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A review on effective removal of pharmaceuticals from aquatic systems: Advanced techniques and scope for future research

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Abstract

Advances in medical research have led to a significant increase in the consumption and usage of pharmaceutical products. The chemical constituents of these products, including chemicals like tetracyclines, salicylic acids, sulphonamides and quinolones, are collectively known as Pharmaceutically Active Compounds (PhACs). Originating from domestic effluents, this micro-pollutant fails to degrade when processed by conventional methods. Accumulation of these treatment-resistant pharmaceutical compounds in soil and water bodies is dangerous, as they act as poisons to the natural environment. The aim of this study is to consolidate the investigated physical, chemical and biological methods for removal of PhACs.

Microorganisms can facilitate the decomposition of PhACs and are often used in conjunction with membrane technology to effect what is known as Biological treatment. With minimal chemical addition and low energy requirements, it is the most cost-effective treatment plan. Emerging Biological treatment processes include: Membrane Biofilm Reactor (MBFR), Nanofiltration, Reverse Osmosis and Integrated Fixed-film Activated Sludge (IFAS) Systems. Addition of chemicals can either break large molecules into smaller simpler compounds, or improve removal efficacies by forming flocs or a heavier particle mass. Physical methods generally do not succeed in complete remediation. Chemical processes include Fenton Oxidation, Photocatalysis and ozonation processes. However, there is a need to control the potential formation of by-products as well as optimization of parameters to become competitive in economic terms. Thus, advanced techniques like Microbial degradation and Phytoremediation were explored.

Keywords: PhACs, Domestic effluent, Physio-chemical, biological wastewater treatment

1. Introduction

Identifying and investigating budding environmental pollution issues early are critical for protecting ecologic and human health. According to the Indian economic survey 2021, India’s domestic pharmaceutical market is estimated as 42 million USD in 2021 and forecasts this valuation to triple by 2030.

The rapid introduction of new medications to the market is expanding the enormous number of chemical classes, each with distinct biochemical action mechanisms, many of which are still poorly understood. Figure 1 illustrates the most prominent pharmaceutical pollutants.

A dosed user metabolizes pharmacological bioactive elements, excreting metabolites as well as unmodified parent molecules. These can subsequently be processed in sewage treatment plants to undergo further changes. Yet, often biodegradation fails, and the unaltered compounds eventually pollute water bodies they are discharged into. There is also a risk of metabolic conjugates being converted back into their free parent forms. PhACs with low persistence can build into really persistent pollutants if they are continuously infused into the aquatic environment, because their rate of transformation or removal may be neutralized by their replacement rates. As unfortunate as often, untreated sewage is discharged into receiving waters. Disposal into the deep ocean from some locales can still possibly remix with upper waters.

Predominantly, municipal wastewater treatment focuses almost exclusively on conventional “priority” pollutants- heavy metals, pathogens, oxygen demanding substances and other broad spectrum organic and halo-compounds. Accumulation of PhAC over multiple water cycles may be remarkably detrimental to human health, making research on and execution of PhAC removal methods quintessential.

Emerging biological, chemical and physical techniques for PhAC removal have been studied over the years. Each method varies with the extent of remediation, cost effectiveness, by-product formation and time to incur removal. Full scale implementation of these mechanisms needs to be effected to curb the eco-toxicity of PhACs.

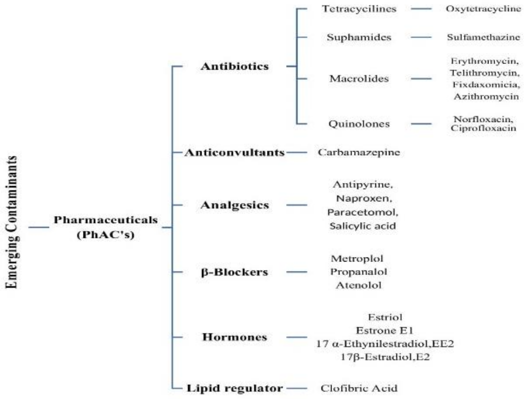

Figure 1 Principle classes of PhACs and representative examples

Environmental Impacts

(PhACs) Pharmaceutically Active Compounds that are profoundly found in alarmingly high concentrations in water discharge systems, fresh water resources and surface waters can cause serious environmental damage due to their toxicological effects. These substances reach water resources through many pathways, a few being- Dumping of household leftover drugs, agricultural facilities, and human excretion after medicinal use and discharge from industries. The global utilization of these PhACs is approximated to exceed (100,00) tons per day. (Randak & Li 2009) Most of these impacts are unknown and hence this issue must be addressed as much as the other major environmental problems. Wastewater Treatment plants (WWTPs) do remove a considerable amount of PhACs, but the others which still remain dissolved-potentially serves to cause a lot of damage to the environment. The slow decomposing of PhACs into the soil makes these soil poisonous and unfit for crop production. The lands furthermore will be abandoned and left barren which leads to soil erosion by wind and water. Exposure to such hazardous waste in the environment causes ill effects to humans, plants and animals. Ill effects can include impaired reproduction, radiation burns, renal failure and Sharps-inflicted injuries. The risk factors and its devasting impacts on the aquatic species which include the impairment of reproduction of the exposed fish populations due to the discharge of these hazardous pharmaceutical Ingredients into the aquatic environment are seen in literature.

2. Techniques for the removal of PhACs:

2.1. Physio-Chemical Treatment Processes:

2.1.1. Fenton Oxidation

Hydroxyl radicals have the unique capability for the oxidation of organic compounds. Under acidic conditions, Fe^{2+} in an environment of H_2O_2 produces OH• radicals by the Fenton process. After Fenton oxidation, the solution's pH is brought up to about 9.0. Subsequently, the Fe^{2+} and Fe^{3+} ions produce nascent compounds of $Fe(OH)_2$, by a rolling sweeping action, hydrogen bond and van der Waals forces, which have a good flocculation effect and help with additional pollutant removal. [1]

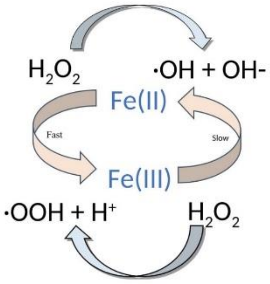

Figure 2 Mechanism of Fenton Oxidation

In the degradation studies of simulated chemical pharmaceutical wastewater by Li et al., the degradation of n-butanol, ethyl acetoacetate, 4, 7-dichloroquinolin and ethyl p-nitro benzoate was effected by 56%, 75%, 100% and 78%, respectively; and 38% of the chemical oxygen demand was expunged by the Fenton process. Analyses also showed that oxidation intermediates of organics significantly improved the biodegradability of SCPW (Stimulated chemical pharmaceutical wastewater).[2]

Excess hydrogen peroxide is commonly employed in the treatment of real wastewater to obtain the desired effect although its availability in Fenton reaction is minimal, it easily decomposes into H2O and O2. However this is expensive.

2.1.2. Photocatalysis

Photocatalysis is becoming a recent attraction in the field of environmental remediation for the removal of PhAC's. The two essential characteristics of photocatalysis to be achieved are: charge separation and effective light absorption (particularly in the visible region). The Photocatalytic experiments are performed in a self-made continuous stirred photochemical reactor, which contains a UV lamp placed in a quartz socket tube with one end closed along with a magnetic stirrer, rotor and reaction vessel with double-jacketed cooling system. The model solution is added inside the reactor and temperature is maintained at 21 ± 2 ºC by continuous circulation of water. Prior to radiation, the measured quantity of photocatalyst is dispersed into the solution and is stirred to achieve equilibrium. After radiation, the model is filtered for analysis. The changes are analyzed using a UV spectrophotometer.

2.1.3. Ozonation

Ozonation and its use in combination with other oxidizing compounds like H_2O_2 (O_3/H_2O_2), and UV (O_3/UV) have been used to efface pharmaceuticals from water. During the treatment, compounds undergo spontaneous transformations, primary degradation products are often degraded further in prolonged treatment.

Variables such as pH, ozone dose, and temperature influence removal and precipitation of pharmaceuticals by ozonation. The ozone required for removing each pharmaceutical varied linearly with DOC (Dissolved organic carbon) content, and suspended solids only had a minor influence on the oxidation efficiency of nonsorbing micropollutants. Due to the short half-life of ozone, this method is expensive and needs high energy for real application. The disappearance of parent compounds does not necessarily indicate successful treatment, since the degraded by-products may be just as biologically active as the parent compounds. Therefore, evaluating the toxicity before and after treatment by ozonation is necessary.

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2.2. Biological Treatment

2.2.1. Nanofiltration

Nanofiltration (NF) a liquid-phase separation process, is one among the four membrane technologies. NF uses a nano pore size of 1 to 5 nm. It usually operates at a pressure of around 7- to 30 for the separation of solutes of compounds with low molecular weight. It is a pressure driven membrane process which is considered a suitable method for its effective in rejecting dyes and in the removal of most of the viruses, organic matter, and salts.[3]

The PhAC elimination shows limited success using traditional treatment processes.[4] Acid cross-linked Polyamide membranes were grafted along with ethylene diamide for the separation of a compound called bisphenol A (BPA). This resulted in 95% rejection of BPA. [5] For the removal of substances like cyclophosphamide, NF membranes with a hollow fibre, a charged surface and cross linked with ED were used.[6] The above which resulted in the highest rejection of ciprofloxacin. A hybrid NF combined with AOP showed the effective rejections of antibiotics, naming a few: norfloxacin (NOR) and azithromycin, up to 98% from a wastewater treatment plant. [7]

NF process feed is passed through a semi permeable membrane where the stream is divided into two major streams- the stream containing the permeate and the stream containing the retentate. The process flow diagram of NF is shown in the below diagram.

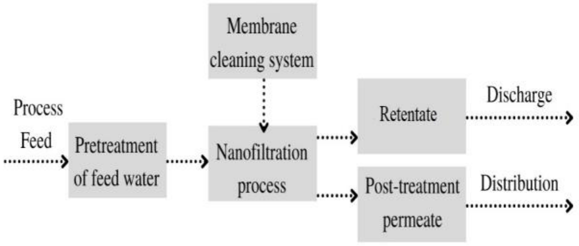

Figure 3 Process flow diagram of NF

Nanofiltration helps with the elimination of calcium and magnesium ions which cause hardness in water with no accumulation of sodium ions during filtration. It also requires no cooling or heating of feed thus making it cost effective. The major advantage of NF is that it handles a massive volume of feed in a continuous manner at a stable rate of permeate.

2.2.2. Reverse Osmosis

Reverse Osmosis is the most widely used membrane filtration method. The system contains a thin semi-permeable membrane with tiny pores of size around 0.1 to 0.0001 micron, which allows passing of only pure water leaving behind contaminants like ionized dissolved salts, dyes, bacteria and viruses- termed concentrate. The driving force behind the process is (100 to 1000 psig) usually by a pump to overcome natural osmotic pressure. Industries use multiple membranes in series for large volumes of water to achieve greater levels of treatment. Cross-flow filtration cleans the membrane's surface at regular intervals keeping it clean and neat.

Rejection of antibiotics from pharmaceutical wastewater was found to be >98% when combined with NF.[8] Cyclophosphamide(CP) is among the most profoundly used medications in chemotherapy, found to affect many aquatic organisms. Following the study of [9] the rejection of CP in feedwater using RO and NF hybrid systems such as MBR-NF and MBR-RO systems, CP retention in RO systems was found to be up to 90% whereas only 20-40% for NF.[10] For the effective removal of PhACs, AOP combined with RO systems was found to be an alternative approach. PhACs were removed by RO and the retentates were furthered treated by AOP. The ozonation of RO brine effluents was also another parallel process for the elimination of some frequently used drugs such as metoprolol and propranolol, classified as powerfully toxic to the living organisms present in water. The below table 1 shows the recovery percentage of the targeted compounds.

RO systems permit recycle/recovery of waste process streams, require very less energy, and operate at considerably ambient temperature hence reducing corrosion issues and formation of scales.RO is considered to be one of the greenest solutions as concentration and separation is achieved without the use of chemical compounds. RO is eco-friendly and also very cost effective. An RO membrane can last up to a period of 2-3 years and requires minimal operator interaction making it maintenance friendly as well.[11]

2.2.3. Activated Sludge

Activated sludge (AS) treatment utilizes an aeration basin and secondary clarifier, to degrade organic matter in wastewater such as food waste and fecal matter. Microorganisms in the wastewater inlet to the aeration tank colonize and metabolize organic waste to ATP for further cell growth, producing CO_2 and water. Since the microorganisms need oxygen to function, the tank is aerated with diffuser systems.

In studies of this treatment by J.A. Mir-Tutusaus, A. Jaén-Gil, Barceló et al., elimination of 73% of the initial pharmaceutical concentrations was effected, 92% disregarding the analgesics and anti-inflammatories. A complete removal of the analgesics and anti-inflammatories acetaminophen, ibuprofen and naproxen was seen, demonstrating the literature regarding these compounds. The antibiotic ciprofloxacin was also effaced by nearly 75% due to activated sludge treatment, by adsorption to the biomass, commonly seen in full-scale wastewater treatment facilities. Gemfibrozil was reported as a recalcitrant. [12]

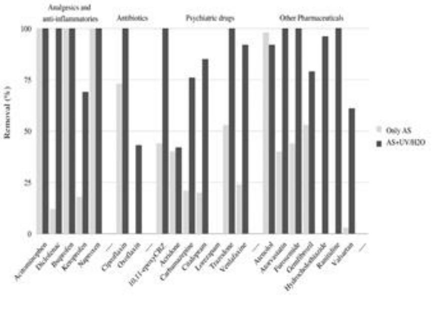

Figure 4 Cumulative removal percentages of PhACs with AS+UV/H_2O

Placing UV/H_2O_2 after the AS, as shown in fig.4, significantly improved the overall PhACs removal. Since OH radicals produced on UV exposure treatment have non-selective reactivity to organic materials, the extent of degradation of PhACs is subpar when treating matrices with high COD or TSS content. Thus, UV/H_2O_2 exposure, when conducted after biological processing, is capable of increasing total PhACs removal by up to 95% when compared to cases without. [13]

2.2.4. Enzyme Bioreactor

An enzymatic membrane bioreactor (EMBR) is a class of enzymatic treatment, which utilizes filtration techniques to prevent loss of enzymes. It retains them in the reactor and enables their replenishment during continuous operation. Amidst the oxidoreductase enzymes, laccases have been in the limelight owing to their ability to degrade phenolic and some non-phenolic compounds. Studies of EMBRs dosed with laccase report removal of a broad spectrum of pharmaceuticals that non-specific biological remediation techniques fail to. Redox mediators form reactive radicals when oxidised by laccase and improve degradation. Any existing effects of steric hindrance can be overcome as these reactive radicals act as ‘electron shuttles’ between laccase and the substrate. [14]

In the work by Sidy Ba et al. 2014, a novel hybrid bioreactor (HBR) of cross-linked enzyme aggregates of laccase (CLEA-Lac) and polysulfone hollow fiber MF membrane was developed to eliminate acetaminophen (ACT), mefenamic acid (MFA) and carbamazepine (CBZ) as model aromatic pharmaceuticals. The study effaced the three drugs in varying degrees from the filtrate of aqueous solution, approximately from 50 to 90% over the course of 8 hours. Synergistic action of the CLEA-Lac during operation was able to eliminate from aqueous solution around 99%, nearly 100% and up to 85% of ACT, MFA and CBZ, respectively. Under continuous operation, the hybrid bioreactor demonstrated removal of the drugs from wastewater CBZ eradication upto 93% after 72 hours, and veritable elimination of ACT and MFA was achieved within 24 hours of treatment.[15].

2.3. Advanced Techniques

2.3.1. Microbial Degradation

The process by which living microbial organisms break down the organic substances into minor compounds is referred to as microbial degradation. Organisms like fungi, bacteria, yeast, algae and protozoa are widely used in this process. These organisms with degradation potential turn contaminants into harmless products.

2.3.1.1. Aerobic Treatment:

The presence of aerobic bacteria along with other microorganisms play an important role in this process. A adequate amount of oxygen must be provided to the bacteria for the treatment to be ensue.

Murphy et al.,1995’s explains the aerobic treatment plant, specifically constructed for the treatment of pharmaceutical wastewater. The construction of the plant included a reactor through which air was allowed to pass in the bottom. The maximum (HRT) hydraulic retention time was estimated to be around 15 days.[16] For the evaluation of the plant’s efficiency, test samples were collected and allowed to test for general parameters. The study resulted in 75% of COD removal in the estimated 15 days of HRT. The other parameters such as color, TSS, TDS, pH, BOD revealed the analyses to be within the prescribed limits. The study also concluded that the major factor that accelerated the biological treatment process was the injection of air in the reactor. Air injection also had an influence on the removal of COD from the wastewater. With such high potential for the removal of both COD and TSS this plant proves to be a budding advancement in treating industrial wastewater. The plant is eco-friendly causing no harm to mother nature with the minimal use of chemicals. The plant can be operated in simple ways for the treatment of industrial effluents and is also cost effective.

The aerobic process takes place in two consecutive steps represented as follows as stated by Ros and Zupancic, 2002:

NH_4 + Carbon-based matter + O_2 + H_2O + Energy + CO_2 ------(1)
Energy + O_2 digested sludge + NO_3 + CO_2 + H_2O --------(2)

As in (1) Organic matter is oxidized straight into decomposable matter and later oxidated to digested sludge. [17]

2.3.1.2. Anaerobic Treatment:

Anaerobic treatment in contrast with aerobic treatment is comparatively inefficient and sluggish. Anaerobic wastewater treatment is the treatment by which degradation of organic matter by the microorganisms take place in the absence of oxygen. Anaerobic wastewater treatment is habitually used in the non-biological wastewater. Wastewater containing high concentrations of organic matter are treated anaerobically as it allows high loading of organic matter, limited sludge production, requirement of a reduced amount of nutrients, provides with biogas energy recovery, acceptable for a widespread range of temperature, limited energy consumption and abundant anaerobic sludge retention time when compared to the aerobic treatment process.

Table 1. Microbial Degradation: Pros and cons of Aerobic and anaerobic processes.

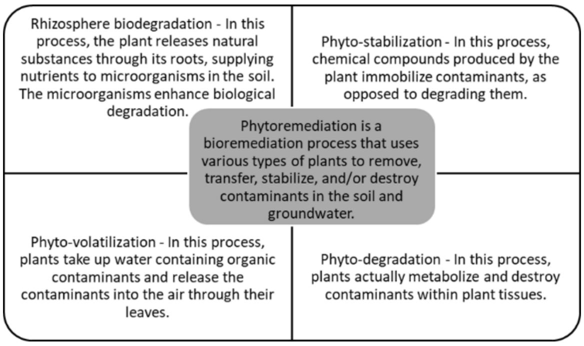

Figure 5 Types of Phytoremediation

2.3.2. Phytoremediation

Phytoremediation is a wastewater treatment technology that relies on florae and their allied microbes in the rhizosphere to remove or convert any harmful chemicals. This technique may be used to purify contaminated soil, the groundwater it holds, polluted water bodies and even atmospheric pollutants. The primary mechanisms which constitute phytoremediation include phytostabilization, phytoextraction, phytoaccumulation, phytovolatilization, phytotranspiration, phytodegradation/transformation, endophytic degradation and rhizosphere remediation. These operations occur at the same time and to fluctuating degrees.

Biotransformation techniques like phytoremediation have been seen to effect degradation of pharmaceutical pollutants into stable intermediates, with only a small fraction being fully mineralized into smaller constituents.

Constructed wetlands are promising in beneficially removing more than 70% of the medical compounds which are counted in the European Union watch list, which include antibiotics and synthetic hormones.[18] Pharmaceutical wastewater flows from the septic tank in which it was stored, or is released subsequently from a primary wastewater treatment system, on topsoil (surface) or through a porous medium such as gravel (subsurface). A dense mat of sorts is formed by interlinked roots and stems of the plants, where chemical, biological, and physical processes occur to treat the wastewater.

Another study by Y.Li et al. studied the action of rhizosphere on scale of six microcosm built wetlands to treat ibuprofen (IBP) polluted wastewater, during the development of *Typha latifolia*. The growth of plants in these wetlands helped in the removal of toxic pollutants especially during the mature stage of plant development when compared with the IBP free wetlands. [19]

In spite of the recent advances (excluding diclofenac that has been scrupulously investigated), only a few studies have been implemented on the metabolic transformation of PhACs in plant tissues, even less is known of their intermediate transformation products.

Phytoremediation is restricted to the surface area of the plants and to the depth engaged by the plant roots. It is also challenging to entirely prevent contaminants leach into the groundwater. The healthy and regular growth and existence of these plants is affected by the harmfulness of polluted lands and the innate fertility of the soil. Heavy metals often become unavailable for phytoremediative extraction, as they bind irremovably to organic soil compounds.

Treatments	Aerobic	Anaerobic
Adaptation Temperature	Adapts high	Room Temperature
Pressure	Atmospheric	Non-Atmospheric
Elimination	Thoroughly	Not entirely
Costs	Low	Low
Abode	Limited hours	Limited hours

Conclusion:

In conclusion, advanced methods of remediation have consistently exhibited better degrees of PhAC removal efficiency, vastly better than physio-chemical methodologies, and slightly better than the adequate biological methods. They also promote a circular economy, with microorganisms and plants thriving naturally whilst recycling toxic PhACs. However, UV exposure is seen to improve the subpar efficiencies of oxidation and ozonation reactions. Hybrid membrane technology has also proved to effect pharmaceutical removal to nearly 98%.

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