



An Overview of Nanofiltration and Nanoadsorption Technologies to Emerging Pollutants Treatment

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Abstract: The presence of emerging contaminants in water resources has been a global concern due to their environmental impact and effects on human and animal health. These compounds include pharmaceuticals and personal care products, pesticides, fire retardants, hormones, and plasticizers, among others. These contaminants are not completely removed during water and wastewater conventional treatment. Thus, alternative processes are necessary to face this issue. In this context, nanotechnology represents a promising strategy for the remediation of emerging contaminants. Nanostructured materials have highlighted properties, such as adsorption capacity and porosity, chemical stability, reactivity, mobility, and antimicrobial activity. The diversity of nanomaterials and the possibility of combining them contribute to their application in contaminant mitigation. Furthermore, they can be used in different removal techniques such as adsorption, and membrane filtration. Thus, this review article provides an overview and advances in the production and application of nanofiltration membranes and adsorbent nanomaterials for the treatment of effluents containing emerging pollutants.

Keywords: carbon-based nanomaterials; metal-based nanoadsorbents; nanofiltration membranes; surface area; surface modification

1. Introduction

The occurrence of emerging pollutants in water resources has generated discussions about the environmental implications and risks to human health. Several activities, agricultural and urban, contribute significantly to stream pollution from the release of contaminants in low concentrations (ng L⁻¹ to mg L⁻¹). As a result, there is an imbalance in the sustainability of the ecosystem, resulting in the scarcity of drinking water [1–3]. Physical factors, nutrient concentration, heavy metals, microbial pollutants, and toxic pollutants determine water quality. Advances in analytical techniques have resulted in the efficient monitoring of water quality, allowing detections at low concentrations of contaminants [4].

Emerging contaminants are natural and/or synthetic substances, and are generally unregulated. These contaminants include various chemical compounds from the pharmaceutical and personal care industries, pesticides, fire retardants, hormones, dioxins, polycyclic aromatic hydrocarbons, plasticizers, sweeteners, and surfactants [4–6]. Most of these contaminants are released into water resources from point sources from industrial, hospital, and domestic effluents [2]. Other occasional activities such as aquaculture are also cited as sources of pharmaceutical products and personal care [7]. In groundwater, these pollutants can be introduced from infiltration processes, leakage from septic tanks, leaching from landfills, problems with drainage, or failures with sewage systems [8]. In



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Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). addition to the discharge of effluents by industries, many compounds from fire retardants can be released into the environment by industrial emissions and undergo atmospheric deposition, reaching water resources [9].

Recent studies have assessed the contamination and ecological risks of contaminants present in water resources in different countries [7,8,10,11]. Even at low concentrations, these pollutants can resist biological and chemical degradation. This feature makes it difficult to treat water and effluents using conventional processes and natural attenuation systems [4,6].

Limitations in treatments are mainly due to the complexity of contaminants, selectivity of treatment technologies, and possible formation of unwanted by-products [2,12]. Physical, chemical, biological, and hybrid technologies can be used to treat emerging pollutants [5]. The use of activated sludge, commonly used to reduce the organic load, is not recommended for removing emerging contaminants, especially those in low concentrations. Concerning activated sludge systems, biological membrane reactors have higher efficiency, from 15 to 42%. However, hybrid systems must be used to remove these pollutants. In this sense, the main techniques used for treating these pollutants are conventional/advanced oxidation processes, adsorption, or their combination with biological processes [13]. Adsorption systems offer good recovery of drugs, pesticides, and personal care products, even when present in low concentrations. Biological treatments such as the construction of wetlands showed good efficiency in removing drugs such as ibuprofen (74–99%), ketoprofen (47–91%), naproxen (76–97%), diclofenac (24–93%), tramadol (54–85%), paracetamol (95-100%), atenolol (58-99%), metoprolol (60-93%), furosemide (80-96%), triclosan (62–91%), gabapentin (53–88%), and caffeine (93–99%) [14]. Although many of these methods present satisfactory performance in emerging contaminants treatment, problems such as high costs, equipment maintenance, treatment time, and energy demand are restrictions that must be addressed. These problems can influence the cost-effectiveness, large-scale application, and the total elimination of these substances from the environment [5,12,15].

In this context, nanotechnology can be considered a quick and efficient option to overcome the challenges of conventional and unconventional water treatment methods [16,17]. Nanomaterials have gained prominence in properties, such as adsorption, porosity, chemical stability, reactivity, mobility, catalytic potential, antimicrobial activity, and recyclability. These characteristics make them promising approaches to remove/treat several organic and inorganic contaminants, biological toxins, pathogens, and dyes [18–20]. Nanofiltration membranes and carbon and metal-based adsorbent nanomaterials have been investigated for water treatment [19]. Treatment processes can occur by adsorption, filtration, and degradation to non-toxic compounds and intermediates [21]. These techniques have been successfully applied in the remediation of a wide range of contaminants such as pesticides, pharmaceuticals, and personal care compounds [22,23].

Nanomaterials can be safely used for wastewater treatment, aiming for reuse of this resource, or for release into water bodies, as long as they comply with current legislation. Thus, the objective of this review was to address an overview and advances in the production and application of nanofiltration membranes and adsorbent nanomaterials for the treatment of effluents containing emerging pollutants.

2. Emerging Pollutants

The increase in the emerging pollutants in water resources has generated numerous concerns. The main implications are related to pollutants emerging in areas of water scarcity or poor management of effluent treatment plants [8]. These pollutants can modify the physical-chemical parameters of the water, considered essential for the maintenance of aquatic life. Problems with eutrophication in water bodies, death, and migration of species are possible interferences that these substances can cause in the ecosystem [3]. Furthermore, these pollutants are susceptible to bioaccumulation and biomagnification in aquatic organisms, contributing to toxicity at higher trophic levels. Bioaccumulation processes occur from the absorption of substances by aquatic organisms or ingestion of

contaminated food. In biomagnification, there is a progressive accumulation of these substances along the food chain [3,24]. When in contact with humans, these pollutants can be associated with cancer risks, hormonal changes, DNA changes, gastrointestinal toxicity, infertility, thyroid diseases, among other problems [25].

Pharmaceutical pollutants include drugs used to treat diseases. Pharmaceutical products such as antibiotics, contraceptives, antidepressants, analgesics, and antipyretics are examples of these substances commonly found in water resources [4]. After consumption, a part of these drugs is metabolized. The remainder is later released into the environment from human or animal excreta. These contaminants are introduced into the environment through direct release from effluent treatment plants, fields fertilized with animal waste, or landfill disposal [3,8].

UV filters, insect repellants, and shampoos are examples of personal care products that can disrupt the aquatic ecosystem. Oxybenzone and octinoxate are found in sunscreens and other commonly used products, such as shampoos, body lotions, and fragrances, soaps, among others. These two compounds are responsible for bleaching coral reefs [26]. Oxybenzone has also been evaluated for toxicological effects on eukaryotic and prokaryotic microalgae [27]. Pesticides are another group of substances commonly found in water resources and are mainly associated with agricultural activities. These contaminants can reach water resources in different ways, persisting in the environment for days or years. The main forms of exposure occur through surface runoff, caused by rain, infiltration, or volatilization, depending on the characteristics of each compound [17,28]. Plasticizers are also considered emerging pollutants [3,29]. Flame retardants are identified in a variety of environmental matrices. This group of emerging contaminants is widely used to prevent the combustion of diverse materials (construction, furniture, textiles, and electronics) [9]. According to Parida et al. [2], many emerging contaminants identified in water bodies exceed the reference values established by the European Union. In this sense, although there is a concern with these compounds in the environment, the regulations of emerging contaminants still need attention from environmental agencies. There are no laws to stipulate the limits of concentrations of pollutants in drinking water, the environment, or wastewater [30]. However, some emerging contaminants were included in lists of priority substances in several environmental bodies, such as The European Union Water Framework Directive, which listed 45 priority compounds with environmental quality standards [31]. Table 1 shows some quality reference values from different regulatory bodies for some commonly identified priority pollutants in water.

Compound	CAS Number	Emerging Pollutants	Occurrence	Maximum Concentration (ng L ⁻¹)	Reference	Recommended Limit U.S EPA	Recommended Limit Europe Union	WHO [32]
Stimulants	58-08-2	Caffeine	Drinking waters	2–5845	[33]		-	-
			Surface water	743 29.62–1104.8 1040 19–127,000	[8] [34] [35] [33]	- -		
			Groundwater	262 2–53	[8] [33]			
		Bisphenol A	Drinking waters	1–178	[33]		Substance subject to review for possible identification as priority substances or priority hazardous substance [36]	-
Plasticizer	80-05-7		Surface water	29.5 2–13,016	[35] [33]			
			Groundwater	2–643	[33]			
	1912-24-9	Atrazine	Drinking waters	1–687	[33]	- 0.003 mg L ⁻¹ [37] -	0.6 μg L ⁻¹ [31,38]	$100~\mu g~L^{-1}$
Pesticide			Surface water	42.1 1–611	[35] [33]			
			Groundwater	2–5	[33]			
Pesticide	121-75-5	Malathion	Surface water	50.4	[35]	-	0.1 μg L ⁻¹ [39]	No established reference value because occurs in drinking water at concentration below those of health concern (<2 μ g L ⁻¹)
Pesticide	330-54-1	Diuron	Surface water	11.7	[35]	Listed as priority pollutant or hazardous substance [40]	0.2 μg L ⁻¹ [31,38]	-

Table 1. Reference values of concentration of priority pollutants and admissible concentrations in water resources.

Table 1. Cont.

Compound	CAS Number	Emerging Pollutants	Occurrence	Maximum Concentration (ng L ⁻¹)	Reference	Recommended Limit U.S EPA	Recommended Limit Europe Union	WHO [32]
Pharmaceuticals	22071-15-4	Ketoprofen	Surface water	245 107	[34] [8]	_	_	_
			Groundwater	23.4 215	[8] [41]		-	-
	15307-86-5	Diclofenac	Surface water	41.4	[8]		Substances should be removed from the watch list [36]	
Pharmaceuticals			Groundwater	1.56 380	[8] [41]			-
		Carbamazepine	Surface water	16.1	[8]		-	
Pharmaceuticals	298-46-4		Groundwater	27.2 136	[8] [41]			-
Pharmaceuticals	439-14-5	Diazepam	Groundwater	35.1	[41]	-	-	-
Pharmaceuticals	15687-27-1	Ibuprofeno .	Surface water	372.1 22.85	[34] [8]	-	_	_
			Groundwater	49.4 988	49.4 [8] 988 [41]			
Pharmaceuticals	3380-34-5	Triclosan -	Surface water	139 8.6 2–289	[34] [35] [33]	Only eligible for specified use [42]	Use is not approved [43]	_
			Drinking waters	2–37	[33]			
			Groundwater	22–284	[33]			
TT.	50-28-2	17β -estradiol -	Drinking waters	18–35	[33]	Listed as priority	$0.4 \text{ ng } \text{L}^{-1}$ [36]	_
Hormones			Surface water	2–6806	[33]	substance [40]		
Hormones	57-63-6	17α- ethynylestradiol	Surface water	4–4390	[33]	Listed as priority	0.035 ng L^{-1} [36]	-
TIOTHIOHES			Drinking waters	32	[33]	substance [40]		
		7-1 Estriol	Drinking waters	0.4–125	[33]	Listed as priority		
Hormones	50-27-1		Surface water	11.9 1–1398	[35] [33]	pollutant or hazardous substance [40]	-	-

Compound	CAS Number	Emerging Pollutants	Occurrence	Maximum Concentration (ng L ⁻¹)	Reference	Recommended Limit U.S EPA	Recommended Limit Europe Union	WHO [32]
Hormones	53-16-7	Estrone	Drinking waters	0.6–20	[33]	Listed as priority	0.4 ng L^{-1} [36]	-
			Surface water	0.8–39	[33]	pollutant or hazardous substance [40]		
			Groundwater	5	[33]			
Hormones	58-22-0	Testosterone	Drinking waters	2–5	[33]		-	-
			Surface water	1–329	[33]	-		
			Groundwater	34–36	[33]	_		

Tabl	e 1.	Cont.

Thus, nanotechnological processes have been developed and are gaining attention for the treatment of effluents, mainly due to the advantages they offer in relation to conventional treatment technologies [20]. Nanotechnology can be applied to treat emerging contaminants in water resources (Figure 1). According to Anjum et al. [44], the main nanomaterials technologies are classified into three main categories (nanoadsorbents, nanocatalysts, and nanomembranes).



Figure 1. Possible sources of emerging contaminants in aquatic resources.

3. Nanotechnological Approaches to the Treatment of Emerging Pollutants

3.1. Nanofiltration Membranes

Nanofiltration is considered an economical process to remove several types of contaminants. Nanofiltration uses membranes with a pore size on the nanometer scale and operating pressures in the range of 5–40 bar. In the nanofiltration (NF) process, the solvent is forced through a selective membrane. The pressure difference generated between the feed and the permeate side makes it possible to retain the material in the nanomembrane [45]. Nanofiltration membranes have been used in several sectors, such as water treatment, biotechnology, foods, chemical field, and pharmaceuticals [46].

Nanofiltration membranes have high permeation for monovalent ions. Moreover, comparing reverse osmosis and nanofiltration membranes, nanofiltration membranes show higher flux [46]. Foureaux et al. [47] found that nanofiltration rejection efficiencies were very close to those achieved by reverse osmosis membranes for active pharmaceutical compounds. In addition, the nanofiltration membranes showed high desalination capacity and rejection of pharmaceutical compounds (>84.1%).

Fouling is one of the main disadvantages of filtration processes. The possibility of reacting with the free chlorine from the water treatment makes the natural organic material the main responsible for the encrustation of the membrane [46]. To overcome the fouling, nanotechnological approaches such as the use of metal oxide nanocomposites and hydrophilic polymer coating have been reported [48]. Chitosan also was an excellent antifouling agent. It provided nanofiltration membranes with performance >97% for diphenhydramine and mebeverine. These results were achieved due to the positive charge of the membrane surface and protonation of drug molecules [49].

Membrane separation processes can be applied to obtain high-quality reuse water, removing all or part of pharmaceutical products. However, conventional treatment processes have shown low efficiency in removing antibiotics. In this sense, Souza et al. [50] evaluated the nanofiltration membranes' performance to remove norfloxacin from a syn-

thetic effluent. In this study, membranes showed high selectivity to norfloxacin (rejection >87%). The pH affected membrane selectivity (highest norfloxacin rejection at pH 6.5). pH 6.5 also contributed to the lowest membrane permeability. The authors associated these results with electrostatic interactions between the membrane surface and the ionic species of norfloxacin.

Several technological applications related to the development of nanofiltration were patented for removing emerging pollutants in aqueous matrices. Among them, the patent US2021252458A1 [51] describes the development of a composite nanofiltration membrane for treating various impurities, including pesticides and precursors of disinfection by-products, among others. Another invention, CN102874900A [52], refers to a nanofiltration method for removing Perfluorooctane sulfonate present in drinking water, or to document CN201458860U [53] concerning a recycling device for glyphosate present in wastewater using nanofiltration.

3.2. Nanoadsorbents

Adsorption is one of the most suitable techniques for removing pollutants from water [54,55]. The interest in this method is due to the relative simplicity of operation, mild conditions for operation, such as room temperature, capacity to remove pollutants in low concentrations (micrograms), cost-energy efficiency, and low impact on the environment, in addition to the possibility to reuse and for the regeneration of the adsorbent [56,57]. Adsorption is a mass transfer unit operation in which a substance is transferred to the surface of a solid through physical or chemical interactions. The surface on which accumulation occurs is called an adsorbent. Adsorbate is a substance that adheres to the surface [54].

Nanomaterials have characteristics of interest for application as adsorbents in the remediation of water contaminated with emerging contaminants. Nanoparticles require lower contact time, faster removal, and lower dosages [54]. Besides their small size, these materials have a high surface area, high reactivity, catalytic potential, and a large number of active sites, enabling the adsorption of several substances. Furthermore, functional groups incorporation improves the adsorption capacity of nanomaterials. There is an increase in the surface area, reactivity, affinity, and selectivity toward the contaminant of interest [54,58,59]. Adsorption capacity is also related to factors such as the nature and size of the contaminant; pH, temperature, and ionic strength of the solution; solid-liquid balance; contact time; and mass transfer rate [54]. The most widely studied nanomaterials as adsorbents are carbon-based nanoadsorbents and metal-based nanoadsorbents.

Technological applications involving nanoadsorbents have been developed and patented. Patent US10143972B2 [60] describes the development of an ultrafiltration membrane comprising a sulfone polymer membrane and an organic polymer sealing layer, in which the pores are filled with nanoadsorbents. Nanoadsorbents are hollow porous nanoparticles of hydrated zirconium oxide. Patent US10245576B1 [61] describes a method for developing a magnetic polymeric nanocomposite aimed at removing bivalent heavy metals such as Pb (II) and Cd (II). Patent CN114471445A [62] describes a process for producing WO3@NiCoP nanoadsorbent for specific removal of tetracycline from water.

3.2.1. Carbon-Based nanoadsorbents

Carbon-based nanomaterials have been widely studied as adsorbents for water treatment. These nanoadsorbents have an ordered structure, high porosity, homogeneous pore size distribution, high specific surface, chemical and thermal stability, and non-toxicity [56]. These characteristics are favorable for their application in the remediation of contaminants. Moreover, the surface of these materials can be modified, converting them into functional materials to increase the ability to remove different contaminants. The adsorption mechanism of carbon-based nanomaterials mainly involves π - π interactions, hydrogen bonds, and electrostatic interactions [54–56]. π - π bonds occur between compounds that accept π electrons and π electron-rich regions on the surface. Electrostatic interactions occur when the pH of the solution is different from the isoelectric point of carbon-based nanoadsorbents. In this way, the material's surface acquires a positive or negative charge, interacting with the contaminant containing electrical charges [59]. Adsorption involving hydrophobic bonds occurs mainly when contaminants are in their molecular state and the nanoadsorbents charge is zero [63,64]. The presence of oxygen-containing functional groups in adsorbents also favors the adsorption phenomenon [65,66]. Hydrogen bonds also participate in adsorption processes and can be formed between the benzene ring on the surface of carbon nanotubes and organic compounds with functional groups containing oxygen (bivalent compounds) [67]. The main carbon-based nanoadsorbents reported are carbon nanotubes and graphene oxide.

The adsorbent properties of carbon nanotubes can be modified by covalent or noncovalent functionalization [54,56,68,69]. Another modification of carbon nanotubes is the treatment with ionic liquids, which increases the hydrophilicity of these nanoadsorbents and their dispersion in the aqueous phase and interaction with contaminants [56]. Carbon nanotubes demonstrated adsorbent potential for removing emerging contaminants such as antibiotics, hormones, and pesticides (Table 2).

Graphene oxide has excellent mechanical, thermal, and electrical properties and chemical stability [55]. Functional groups containing oxygen are introduced in the oxidation of graphene and provide interactions with different inorganic and organic pollutants, thus improving the removal efficiency of this adsorbent [56]. Another technique investigated to increase the adsorption capacity is the reduction of graphene oxide, which improves surface properties and hydrophobicity [55,56]. Graphene oxide is an efficient adsorbent of several compounds, such as aromatic compounds (phenanthrene and biphenyl), dyes, hormones, and pharmaceutical products [55,70–72].

Adsorbent	Modification	Adsorbate (Contaminant)	Adsorption Capacity (mg g ⁻¹)	Mechanism of Adsorption	Reference
Magnetic chitosan/activated		Simultaneous adsorption of cobalt ions (10 mg L^{-1})	44.5 (cobalt ion)	Electrostatic forces,	
carbon@UiO-66 bio-nanocomposite		malachite green dye (15 mg L ⁻¹)	62.1 (azo dye)	hydrogen bonding, π - π , and n- π interactions	[73]
(0.6 g L^{-1})		imidacloprid pesticide (6 mg L ⁻¹)	25.2 (imidacloprid)I	Interactions	
Zero-valent iron nanoparticles/graphene oxide/copper nanocomposite		Bisphenol A $(10 \text{ mg } \text{L}^{-1})$	21.6	Hydrogen bonding	[74]
Polypyrrole doped graphene oxide/covalent		Indomethacin $(5-40 \text{ mg L}^{-1})$	115	Hydrogen bonding, electrostatic	[75]
organic framework nanocomposite (0.2 g L^{-1})		Diclofenac $(5-30 \text{ mg L}^{-1})$	138	interactions, and π - π interaction	
Carbon nanotube (0.5 g L^{-1})	Functionalized with sodium hypochlorite	Ciprofloxacin $(10-160 \text{ mg L}^{-1})$	206	Electrostatic interaction	[76]
		Estrone $(2-50 \text{ mg } \text{L}^{-1})$	50.6		
Multi-walled carbon nanotube (0.8 g L^{-1})	Modify with KOH	17β -estradiol (2–50 mg L ⁻¹)	74.3	Physisorption mechanism	[77]
		17α -ethinylestradiol (2–50 mg L ⁻¹)	39.2		
Multi-walled carbon nanotube (1 g L ⁻¹)	β-cyclodextrin and MnFe ₂ O ₄ nanoparticles	Tetracycline (10–100 mg L^{-1})	89.5	π-π interaction, electrostatic interactions, hydrogen bonding	[78]

Table 2. Application of different nanomaterials in the adsorption of several emerging contaminants.

Adsorbate Adsorption Mechanism of Adsorbent Modification Reference Capacity (mg g⁻¹) (Contaminant) Adsorption Hydrogen bonding, Van der Waals forces, Multi-walled carbon Functionalized with Atrazine herbicide 40.2 [79] nanotubes (0.2 g L^{-1}) magnetic iron oxides $(1-20 \text{ mg } \text{L}^{-1})$ and hydrophobic interactions Functionalized with magnetic iron oxide Caffeine Hydrogen bonding Graphene oxide (0.5 g L^{-1}) 28.9 [70] nanoparticles and $(1-60 \text{ mg } \text{L}^{-1})$ and π - π interaction cobalt oxide nanoparticles Metformin Graphene oxide π - π interaction and 96.8 [80] (0.15 g L^{-1}) $(8-40 \text{ mg L}^{-1})$ hydrogen bonding 17β-Estradiol Graphene oxide nanosheets π - π interaction and estrogen 149 [71] (0.1 g L^{-1}) hydrogen bonding $(0.05-4 \text{ mg L}^{-1})$ Atenolol 136 Carbamazepine 147 $(0.1-100 \text{ mg}^{-1}\text{L}^{-1})$ Electrostatic and π - π Ciprofloxacin 329 Porous reduced graphene interactions. $(0.1-100 \text{ mg L}^{-1})$ [72] hydrogen bonding, oxide (0.25 g L^{-1}) Diclofenac and van der Waals 76 $(0.1-100 \text{ mg } \text{L}^{-1})$ forces Gemfibrozil 40 $(0.1-15 \text{ mg } \text{L}^{-1})$ Ibuprofen 29 $(0.1-21 \text{ mg } \text{L}^{-1})$ p-Chloro-m-xylenol 79 (50 mg L^{-1}) Bisphenol A 97 $(50 \text{ mg } \text{L}^{-1})$ Metal-organic framework Functionalized with Naproxen Hydrogen bonding [69] (MOF) MIL-101 (0.2 g L⁻¹) 156 hydroxyl groups (50 mg L^{-1}) Triclosan (50 mg L^{-1}) 112 Ketoprofen 156 $(50 \text{ mg } \text{L}^{-1})$ Metal-organic framework Carbamazepine Hydrophobic and 82.6 [81] $(5-100 \text{ mg} \dot{L}^{-1})$ (MOF) UiO-67 (1.2 g L⁻¹) π - π interactions Organophosphorus Aluminum-based Electrostatic Functionalized with insecticide 513 metal-organic framework interactions and [68] dimethoate amino groups $(Al-MIL-53) (2.6 g L^{-1})$ hydrogen bond

Table 2. Cont.

3.2.2. Metal-Based Nanoadsorbents

 (20 mg L^{-1})

Metal-based nanoadsorbents are inorganic nanomaterials widely used for contaminant removal. The commonly used are metal oxides such as iron oxide, silica, magnesium oxide, manganese oxide, zinc oxide, aluminum oxide, and titanium oxide [54,55]. In addition to the high surface area, these materials have a specific affinity, low solubility, low environmental impact, and do not cause the formation of secondary pollution [55].

Magnetic nanomaterials, such as magnetic iron oxide nanoparticles, have also been used as adsorbents. These materials are easily separated from the solution due to their magnetic properties [82,83]. Another metal-based nanomaterial investigated is the metal-organic framework (MOF)-based nano adsorbents. Some MOF applied for mitigation of emerging pollutants are Zr-biphenyldicarboxylate (UiO-67), Zn-2-methylimidazolate (ZIF-8), and metal-benzenetricarboxylate (MIL-100). Furthermore, these nanomaterials

have a high capacity for regeneration and reuse, contributing to water and wastewater treatment efficiency [84].

The predominant mechanisms for contaminants adsorption by metal-based nanomaterials are electrostatic interactions, Lewis acid-base interactions, hydrogen bonding, and π - π interactions [84]. Electrostatic interactions were the main mechanism of interaction between MOF and pharmaceutical and personal care products such as naproxen and clofibric acid, and the herbicide 2,4-dichlorophenoxyacetic acid. The interaction occurred between the positively charged adsorbent and the negatively charged contaminant [85]. Abdelhameed et al. [68] investigated the interaction mechanisms between the adsorbent aluminum-organic framework with amino-group and the organophosphate insecticide dimethoate. The authors reported hydrogen bonds between the oxygen of the phosphate and the hydroxyl hydrogen of the MOFs, as well as between the amine hydrogen and the sulfur atom of the dimethoate. Electrostatic interactions were also observed. Caravaca et al. [86] reported that under acidic conditions, the adsorption of amoxicillin onto silverfunctionalized magnetic nanoparticles occurred by the interaction between the π bonds of the amoxicillin rings and the positive ions of the adsorbent. Hydrogen bonding between H of amoxicillin and O of adsorbent also contributed to adsorption. Therefore, the adsorption mechanism of emerging contaminants may vary according to different factors such as process parameters, characteristics of the adsorbent, and the contaminant.

Metal-based nanomaterials have been applied as adsorbents to remove pharmaceutical and personal care products, pesticides, antibiotics, dyes, and heavy metals [58,68,69,84–86]. Table 2 shows some examples of these applications.

3.2.3. Other Nanoadsorbents

Dendrimers are polymeric nanostructures with a three-dimensional configuration that have appropriate characteristics for the adsorption of contaminants. They have a highly branched structure with different functional groups on the surface. Its high specific internal and external area improves the binding capacity to the pollutants of interest [58,83]. Poly(propylene imine) dendrimer was used to modify multi-walled carbon nanotubes for the removal of dyes from textile wastewater. The dye removal efficiency increased from 17.7% to 99.9% after modifying the nanotubes with dendrimer [87].

Studies have developed nanocomposites from nanoscale materials associated with other matrices (Table 2) to improve the adsorbent potential [75,83,88]. A polymer that has been used in the synthesis of adsorbent nanocomposites is the polypyrrole [75,83]. This conductive polymer tends to aggregate. However, its application in nanocomposites may reduce the aggregation and increase the surface area [83]. Nanocomposite from polypyrrole doped with graphene oxide and covalent organic structure was produced to remove indomethacin and diclofenac drugs. The nanocomposite showed higher adsorption capacity than graphene oxide and the combination of covalent organic structure with graphene oxide [75]. Thus, the high surface area of nanomaterials combined with the high number of functional groups of polymers improves the capacity for removing contaminants [83].

Furthermore, recent studies have investigated combining adsorbent materials such as carbon-based nanomaterials and metal-based nanoparticles to increase the adsorption capacity [70,74,78]. For instance, Foroutan et al. [78] synthesized magnetic compounds from carbon nanotubes, manganese ferrite nanoparticles, and β -cyclodextrin and evaluated the capacity to remove the tetracycline antibiotic. The adsorption capacity of this adsorbent (89.53 mg g⁻¹) was higher than that of carbon nanotubes (82.66 mg g⁻¹).

4. Synthesis and Fabrication of Nanomaterials for the Remediation of Emerging Pollutants

The removal of contaminants can be performed by nanomaterials involving adsorption, and filtration, among other processes [89]. The synthesis of materials may vary according to the type of nanomaterial and its application (Figure 2).



Figure 2. Schematic illustration of the synthesis of nanomaterials for application in the remediation of emerging pollutants.

4.1. Nanofiltration Membranes

Wastewater filtration processes using nanofiltration membranes are considered effective in the effluent treatment because they have high density and small pores (less than 2 nm) that allow the application in the retention of small molecules and multivalent ions [45,90]. Nanofiltration polymeric membranes can be manufactured by different methods, including interfacial polymerization, surface modification, and phase inversion. In interfacial polymerization, condensation occurs between the monomers at the interface of two immiscible phases. In this way, the monomer is placed in contact with the porous support, removing the remainder of the aqueous solution. Subsequently, the organic phase is added to the support, forming a layer at the interface [91]. Among the monomers that can act in the formation of the surface barrier, it is worth mentioning tannic acid, phenyl-diamine, and bisphenol A [92]. Nanofiltration membranes produced by the interfacial polymerization method are usually negatively charged, which interferes with the crosslinking process of the material due to charge repulsion [93]. In this sense, positively charged membranes can be obtained through the in-situ introduction or grafting of electropositive monomers [94–96].

In interfacial polymerization, surface, plasma, and layer-by-layer treatments can also be applied, enhancing membrane action [91,97]. Membranes incorporated by nanoparticles gained prominence for being able to increase membrane permeability, in addition to contributing to mechanical properties, hydrophilicity, and selectivity. Titanium dioxide, silica, silver, and zinc oxide nanoparticles are commonly reported in the fabrication of nanofiltration membranes [92]. Song et al. [98] evaluated the ability to remove small-sized cationic contaminants by nanocomposite membranes added with quarternized carbonbased nanoparticles. The incorporation of the nanoparticles in the membranes reached the ideal permeate flux (23.8 L m⁻² h⁻¹ bar⁻¹), high rejection rates (around 95%) for the contaminants (cationic trimethoprim, atenolol, rhodamine B, methylene blue and MgCl₂), and flow rate and recovery 85.5% compared to the pure membrane. In this context, Martins et al. [99] studied the incorporation of Au/TiO₂ (gold and titanium dioxide) and Y₂(CO₃)₃ (Yttrium carbonate) nanoparticles in poly (vinylidene-hexafluoropropylene fluoride) nanomembranes to contaminant removal. The materials added with nanoparticles showed 57% degradation of the norfloxacin.

Regarding surface modification strategies, UV-assisted graft polymerization, electron beam irradiation, plasma, and layer-by-layer approach can be performed. The UV-assisted graft polymerization process occurs due to a chemical reaction involving the active film and substrate present in the membrane [100]. High-energy electron beam irradiation penetrates the polymer layer and contributes to the reduction of membrane pores [101]. In plasma surface modification, nitrogen functional groups increase the hydrophobic characteristic of the membrane [102]. The alternating incorporation of polyelectrolytes (cationic and anionic) occurs in the layer-by-layer method. In the phase inversion, the thermodynamically stable polymer solution is separated into two phases, the first with a high concentration of polymer and the other with a smaller amount of the material [91].

Phase inversion can be used to prepare ceramic nanofiltration membranes for removing emerging pollutants. These ceramic membranes have high thermal, mechanical, and chemical stability, well-defined pore size distribution, and high flux [103]. High dye rejections were achieved with ceramic nanofiltration membrane (>98%) [104,105]. Moreover, Wang et al. [106] present a strategy for developing a robust, high-performance, zero-energy catalytic ceramic membrane to remove micropollutants in wastewater. Membranes functionalized with an active $CoFe_2O_4$ nanocatalyst effectively degraded emerging contaminants. There is a reduction in membrane fouling and a low level of cobalt ion leaching, indicating no environmental risk for the water treatment. Furthermore, according to the authors, ceramic nanofiltration membranes' characteristics (high performance in permeability and removal efficiency and zero energy consumption) promote their application in removing other organic micropollutants from different watercourses.

4.2. Carbon-Based Nanoadsorbents

Carbon-based nanomaterials have been commonly applied in the remediation of pollutants by adsorption processes. The most used nanoadsorbents for this function are nanotubes, fullerenes, nanodiamonds, and graphene nanomaterials [107]. Carbon nanotubes consist of sheets of graphene structured in a cylindrical shape, formed by carbon atoms with sp2 configuration [108]. These nanomaterials can be single-walled or multi-walled (association of single-walled nanotubes) [109].

Carbon nanotubes are commonly produced by the arc discharge method with graphite electrodes. The process consists of passing an electric current between the electrodes (anode and cathode) inside a chamber containing inert gas (helium gas is generally used) [110]. In this way, carbon atoms move from the anode to the cathode, forming carbon nanotubes at the negative electrode [111]. In the chemical vapor deposition method, the production of high purity nanotubes consists of the decomposition of hydrocarbons in the vapor phase or carbon monoxide due to the application of high temperatures (700 and 1000 °C) in the presence of catalysts [112]. The process is carried out in a quartz chamber giving rise to free carbon atoms. These diffuse and deposit on a metallic surface, reorganizing and forming carbon nanotubes [108]. Furthermore, the adsorption capacity of carbon nanotubes can be improved through surface modifications. For this, oxidizing treatments and chemical surface functionalization techniques are used, adding functional groups (polymers, surfactants) [113].

Graphene is composed of a single layer of carbon atoms arranged in a two-dimensional "honeycomb" structure [114,115]. This compound is widely used in the extraction of organic compounds due to the benzene ring in its structure [116]. The functionalization of graphene is performed to improve the pollutant removal capacity of this compound. Generally, it is applied in the form of graphene oxide (functionalized or reduced). Graphene oxide is characterized by the greater predominance of functional groups (such as phenolic hydroxyl (-OH), carboxyl (-COOH), epoxy groups (-COC-), which improve the absorption of pollutants [113].

Graphene oxide can be synthesized by oxidation of graphite or by exfoliation. Staudenmaier, Brodie, and Hummers' methods are commonly used to synthesize this compound. The oxidation of graphite is carried out with potassium chlorate and nitric oxide (Staudenmaier and Brodie) and potassium permanganate and sulfuric acid (Hummers) [117]. In the oxidation of graphite, the stacked sheets of graphene are pulled apart, due to the breakage of the π bond. The GO resulting from this process can also be modified into reduced graphene oxide through chemical, photocatalytic, hydrothermal, or microwave reduction. Furthermore, the functionalization of GO can be performed by doping or by covalent or non-covalent bonds [108].

Carbon nanofibers are mainly developed by electrospinning, chemical vapor deposition, and modeling techniques. Regarding fullerenes, they can be synthesized similarly to carbon nanotubes. Nano-diamonds can be produced by microwave plasma-assisted chemical vapor deposition, laser and ion deposition, blasting, hydrothermal processes and ultrasound. However, the most used technique is chemical vapor deposition [107].

4.3. Metal-Based Nanoadsorbents

Ferromagnetic or ferrimagnetic nanoparticles are also widely studied and applied to remove contaminants. These nanomaterials can be produced by some synthetic routes, which involve chemical, physical, and biological processes [118]. Among the chemical processes applied to the production of these nanomaterials, coprecipitation, thermal decomposition, microwaves, sol-gel, hydrothermal and solvothermal synthesis, microemulsion, sonochemistry, and polyol stand out. Chemical methods are widely used because they present high yield of nanoparticle production with low economic investment [119–123]. Another method used is the wet chemical reduction, which allows the morphological control of the developed nanomaterials and incorporates organic or inorganic ligand [124].

Regarding the production of nanoparticles by physical methods, mechanical milling, electron beam lithography, gas-phase deposition, vapor deposition/standardization, and the electrical explosion of wires are applied [125]. The milling process stands out for its simplicity, in addition to being one of the most used methods for obtaining nanomaterials. In the electron beam lithography method, an electron beam is applied to convert iron particles into iron oxides [126]. Concerning gas-phase deposition, there is the development of a one-dimensional structure of iron oxide through catalyst-assisted chemical vapor deposition on alumina and gold-coated substrates [127]. Physical vapor deposition (evaporation, sputtering and laser ablation) develops thin films by transferring magnetic particles onto a substrate. The material for the development of nanoparticles can also be deposited by electrodeposition processes [128,129]. The electric wire explosion method applies an electric current sufficient to evaporate a metal wire [130].

The biological synthesis of nanoparticles takes place in the bottom-up approach, where metal ion redox processes and stabilization processes control the size of the nanoparticles. Coatings such as proteins, tannins, and peptides as covering agents are incorporated into the nanoparticles, improving the stability and dispersion of the nanomaterials [131]. The biosynthesis of these nanomaterials can be carried out through biological routes by different organisms, including viruses, algae, microalgae, bacteria, and plants. Furthermore, nanoparticles can be synthesized from macromolecules [132].

Regarding biosynthesis by microorganisms, the formation of nanoparticles can occur both intracellularly and extracellularly [133]. In the intracellular mechanism, there is the diffusion of metal ions in the cells that are reduced by enzymes to produce the nanoparticles. The extracellular process is based on the electrostatic interaction of metal ions, which are attracted to the cell wall of the microorganism for enzymatic reduction to occur [134].

Microalgae are commonly applied to produce metallic nanoparticles. These nanomaterials are formed by the heavy metal biosorption process and are potentially suitable for the treatment of effluents [135]. The biosorption process occurs with the binding of heavy metals at specific sites in the microalgal cell structure. The cell walls of these microorganisms are composed of functional groups of carbohydrates and negatively charged polysaccharides (such as hydroxyl, amino, carboxyl, or sulfhydryl) that represent the binding sites for heavy metals in aqueous solution [136].

The contribution of microalgae in the removal of contaminants does not occur only with the production of metallic nanoparticles. Microalgae reduce the contaminants through mechanisms of bioaccumulation, biodegradation, and biosorption. Thus, these microorganisms are applied to mitigate emerging pollutants, including pesticides, hormones, hydrocarbons, dyes, and heavy metal constituents from wastewater [135]. In this context, silver nanoparticles synthesized by microalgae can also remove pathogens in agriculture, contributing to the environment [137].

5. Nanoadsorbent Regeneration Techniques

Adsorbents saturated with emerging pollutants must be regenerated to reuse in new processes. Type of adsorbent and contaminant, adsorbent stability, and toxicity influence the selection of the regeneration method. Thus, thermal, chemical, oxidation, or electrochemical process have been applied for reusing these materials [138,139].

For example, microwave regeneration is a thermal method for carbon-based adsorbents [140]. Shoushtarian et al. [141] applied graphene oxide nanoadsorbents in the adsorption of Basic Red 46 dye and studied the regeneration of the material by the microwave method. The nanomaterial showed good adsorption capacity (360 mg g⁻¹), even after four regeneration cycles (209 mg L⁻¹). Electrochemical regeneration of nanoadsorbents was investigated by Abdul-Ghani et al. [142]. The authors developed two-dimensional terephthalate composite nanosheets with Mxene material intercalated with sodium ions. The ability to remove ciprofloxacin and regenerate the nanomaterial was evaluated. The nanoadsorbent reduced 9 mg L⁻¹ of the contaminant in 15–20 min and was completely recovered by the electrochemical regeneration method in 5 min.

The application of nanoparticles as nanoadsorbents facilitates the recovery process of the nanomaterial, which can be performed by filtration, centrifugation, and magnetic separation methods [143]. Magnetic adsorbents can be recovered from the liquid medium by applying a magnetic field. After, they can be regenerated and reused [139]. The adsorbents' regeneration process can also be carried out under the influence of pH, and NaOH/HCl solution can be applied for desorption of the material [138]. The most commonly used acids and bases are HCl, HNO₃, H₂SO₄, EDTA, Ca(NO₃)₂, NaOH, and NaNO₃, usually in low concentrations (0.1–0.2 M) [144].

The reuse of magnetic composites (CNT/MnFe₂O₄ and CNT/ β -CD/MnFe₂O₄) after the removal of the antibiotic tetracycline was evaluated by Foroutan et al. [78]. The recovery process used NaOH solution and subsequent washing with distilled water. The adsorption efficiency was greater than 90% even after eight cycles of adsorption/desorption. Motaghi et al. [73] investigated the reuse of chitosan/activated carbon@UiO-66 magnetic bio-nanocomposite after simultaneous removal of cobalt ions, azo dye, and imidacloprid pesticide. The adsorbent was separated from the aqueous solutions by applying a magnetic field. Then, it was regenerated by using citric acid and washing with a mixture of ethanol and deionized water. The nanocomposite showed a removal capacity of 88.56%, 93.45%, and 88.45% for cobalt ions, azo dye, and imidacloprid, respectively, even after five cycles of regeneration.

Organic solvents have also been used in the desorption of nanoadsorbents. Andrade et al. [70] investigated the application of different solvents in the desorption of caffeine from graphene oxide functionalized with magnetic iron oxide nanoparticles and cobalt oxide nanoparticles. Ethanol showed the highest desorption capacity with efficiency greater than 70% after five cycles of adsorption/desorption. In this reuse process, an external magnetic field was used to separate the adsorbent. Wu et al. [145] synthesized a magnetic copper-based metal-organic framework and evaluated its potential as a recyclable adsorbent for removing ciprofloxacin and norfloxacin. The material showed high adsorption efficiency (approximately 90%) after reuse for ten cycles, using methanol/ammonia solution as a desorption solvent. Akpinar and Yazaydin [81] evaluated the metal-organic framework UiO-67 for its carbamazepine adsorption capacity and reusability after desorption in acetone and reactivation under vacuum at 90 °C. The adsorbent was reused five times, and the adsorption capacity remained above 90% for all regeneration cycles.

After reaching the limit of reuse cycles, the adsorbent must be disposed of safely. Some examples of procedures used in the management of these adsorbents are incineration, disposal in landfills, or reuse in other applications. The deposition of the adsorbent, containing the contaminant, in landfills requires that the pollutant remain adsorbed for a long period to avoid its release into the environment. The reuse in other applications requires information about the material's toxicity and direct or indirect impact on human health. For the management of the adsorbents used, it is also important to know the concentration of the adsorbed contaminant in case it has not been desorbed [139].

6. Conclusions

Several contaminants from the industrial sector and consumer routine are contaminating water bodies. Emerging pollutants have long-term persistence in the environment without undergoing degradation, in addition to bringing harm to consumer health. Therefore, treatment techniques for these compounds are necessary for the decontamination of wastewater and were addressed by this review article. Emerging contaminants include pesticides, surfactants, plasticizers, fire retardants, compounds from the pharmaceutical industry, and personal care products.

Using the nanomaterials is effective in the remediation of water and effluents due to their mechanical and adsorption properties. The adsorptive capacity of these materials is related to the high surface area and porosity, in addition to the morphological aspect controlled in the manufacturing process. In addition, carbon-based nanomaterials (carbon nanotubes, graphene nanomaterials) and metal-based nanomaterials are technologies used in the adsorption process to remove contaminants. There is a diversity of nanoscale materials and contaminant removal processes to be employed. Therefore, research should continue to develop nanomaterials for effluent remediation to find materials and mechanisms increasingly suitable for different contaminants. Knowledge about the materials and their processing contributes to expanding the process to the industrial scale, allowing companies to apply this technology.

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