




Review

Adsorption and Photo(electro)catalysis for Micropollutant Degradation at the Outlet of Wastewater Treatment Plants: Bibliometric Analysis and Challenges to Implementation

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Abstract: Micropollutants (MPs), which include both natural and manmade substances, are becoming more prevalent in aquatic habitats as a result of the insufficient removal of these compounds in wastewater treatment plants (WWTPs). Advanced remediation techniques are required due to their persistence and potential ecotoxicological hazards. Although adsorption and photo(electro)catalysis exhibit potential in laboratory-scale investigations, the effects of their use in actual WWTP systems are still poorly understood. However, before large-scale application can be implemented, a number of issues need to be resolved, including material limitations, reactor design and optimization, and actual wastewater complexities. This study critically evaluates the application of adsorption and photo(electro)catalysis to actual wastewater, as well as recent advancements in adsorption and photo(electro)catalytic systems for the removal of micropollutants. We also explore the particular difficulties and strategies involved in the large-scale use of adsorption and photo(electro)catalysis in the treatment of wastewater. Emerging trends such as nanocomposites, metal–organic frameworks (MOFs), heterojunctions, and single-atom catalysts (SACs) are highlighted by the bibliometric analysis. We also evaluate MPs' ecological effects in aquatic environments and the incorporation of artificial intelligence (AI) for process optimization. A strategy for transferring nanotechnologies from laboratory-scale research to wastewater treatment implementation is presented in this paper. In this strategy, implementation is proposed based on actual wastewater conditions, focusing on the development of adsorbents and catalysts, reactor design and optimization, synergy between adsorption and catalysis, life cycle analysis, and cost–benefit studies.

Keywords: adsorption; bibliometric analysis; micropollutants; nanotechnology; photo(electro)catalysis; wastewater treatment



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1. Introduction

Micropollutants (MPs) are defined as natural or synthetic compounds that are introduced into aquatic habitats by point and non-point sources, including wastewater treatment plants (WWTPs), and are referred to as emerging pollutants [1]. The persistent nature and propensity for bioaccumulation shown by these contaminants—pharmaceuticals, pesticides, microplastics, and perfluorinated compounds—make them a serious threat to human health and ecosystems [2,3]. Numerous studies have shown that a wide array of MPs have been detected in various water samples, including pharmaceuticals, personal care products, pesticides,

industrial chemicals, persistent organic pollutants, steroid hormones, artificial sweeteners, and nanomaterials [3–6]. According to Terry and Summers [7], non-biodegradable MPs are mainly ignored by conventional WWTPs, which mainly target pollutants that can be biodegraded.

Recent developments in nanotechnology, especially in the areas of adsorption and photo(electro)catalysis, provide innovative solutions. High-surface-area nanomaterials such as graphene and metal–organic frameworks (MOFs) are used in adsorption [8], while photo(electro)catalysis combines photocatalysis with electrochemical oxidation to increase the efficiency of degradation [9]. Even with encouraging lab-scale outcomes, there are still issues with scaling these technologies for practical WWTP applications, for example, reactor design, energy consumption, and catalyst stability [2].

This review fills important gaps by (1) evaluating the suitability of photo(electro)catalysis and adsorption in actual wastewater, (2) mapping trends in nano-adsorbents and photo(electro)catalysis via bibliometric analysis, and (3) making recommendations for AI integration, hybrid systems, and reactor designs/optimization.

2. Emerging Micropollutants: Pollutant Levels, Source, Fate, and Risks

2.1. Current Status of Micropollutants: Global Concentration Levels

Pharmaceuticals, pesticides, industrial chemicals, personal care items, and other micropollutants are found in water systems around the world at trace levels (ng L^{-1} to $\mu\text{g L}^{-1}$), but their persistence and bioaccumulation pose serious dangers to human health and the environment. This section examines their regional differences and global distribution patterns, offering information on environmental exposure and regulatory obstacles.

2.1.1. The Global Distribution of Micropollutants: Example of 17β -Estradiol (E2)

Micropollutant concentrations vary by continent according to recent studies. Research carried out in recent years in different locations around the world investigated the concentration ranges in which 17β -estradiol (E2) was identified in effluents that received conventional treatment. For example, in the United States, the levels of E2 were found to be as high as 250 ng L^{-1} in surface water and as high as 147 ng L^{-1} in groundwater. These levels have been ascribed to both environmental and human factors, including the excessive use of pharmaceuticals and inadequate treatment facilities [10].

Brazil reported the highest concentrations of E2 in drinking water at 33 ng L^{-1} , while China observed E2 levels in surface water as high 17.28 ng L^{-1} . Denmark, on the other hand, reported lower peak concentrations of 14 ng L^{-1} [10].

E2 concentrations in African waters ranged from 0.2 to 4.3 ng L^{-1} [11], and in Europe, they ranged from 0.1 to 85 ng L^{-1} [12], according to a more comprehensive continental analysis. These variations indicate different legal requirements, usage trends, and treatment efficiencies.

2.1.2. Geographical Disparities and Their Implications

Europe: Pollutant Diversity

In current studies (2024) of water quality in European rivers by the Helmholtz Centre for Environmental Research, 610 chemicals whose occurrence or problematic effects are known have been examined in more detail and analyzed to determine whether and, if so, in what concentrations they occur in Europe's freshwater systems. After analyzing 445 samples from 22 different rivers (in 22 European countries), researchers found that 504 out of the 610 compounds were present. They discovered a total of 175 medicinal chemicals, 229 insecticides and biocides, corrosion inhibitors, surfactants, plastic and rubber additives, and per- and polyfluoroalkyl substances (PFASs).

The most frequently detected compounds included the pharmaceutical metabolite N-acetyl-4-aminoantipyrine, metolachlor-ethane sulfonic acid, a transformation product

(TP) of the legacy herbicide metolachlor, the industrial compound m-xylene-4-sulfonic acid, the antiepileptic drug carbamazepine, the sweeteners acesulfame, cyclamate, and sucralose, the vulcanization accelerator/rubber component 1,3-diphenylguanidine, the corrosion inhibitor 5-methyl-1H-benzotriazole, the nicotine TP cotinine, the UV filters phenylbenzimidazole sulfonic acid and benzophenone-4, the herbicide bentazone and herbicide TP metazachlor ESA, and the industrial compound 4'-aminoacetanilide [13].

The measured environmental concentrations (MECs) of the detected pollutants ranged from 1 ng L^{-1} to $74 \text{ } \mu\text{g L}^{-1}$ [13], demonstrating the complex chemical burden on European freshwater systems as well as extensive contamination.

Asia: China and Other Nations

China and Europe exhibit high levels and diversity of MPs, which suggests higher chemical use and enhanced detecting abilities. We will concentrate on China in this section. Organic micropollutants (OMPs) in reclaimed water have been the subject of extensive research in China (24 provincial administrative regions and 4 municipalities). In total, 325 compounds passed pre-selection out of the 369 OMPs found in 11 chemical classes. Among them, pesticides accounted for the highest number of OMPs, totaling 81, followed by pharmaceuticals, organophosphates, and antibiotics, with 55, 53, and 41, respectively. Following these compounds, with quantities of 294.57 ng L^{-1} , 248.18 ng L^{-1} , 133.83 ng L^{-1} , 126.06 ng L^{-1} , and 92.04 ng L^{-1} , respectively, were organophosphate esters (OPEs), PFCs, antibiotics, pesticides, and pharmaceuticals. The average maximum concentrations for polychlorinated biphenyls (PCBs) and polycyclic aromatic hydrocarbons (PAHs) were 1.96 ng L^{-1} and 12.7 ng L^{-1} , respectively [14]. Concerns regarding the dangers of long-term exposure and environmental persistence are raised by these numbers, which represent China's industrial activity and agricultural intensity. According to multiple studies carried out in Malaysia and India, the level of sulphapyridine was found to be 10.2 ng L^{-1} (India) [15], while that of metoprolol was found to be 39 ng L^{-1} (Malaysia) [16]. Some parts of Asia have unique pollution burdens that may be understated due to inadequate infrastructure.

Africa: A Potential Hotspot Location

Significant micropollutant burdens are highlighted by data from nations such as Kenya and South Africa, despite their lack of adequate monitoring infrastructure. For example, the concentrations of medicaments such as paracetamol, metronidazole, chloramphenicol, naproxen, and carbamazepine in aquatic environment are as follows, according to numerous studies conducted in Kenya and South Africa: paracetamol, 107 ng L^{-1} (Kenya) [17]; metronidazole, $7\text{--}10 \text{ ng L}^{-1}$ (Kenya) [17]; carbamazepine, $30\text{--}40 \text{ ng L}^{-1}$ (Kenya) [18]; and naproxen, 360 ng/L (South Africa) [19,20]. These results highlight the critical need for better wastewater treatment and monitoring in developing areas and to identify localized pollution hotspots.

Implications of Geographical Disparities

The presence of micropollutants varies around the world due to geographical variations in wastewater management techniques, industrial activity, regulations, and pharmaceutical use. High quantities and diversity of MPs are seen in China and Europe, indicating greater chemical use and improved detection skills. Although less research has been performed in Africa and some parts of Asia, these areas exhibit specific pollutant burdens that might be understated due to inadequate infrastructure.

2.2. Sources, Effects, and Fate

MPs originate from diverse sources (Table 1), including pharmaceuticals and endocrine-disrupting chemicals (EDCs), pesticides, microplastics, and PFCs. WWTPs

remain a major pathway for the introduction of MPs into water bodies, with effluent concentrations reflecting incomplete removal and the need for secondary treatment.

Over the past decade, microbial hazards to human health and environments have increased due to antibiotic resistance genes (ARGs). Table 1 shows a positive correlation between the horizontal transmission of ARGs mediated by class I integron (*intI1*) and the development of drug resistance in microorganisms in the environment. Bacitracin, sulfonamides, and tetracyclines are dominant ARG types [21]. Their distribution is affected by interactions with other micropollutants in WWTPs.

In biofilters, both the 5-day biochemical oxygen demand (BOD 5) and the type of filter medium positively affect the degradation of pharmaceutical pollutants such as atenolol, propranolol, venlafaxine, citalopram, metoprolol, iohexol, and diclofenac. Biological treatment is typically the main treatment method used in sewage treatment [22].

One common class of environmental endocrine disruptors is steroid hormones. Steroid hormones (such as 17β -estradiol) are considered micropollutants and represent a serious danger to ecosystem health, even at low quantities. 17β -estradiol (E2) is emitted by various sources, such as animal and human excretions, hospital and veterinary clinic effluents, and treatment plants. In aquatic biota, it can cause issues ranging from the feminization of males to inhibiting plant growth. The interaction between dissolved organic compounds, 17β -estradiol (E2), and negative charges on the surface of nanotubes inhibited the adsorption of this hormone [23]. Bisphenol A (BPA) is another example of an EDC. BPA is the most typical representative of the bisphenols (BPs). BPA has been characterized as a “pseudo-persistent” chemical, leading to its spread and potential accumulation in a variety of environmental compartments, including water and sediment, etc. It has been well documented that BPA is related to various adverse health effects in wildlife and humans, such as cancer, infertility, cardiovascular diseases, diabetes, and neurodegenerative diseases [24].

Microplastics can originate from a number of sources, such as the fishing industry, household grey water, and WWTPs. Usually, they are visible as fragments and fibres on the water's surface and in sediments (Table 1). Plastic additives affect the density and source identification of microplastics. The primary constituents of microplastics include polyethylene, polypropylene, polyester and acrylic fibres, nylon, polyethylene terephthalate, and other polymers. Along with treated wastewater, a significant number of plastic pieces measuring between 25 μ m and 50 mm are released into the environment [25].

Mecoprop and diazinon represent pesticides that often come from surface runoff and agriculture. Diazinon is a compound often found at a high concentration in urban wastewater, and its concentration in effluent is also often high. Mecoprop is typically used as an indicator substance because it is absent in background water and is exclusively found in wastewater [26]. Many of the compounds with the greatest potential for risk to the environment and human health are those that were observed with the lowest frequency of occurrence [27].

For decades, the food industry has been using low-calorie sweeteners, of natural or artificial origin, as substitutes for sucrose and other sugars. After being consumed, most of these low-calorie sweeteners are excreted as the parent compound, reaching urban sewage and, when not removed in WWTPs, potentially reaching and contaminating surface waters. It has been widely documented that sweetener concentrations in WWTP outputs differ mainly due to the different treatment setups employed [28].

Perfluorinated compounds (PFCs) such as perfluorooctanoate (PFOA), perfluorohexanoate (PFHpA), perfluorooctane sulfonate (PFOS), and PFC conversion products are transported by means of suspended solids [29]. Sediments are strongly attracted to perfluorinated compounds, mainly due to the long carbon chain and bulky functional groups of PFCs [29]. The contamination level of PFASs in various environmental media fluctuate greatly [30].

Table 1. Micropollutants (MPs), pollutant levels, sources, fates, and their effects.

Class	Example	Source	Pollutant Levels (Concentration)	Fate ^a	Effects ^b	Reference
Pharmaceutical contaminants	Antibiotic resistance genes (ARGs): tetracycline; sulfonamide; bacitracin	WWTPs and animal feces	Jiulong River, China: Bacitracin (22.8% of the total ARGs), multidrug (20.7%), sulfonamide (15.2%) and tetracycline (10.9%) were the dominant ARG types.	Class 1 integrons are genetic elements that carry a variable set of antibiotic resistance genes. Horizontal gene transfer via Class 1 integrons (intI1)	ARGs facilitate the spread of antibiotic resistance, increasing risks to human health and disrupting microbial ecosystems	[21]
	Sulfamethoxazole, tetracycline, and bisphenol A	Industrial wastewater (chemical, pharmaceutical) plants	The pollutant levels were not disclosed.	Pharmaceutical contaminants undergo co-metabolic degradation, driven by carbon-induced enzyme/EPS production	Treatment reduces their persistence and bioaccumulation in the environment	[31]
	Atenolol, propranolol, venlafaxine, citalopram, metoprolol, iohexol, and diclofenac	Stormwater and combined sewer overflow	Bjergmarken WWTPs (Roskilde, Denmark): Metoprolol $1.9 \pm 0.3 \mu\text{g L}^{-1}$ Iohexol $3.9 \pm 2.1 \mu\text{g L}^{-1}$ Diclofenac $0.5 \pm 0.1 \mu\text{g L}^{-1}$ Ibuprofen $0.2 \pm 0.1 \mu\text{g L}^{-1}$	Five-day biochemical oxygen demand (BOD ₅) and biofilter materials influence pharmaceutical degradation pathways	Analysis can be used to determine whether compounds are biodegraded, adsorbed, or converted into metabolites	[22]
Endocrine-disrupting chemicals (EDCs)	Steroid hormones: 17 β -estradiol (E2)	Hospital and veterinary clinic effluents and treatment plants	Europe: 0.1–85 ng L ^{−1} China: 44.5 ng L ^{−1} Africa: 0.2–4.3 ng L ^{−1}	The adsorption of E2 was inhibited due to interactions with dissolved organic compounds and the negatively charged surfaces of nanotubes	In aquatic organisms, E2 exposure may lead to adverse effects such as inhibited plant growth	[23]
	Bisphenols (BPA, BPAF, BPS, DHBP and BPB)	BPA was the predominant BP in both water and sediment samples	Beibu Gulf, South China Sea: BPA 5.26 to 12.04 ng L ^{−1} (water) and 0.56 to 5.22 ng g ^{−1} (sediment) BPAF 0.44–0.60 ng L ^{−1} (water) and 0.08–0.66 ng g ^{−1} (sediment) BPS: 0.07–0.63 ng L ^{−1} (water)	BPs in the Beibu Gulf show sediment partitioning (high log K _{oc} *), with BPA being dominant	BPs display stronger binding in marine versus freshwater systems	[32]
Microplastic particles (MPs)	Black fibres	Greywater/river and fishing	South of Caspian Sea, Iran: 15 units kg ^{−1} (sediments) 710 units m ^{−3} (water)	MPs are widespread in the Caspian Sea, especially fibres	This highlights the urgency of addressing marine MP pollution to protect ecosystems and human health	[33]
	Fragments (main ingredient) and fibres	WWTPs	WWTPs, Madrid (Spain): 12.8 \pm 6.3 particles/L 183 \pm 84 particles/g (sludge)	WWTPs remove most MPs. However, residuals MPs in effluent and sludge contribute to river and soil contamination	Residual MPs pose risks to aquatic and terrestrial ecosystems, potentially impacting biodiversity, soil health, and human food safety	[25]

Table 1. Cont.

Class	Example	Source	Pollutant Levels (Concentration)	Fate ^a	Effects ^b	Reference
Artificial sweeteners	Acesulfame	WWTPs	The pollutant levels were not disclosed.	Acesulfame K is a stable, persistent compound that resists degradation during wastewater treatment, making it a reliable tracer for identifying wastewater contamination in surface and groundwater	Acesulfame K concentrations in aquatic environments are generally low and pose minimal ecological risk	[26]
	Low-calorie sweeteners (LCS): acesulfame (ACE), sucralose (SUC), saccharin (SAC), cyclamate (CYC), aspartame (ASP), neotame (NEO), and stevioside (STV)	WWTPs	WWTPs, Metropolitan region of Campinas (São Paulo State, Brazil): CYC 1–138 µg L ^{−1} ACE 89 µg L ^{−1} (median) SAC 55 µg L ^{−1} (median) SUC 11–42 µg L ^{−1} NEO; ASP; STV: not detected.	CYC and SAC are readily biodegraded, whereas ACE and SUC persist. Due to its stability, SUC serves as an effective tracer for wastewater contamination	Current low-calorie sweetener (LCS) concentrations in Brazilian surface waters pose minimal ecological risk, but continued monitoring is necessary to assess their potential cumulative impacts over time	[28]
Pesticides	Mecoprop (herbicides and fungicides)	Agriculture and surface runoff	The pollutant levels were not disclosed.	Due to its widespread use and relatively high concentrations, mecoprop is proposed as an indicator of urban runoff, though regional variations in pesticide use may affect its reliability	Mecoprop's utility as an indicator is mainly confined to urban areas, limiting its broader applicability in diverse land-use settings	[26]
	Diazinon (organophosphates)	Agriculture and surface runoff	WWTPs of Torroella, Girona, Northeastern Spain: Diazinon 479–607 ng L ^{−1} (influent) 61–93 ng L ^{−1} (effluent)	Diazinon has been frequently detected at the highest concentrations among pesticides in urban wastewater, indicating significant use and persistence	Diazinon enters urban waterways via effluent from sewage treatment plants, contributing to environmental contamination and potential ecological risks	[34]
	Atrazine, nicotine, dinoterb, bentazone, and deethylatrazine (DEA)	Aquifers (groundwater/surface water)	The pollutant levels were not disclosed.	Micropollutants such as thiamethoxam and carbendazim persist in groundwater due to their high leaching potential (GUS index **) and resistance to degradation, highlighting the need for long-term monitoring	Compounds with the highest environmental and human health risks are often those detected least frequently, underscoring the importance of monitoring low-occurrence but high-risk contaminants	[27]

Table 1. Cont.

Class	Example	Source	Pollutant Levels (Concentration)	Fate ^a	Effects ^b	Reference
Perfluorocarburess (PFCs)	Perfluoroalkyl carboxylates (PFCA), especially perfluorooctanoate (PFOA), perfluorohexanoate (PFHpA), perfluorooctane sulfonate (PFOS), and PFC conversion products	Sedimentation was the major sink for PFCs	Marina Reservoir, Singapore: PFCs 4700 ng kg ^{−1} (Sediment). PFOS was dominant PFCs	Urban stormwater runoff introduces PFCs into reservoirs, with suspended solids (SSs) facilitating their transport and leading to significant sediment accumulation. PFOS and 6:2 FtS are predominant due to their strong affinity for sediments	Sediment-bound PFCs threaten benthic ecosystems, while stratification and SS dynamics influence their distribution and bioavailability	[29]
	Perfluoroalkyl substances (PFASs)	Rivers; drinking water sources (reservoirs and groundwater) Location: Qingdao (China)	Qingdao, China: PFASs:28.3–292.2 ng L ^{−1} ; PFB: 256 ng L ^{−1} (max); PFOA: 72.4 ng L ^{−1} (max); PFBA: 41.6 ng L ^{−1} (max);	PFASs are prevalent in Qingdao’s rivers, particularly in suburban and rural areas, with contamination flowing into Jiaozhou Bay. Lower but detectable levels are also found in drinking water reservoirs and tap water	While immediate human health risks are considered low, ecological impacts and concerns over chronic exposure warrant continuous monitoring	[30]

^a Fate: Processes determining the distribution, transformation, and persistence of MPs in the environment (e.g., adsorption, degradation, bioaccumulation). ^b Effects: Direct or indirect impacts of MPs on ecosystems or human health. * Log Koc refers to the logarithm (base 10) of the organic carbon–water partition coefficient (Koc). ** GUS index: Groundwater ubiquity score (GUS) index.

2.3. Ecotoxicological Risk Assessment

Ecotoxicological risk assessments (ERA) often underestimate the cumulative effects of MP mixtures. Traditional ERA methods, such as risk quotient (RQ) analysis, focus on individual toxicants, neglecting the real environment's complex mixtures. Here, we synthesize findings from recent research to propose pathways for advancing risk assessment frameworks.

Conventional ERA methods focus on individual toxicants; for example, the EU Water Framework Directive has set Environmental Quality Standards (EQSs) for substances of national concern [35]. Frameworks that only focus on compounds belonging to a particular use or application class, e.g., pesticides or biocides, are necessary, but seem insufficient in light of the increasing evidence underlining the importance of considering co-exposure to multiple chemicals from different application classes in order to more adequately estimate the risks they pose. Emerging territory-based approaches reveal synergistic risks in aquatic ecosystems [36,37]. Additional papers have shown that toxicants can not only act individually but also collectively and synergistically. An effective national response to mixture toxicity, cumulative risk, and environmental injustice has not yet been established [35].

Emerging approaches address these limitations through mechanistic and predictive models (such as the maximum cumulative ratio and ecological network analysis), effect-based methods, and multi-generation studies. For example, in Tai Lake, China, the maximum cumulative ratio (MCR) was used as an approach to evaluate and prioritize risks of co-exposure to metals. The results of a multi-generation study demonstrated the adverse effects of a mixture of metals (such as copper, lead, and zinc) at environmentally relevant concentrations on the growth and reproduction of *Daphnia magna* and *Moina macrocopa* [38]. A 2025 study demonstrated that effect-based methods were able to assess the potential ecotoxicological risks posed by chemical mixtures present in aquatic ecosystems. These effect-based assessments better capture temporal variations in potential ecotoxicological risks than traditional chemical analyses [39].

The classic mixture toxicity concept of concentration addition was used to calculate the total risk of the analytically determined mixtures. The RQ of a single, randomly selected pollutant is often more than a factor of 1000 lower than the mixture risk. The need for a systematic analysis of the overall risk of all pollutants has been clearly demonstrated. The top 10 mixture components explain more than 95% of the mixture risk in all cases [40].

The most sensitive lifeforms in rivers are fish and invertebrates, whose mixed risk quotient (MRQ) values for micropollutants in rivers frequently surpass 1 due to PAHs and UV filters [41]. Second, the diversity and uniformity of zooplankton levels were significantly negatively correlated with the presence of micropollutants in the water [41].

Sloping continental margins and submarine canyons are important for understanding the continental shelf/deep ocean exchange of particulate pollutants and its impacts on marine ecosystems. Ref. [42] found that the highest concentrations of PAHs, UV filters (EHMC, etc.), and muscone were measured in submarine canyon sediments far from the coast, indicating that priority and emergent pollutants increase the risk to benthic animals.

In the future, the transition from single-substance to mixture-based ERA is imperative to address underestimated ecological risks. Integrating advanced modelling (such as Monte Carlo simulations), effect-based tools, and multi-generational data can bridge current gaps, while policy reforms must prioritize holistic frameworks to safeguard biodiversity and ecosystem services.

3. Systematic Analysis of Micropollutant Removal Technologies

Adsorption. Adsorption on highly porous materials has been proven to be an effective method for removing micropollutants. The adsorption process has been found to be superior to other technologies in terms of simplicity of design, initial costs, operation, and insensitivity to toxic substrates. Adsorbents require energy-intensive thermal regeneration, which raises operational costs and environmental concerns [43]. Research gaps include cell- and molecular-level studies, pilot-scale and field-scale experiments, cost–benefit calculation, the efficiency of adsorbents, microbial interactions in aquatic environments, and the impact of geo-environmental factors on adsorption mechanisms [44].

Membrane separation. Research has investigated the separation of microcontaminants from urban wastewater using ultrafiltration and nanofiltration membranes. Conventional systems offer advantages like low pressure, fouling, and competitive costs. However, the rejection rates depend on temperature, flow, pressure, surface charge, and concentration, making them challenging for complex matrices [45]. Recent material science advancements have improved the implementation potential of different membrane types in industrial processes, particularly wastewater treatment.

Advanced oxidation processes (AOPs). Different advanced oxidation processes have been discussed, including ozonation, Fenton reactions, ultrasound, electrochemical oxidation and photocatalysis. These processes generate reactive oxygen species (ROS) to oxidize pollutants. All of these processes are associated with one another, displaying similar limitations; the findings regarding these processes are summarized in Table 2. Among these processes, photocatalysis stands out for its potential to completely mineralize MPs under ambient conditions without toxic byproducts. [46].

Table 2. Merits and demerits of different AOPs. Reprinted with permission from [46]. Copyright © 2023 IWA.

Method	Merits	Demerits
Ozone-based AOPs	<ul style="list-style-type: none">• High oxidation power: Ozone is a strong oxidant that can effectively degrade a wide range of organic and inorganic pollutants in water and wastewater. This makes it an effective method for the treatment of complex wastewater with recalcitrant pollutants.• Versatile: Ozone can be used as a standalone treatment method or can be combined with other AOPs like UV, hydrogen peroxide, or catalysts to enhance the treatment efficiency.• Fast reaction time: Ozone has a short reaction time, typically in seconds, which makes it suitable for treating large volumes of water or wastewater in a short amount of time.• No residuals: Unlike some other oxidants, ozone does not leave any harmful residual byproducts after treatment, which makes it environmentally friendly.	<ul style="list-style-type: none">• High cost: Ozone production requires a significant amount of energy, making it an expensive treatment option compared to other methods.• Complex system: Ozone-based AOPs require specialized equipment and control systems that can be complex to operate and maintain.• Risk of ozone exposure: Ozone is a respiratory irritant and can be harmful to human health at high concentrations. This makes it important to ensure proper safety measures are in place to protect workers and the environment.• Limited efficiency: While ozone can be effective at degrading many pollutants, it may not be as effective against certain pollutants or wastewater types and may require the use of additional treatment methods.

Table 2. Cont.

Method	Merits	Demerits
Fenton process	<ul style="list-style-type: none"> High efficiency: Fenton's reagent has been shown to effectively remove a wide range of organic and inorganic pollutants in water and wastewater. This is due to the high oxidative power of the hydroxyl radicals generated by the reaction of Fe^{2+} and H_2O_2. Low cost: The reagents used in Fenton's process, including Fe^{2+} and H_2O_2, are relatively inexpensive, making it a cost-effective treatment option. Simple operation: Fenton's process is relatively easy to operate and can be implemented on a small or large scale. Can operate at neutral pH: Fenton's reaction can occur at neutral pH, unlike other AOPs that require an acidic pH. 	<ul style="list-style-type: none"> Sludge generation: Fenton's reaction can produce sludge which requires proper disposal. pH limitation: Fenton's process requires low pH (<3), which can cause equipment corrosion and increase the cost of treatment. Iron impurities: Fenton's reagent is sensitive to iron impurities, which can decrease its effectiveness.
TiO ₂ photocatalyst	<ul style="list-style-type: none"> Effective at low concentrations: TiO₂ photocatalysts are effective at low concentrations and can efficiently degrade a wide range of organic and inorganic pollutants. Versatility: TiO₂ photocatalysts can be used in a variety of treatment processes such as batch or continuous flow processes. No toxic byproducts: TiO₂ photocatalysis does not produce any toxic byproducts during the reaction, which makes it an environmentally friendly treatment method. High stability: TiO₂ photocatalysts are stable and can be reused multiple times, making it a cost-effective treatment option. 	<ul style="list-style-type: none"> UV light requirement: TiO₂ photocatalysts require UV light to activate the photocatalytic process, which increases the cost of treatment. Surface fouling: The surface of the TiO₂ photocatalysts can become fouled with pollutants over time, which can decrease the effectiveness of the catalyst. Narrow wavelength range: The effectiveness of TiO₂ photocatalysis is limited to a narrow range of UV wavelengths, which can limit its applicability in certain situations.
Electron beam	<ul style="list-style-type: none"> High oxidation power: Electron beam irradiation generates reactive species, such as hydroxyl radicals, which can effectively degrade a wide range of pollutants in water and wastewater. Fast reaction time: Electron beam irradiation has a short reaction time, typically in milliseconds, which makes it suitable for treating large volumes of water or wastewater in a short amount of time. No chemicals required: Unlike some other AOPs, electron beam irradiation does not require the addition of any chemicals, which makes it an environmentally friendly treatment method. Low residuals: Electron beam irradiation does not leave any significant residual byproducts after treatment, which reduces the need for further treatment and disposal costs. 	<ul style="list-style-type: none"> High cost: Electron beam irradiation requires specialized equipment and high energy consumption, making it an expensive treatment option compared to other methods. Complex system: Electron beam-based AOPs require specialized equipment and control systems that can be complex to operate and maintain. Possible radiolytic byproducts: The irradiation process can produce radiolytic byproducts, which can pose a risk to the environment and human health if not handled properly. Limited efficiency: While electron beam irradiation can be effective at degrading many pollutants, it may not be as effective against certain pollutants or wastewater types and may require the use of additional treatment methods.

Table 2. *Cont.*

Method	Merits	Demerits
Ultrasound	<ul style="list-style-type: none"> • Low cost: Compared to other physical AOPs such as electron beam, ultrasound is a relatively low-cost treatment option. • Environmentally friendly: Ultrasound does not generate any chemicals or byproducts during treatment, which makes it an environmentally friendly treatment method. • High efficiency: Ultrasound can effectively degrade a wide range of pollutants in water and wastewater and can be used in combination with other treatment methods to enhance the degradation efficiency. • Easy to scale up: Ultrasound can be easily scaled up for large-scale applications and can be used in both batch and continuous flow processes. 	<ul style="list-style-type: none"> • Limited penetration depth: Ultrasound has a limited penetration depth, which can restrict the treatment of deep-seated contaminants in water and wastewater. • Energy consumption: Ultrasound requires energy to generate the sound waves, and the energy consumption can be high depending on the frequency and power used. • Possible formation of toxic byproducts: Under certain conditions, such as the presence of bromide ions in water, ultrasound can form toxic byproducts, such as bromate, which can be harmful to human health. • Equipment limitations: Ultrasound equipment can be sensitive to temperature and pressure variations and can require regular maintenance to ensure proper operation.
Microwave	<ul style="list-style-type: none"> • High efficiency: Microwave irradiation generates heat rapidly and uniformly, which can effectively degrade a wide range of pollutants in water and wastewater. • Fast reaction time: Microwave irradiation has a short reaction time, typically in seconds to minutes, which makes it suitable for treating large volumes of water or wastewater in a short amount of time. • Easy to operate: Microwave-based AOPs are relatively easy to operate and can be controlled by adjusting the microwave power and irradiation time. • Scalable: Microwave-based AOPs can be easily scaled up for large-scale applications and can be used in both batch and continuous flow processes. 	<ul style="list-style-type: none"> • High energy consumption: Microwave irradiation requires high energy consumption and can be expensive compared to other physical AOPs such as ultrasound. • Equipment limitations: Microwave equipment can be sensitive to temperature and pressure variations and can require regular maintenance to ensure proper operation. • Safety concerns: Microwave irradiation can pose a safety risk to operators if not handled properly, due to the potential for electromagnetic radiation exposure. • Formation of byproducts: Microwave irradiation can generate byproducts, such as carbon dioxide and nitrogen oxides, which can contribute to environmental problems if not properly handled.

Coupling technology. Coupled methods such as adsorption–photocatalysis and photo(electro)catalysis are the subject of an increasing number of investigations. Studies have shown that photo(electro)catalysis works better than direct photolysis, photocatalysis, and conventional electrochemical oxidation [9]. In Section 4, we will focus on the advantages of photo(electro)catalysis for degrading micropollutants and the challenges of applying it to large-scale wastewater treatment. We will also briefly introduce the coupling of membrane separation with photocatalysis or photo(electro)catalysis.

4. Photo(electro)catalysis

Photo(electro)catalysis, which combines photocatalysis and electrochemical oxidation, has become a practical method for decomposing non-biodegradable micropollutants (such as microplastics) in wastewater. The photo(electro)catalysis process requires light energy, which may be obtained from sunlight, to function efficiently at room temperature and pressure. This makes it both ecologically benign and energy-efficient [47]. In contrast to

independent photocatalysis, photo(electro)catalysis enhances ROS formation and improves degradation rates by combining photocatalysis with electrochemical processes [48].

Photo(electro)catalysis consists of irradiating a photocatalyst deposited on an electrode with light (UV or light visible) and simultaneously applying a constant anodic potential (E_a) or constant current density (j), thus avoiding the recombination of (e^-/h^+) pairs generated in the photocatalytic process, as shown in Figure 1. When the photocatalyst is illuminated, if the energy of the incident photon is higher than the energy bandgap of the semiconductor photocatalyst ($h\nu > E_{bg}$), the electrons e^- in the valence band VB (e_{VB}^-) are excited and promoted to the conduction band CB (e_{CB}^-), generating a positive vacancy or hole h^+ in the valence band VB (h_{VB}^+), as shown in Figure 1 [49].

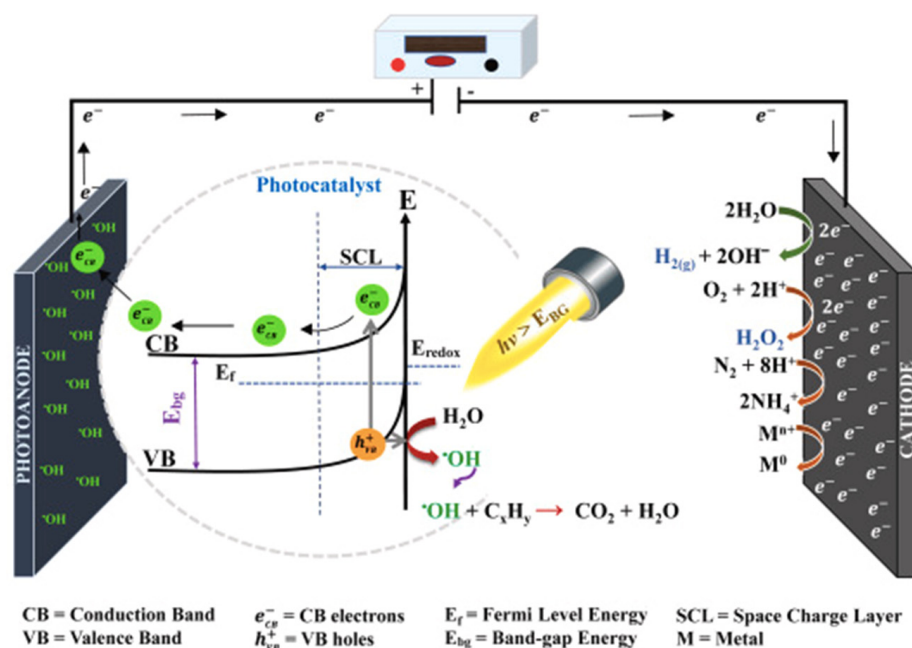


Figure 1. Scheme of photoelectrocatalytic mechanism in organic pollutants degradation in aqueous medium. Reprinted with permission from [49]. Arrows indicate the electron motion and the different reactions that take place. Copyright © 2021 Elsevier.

However, before large-scale application can be achieved, a number of issues need to be resolved, including material limitations, reactor design and optimization, and real wastewater's complexities.

4.1. Material Limitations

Photocatalysis's capacity to harness solar energy further supports its sustainability. However, stability problems in wastewater settings and rapid charge recombination prevent visible-light-active materials from being used in practical applications [50]. Heterogeneous photocatalysis has been extensively studied for the removal of contaminants from aqueous matrices; nevertheless, practical wastewater treatment applications are still limited in number.

New developments in photo(electro)catalyst materials, such as Mxenes and Z-scheme heterostructures, have demonstrated improved stability and charge separation. Mxenes provide enhanced electro transfer rates because of their tunable electronic characteristics [51]. The benefits of a Z-scheme photocatalyst, which mimics natural photosynthesis, include enhanced daylighting, spatial separation of reduction and oxidation active sites, and high redox capacity preservation. These features all help to improve photocatalytic performance [52].

Despite these advances, the industrial-scale synthesis of these materials remains cost-prohibitive. Hydrothermal and sol–gel synthesis methods, while effective, pose scalability challenges [53].

4.2. Reactor Design and Optimization

Three critical factors influence the reactor design: (1) light distribution, (2) mass transfer, and (3) hybrid system integration.

Light distribution. To maximize photon consumption and guarantee steady photocatalytic activity, photocatalytic reactors must have a homogeneous light distribution. Optimal light penetration is particularly difficult in turbid wastewater, where reactor geometries, such as flat-plate designs, often lead to uneven illumination [54].

In order to improve irradiation uniformity, recent developments have concentrated on improving light source designs. For example, it has been shown that better light distribution within reactors can be achieved by strategically placing LED arrays and increasing their number, which will increase photocatalytic efficiency [55]. Furthermore, software simulation is a useful tool to optimize factors that are difficult to measure, such as reactor structure and irradiation uniformity.

Mass transfer. For pollutants and photocatalysts to interact effectively, mass transfer must be performed efficiently. For flow reactors to operate at their best, flow velocity and interelectrode gaps must be precisely controlled. On the other hand, insufficient mixing in batch reactors frequently results in subpar interactions between the catalyst and the pollutant [54].

To improve mass transfer properties, novel reactor designs have been created, such as fluidized bed reactors, static mixers, and spinning disc reactors. By increasing turbulence and decreasing the thickness of the boundary layer, these designs enable better contact between contaminants and photocatalysts [56,57].

Furthermore, mass transfer inside reactors has been examined and optimized using CFD models. For instance, research has looked into how baffle layouts, flow dynamics, and reactor shape affect mixing effectiveness and pollutant degradation rates [58,59].

Hybrid systems. The overall effectiveness of micropollutant removal can be increased by combining photocatalysis with other treatment techniques like membrane filtration or adsorption. The benefits of membrane separation and photocatalysis are combined in photocatalytic membrane reactors (PMRs), which provide enhanced pollutant removal and continuous operation. However, the interaction of several processes leads to problems including intricate system tuning and membrane fouling. Recent studies have concentrated on creating innovative PMR designs and materials in order to address these issues. For example, it has been demonstrated that adding photothermal effects to PMRs increases their longevity and efficiency. In order to speed up the treatment of water and wastewater, hybrid systems that combine membrane filtration and photo(electro)catalytic have also been investigated. In the design of compact reactors and wastewater remediation, where sieving and reactivity are crucial, the close and cooperative interactions among the stacked metal oxide nano-sheets allow for high catalytic stability and efficiency, creating new opportunities for the upscaling and application of stimulus-responsive membranes [60,61]. The integration of CFD modelling in hybrid system design has further improved performance prediction and process control, enabling the optimization of complex reactor configurations [59].

4.3. Actual Wastewater Complexity

Actual wastewater matrices contain diverse pollutants, including pharmaceuticals (e.g., antibiotics and analgesics), pesticides (e.g., glyphosate), microplastics, and endocrine-

disrupting compounds (EDCs). These contaminants coexist with competing ions (e.g., Cl^- , SO_4^{2-} , and NO_3^-) and dissolved organic matter. This complexity severely impedes the efficiency of photocatalytic and photo(electro)catalytic systems, which rely on reactive oxygen species (ROS) such as hydroxyl radicals ($\bullet\text{OH}$) and superoxide ($\text{O}_2\bullet^-$) to degrade pollutants [49,53,62]. For example, the inhibitory effect of dissolved organic matter on anilines and sulfonamide antibiotics in $\text{SO}_4\bullet^-$ -based advanced oxidation processes has been reported, which is thought to be mainly caused by the reduction of intermediate radicals by phenolic groups in the dissolved organic matter [63]. $\text{OH}\bullet$ can be scavenged by nitrobenzene (NB) [64], while $\text{OH}\bullet$, $\text{Cl}\bullet$, and $\text{ClO}\bullet$ can be scavenged by tertiary butyl alcohol (TBA) [65].

Recent advances in catalyst engineering aim to mitigate these challenges. Heteroatom doping (e.g., N, S) into TiO_2 or ZnO lattices enhances visible-light absorption and charge carrier mobility. With the aim of improving TiO_2 photocatalyst material activity performance, nitrogen (N) is one of the most studied non-metal atoms for titania. For example, research has realized the fabrication of nitrogen-doped microspheres with enhanced performance under visible light for the photoreduction of CO_2 [66]. Another study deployed N-doped TiO_2 immobilized on glass spheres for the degradation of the antibiotic ciprofloxacin; a 20 mg/L initial concentration of ciprofloxacin was removed at a rate of 90% by 3 g/L of the catalyst after 90 min of visible irradiation [67]. Sulfur (S) is another type of non-metal element that researchers have used to create N and S co-doped TiO_2 as an active mesoporous material, reporting its deployment for the photocatalytic degradation of reactive oxygen [68]. Co-catalysts such as magnetic Fe_3O_4 or Ag can also be added to improve stability and selectivity. For example, Fe_3O_4 -based photocatalysts are found to be effective as simple recyclable photocatalytic materials, not only preventing the excessive use of the catalyst but also allowing easy recovery of the deactivated photocatalyst, making the process fruitful [69].

Furthermore, in situ monitoring methods like electrochemical sensors and real-time spectroscopy might enhance comprehension of reaction kinetics under actual wastewater circumstances [70]. For example, operando spectroscopy (such as operando FTIR and operando XAS) allows for the simultaneous monitoring of the catalyst's surface and the gas-liquid phase on the same sample under real reaction conditions [71]. The electrochemically boosted trace co-PMS catalytic system was proposed using mass spectrometry and DFT calculation [72].

Despite progress, future research must prioritize long-term durability testing under variable wastewater compositions and standardized protocols for comparing matrix effects.

5. Adsorption

5.1. Material Limitations

High-performance adsorbents, such as graphene-oxide-based magnetic materials and nitrogen-doped core-shell mesoporous carbonaceous nanospheres, necessitate multi-step production procedures (such as sol-gel techniques and co-precipitation) that are difficult to scale industrially [73]. Advanced nanomaterials (including TiO_2 , graphene oxide, and carbon nanotubes) pose possible toxicity hazards via leaching in actual wastewater circumstances [74]. Additionally, because of competition for active sites from ions and organic matter, traditional adsorbents show decreased efficiency in complex wastewater matrices [75].

Researchers are investigating innovative adsorbents including covalent organic frameworks (COFs) and composites based on biochar to overcome these issues. COFs are very successful at removing micropollutants because of their large surface area and adjustable porosity, whereas materials based on biochar offer more affordable options with better adsorp-

tion capabilities [76,77]. Pollutant selectivity and adsorption efficiency are further improved by surface functionalization processes, such as amine or carboxyl group modifications.

5.2. Technological Challenges

Current adsorption system optimizations rely heavily on trial-and-error methods, which are time-consuming and inefficient. Water treatment has made extensive use of artificial intelligence (AI) techniques such as decision trees (DTs), random forests (RFs), artificial neural networks (ANNs), and support vector machines (SVMs). These techniques greatly improve the efficacy of adsorbents for pollutant removal by excelling in tasks such as regression, classification, and pattern recognition. Artificial neural networks (ANNs), for instance, are adept at modelling complex systems with nonlinear relationships, while support vector machines (SVMs) are effective in handling high-dimensional datasets. Despite the potential of machine learning (ML) for predictive modelling, its use in adsorption systems is still in its infancy [78,79]. The adsorption efficiency is determined by the adsorbent, contaminant, and wastewater matrix parameters. Optimizing these parameters is crucial for effective treatment. Adsorption processes may be optimized, pollutant removal efficiency could be predicted, and intelligent adsorption systems that adjust to shifting water conditions and pollutant compositions could be developed through the use of data analytics and machine learning algorithms [78]. Proactive maintenance and performance optimization are made possible by machine learning techniques, which may evaluate complex datasets to find trends in adsorption efficiency. However, issues including computing requirements, data gathering, and model interpretability must be addressed. The most popular model for forecasting the assessment of these contaminants' adsorption performance is the artificial neural network (ANN). The findings of a comparison between modelling and experimental data indicate that the AI models could safely forecast the adsorbents' removal or adsorption capacities of organic chemicals, nutrients, pharmaceuticals, medications, pesticides, and PCPs that are frequently investigated include cephalexin, triamterene, paracetamol, phenol, phosphate, heptachlor, chlorophenol (CP), and the insecticide chlorothalonil [80].

Furthermore, issues such as variable wastewater flow rates and different pollutant concentrations make application difficult. The majority of adsorption research focuses on artificial wastewater, failing to adequately represent the intricacy of actual wastewater systems [73]. Researchers have used historical data to train an artificial neural network (ANN) model to forecast the water quality and quantity of inflows and outflows, achieving relatively good prediction accuracy. The model was trained using the inflow and outflow flow rates and waste concentration of WWTPs as an example [81]. To lessen these operational difficulties, researchers are looking into sophisticated monitoring technologies such as online sensors and real-time process control algorithms. Real-time chemical sensing in wastewater has been shown to be effective for process monitoring and control. Based on this technology, there was a 10% improvement in terms of nutrient removal rates and energy-saving benefits [82].

5.3. Actual Wastewater's Complexity

Adsorption can necessitate secondary treatment processes due to the formation of hazardous intermediates, such as chlorinated organics (e.g., trichloroethylene) from solvent-laden industrial effluents [78]. Due to the possibility of secondary pollution, the proper disposal of used adsorbents, particularly those saturated with dangerous pollutants, is an important issue that needs to be taken into consideration in practical applications. Hybrid adsorption–oxidation technologies are emerging as a solution, with integrated adsorption/advanced oxidation process (AOP) systems effectively removing pollutants from wastewater by combining adsorption and AOP, benefiting from simple design, mild

conditions, and low costs. The process involves two stages: first, a solid adsorbent captures pollutants, reducing their concentration. Second, AOP treatment generates reactive oxidizing species that break down remaining pollutants into less harmful byproducts such as carbon dioxide and water [83,84]. The effectiveness of adsorption/AOP systems depends on factors such as the type of adsorbent, the characteristics of the contaminants, and the AOP technique used. In the upcoming section, we will introduce a range of water treatment adsorbents, including carbon nanotubes and activated carbon, as well as a class of sustainable adsorbents derived from industrial and agricultural waste. Chitosan and chitin are the sources of this class of adsorbents. There are also new materials such as metal–organic framework (MOF) materials. Oxidative adsorption was introduced as a new technique for removing phenolic contaminants from water and wastewater, and MnO₂ nanodiscs were synthesized on sulphur-enriched biochar as a low-cost environmentally friendly adsorbent. This research shows that a maximum sorption capacity of 91% at the equilibrium time of 18 h with an adsorbent dosage of 1.0 mg/mL and pH = 4 was obtained [85]. However, for the procedure to be economically feasible, the spent adsorbent must be regenerated and reused. The researchers wanted to find out if clay-type materials may be regenerated using electrochemistry. Clay stability during regeneration was investigated via four successive cycles in different aqueous matrices (ultrapure water, synthetic urine, and river water). The findings showed that during the photo-assisted electrochemical regeneration process, the CVL clay was comparatively stable. Additionally, even when natural interfering agents were present, CVL clay was still able to eliminate antibiotics [86].

The benefits of composite materials have drawn a lot of attention. For example, Fe-Mn oxide-based composites have been widely used in the removal of organic pollutants, which can not only show excellent adsorption/oxidation performance but also show catalytic activity for common oxidants. The physicochemical properties that determine how effective Fe-Mn oxide materials are at removing contaminants may differ as they perform different functions. In short, the removal rate of pollutants is often affected by basic physicochemical properties such as SSA and pore volume, while the material composition, surface functional groups, and crystal structure usually determine the removal mechanism of pollutants [87]. This information is conducive to the further improvement and development of the material for the engineering application.

Periodic regeneration employing low-energy desorption techniques is a new way to address these problems [88,89]. The economic feasibility of multi-stage treatment systems is one of the issues that still arise when scaling hybrid systems, in spite of these advancements. Life cycle assessments must be given top priority in future studies in order to analyze the environmental trade-offs of hybrid technologies.

5.4. Economic and Regulatory Obstacles

While preliminary studies suggest that nanomaterial-enhanced adsorption systems (NM-ESs) could reduce energy consumption by up to 50%, their integration into existing wastewater treatment infrastructure remains expensive [74]. Advanced adsorption materials such as MOFs and graphene oxide are expensive to produce on a large scale, and costly precursors are needed to synthesize magnetic graphene oxide [73,78].

Furthermore, the disposal of wasted adsorbents that are loaded with pollutants poses serious environmental problems. Secondary pollution from improper disposal could raise operating expenses even more [78]. In order to ascertain the long-term economic and environmental viability of developing adsorption technologies, life cycle evaluations, or LCAs, are essential. Additionally, incentive schemes and regulatory laws may be crucial in encouraging the use of sustainable adsorption technology.

6. Bibliometric Analysis of Nanomaterials

6.1. Methodology

Bibliometrics is a valuable method for analyzing and predicting research trends. A study's main concepts are summarized in the keywords. As a result, we can use the co-word (co-occurrence) analysis method to identify research trends and hotspots [8] (Zhao et al., 2018). Bibliometric reviews, in contrast to systematic literature reviews, give information on any study topic using a variety of bibliometric and bibliographic data [90].

A bibliometric analysis was conducted using Scopus to map research trends in nanomaterials applied to environmental remediation, with a focus on nano-adsorbents and photo(electro)catalysts. Our Scopus-based bibliometric analysis (2011–2024) identified 930 relevant articles using keyword such as “nano-adsorbent” and “photo(electro)catalysis”. Data were exported as .csv files and analyzed using VOSviewer (version 1.6.19) to construct co-occurrence overlay visualizations.

6.2. Trends in Nano-Adsorbents

The terms we gathered from 297 article titles and keywords related to nano-adsorbents are shown in Table 3.

Table 3. The evolution and development of nano-adsorbents over time (2011–2024).

Year	Nanoadsorbents	Reference
2011–2014	Activated carbon; Titania Nanoparticles	[91] [92]
2015–2018	Heterogeneous Core-shell nanotubes Co-doped Fe ³⁺ -TiO ₂ -xNx catalyst ZnO/MMT nanocomposite Tetra-amido macrocyclic ligand (TAML)/hydrogen peroxide (H ₂ O ₂) Zero-valent iron nanofibers/reduced ultra-large graphene oxide (ZVINFs/rULGO)	[93] [94] [95] [96] [97] [98]
2019	Anion exchanger; Hybrid ion exchanger Nano zirconium carbide reduced graphene oxide (rGO)—titanium dioxide (TiO ₂) nanocomposite	[99] [100] [101]
2020	Ternary heterojunction MWCNT/N-TiO ₂ /UiO-66-NH ₂ Cobalt-doped graphene powdered sand composite (Co@graphene) Vanadium–titanium magnetite Yolk/shell Fe ₃ O ₄ @MgSiO ₃ nanoreactor Graphene oxide and MOFs co-modified composites Spherical cuprous oxide nanoparticle-hybrid anion exchanger EuxTi1-xO ₂ -yNy/CoFe ₂ O ₄ (Eu/N-doped titania coupled with cobalt ferrite) Iron-porphyrin-loaded biochar (Fe (TPFP)/BC)	[102] [103] [104] [105] [106] [107] [108] [109]
2021	Graphene aerogel composite UiO-66(Zr) Perylene diimide functionalized g-C ₃ N ₄ @UiO-66 supramolecular photocatalyst Porous g-C ₃ N ₄ /calcined-LDH Graphene/β-cyclodextrin membrane Carbon fibre cloth-UiO-66-NH ₂ /AgI Z-scheme Bi ₂ O ₃ /TiO ₂ @reduced graphene oxide ZnO nanorods/Fe ₃ O ₄ -graphene oxide/metal-organic framework nanocomposite g-C ₃ N ₄ /MOFs composite Hydrophilic 3D N-doped carbon/CuO composites AQ2S@rGO nanocomposite Nickel ferrite nanoenabled graphene oxide (NiFe ₂ O ₄ @GO) Cyclodextrin-modified polyacrylonitrile nanofiber membranes Ag-P25 photocatalysts CuS@carbon nanocomposites Pd,ÄiZnO/N-doped carbon nanofibers electrode	[110] [111] [112] [113] [114] [115] [116] [117] [118] [119] [120] [121] [122] [123] [124] [125]

Table 3. Cont.

Year	Nanoadsorbents	Reference
2022	CuAl-layered double hydroxide/MgO ₂ nanocomposit	[126]
	Ruthenium-modified composite electrode	[127]
	CuFe ₂ O ₄ -Ti and CuFe ₂ O ₄ -Ti-GO nanocomposite	[128]
	Chitin biochar	[129]
	An electronegative silanized β -cyclodextrin adsorbent	[130]
	BiOI/TiO ₂ flexible and hierarchical S-scheme heterojunction nanofibers membranes	[131]
	Modified cellulose/poly(3,4-ethylenedioxythiophene) composite	[132]
	TiO ₂ /carbon heterostructure fibres	[133]
	g-C ₃ N ₄ -x/Bi/Bi ₂ O ₂ (CO ₃) ₁ -x(Br, I)x heterojunction	[134]
	TiO ₂ -La 0.05%-carboxymethyl- β -cyclodextrin (CMCD)	[135]
	Collagen fibrous aerogel cross-linked by Fe (III)/silver nanoparticle complexes	[136]
	Porous polymer layer on TiO ₂ -graphene	[137]
	Composite sponge	[138]
	Bi ₂ WO ₆ and Bi ₂ O ₃ -ZnO heterostructure	[139]
	n-n heterojunction; TiO ₂ /AgBiS ₂	[140]
2023	MgO/Al ₂ O ₃ - modified rice husk biochar	[141]
	Heterostructured zeolitic imidazolate framework (ZIF)-based materials	[142]
	Iron oxide nanoparticles (IONPs)	[143]
	Biochar-based geopolymer nanocomposite	[144]
2024	Magnetic porous Fe ₃ O ₄ /HCP hybrid microparticles (HSF and HSFV)	[145]
	TiO ₂ /biochar; ZnO/biochar	[146]
	ZnS:Ni loaded on sponge-activated carbon	[147]
	Granular graphene oxide-activated carbon (GGA)	[148]
	Oil palm waste-derived adsorbents	[149]
	Na-jarosite	[150]
	Zero-valent iron nanoparticles (nZVI)	[151]
	CoFe ₂ O ₄ @Ti ₃ C ₂ @MIL101(Cr)	[152]

Early research (2011–2018) prioritized activated carbon and carbon nanotubes, while recent studies (2018–2022) focused on nanocomposites (reduced graphene oxide (rGO)–titanium dioxide (TiO₂) nanocomposites, graphene oxide and MOFs, 3D aerogels, and biochar (Table 2)). MOFs, biochar, and heterojunctions are emerging hotspots, offering tunable porosity and enhanced adsorption kinetics.

- MOFs

Because of their rich surface chemistry, customizable pore shape, and flexible synthesis methods, MOFs are more appealing in the adsorption field than other porous materials such as silica gel, zeolite, and active carbon. These materials cannot, however, be used exclusively as photocatalysts due to their weak photocatalytic activity. Researchers have developed composite photocatalysts by co-modifying TiO₂ with reduced graphene oxide (RGO) and MOFs (ZIF-8, UiO-66, and Cu-BTC) to enhance pollutant removal via adsorption and photocatalysis under UV and visible light [106]. The composites exhibit superior performance due to synergistic effects. Optimized composites achieve rhodamine-B degradation rate constants of $1.65 \times 10^{-1} \text{ min}^{-1}$ (UV) and $1.12 \times 10^{-1} \text{ min}^{-1}$ (visible light), demonstrating dual-light efficiency. This work highlights the role of MOF pore size/BET area and TiO₂–RGO interfaces in designing high-performance environmental remediation materials [106]. Another study synthesized a binary composite of g-C₃N₄, MIL-101(Fe), and g-C₃N₄ nanosheets for water remediation. The composite achieves a 99.3% degradation efficiency, outperforming pristine g-C₃N₄ by 40%. The enhanced performance is attributed to improved optical properties and quenched recombination rate. The g-C₃N₄ nanosheets significantly affect the kinetics of RhB degradation under visible light, with the Z-scheme mechanism being the primary pathway [118].

- Biochar

Biochar has shown excellent micropollutant removal properties. Recently, biochar has been used to remove contaminants such as aqueous pharmaceuticals [141,146] and phenols [144]. Surface modifications improve biochar selectivity and adsorption ability. A hybrid sorbent (MgO/Al₂O₃-modified rice husk biochar) demonstrated higher pharmaceutical removal capacities and a higher surface area than pristine rice husk biochar prepared at the same pyrolysis temperature. On the other hand, biochar-based geopolymers (bio-sorbents) represent a sustainable solution and are often noted to have unique size-dependent characteristics, such as a large specific area, facile synthesis, a beneficial surface chemistry, and high scalability.

- Bibliometric analysis

Co-occurrence overlay visualization (Figure 2) maps the frequency and temporal evolution of author keyword associations. This allows for the analysis of a large volume of collected literature and provides insights into research hotspots and technology trends over the course of a decade. The key methodological steps included the following: 1. Term Extraction: Keywords with five or more occurrences were retained. 2. Normalization: A relevance score was applied to exclude generic terms (e.g., “water”, “analysis”). 3. Clustering: The Linlog/modularity algorithm grouped terms into thematic clusters. 4. Overlay: Nodes were colour-coded by average publication year to highlight temporal trends.

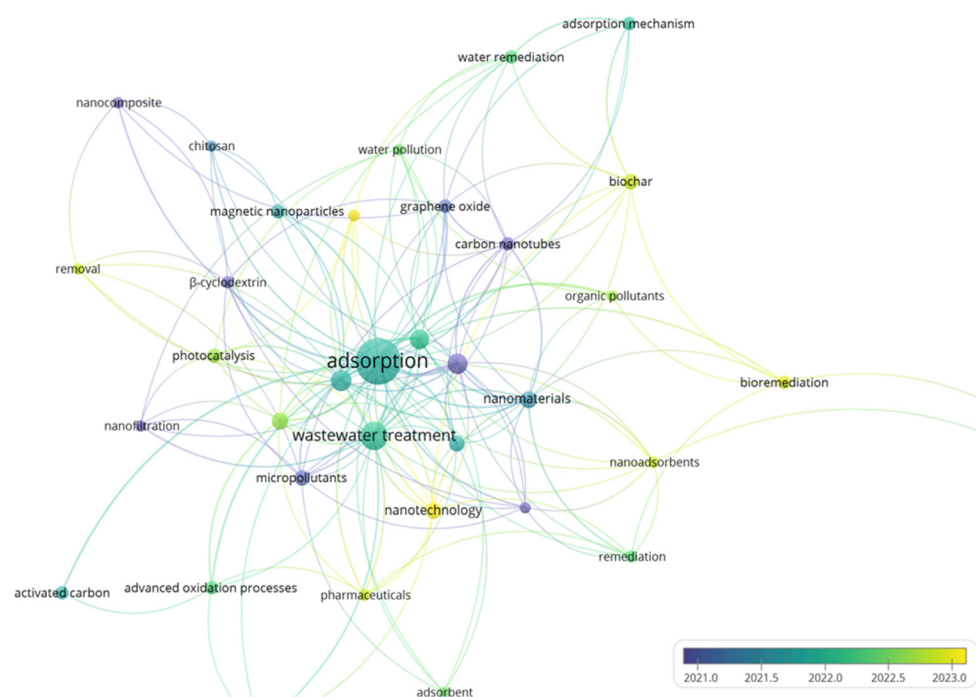


Figure 2. Co-occurrence overlay visualization was created using bibliographic data obtained from the combination full-text search of micropollutants and nano-adsorbents.

Overall, 35 items out of 912 keywords met this threshold. These 35 items were chosen, arranged in 24 clusters with 161 links, and given a total link strength of 245. The Supplementary File contains the links and statistics on the total link strength for 35 items (Table S1).

Blue-coloured items are research hotspots in the past decade; these terms are related to “nano-adsorbents”, and their items labels and total link strength are as follows: adsorption (81), carbon nanotubes (16), graphene oxide (15), and β -cyclodextrin (10).

Yellow-coloured items are those whose average publication year is the most recent. The items with a yellow colour related to “nano-adsorbents” include nanotechnology and

biochar. These items are recent new research directions for the design and synthesis of nano-adsorbents based on photo(electro)catalysis.

6.3. Trends in Photo(electro)catalysts

Co-occurrence overlay visualization (Figure 3) maps the frequency and temporal evolution of keyword associations. This allows for the analysis of a large volume of collected studies and provides insights into research hotspots and technology trends over just a decade.

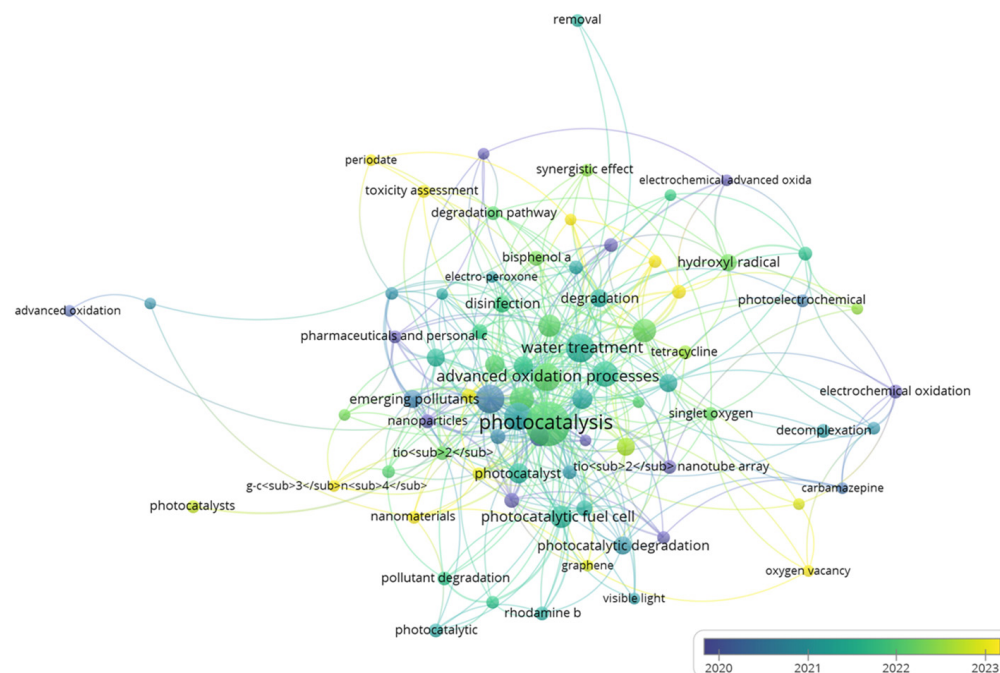


Figure 3. Co-occurrence overlay visualization was created using bibliographic data obtained from the combination full-text search of micropollutants and photo(electro)catalytic.

The key methodological steps included the following: 1. Term Extraction: Keywords with five or more occurrences were retained. 2. Normalization: A relevance score was applied to exclude generic terms (e.g., “water”, “analysis”). 3. Clustering: The Lin-log/modularity algorithm grouped terms into thematic clusters. 4. Overlay: Nodes were colour-coded by average publication year to highlight temporal trends.

Overall, 78 items were selected from 1883 keywords, with 412 links and a total link strength of 543. Keywords coloured yellow had the most recent average publication year. The links and total link strength information for the 78 items are provided in the Supplementary File (Table S2).

Colours indicate the average publication year of keywords. Blue-coloured items are research from earlier publications, while items coloured yellow have the most recent publication year. TiO_2 dominated early research. Recent advancements emphasize graphitic carbon nitride ($\text{g-C}_3\text{N}_4$) and heterojunctions. Scalable reactor design and defect engineering are critical for industrial adoption.

7. Technological Advancements

7.1. Critical Analysis of Implementation Challenges

7.1.1. Adsorption

Adsorption on highly porous materials has been proven to be an effective method for removing micropollutants. The adsorption process has been found to be superior to other

technologies in terms of its simplicity of design, initial costs, operation, and insensitivity to toxic substrates. **Activated carbons (ACs)** are the most widely used adsorbents, and they are highly effective due to their high adsorption capacity [153], demonstrated for a large number of pollutants of various nature and properties [153,154]. ACs exhibit declining efficiency in real wastewater due to matter (e.g., natural organic matter (NOM)) and require energy-intensive thermal regeneration, which increases operational costs and environmental concerns [43].

The adsorption process is suitable for removing contaminants from wastewater due to the number of aromatic rings and the chemical structure of these emerging pharmaceutical contaminants. In this process, **graphene nanomaterials** are dominated by π - π interactions between aromatic rings, hydrophobic interactions, and H bonding [155–158]. **Graphene** and its nanocomposites are considered as good candidates as water treatment materials due to their large surface-area-to-volume ratio and other beneficial physiochemical properties, such as electrostatic interaction with contaminants. Researchers aimed to create and **characterize graphene oxide (GO)** and evaluate the adsorption of butylparaben (BP) on GO as an adsorbent for wastewater treatment. GO was characterized using various methods, and the removal efficiency was found to be 84.3% at a BP concentration of $600 \mu\text{g L}^{-1}$, an adsorbent content of 5 g L^{-1} , and a pH of 7 in the solution. The adsorption rate followed the PFO model, with a spontaneous process with a negative Gibbs energy value. The ionic effect showed that increasing salt concentration increased repulsive forces but did not decrease adsorption capacity. The regeneration cycle showed an efficiency of 85% up to the second cycle [159]. Due to its numerous potential uses, graphene oxide (GO) has recently attracted a lot of attention from both academia and industry. GO may be incorporated into subsequent products or reduced to chemically transformed graphene. However, there are scientific, engineering, and financial obstacles to the industrial-scale production of GO. These include the need to choose and acquire the right graphite and reaction media, oxidize graphite in a safe and economical manner, purify and isolate products, remove and treat wastewater, handle and store products, and assess the quality of the final product [160].

7.1.2. Photo(electro)catalysis

Graphitic carbon nitride (g-C₃N₄) photocatalysts. Among the different photocatalysts, graphitic carbon nitride (g-C₃N₄) stands out due to its low cost, excellent chemical and thermal stability, adequate bandgap, and optimal band locations. However, g-C₃N₄'s poor light responsiveness and high rate of photoinduced charge recombination have an impact on its photocatalytic performance [161].

Lead-free halide perovskites. Due to their exceptional photophysical characteristics, including their long carrier diffusion lengths, adjustable band structures, and high optical absorption coefficients, metal halide perovskites have garnered a lot of interest in the field of photocatalysis. Despite the exciting progress made in this research field, there are still critical issues and challenges to overcome. Their stability in aqueous environments and polar solvents is limited [162], and their photocatalytic performance remains unsatisfactory due to inadequate light utilization and undesired electron-hole recombination [162]. Further understanding of the photocatalytic mechanisms at the molecular or atomic levels is needed to improve efficiency. Further theoretical calculations and exploration of this promising field will lead to further improvements in efficiency and stability.

Our energy future relies on sustainable energy conversion technologies, and **single-atom catalysts (SACs)** have emerged as a promising solution. These atomic-level electrocatalysts enable high metal utilization and low-cost catalyst engineering coupled with high catalytic activity. However, there are challenges in their large-scale production due to their low production efficiency. Most SAC-based electrocatalysts are non-noble metals that need

anchoring using heteroatoms via coordination or covalent bonding [163]. SAC research should focus on balancing loading, migration, and agglomeration.

7.1.3. Systemic Limitations

Photocatalysis is a promising advanced oxidation process with advantages such as scalability, continuous reaction monitoring, and efficiency. Despite these advantages, continuous photoreactors are not widely used in industry due to challenges in reactor design and scale-up issues related to photo-degradation efficiency, reductions in active sites, economic feasibility, and catalyst recovery [164]. Optimizing system parameters such as catalyst loading, pH, reactor volume, light intensity, and dye concentration can lead to scalable designs for industrial-scale implementation.

7.2. Research Gaps and Future Directions

7.2.1. Innovative Materials

Researchers have developed an innovative RGO/AC composite using steam activation of GO/BC cross-linked with waste cassava peels. This method eliminates the need for secondary AC coating processes and enhances activation efficiency, pore development, and hydrophobicity. The composite outperforms conventional AC with regard to adsorption capacity and affinity for PFAS compounds. The composite's unique adsorption mechanism combines hydrophobic interactions, electrostatic forces, and hydrogen bonding. This eco-friendly approach offers a promising solution for in situ water treatment [165]. g-C₃N₄ is a suitable candidate for dual-purpose photocatalysts engineered to enhance charge separation and visible-light utilization. Researchers aimed to improve the photocatalytic activity of g-C₃N₄ in various reactions, including photocatalytic H₂ generation, mineralization, removal of organic pollutants and heavy metals, reduction of H⁺ and CO₂, oxidation of organic substrates/biomass, and simultaneous H₂ and H₂O₂ production. Combined approaches could provide efficient and sustainable solutions for energy and environmental issues. The key to successful advancement is that the photocatalyst's valence band energy level should be more positive than the pollutant's oxidation potential [166]. Wide-bandgap semiconductors are suitable for dual-functional photocatalytic systems due to their negative CB and positive VB energy levels.

7.2.2. Process Optimization

Hybrid adsorption–oxidation technologies are emerging as a solution, and integrated adsorption/advanced oxidation process (AOP) systems effectively remove pollutants from wastewater by combining adsorption and AOP, benefiting from simple design, mild conditions, and low costs [83,84].

The chemical composition and properties of environmental media determine nanomaterial (NM) transport, fate, biouptake, and organism response. The nanomaterial environmental, health and safety (NanoEHS) field has started to undergo harmonization to allow data comparison and re-use, but there is limited guidance for standardizing test media. A workshop at Duke University identified five categories of test media: aquatic testing media, soil and sediment testing media, biological testing media, engineered systems testing media, and product matrix testing media [167]. For each category, a minimum set of medium characteristics to report in all NM tests is recommended. The media surrounding NMs are key determinants of the transformations they undergo and the ultimate characteristics of the resulting materials that move through environments and are taken up into the biota.

7.3. Concrete Proposals for Overcoming Limitations

7.3.1. AI-Driven Advancements

Artificial intelligence (AI) models such as random forest (RF) and artificial neural networks (ANNs) are used to forecast the adsorption efficiency of contaminants on materials. AI-aided adsorption processes have the potential to enhance wastewater treatment by providing accurate predictions of adsorption performance, reducing costs and time, and optimizing resource utilization [79]. For example, the rapid prediction of adsorption capacities at random sites on materials such as g-C₃N₄ has been made possible by the combination of transfer learning and deep neural networks (DNNs). The proposed AI method has the same prediction accuracy as the ab initio DFT calculation, but is millions of times faster than DFT in predicting adsorption abilities at arbitrary sites and only requires one-tenth of the dataset compared to training from scratch [168].

Both photocatalytic and electrocatalytic processes can be optimized using AI models. In order to map dynamic catalytic pathways, such as dipole coupling in CO₂ reduction, AI analyzes time-resolved spectroscopic data, offering insights into intermediate states and efficiency drivers [169]. For CO₂ reduction, ML optimizes reaction parameters and nanoscale catalyst geometries. This improves catalytic activity/selectivity and lowers experimental expenses [170].

Regarding direct synthesis, machine learning methods examine the connections between photoactivity and structural characteristics [171]. Moreover, photocatalytic activity (such as CO₂ reduction) is predicted by neural networks using genetic, whale, or particle swarm methods that have been tuned. In order to construct heterojunction catalysts and optimize absorbance curves, these models can be combined with density functional theory (DFT) [171].

7.3.2. Environmental and Economical Benefit Analysis

The application of nanometal catalysts in Fenton-like processes has made significant progress, facilitating the practical application of advanced oxidation processes (AOPs) for these highly active catalysts. However, challenges such as managing production costs, upscaling production, developing cost-effective catalyst modules, the overuse of oxidants, treating residual sulphates and excessive oxidants in wastewaters, the disposal of spent catalysts and by-products, and the complexity of wastewater substrates still exist.

Combining life cycle assessment (LCA) and techno-economic analysis (TEA) can provide useful support for engineering-scale Fenton-like applications. Current research focuses on improving the removal performances of pollutants and their operational stabilities but rarely considers the costs of catalysts/catalytic modules. Cost analysis is crucial for pilot-scale and large-scale Fenton-like systems, and pursuing the balance between costs and performances of catalysts/catalytic devices is a key issue to realize the application of Fenton-like system engineering [172]. The integration of LCA and TEA as guidance tools for selecting the best Fenton-like technologies for the treatment of targeted wastewaters can better realize the evaluation and optimization of process/technology, cost assessment, and the analysis of the environmental effects of engineering applications from multiple perspectives, such as technology, economics, and the environment.

7.3.3. Policy and Industry Collaboration

Through qualitative research, literature reviews, and interviews with adsorbent material producers, consultants, and end users, researchers have diligently laboured to create a production platform. Clarifying the product and creating a business plan that facilitates commercialization are the goals of this platform. This highlights how crucial a results-driven strategy and a product-focused approach are to the commercialization of innovative

adsorbent materials [173]. Building manufacturing platforms facilitates the development of direct relationships with industry partners.

8. Conclusions and Future Scope

Although micropollutants are a serious threat to human health and ecosystems, conventional WWTPs still encounter difficulties in eliminating them. In order to address these persistent pollutants, this review emphasizes the promise of cutting-edge technologies such as photo(electro)catalysis and adsorption, especially when combined with nanomaterials. While adsorption provides targeted removal using high-surface-area materials such as graphene oxide and MOFs, photo(electro)catalysis combines light-driven and electrochemical processes to provide greater efficiency in degrading non-biodegradable micropollutants. Material instability, reactor design limitations, and interference from intricate real wastewater matrices are some of the challenges that practical implementation encounters. Emerging developments that are changing research objectives, such as heterojunctions, single-atom catalysts (SACs), and AI-driven optimization, have been highlighted by the bibliometrics analysis. There is potential for improving sustainability using hybrid systems that combine adsorption with catalytic reactions and in situ regeneration techniques. However, cost and regulatory issues continue to be major roadblocks to industrial use.

Future scope includes testing actual conditions, developing stable photocatalysts and adsorbents, engineering reactors for actual wastewater treatment system, exploring synergies between adsorption and catalytic processes, expanding machine learning models for predictive optimization, and conducting lifecycle analyses and cost–benefit studies to evaluate nanomaterial-based systems' feasibility.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/pr13061759/s1>, Table S1: Links and total links strength information for 135 items from nanosorbent topics, obtained using VOSviewer application (version 1.6.19) note: we set minimum number of keyword occurrence of 5; Table S2: Links and total links strength information for 359 items from photo(electro)catalyst topics, obtained using VOSviewer application (version 1.6.19) note: we set minimum number of keyword occurrence of 5.

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