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Review

Integrated and Hybrid Processes for the Treatment of Actual Wastewaters Containing Micropollutants: A Review on Recent Advances

Mina Asheghmoalla and Mehrab Mehrvar *

Department of Chemical Engineering, Toronto Metropolitan University, 350 Victoria Street, Toronto, ON M5B 2K3, Canada; mina.asheghmoalla@torontomu.ca

* Correspondence: mmehrvar@torontomu.ca; Tel.: +1-(416)-979-5000 (ext. 556555); Fax: +1-(416)-979-5083

Abstract: The global concern regarding the release of micropollutants (MPs) into the environment has grown significantly. Considerable amounts of persistent micropollutants are present in industrial discharges. Depending solely on a singular treatment approach is inadequate for the effective removal of MPs from wastewater due to their complex composition. The performance of different treatment methods to meet the discharge standards has been widely studied. These efforts are classified as hybrid and sequential processes. Despite their adequate performance, the optimization and industrial application of these methods could be challenging and costly. This review focuses on integrated (sequential) and hybrid processes for MP removal from actual wastewater. Furthermore, to provide a thorough grasp of the treatment approaches, the operational conditions, the source of wastewater containing MPs, and its characteristics are detailed. It is concluded that the optimal sequence to achieve the removal of MPs involves biological treatment followed by an advanced oxidation process (AOP) with a final passage through an activated carbon column. To refine this process further, a membrane unit could be added based on the desired effluent quality. Nevertheless, considering practical feasibility, this study identifies specific areas requiring additional research to implement this integrated treatment strategy effectively.

Keywords: wastewater treatment; micropollutant removal; integrated treatment technologies; advanced treatment methods; advanced oxidation process; adsorption; membrane bioreactor

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1. Introduction

Global population growth, coupled with an inadequate discharge of wastewater, has greatly increased freshwater pollution. Hence, access to clean water has become the most pressing issue facing people in many parts of the world and is predicted to worsen with the diminishing availability of freshwater resources. For this reason, recycling, reusing, and minimizing wastewater have become the new focus of treatment plants to minimize the discharge of waste to the environment at the point of generation [1,2].

Further, the micropollutants (MPs) in water, even at a trace level, may have a toxic effect on aquatic life and cause antibiotic (Ab) resistance [3]. These so-called emerging contaminants (ECs) comprise a diverse array of pollutants classified into four categories. The first category encompasses metals and metalloids, such as lead, mercury, arsenic, and cadmium, originating from industrial discharges and agricultural runoff. The second category involves organic micropollutants, including synthetic and naturally occurring compounds like pesticides, herbicides, and industrial chemicals. Their resistance to conventional wastewater treatment processes makes their removal particularly challenging, raising concerns for environmental and human health [4–7]. The third category of MPs comprises steroid hormones, such as estrone and testosterone, known for their endocrine-disrupting effects. These hormones enter water bodies through agricultural runoff and wastewater discharge [8,9]. The fourth category includes pharmaceuticals, arising from widespread

medication use, that enter water sources through industrial and sewage discharges. Antibiotics, painkillers, and various therapeutic drugs contribute to concerns about antibiotic resistance and potential health risks [10–13]. Many of these MPs are removed by conventional treatment; however, some remain unaffected and enter the environment [14,15]. Conventional wastewater treatment plants effectively reduce suspended solids and remove nutrients and biological oxygen demand. Thus, their poor performance in MP removal is justifiable, considering the nonbiodegradable nature of many MPs [16]. Therefore, developing advanced wastewater treatment technologies with high levels of treatment efficiency has become increasingly necessary [17,18].

Conventional treatment plants mainly consist of physical, chemical, and biological unit operations. Physical treatment technology does not degrade the pollutants, resulting in pollutant removal only from water and includes processes like screening, mixing and flocculation, gravity sedimentation, grit removal, floatation, and clarification [19,20]. During chemical treatment, the structure and properties of pollutants change because of the chemical reaction between an oxidizing agent and the pollutants [21]. This treatment includes coagulation, precipitation, chemical oxidation, chemical neutralization, chlorination, UV oxidation, catalysis, ozonation, electrochemical oxidation, and wet oxidation [22]. Conventionally, biological processes, in the form of aerobic and anaerobic processes with the suspended or attached growth of microorganisms, are widely used to treat wastewater, in which organic pollutants are consumed as a carbon source. Moreover, these biological processes are cost-effective and well-established methods in water and wastewater treatment [23,24].

Biodegradation is the degradation of contaminants by naturally occurring microorganisms in the environment. During biodegradation, the organic pollutants are first subjected to reactions like hydrolysis, oxidation, alkylation, and dealkylation; then, the formation of conjugates facilitates digestion [25]. However, due to the toxicity and nonbiodegradable nature of many industrial chemicals, conventional biological treatment does not always provide desirable outcomes. Among aerobic biological processes, activated sludge (AS), trickling filters, and membrane bioreactors are the most prevalent treatment techniques [26].

The performance of biological processes varies significantly based on the temperature, aeration rate, nutrient supply, treatment time, acclimation period, type of microorganism, characteristics of influent, and initial concentration of pollutants. The biodegradability of wastewater is the main factor for determining the usage of biological treatment. The biodegradability of wastewater depends on the types of pollutants and is determined by the ratio of the 5-day biological oxygen demand to the chemical oxygen demand (BOD_5/COD). Wastewater with a BOD_5/COD ratio of 0.5 or greater is considered biodegradable.

Advanced treatment methods with a high rate of micropollutant removal have proved their superiority in various studies and can be added to a conventional treatment plant [27,28]. Although advanced technologies surpass conventional treatment techniques, increasingly stringent regulations for the discharge of wastewater containing micropollutants have made the application of a single advanced treatment technique insufficient to reduce micropollutant concentrations to meet discharge limits. Moreover, even if a single treatment achieves the desired removal, it might not be economically feasible to upgrade a treatment plant, which imposes the use of hybrid or integrated (sequential) advanced processes so that the effluent has an acceptable quality, and the process becomes cost-effective [29–31]. Hybrid processes are those treatment technologies whose performance has been intensified by fusing one or more treatment technologies into one unit. In contrast, integrated processes are defined as a sequence of two or more advanced treatment techniques [21].

Thus, due to the growing concern regarding the release of micropollutants and the inefficiency of conventional treatment techniques, numerous advanced treatment technologies have been studied by researchers using synthetic wastewater. Synthetic

wastewater provides sufficient insight into the mechanisms of the treatment process itself and the degradation pathway that certain pollutants go through; nonetheless, the true performance of the process and operating challenges are only discovered when actual wastewater is used [21,32,33]. Actual wastewater is a complex matrix with many unknown pollutants; therefore, each high-efficiency treatment method needs to be further studied for its performance using these types of wastewaters to validate its large-scale feasibility.

To date, numerous studies have compiled the latest advancements in the removal of pharmaceuticals, antibiotics, and micropollutants from wastewater with various advanced treatment technologies, including advanced oxidation processes, activated carbon adsorption, membrane filtration, advanced biological processes, and combined approaches [13,22,34–42]. Moreover, Bui et al. [18] reviewed the removal of micropollutants with various industrial-scale treatment technologies, providing a foundational understanding. Motivated by their work, this study specifically focuses on the state of the art from 2016 onwards. Despite several studies focusing on MP removal from wastewater, limited research has been dedicated to treating actual wastewaters containing micropollutants by hybrid and integrated treatment technologies. Hence, this study aims to review recent trends in hybrid and integrated treatment technologies for the removal of micropollutants from real wastewater and to provide a base for further studies. This paper first goes through the characterization of micropollutants containing wastewater from different sectors. It then discusses current advanced treatment technologies used in wastewater treatment and later reviews various case studies that have used hybrid and integrated treatment technologies to remove micropollutants. Finally, the prospects of treatment techniques, findings, and recommendations for further studies are discussed to serve as a guide for future research areas.

2. Regulations for MP Removal

Various organizations and regulatory bodies, such as the Environmental Protection Agency (EPA), U.S. Food and Drug Administration (FDA), World Health Organization (WHO), Canadian Ministry of Environment and Climate Change, and the Water Framework Directive, play a role in establishing limits and guidelines for water and wastewater quality. These limits aim to control the quantity and impact of pollutants released into sewer systems and surface water [43–45]. In Europe and the United States, regulations are set around the pollutants affecting drinking water. However, Australia has implemented comprehensive guidelines specifically targeting the discharge of secondary-treated wastewater, including 34 priority micropollutants, mainly pharmaceuticals. On the other hand, Switzerland has taken a pioneering approach by setting a limit for the removal of micropollutants at the point of generation, requiring treatment processes to achieve an 80% removal in their effluent by monitoring the concentration of five indicators, including benzotriazole, carbamazepine, diclofenac, sulfamethoxazole, and mecoprop [18]. These initiatives aim to safeguard water quality and mitigate the environmental impact of industrial wastewater and wastewater treatment plants [46,47].

Table 1 shows the effluent quality required for wastewater disposal into surface water. In Europe, effluent quality standards for urban wastewater treatment plants are determined according to the plant's size. Smaller treatment plants serving a limited population can maintain a lower effluent quality, while larger treatment plants are subject to more stringent standards. For properties such as TP, TN, and TSS, two thresholds are defined based on the population equivalent (p.e.), a term used to express the strength of pollutants in the wastewater produced by industry. It denotes the quantity of substances whose biological oxygen demand matches the average BOD of the wastewater generated by an individual. In other terms, one p.e. is equal to the BOD_5 of the wastewater with respect to 60 g of oxygen in 24 h [29]. Moreover, as per the Urban Wastewater Treatment Directive, a minimum removal of 70–90% in BOD_5, 75% in COD, 80% in TP (total phosphorous), 70–80% in

TN (total nitrogen), and 70–90% in TSS (total suspended solids) is required prior to the discharge into surface water [48].

Table 1. Discharge requirements to surface water based on the maximum monthly average from urban wastewater treatment plants (Canada and European Union) and pharmaceutical manufacturing industry (USA) [29,43–45,49].

Composition	Canada	USA	European Union	India
BOD_5 $(\frac{mg}{L})$	25	111	25	30
COD $(\frac{mg}{L})$	-	856	125	250
TP $(\frac{mg}{L})$	1 $\frac{mg}{L}$	-	2 [1] 1	-
TN $(\frac{mg}{L})$	3–5 $\frac{mg}{L}$	29.4 $\frac{mg}{L}$ [2]	15 [1] 10	100
TSS $(\frac{mg}{L})$	25 $\frac{mg}{L}$	166 $\frac{mg}{L}$	60 [1] 35	100
pH	6–9.5	6–9	6–10	5.5–9

[1] The higher limit corresponds to population equivalent (p.e.) $= 10^4 - 10^5$, and the lower limit corresponds to p.e. $> 10^5$. [2] Equivalent to ammonia nitrogen ($N\text{-}NH_3$).

As indicated in Table 1, the discharge limit significantly varies based on the country and environmental policies with the US having the highest maximum discharge tolerance compared to all other listed countries and the European Union defining the maximum discharge limit for all the wastewater surrogate properties based on the population equivalent. The criteria for BOD_5, TP, and pH align between Europe and Canada. Furthermore, all countries listed in Table 1, except for Canada, monitor and regulate COD as a parameter. The United States has established comprehensive regulations for wastewater discharge, categorically defining limits based on the pollution source. The Code of Federal Regulations outlines discharge limits for various industries, encompassing, among others, pharmaceutical manufacturing, hospitals, and organic and inorganic chemical and pesticide manufacturing [50].

In summary, effective regulation for wastewater discharge is crucial for maintaining environmental standards and safeguarding water resources. However, beyond adhering to regulatory frameworks, it is equally important to characterize the wastewater. The nature and composition of wastewater from diverse sources vary significantly, affecting the selection of appropriate treatment technologies. Understanding and characterizing wastewater, including its strength, pollutants, and unique properties, become important in ensuring the success of treatment processes. By focusing not only on regulatory compliance but also on the wastewater properties, a more comprehensive and sustainable approach can be adopted for wastewater management. This shift in perspective acknowledges the challenges posed by variable wastewater compositions and emphasizes the importance of solutions for effective environmental protection.

3. Characterization of Actual Wastewater Containing MPs

Industrial wastewater, such as from pharmaceutical production plants, contains a considerable amount of micropollutants; however, the nature of the wastewater varies significantly. The treatment technique is selected based on the wastewater properties, source of generation, and point of discharge. Although many processes treat a certain type of wastewater well, they might perform poorly in the treatment of wastewater with properties other than the tested original, and this discrepancy comes from variations in the strength of wastewater and pollutants in the water matrix. The assessment of wastewater strength has been overlooked in many regions when establishing guidelines for designing onsite treatment. The conventional design and sizing of onsite wastewater treatment

systems usually rely on standard ranges set for domestic wastewater, if wastewater strength universally falls within these limits. However, this assumption may not hold for industrial establishments, where higher wastewater strengths or the presence of harmful chemicals used in processing or cleaning activities may be prevalent [51,52].

Key parameters such as BOD_5 and TSS serve as essential indicators of wastewater strength and biodegradability. A failure to address high-strength wastewater, defined by BOD_5 exceeding 1000 mg/L and TSS more than 350 mg/L, has been associated with early failures of onsite treatment systems [52]. Therefore, understanding the strength and characterizing the wastewater are inevitable for ensuring the effectiveness of treatment processes. Various types of wastewaters containing MPs include but are not limited to those originating from hospitals, agricultural and pharmaceutical manufacturing industries, and municipal wastewater [4,10,11,13]. As the effluents of these sectors are some of the main contributors of micropollutants in water, this study has characterized their effluents based on surrogate properties reported in the open literature [3,16,53,54]. Table 2 shows the properties of wastewater from various sources. From this table, it is observed that characteristics of wastewater vary vastly, even in one category.

Table 2. Characterization of actual wastewater containing micropollutants based on the point of generation and their bulk properties.

Surrogate Properties	Municipal	Agricultural	Pharmaceutical	Hospital
COD $\left(\frac{mg}{L}\right)$	250–1000	27–2750	128–65,000	74–7800
BOD_5 $\left(\frac{mg}{L}\right)$	1–400	90–2000	4–3100	56–2900
TOC * $\left(\frac{mg}{L}\right)$	6–168	10–167	850–17,000	30–3100
TSS $\left(\frac{mg}{L}\right)$	7–1220	3–700	19–450	97–3260
TP $\left(\frac{mg}{L}\right)$	0.09–29	73–392	0.4–220	1–27
TN $\left(\frac{mg}{L}\right)$	6.5–22	24–200	8–4000	9–340
pH	6–8	6–9	0.34–14	6–9
References	[55–61]	[62–66]	[40,67–72]	[73–79]

* Total organic carbon.

Using Table 2, the following conclusions are drawn:

- Wastewater from hospitals and agricultural activities stands out as having high concentrations of suspended solids and biological oxygen demand. The maximum COD level for these specific wastewater types is considerably lower compared to that of wastewater originating from the pharmaceutical industry. This implies that integrated filtration and biological treatment could potentially treat the wastewater from hospitals and agricultural activities.
- The pharmaceutical industry produces wastewater with high chemical oxygen demand, total nitrogen (TN), and total organic carbon (TOC); as well as a wide range of pH, indicating that advanced treatment technologies along with conventional methods might be the only option for effective treatment.
- Municipal wastewater has the mildest properties among all other classes in terms of total nitrogen, phosphorus, and chemical and biological oxygen demand, leaving the choice of treatment techniques to local authorities and owners with the desired removal level.

In summary, the characterization of wastewater containing micropollutants underscores the considerable variation in the physical and chemical properties of the wastewater. The wide range of characteristics presents a considerable challenge in efficiently eliminating MPs. Numerous review studies provided insights into the limitations of the conventional treatment approaches and underscored the need for advanced treatment methods capable of managing the complex nature of wastewater containing MPs [14,25,40,42,79,80].

The next section explores various advanced treatment approaches that have proven effective in addressing micropollutant removal from various sources of wastewater and contributes to a thorough comprehension of the challenges faced by the application of each treatment method.

4. Advanced Wastewater Treatment Technologies

To date, conventional treatment has been successful in removing nutrients and basic organic matter; nevertheless, their poor performance in removing MPs enabled the development of low-cost treatment methods as a complementary step for the current treatment plants [1,18]. Part of the emerging contaminants are removed during conventional treatment by sorption to sludge or stripping, coagulation, and flocculation; however, some will escape the treatment and accumulate in the environment [3,81,82]. As a result of the versatile characteristics of wastewater, different advanced treatment processes, including membrane technology, adsorption, advanced oxidation processes (AOPs), and advanced biological treatment, are used for MP removal from wastewater [18,83–87]. The raw wastewater characteristics, desired final effluent quality, process cost and performance, and the possibility of upgrading the current treatment plants are factors affecting the selection of the treatment methods. The following sections summarize the mechanism of these treatment methods, and the parameters affecting their performance are discussed.

4.1. Membrane Processes

Membrane processes are used for the removal of pollutants from wastewater. Various pollutants and solid particles are effectively eliminated by this process. The separation is achieved by employing semipermeable materials that separate water from contaminants by filtration and sorption to the surface. The two streams produced after the separation are treated water and the concentrated stream which is held by the membrane module. Various polymer and polymer blends, as well as metals, ceramics, and glasses, can be used for membrane production, creating distinct membrane properties. The types of contaminants that can be retained by the membrane relies on the membrane characteristics. The module pore size can range from 0.09 to 0.2 µm, providing a total filtration area of about 45 m^2 [29,80,88].

The driving force in membrane separation processes can be based on pressure, including microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO), with the latter as the most effective technology, mainly used in water reclamation plants as it filters pathogens and viruses along with inorganic salts and other organic compounds. Furthermore, applying an electric field also separates the anion and cation in the water matrix in electrodialysis with the aim of water desalination [22,40].

Recently, the application of membrane processes for the removal of micropollutants is of interest due to their improved performance in water treatment. Studies have shown that UF and NF could achieve up to 80% and 96.5% of antibiotic removal, resulting in their popularity for industrial applications in removing MPs [89–91]. Nonetheless, they fail to remove some pollutants such as Bisphenol A, NP, NDMA, B-estradiol, and caffeine [18].

Upgrading wastewater treatment plants with membrane processes for water decontamination eliminates the need for a secondary clarifier, tertiary filtration, and disinfection. It reduces the overall treatment cost, total suspended solids, required process space, and aeration tank footprint. Moreover, it is advantageous in terms of less energy consumption and environmental pollution, process simplicity, and ease of automation [29,40]; however, the membrane processes only remove the pollutants from water. The contaminants in the concentrate stream still need further processing to be eliminated without returning to the environment. In addition, the water needs to be pretreated before the membrane process to reduce solids in water to avoid membrane blockage. Hence, in the design of the membrane treatment stage, the adequate membrane flux, easy module disassembly, replacement, and cleaning procedure need to be considered [22,29,40]. Due to these disadvantages, reducing membrane fouling by modifying its surface properties, and using destructive

treatment techniques have been extensively studied. To address these challenges, hybrid systems incorporating photocatalysts and membranes called photocatalytic membrane reactors (PMRs) were developed with ceramic membranes and UV light with TiO_2 as an AOP [92,93].

4.2. Advanced Oxidation Processes (AOPs)

AOPs, generators of hydroxyl radicals, are effective methods for the degradation of organics in wastewater. Their efficiency is based on the rate at which radicals are being generated, where, as a result of their reaction with the pollutants, simpler organic matter is produced, or complete mineralization is achieved [94].

In AOPs, radicals are generated in different ways, including H_2O_2, O_3, UV light, ultrasound irradiation, and catalysis, which produce products that are simpler in their chemical structure, and consequently biodegradable compounds like acetic acid and maleic acid [21,95]. The process efficiency depends on pH, the presence of background natural organic matter and radical scavengers in the water matrix, lamp technology, the concentration of oxidants, the reactivity of pollutants with an oxidizing agent, the initial concentration of pollutants, treatment time, the geometry of the reactor, and the type of catalyst [96,97]. AOPs can be categorized as Fenton-based, ozone-based, UV-based, and catalysis and include energy-assisted processes, as shown in Figure 1.

The effectiveness of this treatment technique in removing micropollutants has been proven in various lab-scale and large-scale applications. Reaction times ranged from 5 to 180 min, achieving a removal degree of 70 to 100% for ozonation, 40 to 100% for chemical-based processes, 90 to 98% for ultrasound-assisted photochemical oxidation, and 80 to 100% for Fenton-based processes [24,34,36,98–100]. Although the removal efficiency of micropollutants through AOPs is significant, many challenges limit their application at the industrial scale. Among these challenges, the possible increase in ecotoxicity and mutagenicity because of the formation of oxidation by-products such as bromate, phenolic intermediates, and nitrosamines could be mentioned [16,101,102].

The main advantage of the AOP is its capability to degrade various types of pollutants with minimal or no solid waste generation, improving biodegradability and reducing toxicity [95]. However, the complete degradation of contaminants in AOPs requires high energy consumption, which results in using this method prior to biological processes for biodegradability enhancement [103]. Moreover, the partial reaction during the oxidation process may produce more toxic intermediates than their original form; hence, thorough knowledge of the possible generated intermediates is needed. In a real water matrix with a complex structure, achieving full degradation pathway knowledge is unlikely. Thus, the need for another treatment method after AOPs seems inevitable [104]. Recent studies in AOPs focus on a more sustainable process by minimizing energy consumption, reducing waste, and improving catalytic activity [105,106].

4.3. Adsorption

Adsorption is a process through which pollutants are separated from a liquid phase and accumulated on a solid surface. The process can be physical, in which the matter adsorbs on the adsorbent with Van der Waals forces or electrostatic interactions. Various regeneration methods are used in physical adsorption to recover the adsorbent, while in chemical adsorption, the adsorbate is attached to the adsorbent's surface with strong covalent or ionic bonding, which is an irreversible phenomenon [107]. Adsorbents in wastewater treatment can be classified as polymeric resins, natural adsorbents like sand and zeolites, and carbon-based materials. Adsorption is mostly used after a secondary biological treatment to separate the suspended solids and reduce the biological oxygen demand (BOD) in the effluent [29,108].

Activated carbon (AC) is widespread in water works due to its nonselective nature, well-developed porous structure, and high adsorption capacity [31,109]. Therefore, it removes various types of contaminants, including refractory and inorganic com-

pounds [32,110]. In water reuse applications, it is mainly used to remove odour, taste, and turbidity from water, and in industrial effluents, it is used to remove toxicity and natural organic matter and to meet environmental discharge standards [40,111].

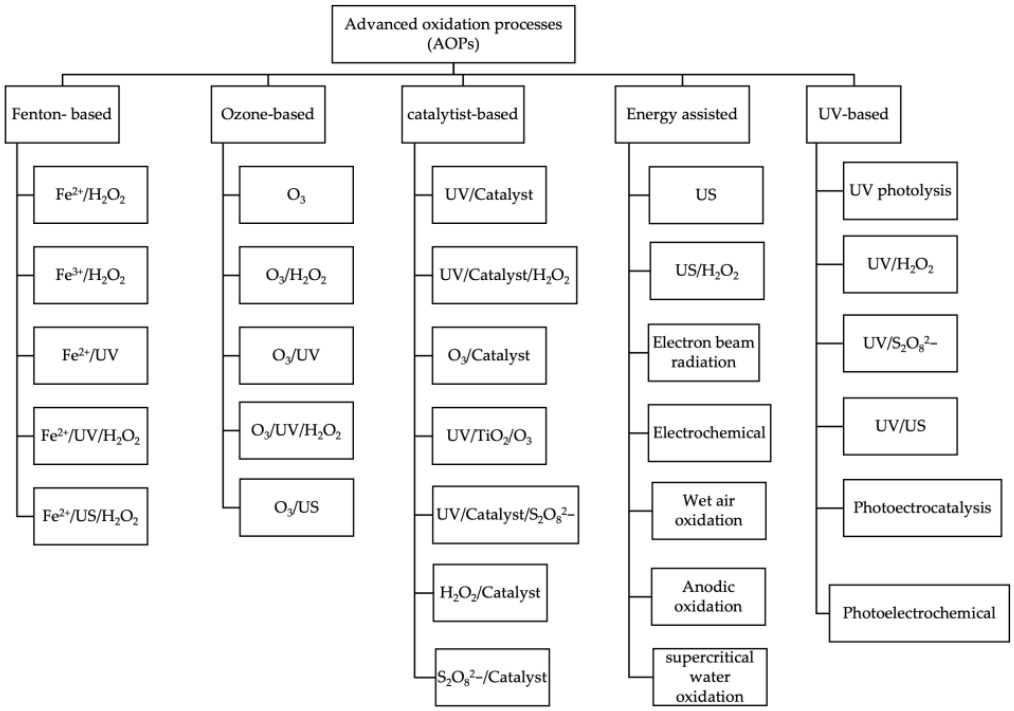

Figure 1. Classification of advanced oxidation processes (adapted from [105]).

Activated carbon is produced in two stages from various carbonaceous materials; first, the organic material is carbonized, and the char is made at a high temperature of up to 800 °C; then, it is activated by gases such as CO_2 and steam at 900 °C when its porous structure is developed [37]. This adsorbent is classified into two size categories: powdered activated carbon (PAC) with a particle size of less than 0.2 mm and granular activated carbon (GAC) with sizes ranging from 0.2 to 5 mm. The process benefits from the recyclability of the spent adsorbent, the high treatment efficiency, and the wide application range; however, it can be expensive and complex in terms of the preparation of activated carbon and its regeneration. Moreover, the regeneration efficiency is usually low; therefore, the adsorption capacity constantly deteriorates and generates secondary waste, which needs further waste management [38,40].

When all the adsorption sites are saturated with pollutants, no more pollutants will be removed from the wastewater, and the adsorbent should be replaced with a virgin one or regenerated. The regeneration of spent activated carbon takes place by using chemicals to oxidize the pollutant on active sites, solvents to desorb the adsorbate, and thermal and microbiological regeneration with the simultaneous degradation of pollutants. Even though the thermal regeneration of GAC accounts for 75% of operational costs, it remains the most popular regeneration method. In contrast, the regeneration of PAC is not yet fully established, and it is often disposed of as solid waste or returned to a bioreactor to improve the biological treatment's efficacy by simultaneous adsorption and biodegradation [41,111,112]. The use of solvents for the regeneration of GAC, on the other hand, requires the recovery or disposal of spent chemicals, which adds another separation unit to the treatment plant [113].

The adsorption efficiency depends on the characteristics of the wastewater and adsorbent, as well as the operating conditions. Adsorbent characteristics include surface morphology and functional groups, pore size of the adsorbent, ash, and mineral matter content. Among pollutants, properties that determine the efficacy of adsorption are solubility,

molecular size, charge, and pollutant structure. The pollutants' initial concentration, pH, the wastewater temperature, and the dose of the absorbent used are some of the important operating conditions in determining the performance of the process [109,114,115].

Adsorption by AC shows a moderate-to-high removal efficiency (more than 50%) for persistent pharmaceuticals such as azithromycin, carbamazepine, citalopram, hydrochlorothiazide, ofloxacin, oxazepam, trimethoprim, sulfamethoxazole, amoxicillin, penicillin, ciprofloxacin, and tetracycline [17,31,37,116]. For surrogate wastewater properties under normal operating conditions, activated carbon adsorption provides an effluent with a BOD of 2–7 mg/L and COD of 10–20 mg/L. However, low-molecular-weight ketones, acids, aldehydes, sugars, aliphatic, and high-molecular-weight compounds are poorly removed by activated carbon [23]. Moreover, the insufficiency of one-stage adsorption was observed by Guillossou et al. [116]; although substantial organic micropollutant removal was reported through tertiary treatment of municipal wastewater, the treatment failed to meet the environmental discharge regulation.

The recent studies on MP removal by adsorption processes have focused on developing low-cost adsorbents from biomass, agricultural waste, and soil and ore materials to make the process more sustainable while minimizing the adsorbent cost [117–120]. Surface modification and the impregnation of activated carbon for efficiency enhancement [121–125], and the integration of the adsorption process with biologically activated carbon (BAC) filters to remove pharmaceuticals and nutrients have simultaneously gained lots of attention [126]. The effectiveness of this method has been proven by many researchers, and dos Santos and Daniel [127] have reviewed the studies on the application of BAC filters for ammonia and organic matter removal.

4.4. Advanced Biological Treatment

In wastewater treatment, advanced biological processes utilize specialized microorganisms and biofilm-based systems to enhance the removal of micropollutants. These processes include two-phase bioreactors, moving-bed biofilm reactors (MBBRs), and immobilized cell biofilm reactors [128]. The enhancement techniques involve bioaugmentation, introducing specialized microbial cultures [129,130]; bioacclimation, adapting microorganisms to degrade novel or toxic compounds [131,132]; and biostimulation, optimizing environmental conditions to increase microbial activities [133,134].

Factors such as the specificity of microbial consortia, bioreactor design, and operational conditions affect the efficacy of advanced biological treatments. While these methods offer the advantage of targeting persistent micropollutants more effectively than conventional biological treatments, they also face challenges, including increased operational costs, system complexity, and maintenance demands [129,134–136].

Although conventional biological treatments, such as activated sludge and trickling filters, are well established and effective for the removal of bulk organic matter, they have limited MP removal capacity [137]. Research has shown variable micropollutant removal efficiencies with these advanced systems [136,138]. Removal efficiencies for MPs such as caffeine, ibuprofen, and acetaminophen have been reported to exceed 90%, which are poorly removed by conventional methods [139]. However, other micropollutants such as carbamazepine, metoprolol, diclofenac, citalopram, and tramadol are less amenable to removal, with efficiencies below 40% even when using advanced treatments [135,140,141].

Grandclement et al. [140] and Kanaujiya et al. [128] have reviewed the challenges of advanced biological treatments and have suggested further exploration of the hybrid systems. Such systems may include biofilm carriers, enzyme aggregates, and powdered and granular activated carbon. Enhanced membrane bioreactor (MBR) systems with powdered activated carbon (PAC) and hybrid MBBR-MBR systems have been identified as promising advancements [138,141]. Future studies are recommended to optimize these systems, addressing their economic and operational challenges to efficiency for widespread application and broadening the range of treatable MP by various bioaugmentation and bioremediation techniques.

Analyzing the mechanisms of various processes covered in this section, the efficacy of advanced treatment technologies can generally be categorized as low, medium, and high. A removal rate exceeding 80% is deemed highly effective, while a rate below 40% is classified as low, and a moderate range of 40 to 80% falls within the medium category. Table 3 shows how effectively conventional and advanced biological, adsorption, AOP, and membrane filtration processes could remove BOD, organic micropollutants (OMPs), total solids (TSs), and total organic carbon (TOC) from the wastewater.

Table 3. Performance of wastewater treatment technologies based on surrogate properties.

Surrogate Properties	Conventional Biological	Advanced Biological	Adsorption	AOPs	Membrane Technology	References
TS removal	Moderate	Moderate-high	High	Low	High	[23,129,142]
OMP removal	Low-High	Moderate-high	Moderate-High	High	High	[81,116,137,138,143,144]
BOD_5 removal	High	High	High	High	High	[81,83,132,145]
TOC removal	Moderate-High	High	Low-High	High	High	[28,70,83,132,141]

When opting for the most suitable treatment approach, factors such as process efficiency, operational simplicity, and adaptability must be considered [104,146]. Table 4 summarizes the advantages and disadvantages of some wastewater treatment processes. This table demonstrates that one or more treatment technologies must be coupled to achieve the desirable effluent quality and overcome the limitations coming from the application of a single treatment technique.

Table 4. Advantages and disadvantages of selected wastewater treatment processes.

Treatment Methods	Advantages	Drawbacks	References
Conventionalbiological treatment	• Efficient enough; • Most widely used and established process; • Environmentally friendly; • Nutrient removal; • Low cost.	• Limited temperature range; • Energy-consuming; • Requires solid waste disposal; • Requires a long time for sludge acclimation; • Low efficiency for nonbiodegradables; • Slow process.	[34,40,83]
Advanced biological treatment	• Enhanced removal efficiency for a wide range of MPs; • Targeted contaminant removal; • Reduced space occupation; • Greater adaptability to varying wastewater characteristics; • Enhanced biomass settling properties.	• Increased complexity of treatment system; • High-cost process; • Potential clogging in some systems; • Difficulties in degrading persistent MPs; • Requirement for specialized knowledge to control the microbial activities; • Potential detachment of biofilm.	[135,138,147–149]
Membrane technology	• Low space occupation; • Easy and continuous operation; • Simple process; • No post-treatment and chemical required; • Wide range of application for various wastewater types.	• High maintenance cost; • Membrane fouling; • Require frequent membrane cleaning; • Limited flow rate; • Requires high energy; • Requires brine disposal and brine toxicity assessment.	[67,81,88]

Table 4. *Cont.*

Treatment Methods	Advantages	Drawbacks	References
AOPs	• Fast process; • Sustainable and efficient; • Pollutant mineralization possibility; • Efficient for removal of a wide range of organics.	• Unknown reaction mechanism and pathways; • Requires post-treatment due to possible toxicity increment and by-product formation; • Energy-consuming; • High-cost process; • Requires chemical supply and/or pH adjustment.	[22,80,105]
Adsorption	• Ease of operation; • Low-cost and adaptive to current treatment plants; • Efficient in removing a wide range of contaminants; • Low-dissolved solid effluent; • Fast kinetics; • Greatly reduce the concentration of disinfection by-product precursors.	• Requires solid waste disposal or cyclic regeneration; • High cost of regeneration and material loss; • Non-destructive for contaminants; • Column blockage and needs backwashing; • Progressive capacity reduction after a couple of treatment cycles.	[31,38,39]

5. Hybrid and Integrated Treatment Methods

Tables 3 and 4 show that AOPs successfully degrade pollutants from wastewater; however, they are not economically viable. Bioprocesses are low-cost but have low removal efficiency and are inefficient for removing refractory compounds. Adsorption and filtration processes are very effective in reducing solid contents, yet they have high maintenance requirements and do not degrade the pollutants, which raises another issue in solid waste disposal. Hence, pre-treatment/post-treatment and hybrid techniques have been studied by many researchers to convert pollutants into more biodegradable molecules or to meet discharge regulations [59,144,150]. The integrated and hybrid methods help to reduce the total cost and chemical usage, as well as the indirect environmental implications of chemical treatment, and they lead to the degradation of pollutants and avoid organic shock or inhibitory effects on microorganisms in a bioreactor [104].

5.1. Sequential Treatment Strategies

The main objective in a sequence of two or more treatment techniques is to remove the MPs, suspended solids, toxicity, and organic content. The sequential treatment of wastewater with integrated biological and AOPs, the biological treatment and adsorption process, and AOPs with adsorption are frequently seen in the open literature. Each of these integrated processes is discussed separately in the following subsection.

5.1.1. Biological Treatment and AOPs

The most common type of integrated treatment process, as seen in Table 5, is the sequence of AOPs and biological treatment. AOPs are effective in degrading pollutants; however, the unknown reaction pathways and the formation of oxidation transformation products (OTPs) are the primary reasons for adding another polishing treatment stage. Many studies have focused on the application of AOPs as a pre-treatment stage to increase the wastewater biodegradability, so that it can be sent to a biological process for further pollutant removal [24,99,102,151]. Given that approximately 60% of the overall operating cost of AOPs is attributed to electricity, it is important to keep the pre-treatment as short as possible to avoid mineralization and only increase biodegradability. This approach makes the pre-treatment practical while maintaining the use of chemicals to a minimum level [152,153].

While AOPs as the pre-treatment stage have shown their efficiency in increasing biodegradability, some studies have shown that not only do the AOPs added to currently conventional biological treatment reduce the costs associated with chemical supply, but also, the effluent does not need a further polishing stage, as it is safe to discharge [154–156]. The same results were observed by Jose and Philip [150], where the degradation and toxicity of seven volatile organic compounds present in wastewater from a pharmaceutical manufacturing plant were studied in a plasma reactor. The results showed 90.9% and 91.8% of total BOD and COD removal, respectively. Moreover, it was observed that the BOD/COD ratio was not constantly increasing, and the final effluent from treatment was nontoxic to E. Coli. On the contrary, it has been reported that the only treatment train that decreased effluent toxicity was using AOP as a pre-treatment [157,158].

From Table 5 and studies on sequential AOP and biological treatment aiming for organic, micropollutant, and toxicity removal from wastewater, the following can be concluded:

1. The efficiency of the integrated process for removing MPs ranges between 70 and 100% depending on the operating condition, the treatment technique used, and the nature of the wastewater.
2. The degree of mineralization and organic content removal varies between 26 and 97%, and 38 and 100%, mainly due to applying AOPs as a pre-treatment stage to degrade the pollutants to simple organic compounds, but it fails to fully mineralize them.
3. The solar Fenton and ozonation processes are studied extensively at full-scale and pilot-scale, showing their potential to be added to a current WWTP; however, the implication regarding the ozonation process needs further investigation.
4. Sequential batch reactors (SBRs) and activated sludge bioreactors are the most common types of biological treatment used.
5. The application of AOP before biological treatment increased biodegradability significantly; however, it is not preferred, due to the added cost of chemicals and power supply.

5.1.2. Biological Treatment and Activated Carbon Adsorption

Biologically treated wastewater can be treated with an adsorption process to remove micropollutants, other intermediates, and nonbiodegradable compounds before discharging to surface water. In this way, the environmental disposal requirements are met, and discharged wastewater has the minimum environmental impact, or the water from this tertiary treatment could be recycled and reused for another process [144,159]. Since both biological treatment and activated carbon adsorption are well-established treatment techniques, studies concerning the combination of these two techniques are mainly at pilot- and full-scale, which analyses the adsorption efficiency in removing MPs and column blockage due to the presence of background organic matter and solids from previous biological treatment [127,160,161]. Studies listed in Table 5 show that the sequential biological/filtration and activated carbon treatment as a tertiary treatment technique can achieve up to 90% MP removal with a COD reduction in the range of 40–74%, which is on average less than the organic removal observed in the combination of biological and AOPs. This lower efficiency could be attributed to the scale of this study, as the application of AOPs is mostly on a lab scale under controlled experimental conditions. Although this sequence serves well for the removal of MPs, activated carbon is reported to release soluble microbial products (SMPs) into the wastewater, and competition for adsorption led to a low removal of ammonia, colour, and antibiotic resistance genes (ARGs); therefore, it is important to remove the biodegradable pollutant, ammonia, as well as solids before sending the wastewater to the adsorption column [127,162,163].

Table 5. Sequential treatment processes for the removal of MPs from actual wastewater.

Source of Wastewater	Treatment Sequence	Study Specification	Findings	Reference
Hospital	AS-Sonochemical, AS-Sono_PF	Lab-scale, Biological process: RT = 36 h MLSS dose= 25/400 $\frac{mL}{mL_{WW}}$ AOP: RT = 90 min $[Fe^{2+}]$ = 5 $\frac{mg}{L}$ power density = 88 $\frac{W}{L}$	Pharmaceutical removal: 58.82% (sonochemical), 82.86% (sono_PF), No inhibitory effect of effluent from the sonochemical process, pharmaceuticals removal by AS+ sono_PF: 91.13%.	[75]
	CF-UV/H_2O_2-AS, CF-UV/H_2O_2-Fungal biological	Lab-scale, Fungal treatment: FBR reactor, RT =7 d, AS: CSTR reactor, HRT = 48 h, SRT = 20–22 d, AOP reactor: immersion-type, RT= 10 min, $[H_2O_2]$ = 15 $\frac{mg}{L}$.	AOP+ fungal treatment: 94% removal efficiency, AOP+ AS treatment: 83% removal efficiency, Biological treatment+ AOP: 93–95% removal efficiency, saving energy and chemicals, AOP+ AS is the only sequence reducing toxicity,	[158]
	SPF-AC	Lab-scale, Fe^{2+}: single shot and three-stage addition strategy, Fe^{2+} dose = 5 $\frac{mg}{L}$ and 15 $\frac{mg}{L}$, H_2O_2 dose = 50 $\frac{mg}{L}$ and 150 $\frac{mg}{L}$, AC dose = (0.05–1) $\frac{g}{L}$, EBCT = (5–65) min.	Single Fe^{2+} addition: 20% primary degradation, 3.05% mineralization, Consecutive Fe^{2+} addition: 58% primary degradation, 12.07% mineralization, Low removal of highly polar TPs, High aromatic product removal during the adsorption.	[164]
Pharmaceutical manufacturing industry	SBR-EF, EF-SBR	Lab-scale, 0.1 mM caffeine and 5-fluorouracil, SBR: RT = 1 d, COD/N/P = 100:5:1, AOP: pH = 2.9, $[Na_2SO_4]$ = 0.05 M, $[Fe^{2+}]$ = 0.2 mM, RT = 2 h and 4 h, I = 200 mA, TSS_0 = 19 $\frac{mg}{L}$, COD_0 = 120 $\frac{mg}{L}$, BOD_5/COD = 0.03.	SBR pre-treatment: 57% caffeine, 82% 5-fluorouracil, and 50% COD removal, AOP post-treatment: 100% removal of pharmaceuticals, 99% COD removal, AOP pre-treatment: 60% COD removal, 100% pharmaceutical removal, Biodegradability= 0.33, SBR post-treatment: 100% COD and pharmaceutical removal.	[71]
	EF-AO	Lab-scale, Cathode: carbon fiber, Anode: DSA mesh or Nb/BDD plate, Electrolyte: K_2SO_4 0.05 M, COD_0 = 1200 $\frac{mg}{L}$, TOC_0= 431.55 $\frac{mg}{L}$, BOD_5/COD = 0.14.	Observed 97.1% TOC removal, Observed similar biodegradability increment in EF-BDD and EF-DSA, Achieved nearly complete mineralization by EF-BDD, EF-DSA process cost: 2.20 $\frac{\$}{m^3}$, EF-BDD process cost: 2.54 $\frac{\$}{m^3}$.	[153]
	Fenton-AC	Lab-scale, AC column Q = 0.3 $\frac{mL}{min}$, Fenton: $[H_2O_2]$ = 8.5 $\frac{g}{L}$, RT = 25 min, TOC_0 = 43,595 $\frac{mg}{L}$, COD_0 = 162,933 $\frac{mg}{L}$, BOD_5/COD = 0.29, $[CBZ]_0$ = 442 $\frac{mg}{L}$.	Carbamazepine removal: 49.39% by Fenton process, Total carbamazepine removal: 99.51%, Complete detoxification, Zero-discharge process for high-strength wastewater on-site treatment.	[165]

Table 5. *Cont.*

Source of Wastewater	Treatment Sequence	Study Specification	Findings	Reference
Pharmaceutical manufacturing industry	Flocculation-AC	Pilot-scale, Secondary effluent, TOC_0 = 26.7 $\frac{mg}{L}$, P-$PO_4{}^{3-}$ = 1.5 $\frac{mg}{L}$, N-$NH_4{}^{+}$ = 0.95 $\frac{mg}{L}$.	Increased bacterial diversity at each stage, Total removal percentages: 95% TOC, 98% P-$PO_4{}^3$, 83% N-$NH_4{}^+$, Total pharmaceutical removal of 90%.	[166]
	Coagulation-E-beam irradiation-AS	Lab-scale, HSWW and LSWW from equipment washing, Coagulants: 4 and 3 $\frac{g}{L}$ $FeCl_3$, $Al_2(SO_4)_3$, $Ca(OH)_2$, Irradiation dose rate = 100 $\frac{kGy}{min}$ H_2O_2 dose = (0.01–0.75) $\frac{mg}{L}$ $K_2S_2O_8$ dose = (0.01–0.2) $\frac{mg}{L}$ MLSS = 3000 $\frac{mg}{L}$.	COD removal: 89% (HSWW), 94% (LSWW), E-beam effluent biodegradability: 0.77 (LSWW), 0.74 (HSWW), Complete detoxification, Best coagulant: $FeCl_3$, Best oxidant: H_2O_2, Process cost: 1.37 $\frac{\$}{m^3}$ (HSWW), 2.85 $\frac{\$}{m^3}$ (LSWW).	[167]
	DF-AS, SPF-AS, EF-AS	Lab-scale, HSWW and LSWW from equipment washing, Solar intensity = 900 $\frac{W}{m^2}$, HRT = 120 min, LSWW: [Fe^{2+}] = 0.05 $\frac{mol}{L}$ [H_2O_2] = 0.25 $\frac{mol}{L}$ HSWW: [Fe^{2+}] = 0.1 $\frac{mol}{L}$, [H_2O_2] = 1 $\frac{mol}{L}$ MLSS = 3200 $\frac{mg}{L}$, RT = 8 d.	Best performance: SPF, Complete detoxification by sequential method, Total COD removal: LSWW: 78% (DF), 84% (SPF), and 70% (EF), HSWW: 67% (DF), 82% (SPF), and 54% (EF), Eliminated coagulation stage before AS treatment by using Fenton process.	[168]
	SPF-Ozonation (SPFO), Fenton-Ozonation (FO)	Lab-scale, TOC_0 = 5000 COD_0 =18,300 $\frac{mg}{L}$ pH = 9.2, O_3 dose= 0.1 $\frac{g}{min}$ Recirculation flow rate = 50 $\frac{L}{h}$ SPFO: RT = 2 h, [Fe^{2+}] = 10 $\frac{mg}{L}$ FO: RT = 3 h, [Fe^{2+}] = 100 $\frac{mg}{L}$ [H_2O_2]/COD = stoichiometric.	SPFO COD, TOC, and TN removal: 60%, 11%, 10%, FO COD, TOC, and TN removal: 65%, 25%, 26%, Estimated SPFO cost: 12.69 $\frac{€}{m^3}$, Estimated FO cost: 14.97 $\frac{€}{m^3}$, The effluent did not meet the standard discharge limit.	[169]
	Fenton-SBR	Full-scale and continuous Fenton, Lab-scale SBR, Fenton: Q = 3 $\frac{m^3}{h}$, HRT = 60 min, [$FeSO_4$] = 0.024 $\frac{mg_{Fe}}{mg_{TOC}}$, [$CuSO_4$] = 0.0021 $\frac{mg_{Cu}}{mg_{TOC}}$, [H_2O_2] = 21.5 $\frac{mgg_{H_2O_2}}{mg_{TOC}}$, P = 1.5atm, T = 120 °C, SBR: HRT = 2 d, SRT = 20 d, V = 15 L, TOC_0 = 1440 $\frac{mg_C}{L}$, COD_0 = 7010 $\frac{mg_{O_2}}{L}$, BOD_5/COD = 0.17.	Fenton: COD and TOC removal: 85–90%, 90%, Sequential process: TOC removal: >90%, 65% less use of oxidant, Decreased operating temperature from 120 °C to 70 °C, Biological process cost: 11% of overall cost, Estimated cost for Fenton: 25 $\frac{€}{m^3}$, Estimated cost for sequential process:16 $\frac{€}{m^3}$.	[170]

Table 5. *Cont.*

Source of Wastewater	Treatment Sequence	Study Specification	Findings	Reference
MWWTP	Ozonation-SF, Ozonation-MBBR, Ozonation-FBBR, Ozonation-AC	Full − scale ozonation, Pilot − scale post − ozonation, Secondary effluent, O_3 dose = 0.55 $\frac{g_{O_3}}{g_{DOC}}$, HRT = 43 and 13 min, $EBCT_{MB}$ = (14.5–25) min.	Removal of more than 79% for 200 MPs. NDMA reduced by 41–83%, SF performed the best for removal of TSS, DOC, Biological treatment failed to remove the TPs. GAC removed most of MPs, Estimated upgrading cost with ozonation: 0.06 $\frac{CHF}{m^3}$.	[16]
	SBR-SPCO, SBR-SPFO, SBR-Ozonation	Lab − scale, Primary effluent, Initial concentration = 200 $\frac{\mu g}{L}$, SBR setup: HRT =7 h, DO =3 $\frac{mg}{L}$, AOP setup: $[Fe]^{3+}$ = 2.8 $\frac{mg}{L}$, $[TiO_2]$ = 250 $\frac{mg}{L}$, O_3 reactor flow rate = 0.58 $\frac{L}{min}$, $[O_3]$ = 13 $\frac{mg}{L}$.	SPCO removal efficiency: 95% ECs, 40% TOC, 59% COD removal, SPFO removal efficiency: 100% EC, 26% TOC, 38% COD, Ozonation removal efficiency: 100% EC, 35% TOC, 58% COD, Best performance: SPFO and ozonation for pharmaceutical removal.	[58]
	MBBR-SPF, AS-SPF, CF-SPF, MBBR-UV/H_2O_2, ASUV/H_2O_2, CF-UV/H_2O_2	Lab-scale, Primary effluent, [Venlafaxine] = 200–1000 $\frac{ng}{L}$, T = 22 °C, $[H_2O_2]$ = 25 $\frac{mg}{L}$, SPF intensity = 900 $\frac{W}{m^2}$, Fe/H_2O_2 = 5:25.	Venlafaxine removal: 90%, Achieved venlafaxine removal in 5 min with UV/H_2O_2, Required 120 min of operation for venlafaxine removal in SPF, Best effluent quality order: MBBR > AS > CF.	[60]
	Ozonation-BAF, Ozonation-Nonaerated biofilter, Ozonation-Aerated GAC, Ozonation-Nonaerated GAC	Pilot-scale, Secondary effluent, O_3 dose = 0.33 $\frac{g_{O_3}}{g_{DOC}}$, HRT = 12.6 min, EBCT = (26.7–36.4) min, Filter column height = 4 m, D ^{2}= 0.19 m.	Increased anti-estrogenic and mutagenic effects by ozonation, Decreased anti-androgenic activity by ozonation, Better performance of aerated/nonaerated AC in pollutant and toxicity removal.	[157]
	Ozonation-BAC-Coagulation-CMF	Pilot - scale ozonation, Lab - scale CMF, Secondary effluent, Coagulant: PACl, O_3 reactor: V ^{3} = 35 L, RT = 5 min, O_3 dose = 6 $\frac{mg}{L}$, BAC EBCT = 4.5 min.	Accelerated membrane fouling due to released SMP in BAC, Increased permeate quality by reducing residual O_3 and DBPs, Decreased permeability by 20% in the first 305 h of treatment by BAC.	[162]
	AS-SF, AS-AC Aerobic biomass-SF	Full − scale, Primary effluent, [Ab] = (25–60) $\frac{\mu g}{L}$, Monitored 52 Pharmaceuticals and 6 ARGs.	Antibiotic (Abs) removal in all treatment plants: 80–88%, COD removal in treatment plants: 55–74%, Increased concentration of some antibiotics after AS treatment.	[171]

Table 5. *Cont.*

Source of Wastewater	Treatment Sequence	Study Specification	Findings	Reference
MWWTP	SPF/H_2O_2-GAC	Pilot-scale AOP, SE from CAS and MBR, [Ab] = 100 $\frac{mg}{L}$, $[H_2O_2]_{CAS}$ = 100 $\frac{mg}{L}$, RT_{CAS} = 115 min, $[H_2O_2]_{MBR}$ = 50 $\frac{mg}{L}$, RT_{MBR} = 111 min, AC dose = 500 $\frac{mg}{L}$.	Achieved full disinfection after 60 min of treatment by SPF, Increased effluent toxicity by SPF, Achieved full toxicity and >95% Ab removal by sequential SPF and AC.	[172]
	Multilayer filter column-IEX-UV/H_2O_2, Multilayer filter column-O_3/biofiltration-UV/H_2O_2	Full-scale IEX, Pilot-scale UV/H_2O_2, $[C]_0$= (25–70) $\frac{\mu g}{L}$, DOC_0 = (10–20) $\frac{mg_C}{L}$, Filter rate = 25 $\frac{m}{h}$, IEX: Q = 750 $\frac{L}{h}$, EBCT = 2 min, UV/H_2O_2: $[H_2O_2]$ = 10 $\frac{mg}{L}$, UV dose = 1500 and 300 $\frac{mJ}{cm^2}$, O_3/biofilter: RT = 15 min, $[O_3]$ = 15.3 $\frac{mg_{O_3}}{L}$.	Removal of humic fraction of organic matter in Ion exchange, Removal of hydrophobic fraction of organic matters in O_3/biofiltration, Organic matter removal in IEX and O_3/biofiltration: 45–65%, Reduced energy consumption in sequential IEX and UV/H_2O_2 by 84%, Estimated cost of sequential IEX and UV/H_2O_2 0.34 $\frac{€}{m^3}$.	[173]
Laundry WW	UF permeate-AC, UF permeate-Polymeric resin filtration, UF permeate-EO, UF permeate-NF	Lab-scale, UF TMP = (10–18) psi, $[Na_2SO_4]$ = 3 $\frac{mg}{L}$, Cathode: graphite, stainless steel, Anode: BDD, Ti/Pt, and Ti/IrO_2, RT = 120 min, NF TMP = (100–200) psi, AC and resin HRT = (6–30) min.	UF removal efficiency: 50% COD, 95% TSS, 97% turbidity, 75% nonylphenol ethoxylates, EO removal efficiency: 73% COD, NF removal efficiency for COD, turbidity, and nonylphenol ethoxylates = 80%, AC removal efficiency: (40–45)% COD, 80% nonylphenol ethoxylates, and no turbidity removal.	[163]
	Filtration-IEX-AC-Ozonation	Lab-scale, Primary effluent, Q [1] = [0.1–5] $\frac{L}{min}$, O_3 dose = (1–5) $\frac{g}{h}$,	Best filter arrangement: Polypropylene + IEX + AC, TSS and BOD_5 removal of 80%, Removal of all coliforms by disinfection.	[174]
Agricultural industry	PCO-AnBT	Lab-scale, PCO: SCPC reactor, Catalyst: TiO_2-P25, Oxidant: H_2O_2, TiO_2 dose = 100 $\frac{mg}{L}$, RT= 3 h, RT in bioreactor = 28 d.	AOP removal efficiency: 59.26% COD, 44.18% TOC, Biological treatment removal: 46.4% COD, 86.6% TOC, Sequential process removal efficiency: 72.2% COD, 53% TOC.	[65]

[1] flow rate (Q), [2] diameter (D), [3] volume (V).

5.1.3. Activated Carbon Adsorption and AOPs

The integration of AOPs and adsorption presents a novel approach to wastewater treatment. This method involves degrading pollutants through AOP and subsequently eliminating potential toxic by-products by adsorption in a step-by-step treatment process. Some research has also explored the concept of simultaneous oxidation and adsorption, which allows for the recovery of the adsorbent while pollutants are degraded within a reactor vessel [175,176].

A study by Mostafazadeh et al. [163] for the removal of nonylphenol ethoxylates showed that an integrated process using ultrafiltration (UF), electro-oxidation (EO), and

electro-coagulation (EC) can recover 80% of laundry wastewater. This approach reduced the COD of raw wastewater to below 80 mg/L, meeting environmental discharge water quality standards. Additionally, this study explored nanofiltration (NF) for water reuse and also assessed adsorption by activated carbon and polymeric resin, achieving high pollutant removal (90%), but these methods were less efficient in meeting environmental limits compared to the other techniques studied. On the contrary, Dwivedi et al. [166] and Naghsh Javaheri et al. [174] revealed that the effluent from the activated carbon process can be repurposed for irrigation or discharged into surface water. They found that a combination of the Fenton process/ozonation followed by activated carbon adsorption achieved full toxicity removal and an 80% removal of MPs.

Interpretation of the data from Table 5 shows that activated carbon, as the last polishing step, can achieve up to 95% MP removal, 80% TSS and BOD removal, and complete toxicity removal; however, as during the oxidation stage, highly polar oxidation by-products (DBP) are formed, the activated carbon column fails to remove all of them, and therefore, they can pass the treatment train and accumulate in the environment.

5.2. Hybrid Treatment Strategies

A potential solution for effective wastewater treatment lies in hybrid treatment strategies where the drawbacks of one treatment are mitigated, and the efficiency of MP removal is enhanced simultaneously. Hybrid technologies encompass the combination of multiple technologies into a single approach. For instance, ultrafiltration and powdered activated carbon were merged into a unit to remove ARGs and DOC before releasing the wastewater to the environment, and it effectively increased the membrane flux and reduced the membrane fouling [177]. Hybrid processes usually include a biological treatment stage in conjugation with a physical/chemical treatment followed by a membrane filtration step [178–184]. Therefore, the two main hybridized treatment techniques, including MBR and biological activated carbon filters (BACs), are separately discussed in this section and an overview of recent state-of-art on MP removal utilizing hybrid processes is presented in Table 6.

5.2.1. Membrane Bioreactor

A membrane bioreactor (MBR) is the combination of a biological process with membrane filtration in hybrid mode. Compared to the conventional activated sludge (CAS) process, better effluent quality can be achieved by MBR, making it a promising technology in wastewater reuse. MBRs excel in eliminating pathogenic microorganisms and micropollutants through biodegradation, the absorption of sludge, and the membrane retention mechanism [185–187].

Despite the benefits outlined for MBR for removing phenolic compounds and estrogens, significant challenges persist, including low hydrophilic trace organic contaminant (TrOC) removal, limited flow rate, membrane blockage, and high operating and capital cost. These challenges limit the application of MBR, and therefore, their performance of the process needs to be intensified [2,88,187]. Intensification of the MBR performance may take place by adding PAC for better organic removal or passing the effluent through reverse osmosis (RO) such that the mineral and all pathogens are removed [188]. Recent trends in activated carbon-assisted MBRs focus on optimizing the process performance in terms of PAC dosage and sludge retention time (SRT) to avoid membrane fouling, quantifying the micropollutant removal through sorption and biodegradation, developing new membrane material to limit membrane blockage, and lastly determining the fate of pollutants in the concentrated sludge and its disposal [19,88,160,189].

The improvement in the performance of MBR by the pre-treatment of wastewater through the addition of PAC or ozonation was studied by Kaya et al. [70,190], and improvements in COD and pharmaceutical removal in both cases were reported. However, membrane fouling remained a significant challenge, particularly in highly polluted wastewater. Ozonation did not alleviate membrane fouling, leading to an operational halt after

approximately 168 days. In contrast, membrane fouling was temporarily reduced by adding PAC due to activated carbon's adsorption capacity, resulting in a transient increase in permeate flux. This effect was also noticed in COD reduction, where PAC saturation led to decreased removal. Despite stable etodolac removal with increased OLR, maintaining a consistent membrane flux and reducing fouling with high organic removal efficiency remained a challenge.

Improved permeate flux by the addition of PAC to MBR or enhanced filtration is reported by many researchers. Lee et al. [191] found that adding PAC to MBR not only improved water quality but also increased the permeate flux in a hybrid system. However, this study noted a reduced removal in TP due to higher MLSS concentration and longer SRT, leading to decreased removal rates. Another finding was a sharp decrease in flux in the MBR/PAC system followed by the NF system after five days of operation. Rasouli et al. [192] also confirmed that the membrane flux increases by enhancing the MF process by adding PAC. Although these studies showed improvement in the flux by the addition of PAC, other studies observed opposite results [193,194]. Amaral et al. [193] reported that background natural organic matter (NOM) caused a decline in the flux and MP removal, which could be avoided only by adding calcium to capture the NOM before causing the membrane fouling. This highlights the necessity to study the actual wastewater matrix for the process performance evaluation.

Table 6. Hybrid treatment strategies for the removal of MPs from actual wastewater.

Source of Wastewater	Treatment Process	Study Specification	Findings	Reference
Pharmaceutical manufacturing industry	MBR/PAC	Lab-scale, OLR = 1–2 $\frac{kg_{COD}}{day.m^3}$, RT = 145 d, COD_0 = 18,000 $\frac{mg}{L}$, TMP = 200 mbar, Q_{air} = 8–10 $\frac{L}{min}$, DO = 4.6 $\frac{mg}{L}$, T =25 °C, PAC dose = 2 $\frac{g}{L}$.	Increased removal from 73% to 97% in hybrid method, Observed Cake adsorption as the main fouling mechanism, COD removal = 90%.	[190]
	Fenton/AS	Lab-Scale, Effluent of CBZ production plant, [CBZ] = 50 μM, pH = 3, $[Fe^{2+}]$ = 100 μM, $[H_2O_2]$ = 200 μM, RT = 4 h.	CBZ degradation: 97.32% by Fenton, TOC removal: 10.07% by Fenton, Achieved total removal of 97.90% (CBZ) and 28.87% (TOC).	[195]
	Electro-flocculation/catalytic ozonation	Lab − scale, Fe - Zn - loaded zeolite as a catalyst, O_3 reactor: Q = [0.8–1.55] $\frac{mg}{min}$, catalyst dose = (5–15) $\frac{g}{L}$, DC volts = (5–15) V, $[Abs]_0$ = (50–100) $\frac{mg}{L}$.	Observed 41% and 77% BOD and COD removal, Abs removal = (55–100)%.	[196]
	MFC	Lab-Scale, Two-chambered MFC, Anode: stainless steel, Cathodes: Pt-Ti or Pd/Ir-Ti, COD_0 = 7440 $\frac{mg}{L}$, pH = 9.2, HRT = 30 h.	Best cathode: Pt − Ti, COD removal = (91–93)%, Biodegradability increased from 0.26 to 0.54, Maximum power density: 20.5 $\frac{W}{m^3}$.	[197]
MWWTP	UF/PAC, UF/BPAC	Lab − Scale, Secondary effluent, Dead − end UF filtration process. V = 400 mL, Membrane area = 41.8 cm^2, TMP = 0.1 Mpa, Q_{air} = 3 $\frac{L}{min}$.	DOC removal: 63.2% (PAC), 54.1% (BPAC), Optimum dosage of PAC and BPAC: 40 $\frac{mg}{L}$, Increased membrane flux by 15.6% (PAC) and 25.1% (BPAC).	[177]

Table 6. *Cont.*

Source of Wastewater	Treatment Process	Study Specification	Findings	Reference
MWWTP	O_3/CM	Lab − scale, Secondary effluent, Ceramic membrane doped with MnCe, Operation time = 70 d, Flux = 60 $\frac{L}{m^2 h}$, O_3 dose = 5 $\frac{mg}{L}$.	MP removal ranged from 89.33% to 99.49%, Achieved DOC removal of 30%, Reduced irreversible membrane fouling by 10.17%.	[198]
	Catalytic ozonation	Lab − scale, Secondary effluent, Catalytic ozonation with volcanic rock, Catalyst dose = 0.5 $\frac{g}{L}$, O_3 reactor: Q = 0.2 $\frac{L}{min}$, T = 25 °C.	Permanent disinfection of wastewater, Full pathogen removal in the hybrid process, Reduction in energy consumption: 12%.	[199]
	PAC/DBF	Full − scale, Secondary effluent, PAC dose = 10 $\frac{mg}{L}$, V = 180 m^3, Iron dose = 0.1 $\frac{mg_{Fe^{3+}}}{mg_{PAC}}$, $Q_{filtration}$ = 30 $\frac{L}{s}$.	80% MP removal, TSS removal = 63.8%, Turbidity removal = 91.2%, DOC removal = 31.3%, P removal = 52.6%.	[200]

5.2.2. Biological Activated Carbon

Among the suggested alterations and techniques to enhance the efficacy of water treatment, the biological activated carbon method stands out as an ecologically sound and economically viable approach. The treatment method exhibits the potential to surpass various constraints linked to both treatments by utilizing the synergetic effect of biological activity and adsorption. AC is used as a medium for microorganism immobilization and the elimination of organic substances in water, while the adsorption capacity of AC is restored through microorganism activity which reduces the operating cost of supplying fresh AC [126,201].

BAC systems have proven their superiority in effective water treatment with more than 70% of removal of natural organic matter (NOM), ammonium, and MPs [169,183,188,202–205]. BAC systems have been studied for process improvement through surface modification of activated carbon, advanced oxidation post-treatment, coagulation–flocculation, and membrane filtration pre-treatment, showing a high removal of DBP and low-molecular-weight compounds, increased adsorption capacity, and a reduced need for chemical supply [206–210]. Although the efficiency of the BAC treatment method is observed through full-scale applications, the process is slow, and removal occurs through the long empty bed contact time (EBCT). Moreover, the reduced adsorption capacity as a result of occupancy of the AC surface by microorganisms and the diffusion of adsorbed soluble microbial products (SMPs) back into the water matrix are challenges ahead of the widespread application of BAC systems [211]. Therefore, further studies are required to optimize the BAC performance for contact time and temperature, analyze the effect of adsorbed toxic pollutants on biofilm, and control and maintain microbial activity under a high organic load [126,127].

Table 6 lists the studies regarding MP removal from actual wastewater through hybrid treatment techniques, in which their performance has been intensified by combining one treatment technique with another. Hybrid MBR, BAC, filtration, and AOPs are various techniques that can be seen in this table, with an MP removal efficiency of more than 90% in most cases. Table 6 shows that the studies tested low-strength pharmaceutical wastewater and municipal secondary-treated wastewater where the solids and major parts of biodegradable pollutants are removed. In general, combining a biological process with activated carbon and a filtration stage provides high-quality water that can be discharged

into the environment. This method benefits from sustainability due to the regeneration of activated carbon, which reduces the frequency of fresh adsorbent supply and the treatment operating cost by 10% compared to the solo biological treatment [174].

5.3. Hybrid and Sequential Treatment Strategies

Hybrid wastewater treatment methods taking advantage of the biological activated carbon filters and membrane bioreactors integrated with some other efficient techniques have been studied by many researchers for the effective removal of organic compounds, micropollutants, ammonia, toxicity, and oxidation by-products [102,115,144,212–214]. As discussed, sequential and hybrid technologies efficiently treat wastewater; however, their performance can be intensified to achieve better water quality for reuse with minimal environmental effect and sustainability. Table 7 lists the studies that used hybridization and sequential treatment to increase wastewater quality such that environmental discharge limits are met, and the treated wastewater is safe to reuse.

Refractory pharmaceuticals such as carbamazepine, diclofenac, iohexol, and metoprolol cannot be removed through biological processes, even with an efficient process like MBR. Therefore, post-treatment of the biological process effluent with an AOP step, which can effectively degrade the nonbiodegradable compounds, is essential. Ooi et al. [215] studied the performance of six pilot-scale moving-bed biofilm bioreactors (MBBRs) prior to the ozonation step for hospital wastewater treatment, where it was found that 17 pharmaceuticals out of 22 monitored ones were removed with an efficiency of more than 20%. Although 90% TOC removal was achieved in biological treatment, the low removal of pharmaceuticals during this stage indicated the necessity of an effective ozonation post-treatment. Similar results were observed in other studies that used an SBR, MBBR, and CAS pre-treatment to reduce the organic concentration so that the advanced oxidation post-treatment can achieve high MP removal. This sequence not only decreased the treatment time and cost but also increased the organic removal and contributed to the effluent disinfection [216–220].

The utilization of AOPs in wastewater treatment reduces the toxicity in many cases and improves biodegradability; however, contradictory results in different studies suggest that a membrane filtration step or an adsorption stage after AOP ensures toxicity, TrOCs, and DBP removal [221–223]. The use of BAC after an oxidation step for the removal of reaction intermediates and complete pharmaceutical removal has been investigated by various researchers, with MP removal in the range of 60–100% [57,209,224–229].

The effect of toxicity and the formation of by-products through the catalytic oxidation of oxytetracycline (OTC) was studied by Liu et al. [209], where seven transformation by-products were detected after the oxidation. Although the process effectively removed 92.6% of OTC and 74% of COD, the toxicity tests showed that the effluent of the oxidation process was 100% toxic to tested zebrafish embryos. In another study of the toxicity assessment after an oxidation step, GilPavas et al. [230] evaluated the treated industrial effluent by Fenton (F) and Photo-Fenton (PF) processes. They reported that the maximum acute toxicity removal was 20% and 60% for the Fenton and Photo-Fenton processes, respectively, while the activated carbon process achieved 90–100% toxicity removal, ensuring a safe effluent. The formation of toxic oxidation by-products and residual oxidants in the medium was the main reasons for the toxicity in the effluent. In the same study, it was also observed that the F/PF was the main contributor to COD reduction, where 36–45% of COD removal occurred in the oxidation reactor, with AC contributing to a further 4–5% reduction in COD.

Table 7. Integrated and hybrid treatment technologies for the removal of MPs from actual wastewater.

Source of Wastewater	Treatment Sequence	Study Specification	Findings	Reference
Pharmaceutical manufacturing facility	O_3-Anaerobic MBR	Lab − scale, Batch mode, Ozonation: pH = 7, $[O_3] = 2\ \frac{g}{h}$, HRT = 60 min, T = 35 °C, $COD_0 = (20,000–23,000)\ \frac{mg_{O_2}}{L}$, MBR: submerged flat sheet/hallow fiber module, operation time = 167 d.	O_3 pre-treatment reduced inhibition of MOs. Ozonation COD removal = 26%, Best membrane: hollow fiber module, Total COD removal: 80–90%, Total etodolac removal: 90–99%, After 168 days, no flux was observed.	[70]
	O_3/GAC-PS-TiO_2 photocatalysis	Pilot − scale, Circulation flow rate = (10–15) $\frac{L}{min}$, PS-TiO_2 dose = (200–300) g, RT = 4 h, COD_0 = 1000 ppm, $[phenol]_0$ = 4.75 ppm.	Total COD removal = 71%, Total phenol removal = 100%, Achieved 95% COD removal after 12 h operation.	[151]
	Chlorine dioxide oxidation (ClO_2)-BAC	Lab − scale, Batch mode, pH = 2, $[ClO_2]/[OTC] = 1.5:1$, T = 25 °C, HRT = 8 h, $[COD]_0 = (249.5–345.7)\ \frac{mg}{L}$.	OTC removal = 98.3%, COD removal = 68.1%, Biodegradability increased from 0.04 to 0.23.	[231]
MWWTP	MBR without phosphorus precipitation-NF, MBR with phosphorus precipitation-PCD	Pilot − scale, Primary effluent, NF process: Flux = 22 $\frac{L}{m^2h}$, TMP = 5.4 bar, recovery = 84%, PCD: $[Fe_2(SO_4)_3] = 300\ \frac{mg}{L}$, Pulse frequency = 840 $\frac{pulse}{s}$, Recirculation flow rate = 15 $\frac{L}{min}$, MBR: Flux = 10 $\frac{L}{m^2h}$, $[MLSS] = 10\ \frac{g}{L}$, HRT =21 h.	MBR +NF reached 84% MP, 99% COD, and 99% DOC removal, MBR+ PCD reached 90% MP, 92% COD, and 86% DOC removal, MBR+ NF allowed recovery of phosphorus, MBR without precipitation had a better MP removal.	[14]
	BAC-UF	Pilot − scale, Secondary effluent, BAC: V = 2 m^3, EBCT = 50 min, Q = 48 $\frac{m^3}{d}$, UF concentrate: Q = 60$\frac{m^3}{d}$,	Total pharmaceutical removal of 78 to 89%, GAC played the main role in removal, 35% sulfamethoxazole, 22% ciprofloxacin, and 32% azithromycin removal due to biofilm, TOC removal = 11.9%, COD removal = 37.9%.	[61]
	Ozonation-BAC-MF	Lab − Scale, Secondary effluent, Continuous mode, O_3 reactor: Q = 1.4 $\frac{L}{min}$, $[O_3] = 0.11\ \frac{g}{L}$, RT = 2 h, BAC column: Length = 180 mm, D = 50 mm, EBCT = 20 min, MF module: hollow fiber, flux = 180 $\frac{L}{hm^2}$.	Reduced fouling index by 4 times, Removal of Humic substances: 57%, biopolymers: 100%, DOC: 10%, Decreased water quality but increased permeate flux by BAC addition.	[232]

Table 7. *Cont.*

Source of Wastewater	Treatment Sequence	Study Specification	Findings	Reference
MWWTP	MF-RO-UV/H_2O_2, O_3-BAC-UV/H_2O_2, MF-RO-UV/HOCl, O_3-BAC-UV/HOCl	Pilot − scale, Tertiary effluent, $[H_2O_2] = 6\ \frac{mg}{L}$, UV lamp = 254 nm, UV dose $= 1000\ \frac{mJ}{cm^2}$, $[HOCl] = (2–3)\ \frac{mg}{L}$, EBCT = 15 min, $[O_3] = 0.7\ \frac{mg}{mg_{DOC}}$.	Best sequence: O_3 > BAC > MF > RO > UV/H_2O_2. Total removal: 97%, O_3 > BAC sequence: Higher NDMA removal, MF/RO sequence: Higher NMOR removal, UV/H_2O_2 was more effective in removal of –NDMA and NDMA precursors and controlling toxicity.	[233]
	MBR-UV/H_2O_2	Pilot − scale MBR, Lab − scale AOP, MBR: hollow fiber UF membrane, HRT = 16 h, Recirculation flow rate $= 90\ \frac{L}{min}$, MLSS $= 4250\ \frac{mg}{L}$, SRT = 13.56 d, AOP: $[H_2O_2] = (25–100)\ \frac{mg}{L}$, RT = [10–40] min, $[MP] = (10–5000)\ \frac{\mu g}{L}$.	At high pharmaceutical concentrations, complete removal can be achieved only by increasing the dose of oxidant and treatment time, The removal at various pharmaceutical concentrations ranges from 95 to 100%.	[234]
	Ozonation-MF-BAC-RO-UV-Chlorination	Full − scale, Secondary effluent, Feed water was treated with MBR as sec ondary treatment method, The treatment plant capacity: 20 $\frac{m^3}{d}$.	Out of 109 TrOCs found in feed water, 34 were in product water, Full toxicity and TrOC removal, Nitrosamine removal: 100%, The presence of NDMA in reject water had no environmental impact.	[143]
	Enhanced coagulation-UF (CUF), Coagulation-adsorption-UF (CAUF), Coagulation-oxidation-UF (COUF)	Lab − scale, Secondary effluent, CUF coagulants: PAC, K_2FeO_4, ploy aluminum chloride (PACl), Densadeg tan k: V = 15 L, sludge reflux ratio: 50%, UF: flux $= 2\ \frac{L}{m^2h}$, submerged module, $[MP] = (1–10.4)\ \frac{\mu g}{L}$, COD $= 32.3\ \frac{mg}{L}$, $[PACl] = (10–55)\ \frac{mg}{L}$, PAC dose $= (10–100)\ \frac{mg}{L}$, Ferrate dose $= (1–40)\ \frac{mg}{L}$, RT = 60 min.	COD removal efficiencies: 22.4% UF, 27.3% CUF, 54% CAUF, 57.9% COUF, Observed minimum toxicity for CAUF, Transmembrane pressure increase rate: highest for UF, lowest for CAUF, CAUF showed the highest MP removal ranging from 74 to 96%, Enhanced coagulation alleviated membrane fouling.	[235]
	Anaerobic AS-Aerobic AS-MBBRs/AS (Hybas)-Anaerobic MBBR-Aerobic MBBR	Pilot − scale, Total RT = 330 min, COD $= 469\ \frac{g}{L}$, $[MP] = (0.12–10)\ \frac{\mu g}{L}$, $Q_{WW} = 300\ \frac{L}{h}$, $Q_{sludge\ return} = 500\ \frac{L}{h}$.	Removal efficiency of 14 out of 21 pharmaceuticals exceeded 50%, Achieved 92% COD and 97% NH_4^+ removal, Faster pharmaceutical degradation rate in aerobic reactors, Intermittent biomass feeding strategy increase pharmaceutical removal.	[236]

Table 7. *Cont.*

Source of Wastewater	Treatment Sequence	Study Specification	Findings	Reference
MWWTP	UV/H_2O_2-BAC	Lab − scale, RO brine, UV/H_2O_2: HRT = 30 min, $[H_2O_2]$ = 3 mM, lamp 254 nm and 39W, BAC: Column height = 12 cm, D = 1.5 cm, EBCT = 60 min, $[COD]_0 = 150 \frac{mg}{L}$, $[TN]_0 = 23 \frac{mg}{L}$, $[DOC]_0 = 43 \frac{mg}{L}$, Salinity level = (7–16) $\frac{g}{L}$.	Achieved DOC removal of 45–49%, No salinity effect on nitrification was observed, Achieved ammonia and nitrite removal of 90% and 80%, TN removal ranged from 30 to 47%, Maximum Nitrate removal: 39%.	[237]

The poor performance of the AC column for the reduction in organic matter was reported by many researchers [61,231,232]. However, adsorption seems to perform better in the removal of MPs, while AOPs are more efficient in reducing the TOC and COD. This shift in the performance is due to the competition of background dissolved organic compounds (DOCs) in wastewater or a low ratio of biodegradable TOC to total TOC [225,238]. In addition, the larger particles of activated carbon tend to facilitate pore blockage, which results in a lower organic removal [176,239].

Among various AOPs, UV/H_2O_2 and ozonation processes are the most studied ones in the integrated and hybrid processes, possibly due to scale-up feasibility and good performance in the MP removal [240–244]. In a pilot-scale study, Du et al. [176] compared UV/H_2O_2-BAC and O_3-BAC systems in a drinking water treatment plant. The BAC columns had different properties but worked under a consistent EBCT of 10.6 min. This study found that younger activated carbon with smaller particle sizes in the UV/H_2O_2-BAC system more effectively removed dissolved organic matter (DOM), especially those with lower molecular weights. The UV/H_2O_2-BAC effluent achieved a higher DOM removal than O_3-BAC, and it was considered safer than pre-ozonation.

From Table 7, the sequential use of AOPs and BAC followed by a membrane separation stage showed more than 80% of MP removal. However, BAC columns showed the release of humic substances and SMPs while contributing to the removal of MPs [17,245]. A major reduction in organic compounds was found to take place in the AOP stage, and partial consumption of the oxidants that enter the BAC column made them react with the BAC particles and resulted in their release into the water matrix [232]. Even with the chance of release of SMPs, the utilization of BAC after an AOP stage is suggested as not only does BAC remove the oxidation by-product but it also alleviates the membrane fouling [178,210,246–248].

A biological activated carbon filter used for pre-treatment of a membrane module influent was evaluated by Hamid et al. [232] and Im et al. [162] on a secondary-treated effluent of municipal wastewater. Both scientists concluded that the use of BAC increases the effluent quality; however, Hamid et al. [232] found out that the use of BAC in pre-treatment increased permeability and decreased the membrane fouling index. In contrast, Im et al. [162] observed that BAC increased the soluble microbial product in the membrane module, caused irreversible fouling, and decreased permeability. These contradictory results are related to the column's empty bed contact time and the ozone dosage used in the pre-treatment stage. In the same study, the EBCT of 4.5 min resulted in a very high flow rate and risked the wash-out of microorganisms without fully using the adsorption capacity. Xu et al. [211] reported the potential increased risk of ARG contamination by using the BAC process, suggesting a change in the backwashing method to reduce the secondary pollution by cyanobacteria on the activated carbon.

All in all, it is concluded that operating conditions are the main elements in determining the process efficiency and that biological-based processes are slow, with an average retention time of 20 h or more, whereas AOPs can reach proper removal within 2–12 h of

operation. Moreover, the integrated systems taking advantage of hybrid and sequential processes can successfully reduce the MPs in the wastewater matrix to below the value set by regulation. However, the selection of the treatment technique relies on the nature of MPs in the wastewater. Hence, based on reports and results from the studies reviewed, the potential best treatment approach for removing MPs is the sequential use of activated sludge biological-AOP-MBR-RO or activated sludge biological-AOP-BAC-MF. Only through this integrated process can the safety of discharged wastewater be guaranteed where no or minimum environmental effect is observed.

6. Prospects of Integrated Treatment Processes

The combination of multiple advanced treatment methods has displayed auspicious outcomes in dealing with wastewater containing micropollutants. Numerous researchers are focusing on this emerging domain of wastewater treatment. However, further investigations are necessary to comprehensively understand the breakdown pathway that occurs during the merged processes, resulting in improved treatment outcomes. For specific MPs, mainly pharmaceuticals such as metformin, paracetamol, penicillin, and diclofenac, the degradation pathways and reaction rates have been established and demonstrated in the adsorption, biodegradation, photocatalytic membrane reactor, and catalytic oxidation [32,33,89,92,249].

By identifying the degradation pathway of MPs, the underlying process mechanism becomes clearer, aiding in the prevention of the generation of harmful by-products during degradation. Additionally, it is crucial to define the degradation routes of MPs at each phase of the sequential/hybrid process. This allows for identifying the roles and contributions of each method to the overall integrated/hybrid approach. This, in turn, unveils the mechanism of integrated processes, enabling their optimization to achieve the most effective results with minimal toxic by-products and time investment.

The biological treatment showed that it could reasonably reduce the COD and TOC; however, the adsorption of MPs on sludge cannot be neglected, as some non-biodegradable MPs like sulfonamides and quinolones absorb on the sludge and desorb to the wastewater matrix, making the effluent more polluted than it was [65,171].

On a positive note, methane production in anaerobic bioreactors could serve as a valuable contribution to renewable energy resources; nonetheless, the treatment proves to be economically inefficient as it exclusively targets the biodegradable MPs. On the other hand, the utilization of MBR and BAC columns showed promising results in MP removal in a shorter time compared to biological processes. Still, the production of secondary waste in membrane technology, membrane fouling, and the possible release of SMPs to wastewater are the main challenges facing the application of these technologies. Further studies focusing on alleviating membrane fouling, the modification of activated carbon and the membrane surface with nanoparticles, and the fate of MPs after the treatment in the concentrated stream are essential.

Pilot- and full-scale studies for MP and TrOC removal from water and wastewater through the sequential use of AOPs and BAC columns and hybrid ozonation-activated carbon adsorption have proven their efficiency [126,250]. Subsequent research endeavours should prioritize enhancing the performance of the BAC column to achieve more dissolved organic carbon (DOC) removal in a shorter time. Additionally, investigations should delve into the potentially toxic effects of absorbed metals and other pollutant chemicals on biofilm, along with their impact on the removal mechanisms. Managing microbial activity within the BAC column is essential, which potentially establishes a foundation for maintaining stability in DOC removal over an extended period.

Finally, investigating integrated/hybrid treatment approaches using actual effluents, expanding processes alongside cost evaluations, creating economically viable alternatives for industrial acceptance, and reducing water consumption through reuse constitute key research areas. Furthermore, thorough exploration is imperative to ensure the strength

and viability of large-scale integrated and hybrid processes for effectively eliminating MPs while mitigating the formation of harmful by-products.

7. Conclusions

Conventional wastewater treatment plants encounter challenges in effectively addressing wastewater containing micropollutants (MPs). While a significant portion of MPs can be eliminated through biological processes, they fall short of completely eradicating these emerging contaminants. The remaining non-degradable MPs might exit the treatment process and contaminate surface water. In response to this issue, the utilization of AOPs, membrane technologies, and activated carbon adsorption as polishing steps has been introduced. Despite their efficacy in degrading/removing a broad spectrum of MPs from wastewater, using these methods as standalone treatments is limited due to inherent drawbacks associated with each technique.

In recent years, the focus of studies has shifted towards developing sustainable wastewater treatment approaches that utilize and integrate various processes for effective MP removal while counteracting the limitations of individual treatment techniques. Developing such integrated treatment processes holds increasing significance in the wastewater treatment field. This study aimed to compile various integrated and hybrid treatment techniques utilized to remove MPs from real wastewater.

Multistage sequential and hybrid treatment strategies showed that MBR and BAC are the most promising technologies for removing MPs from wastewater. Nonetheless, the inconsistent removal of organic contaminants, attributed to issues like membrane blockage, activated carbon saturation, and the generation of secondary waste, prompted the enhancement in treatment performance by incorporating pre/post-treatment stages. Furthermore, the wide application of AOPs to mineralize and destroy the MPs from the wastewater matrix has been reported; however, the complications through oxidation reactions and the production of unknown by-products suggested the post-treatment of the effluents by AOPs.

Having thoroughly examined the existing body of open literature regarding the assessment of integrated treatment methods for actual wastewater containing MPs, the following suggestions concerning potential combinations of treatment choices are made:

The sequential treatment of biologically activated carbon followed by membrane technologies such as reverse osmosis, ultrafiltration, and microfiltration ensures effective MP and total nitrogen removal and offers an economical solution.

Ozonation or UV/H_2O_2 processes followed by an activated carbon adsorption column can effectively degrade the MPs and reach COD removals of up to 70%. This sequence eliminates the possible toxic by-products formed during oxidation and increases the biodegradability of the wastewater, while the reaction of residual oxidants with adsorbed organic pollutants can recover the adsorption capacity of activated carbon.

The pre-treatment of wastewater with biologically activated sludge to remove the biodegradable organics in the wastewater and the further removal of MPs through an effective oxidation process like ozonation or UV/H_2O_2 not only shorten the treatment time but also reduce the energy consumption of AOP while minimizing the operating cost of the integrated approach.

Hence, based on this evaluation, the optimal method consists of a sequential utilization of biological treatment, the AOP system, AC columns, and finally membrane treatment. This sequence is favoured due to the membrane treatment's capacity, in the final stage, to eliminate pollutants that persist through earlier treatment phases. The application of such a system necessitates a comprehensive assessment of its sustainability, economic viability, and ecological impact, supported by appropriate public policies.

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Abbreviations

Abs	Antibiotics
AC	Activated Carbon
AnBT	Anaerobic Biological Treatment
AO	Anodic Oxidation
AOP	Advanced Oxidation Process
ARGs	Antibiotic Resistance Genes
AS	Activated Sludge
BAC	Biological Activated Carbon
BAF	Biological Aerated Filter
BDD	Boron-Doped Diamond
BPAC	Biological Powdered Activated Carbon
CAS	Conventional Activated Sludge
CAUF	Coagulation–Adsorption–Ultrafiltration
CBZ	Carbamazepine
CF	Coagulation–Flocculation
CI	Current Intensity
CM	Catalytic Membrane
CMF	Ceramic Membrane Filtration
COD	Chemical Oxygen Demand
COUF	Coagulation–Oxidation–Ultrafiltration
CSTR	Continuous Stirred Tank Reactor
CUF	Enhanced Coagulation
D	Diameter
DBP	Disinfection By-Product
DOC	Dissolved Organic Carbon
DOM	Dissolved Organic Matter
DSA	Dimensionally Stable Anode
EBCT	Empty Bed Contact Time
EC	Electro-Coagulation
ECs	Emerging Contaminants
EF	Electro-Fenton
EO	Electro-Oxidation
FBR	Fluidized Bed Reactor
FBBR	Fluidized Bed Bioreactor
FLUT	Flutamide
GAC	Granular Activated Carbon
HRT	Hydraulic Retention Time
HSWW	High-Strength Wastewater
IEX	Ion Exchange
LSWW	Low-Strength Wastewater
MBR	Membrane bioreactor
MBBR	Moving-Bed Bioreactor

MF	Microfiltration
MFC	Microbial Fuel Cell
MLSS	Mixed Liquor Suspended Solids
MO	Microorganisms
MPs	Micropollutants
MWCO	Molecular Weight Cut-Off
MWW	Municipal Wastewater
MWWTP	Municipal Wastewater Treatment Plant
NDMA	N-nitrosodimethylamine
NF	Nanofiltration
NMOR	N-nitrosomorpholine
NOM	Natural Organic Matter
OLR	Organic Loading Rate
OM	Organic Matter
OMP	Organic Micropollutant
OTC	Oxytetracycline
OTP	Oxidation Transformation Products
PAC	Powdered Activated Carbon
PAlCl	Polyaluminium Chloride
PCD	Pulsed Corona Discharge
PCO	Photocatalytic Oxidation
PE	Primary Effluent
p.e.	Population equivalent
Q	Flow rate
RO	Reverse Osmosis
RT	Retention Time
SBR	Sequencing Batch Reactors
SCPC	Solar Compound Parabolic Collector
SE	Secondary Effluent
SF	Sand Filtration
SMP	Soluble Microbial Products
Sono_PF	Sono-Photo-Fenton
SPCO	Solar Photocatalytic Ozonation
SPF	Solar Photo-Fenton
SPFO	Solar Photo-Fenton Ozonation
SRT	Sludge Retention Time
SWW	Sewage Wastewater
TE	Tertiary Effluent
TMP	Trans Membrane Pressure
TN	Total Nitrogen
TOC	Total Organic Carbon
TP	Total Phosphorous
TPs	Transformation Products
TrOC	Trace Organic Contaminant
UF	Ultrafiltration
V	Volume
WW	Wastewater
WWTP	Wastewater treatment Plant

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