasco-Correa, X. Ge, Y. Li, copyright (2016) Elsevier (Licence Number: 4500630743190))

12.3 Ways of Reduction in Pollution of Pharmaceuticals in Environment

An important step in pharmaceutical pollution prevention includes reduction of hazardous wastes from source. Reduction in pollution at the source can be possible through the modification in process, replacement of material and good operating practices. The reach of the pharmaceutical industry is increasing day by day, and this makes the industry highly competitive. Each company's confidential policy and high specificity leads to small general discussions of material substitution and process modification. The aim is to target the thinking of manufacturers about their ways of manufacturing processes. One of the best ways to reduce the pharmaceutical pollution is to control it at its source (Fig. 12.6 (Licence Number: 4481750472810)).

There are some industries working successfully in improving efficiency and profit and also in minimizing environmental impacts. Among all, source reduction method is one which serves the primary aim of industries to reduce the wastes. Implementation of source reduction methods is generally quite difficult in pharmaceutical manufacturing units as in other manufacturing sectors. Looking at future aspects, many pharmaceutical companies are finding ways to minimize waste in future production processes by investing in research and development. Using techniques for pollution prevention at the start of a new drug development is more economical, efficient and environmentally favourable.

12.4 Lignocellulosic Materials as Adsorbent for Pharmaceuticals

The adsorption process carried out by biomass is called biosorption (Siddiqui et al. 2017). It contains waste of microbial origin and organic plant materials. These materials have capability to remove the substance dissolved in aqueous medium.

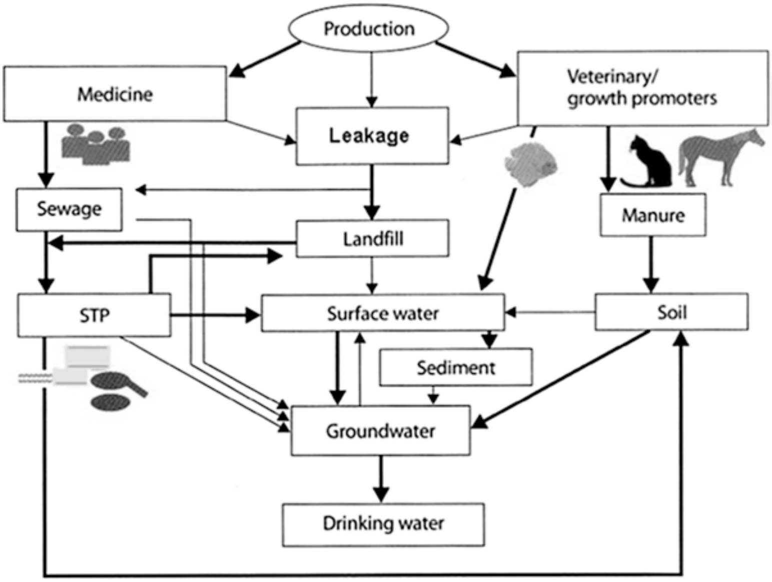

Fig. 12.6 Image showing entry of pharmaceutical into the environment. (Reprinted by permission from Elsevier Zhenxiang He et al. 2016 copyright (2016) Elsevier (Licence Number: 4481750472810))

Lignocellulosic materials serve to be the best material as it involves low cost and has good adsorbent capacity (Fig. 12.7); therefore it can be used for detoxification (Magalhaes and Neves 2006). Biomass is known to be an enhancing agent for adsorption process (Cristavo et al. 2011). For example, sugarcane bagasse and its various polymers like cellulose, hemicellulose and lignin contains functional groups like hydroxyl and/or phenolic, carbonyl groups and amines, and these groups can be modified chemically to form new compounds with various new properties.

Another example is coconut tree which is also an important lignocellulosic material; almost whole coconut tree is used for deriving lignocellulosic material, even the leaves and the fruits. The adsorption capacity of coconut fibre in liquids containing organic contaminants such as gasoline, diesel and lubricants was studied.

Kyzas in the year 2014 said that there is limited research on adsorption of organic compounds such as pesticides, petroleum derivatives and pharmaceuticals using green organic residues, whereas these organic derivatives have been proven to be good adsorbent for removal of dyes and metals.

'Chromatography' is an analytical technique commonly used for separating a mixture of chemical substances into its individual components. There are many types of chromatography, e.g. liquid chromatography, gas chromatography, ion-exchange chromatography and affinity chromatography, but all of these employ the same basic principles; therefore the chromatographic technique serves to be the best method among all the processes known (Boix et al. 2016).

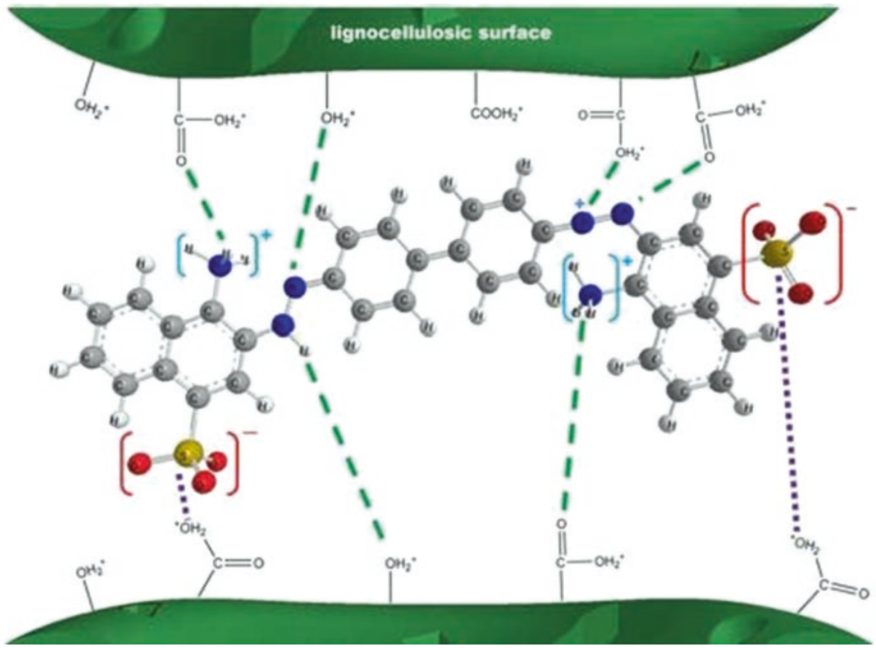

Fig. 12.7 Adsorption phenomenon on the surface of lignocellulose. (Adopted from Yaneva and Georgieva (2012))

Among various tests, one important test of toxicity provides results which give very sensitive values because it not only detects presence or absence of a particular molecule but also provides information about ecotoxicological effects produced after the removal. Among various toxicity tests, the commonly used for the observation of environmental genotoxicity is the *Allium cepa* which is specifically used for wastewater and soil testing (Mazzeo et al. 2015) because it serves as an excellent biomarker of cell mutagenic effects. This test tells about various mutagenic points of the chromosome and also identifies sensitive pollutants, and moreover it has low cost and is easily implementable (Kumari et al. 2011).

Genotoxic effect is estimated in meristematic cells which provide information about genotoxic effects; and changes in the mitotic index indicate cytotoxicity (Mazzeo et al. 2015). The breaks in chromosome and micronuclei in meristematic cell helps to determine the mutagenic potential. **Mazzeo et al**. (2015) tested *Allium cepa* and found that sludge of sewage was mutagenic and genotoxic, even at low concentration. It was found by this test that both pharmaceuticals **thiabendazole** and **griseofulvin** can cause damage to the meristematic cell, which also leads to problems in microtubule like metaphase C, breakage in chromosome of anaphase, multipolar division, bridged anaphase and disorganized anaphase. Therefore, it can be easily said that these tests are important for the collection of ecotoxicological data (Andrioli et al. 2014).

We can also use sugarcane and coconut fibres for the removal of pharmaceuticals from contaminated water, under the hypothesis that the adsorption of pharmaceuticals by these fibres may reduce the toxicity of contaminated water.

12.4.1 Discussion on Adsorption Mechanism of Pharmaceuticals onto Adsorbents

12.4.1.1 Silanol Functional Groups

pH is an important factor in the adsorption of pharmaceuticals such as ketoprofen, carbamazepine, ibuprofen, diclofenac and clofibric acid because it contains various functional groups of silica. The presence of hydroxyl group in –COOH and silanol groups (SiOH) leads to hydrogen bonding, and there is possible interaction between these two groups. The hydrogen bonding can be shown in Fig. 12.8.

These materials possess silanol groups (surface active groups) which explain cationic exchange mechanism and carboxylate group (–COOH) which explains the ligand exchange mechanism. The presence of these groups (silanol and carboxylate groups) leads to a great contribution towards adsorption tendency of adsorbents. Activated carbon can also be replaced by phenolics, carboxyl and lactone functional groups (Putra et al. 2009; Pocostales et al. 2011) that contain acidic and basic groups which have strong influence on the surface charges and enhance the adsorption properties of activated carbon. Adsorption phenomenon, therefore, not only depends on pore structure but also on surface charge because change of surface charges is also an important factor which affects adsorption.

12.4.1.2 Carbonyl Functional Groups

Carbonyl group (C=O) is also a good adsorbing site for binding of pharmaceuticals (Chang et al. 2009). The interaction of cation with the adsorbent can be easily predicted by the shifting of absorption spectrum for C=O towards the higher or lower

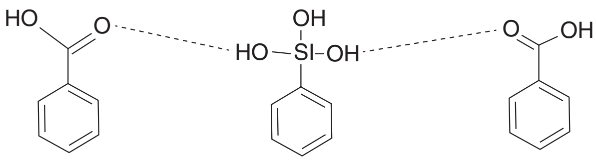

Fig. 12.8 Hydrogen bonding interaction

frequency. For example, adsorption of oxytetracycline onto montmorillonite clay lowers its value from 1685 to 1665 cm^{-1}. For flurbiprofen antibiotic, C=O band is shifted from 1700 to 1708 cm^{-1} after adsorption onto the adsorbent. The binding of C=O group through their charged surface can be explained using cationic exchange mechanism or also through H atom bonded with OH group of water connected to cations on adsorbent surface (Kulshrestha et al. 2004).

The affinity of deprotonated OH group to bind with C=O group is rather greater than the protonated OH group. It was also observed that feldspar or quartz surfaces have deprotonated hydroxyl group attached easily with C=O group of cephapirin antibiotic than to protonated form of hydroxyl group.

12.5 Conclusion and Future Prospects

The environmental impact of pharmaceuticals is not so much clear, and the issue of resolving this problems is quite difficult because science and technology required to fully counter this risk is still in the earliest stages of its development. Human beings are an integral part of this environment. Earlier research has showed that there is inseparable connection between human health and the environmental quality. However, at this moment we need to follow the precautionary principle which signifies that "Any activity which raises threat to human health or the environment, precautionary measures should be taken".

Numerous proactive measures should be taken to reduce the amount of pharmaceuticals introduced to the environment by various actions of general public. Lignin, being a green material, can serve as a better adsorbent for various harmful pollutants (Fig. 12.9; Order Number: 501453669).

Safety and well-being of patients should not be carried out at the expenses of the safety of communities and the ecosystems. High-quality healthcare and environmental protection are intimately linked. Prevention of pollution establishes a hierarchy in the type of measures that should be taken when dealing with environmental risk. In case of hazardous waste, the following hierarchy needs to be followed:

Fig. 12.9 Pictorial diagram of lignin as adsorbent for various pollutants (Reprinted with permission from Supanchaiyamat et al. 2019 copyright (2019) Elsevier (Order Number: 501453669))

- First: minimization/reduction
- Second: reuse
- Third: recycling
- Last: proper disposition (incineration—waste to energy facilities)

Green pharmacy will be a better alternative for the reduction of pharmaceutical pollutants in the coming future.

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