

Emerging organic pollutants in aqueous environments: Detection, monitoring, and removal techniques

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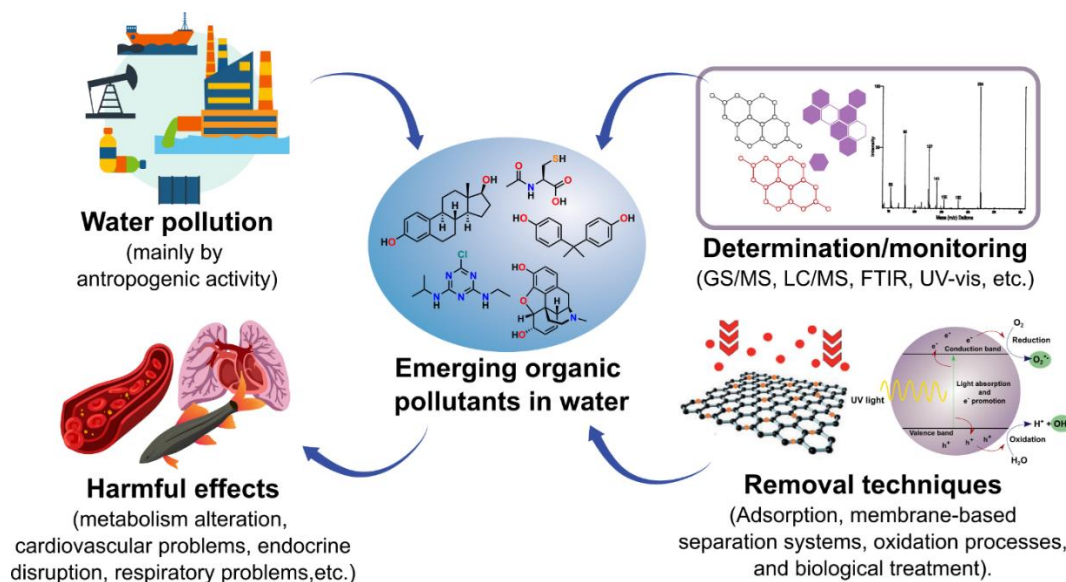
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Graphical Abstract



Abstract. Currently, human beings face different problematics associated with environmental pollution, including the continuous degradation of the quality of water and, in this sense, its availability to be used for many purposes. Various factors are responsive for water pollution; however, anthropogenic activity (industries, agriculture, domestic processes, etc.) has the highest contribution. Specifically, anthropogenic activity has caused a vast pollution of almost all the water environments around the world by different types of chemical substances and materials, also called “emerging pollutants/contaminants”. Among these, those with an organic nature result to be of great interest due to their relatively high concentrations in waters, their structural and behavioral diversity, as well as, their potential risks to biota. To deal with this, different analytical techniques and removal methodologies to detect/monitor and eliminate emerging organic pollutants from water, respectively, have been well-studied and developed through time. Thus, due to the large quantity of works and advances carried out in this topic, it is important to take a look and review the different aspects involved in such techniques

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Review



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and methodologies, which is the aim of this review. Here, an introduction to the concept of emerging organic pollutants is firstly done, including a bit of history about water pollution, the description of the most important classes of organic pollutants, their main sources and effects on biota. Then, sampling, sample pretreatment techniques (mainly liquid-liquid phase extraction and solid phase extraction), and the analytical determination of these compounds (e.g., employing gas chromatography, liquid chromatography, and mass spectrometry) are discussed and exemplified through relevant, most recent works. Finally, both conventional and non-conventional water treatment techniques, like adsorption processes, membrane-based separation systems, and advanced oxidation, are approached. Throughout the review, the discussion of different important aspects, such as advantages, disadvantages, and variants of every technique or methodology, are also included.

Keywords: Water pollution, emerging pollutants, contaminants, removal techniques, analytical determination.

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1. Introduction

Liquid fresh water represents almost 0.8 % of all the water in the world, i.e., approximately 11 million km³, which results to be a relatively small quantity that needs to be cared for and preserved to a high extent. However, that task has not been apparently easy due to various factors, including population growth, urbanization, and industrial development, which have been contributed to the pollution of fresh water through time. Thus, the scarcity of fresh water because of its high pollution rate and overexploitation is one of the biggest environmental problems that humanity faces currently (Borrull et al., 2020; Schwarzenbach et al., 2010). In this sense, more than one thousand chemical organic substances have been identified in different water environments, which due to their concentrations, structural and behavioral diversity, harmful effects on human health and biota, and their persistence in the environment are of emerging concern to environmentalists and governmental agencies (Taylor et al., 2021; Yang et al., 2020). These substances have been also called “emerging organic pollutants” (EOPs) and they are the objective of this review.

More specifically, EOPs comprise a high quantity of organic substances that have been persistently found in water (and other environments), which have a perceived or veridical threat to living

beings, but with a lack of published health criteria, in almost all cases (Sauvé and Desroires, 2014). Different types of EOPs have been identified in water, among which there are pharmaceuticals, biocides, personal care compounds, industrial chemicals and additives, illicit drugs, biological toxins, and even nowadays nanomaterials (Chander et al., 2016; Chaturvedi and Dave, 2021; Davoli et al., 2019; Esposito et al., 2020; Joseph, 2017). These can reach water bodies through different sources, for example, agricultural practices, industry, daily household activities, etc., and processes like discharges, surface runoff, atmosphere precipitation, or subsurface infiltration (Ahmad et al., 2020; Parra-Saldivar et al., 2020). In terms of their toxicity, EOPs can cause affectations in aquatic organisms by metabolism alterations, as well as, many harmful effects on humans, such as gastrointestinal, kidney, liver, nervous system, endocrine, and immune system disorders (Alharbi et al., 2018; Bhavya et al., 2021; Saaristo et al., 2018).

In order to deal with this environmental problem, it has been necessary, first, utilizing proper analytical techniques to measure and monitor the levels of EOPs in water, such that it is possible to obtain relevant information about their occurrence, fate and environmental behavior. This is the base for further regulations and the establishment of proper solutions. To accomplish it, different analytical methodologies have been implemented, including gas chromatography, liquid chromatography, and mass spectrometry, as well as, Fourier transform infrared spectroscopy, Raman spectroscopy, UV-Vis spectroscopy, and fluorescence measurements (Quintelas et al., 2020; Warner et al., 2020; Yang et al., 2020). Among these, chromatographic techniques coupled with mass spectrometry as detector have had the best results to quantify EOPs in water and, thus, they have been widely accepted and implemented for this purpose. However, various aspects associated to the targeted EOPs have to be taken into account to a proper choice between those techniques, e.g., volatility, polarity, functional groups, thermal stability, etc. In addition, some sample pretreatment steps are carefully choice with the aim to achieve a correct determination of EOPs in complex matrixes (Alvares and Jones-Lepp, 2010; Hartmann et al., 2020; Tang et al., 2019).

On the other hand, various removal methodologies of EOPs from water have been developed and studied as a part of a complete water treatment process since the end of the last century. They can divide in conventional water treatments and non-conventional water treatment. For its part, conventional water treatment refers to the commonly used techniques for decontaminating wastewater, for example, filtration, coagulation/flocculation, sedimentation, and biological treatment, which are widely found in wastewater treatment plants (Golovko et al., 2021; Kerasi et al., 2021; Turan et al., 2021). In terms of removal efficiency, conventional treatments remain limited for EOPs removal, such that, high concentrations of these pollutants are usually found in the obtained effluents (Cristaldi et al., 2020; Di Marcantonio et al., 2020; Khasawneh and Palaniandy, 2021). For this, it has been necessary to implemented additional, non-conventional treatment techniques to achieve a higher removal efficiency. In this case, adsorption processes (e.g., silica adsorbents, polymeric adsorbents, clay adsorbents, nano-adsorbents, etc.), membrane-based separation systems (e.g.,

microfiltration, ultrafiltration, nanofiltration, reverse osmosis, among others), and advanced oxidation processes (e.g., UV irradiation, ultrasonics, TiO₂ photocatalyst, H₂O₂, O₃, etc.) are the most promising methodologies (Bouyarmane et al., 2021; Ding et al., 2020; Lopera et al., 2019; Sun et al., 2021; Tak and Vellanki, 2019). Nowadays, this is an important topic in different research field, such as environmental sciences, chemistry, engineering, physics, etc., since various aspects remain as optimizable parameters and some drawbacks of the techniques need to be overcome for their full application.

In this sense, this review seeks to provide a more accurate view about EOPs in terms of the most employed analytical techniques for their determination and monitoring, as well as, the most utilized and explored methodologies for their removal. Here, general aspects associated with EOPs, such as a bit of history about water pollution, established definitions, types, and reported harmful effects are firstly covered. After that, a deeper discussion about the implementation of chromatographic techniques and mass spectrometry in the determination of EOPs in water is provided. Other less implemented techniques, such as FTIR, UV-Vis, and fluorescence are also included. Finally, an approach to conventional and non-conventional water treatment techniques is carried out, exemplifying them with relevant and recent works published in literature. Aspects such as basic principles, advantages, disadvantages, and some characteristics to be explored of each technique or methodology are discussed throughout the review.

2. A brief historical summary of water pollution by organic substances

Although environmental pollution has been an evident fact since the establishment of human beings as a society, this problem has only recently been taken into account. In the case of water pollution, the research began in the early 19th century and it was due to the emergence of different deadly conditions and their spread through this medium (Vasilachi et al., 2021). An example of this is the increase of deaths by cholera in London in the nineteenth century due to the consumption of high-polluted water from the Thames River. In the same way, typhoid fever claimed many lives in the United States in the last century due to its spread by water, for which there was no appropriate treatment. For this, the development of water treatment methodologies, such as filtration and chlorination, was promoted and carried out (Okun, 1999).

Initially, water treatment was focused mainly in the removal of bacterial pathogens through which those infections were produced. However, the chemical revolution that accompanied the technological advance of World War I and World War II led to the production of thousands of different synthetic organic substances, which began to contaminate water bodies. At first, these chemicals were produced for different purposes, including chemical weapons (e.g., bromine ethyl acetate, chlorine, diphosgene), pharmaceuticals (e.g., salicylic acid, oxytetracycline, phenazone), pesticides (e.g., chlorophenols, carbamates, naphthalene), among others (Larsson, 2014; Ongley, 1996; Özkara et al., 2016); however, all of them had a particular and important characteristic: they were synthesized to

be long lasting to achieve economy in their application. In this sense, the wide use of these chemical agents linked to industrialization and the increase of world population, their long permanence in the environment and their propagation through different media, including water, led not only to their contamination, but also to the direct affectation of different species, including human beings (Peplow, 2020; Vilches et al., 2016).

It was not until the mid-20th century that the National Cancer Institute of the United States made a statement on this issue, mentioning the relationship between potential future cancer cases and the high consumption of water highly contaminated with chemical agents (Hueper, 1960). Later, the Environmental Protection Agency, or EPA, recognized the presence of many synthetic organic substances in different drinking water sources, particularly in the Mississippi River. At the same time, some epidemiological studies revealed higher rates of some forms of cancer in the population using the untreated Mississippi River water. Another manifestation of the impact of organic substances on the quality of water emerged from Europe at the end of 20th century, where it was determined various pharmaceutical compounds in drinking water from sources that had received human wastewaters. Curiously, the people who used this water presented a wide range of illnesses, from cardiac to mental (Shraim et al., 2017; Stan and Heberer, 1997). By that time, some widely used pesticides were detected in fish samples of a drinking water source in San Francisco, United States. What was important about this case not only was the detection of organic synthetic substances, but their presence in an important human food source such as fish. This was one of the first indications of the bioaccumulation of organic pollutants (Pico et al., 2019; Rocha et al., 2018). In Latin America the studies about water pollution started shortly after, detecting initially different pharmaceutical compounds and their metabolites in natural waters in Rio de Janeiro, Brazil (Stumpf et al., 1999). In this sense, these facts were the basis of a series of laws and projects focused on the control and regulation of organic substances in water by different organizations, for example the World Health Organization and the European Community (Okun, 1999). Also, the development and establishment of new technologies for the treatment of polluted water prioritized, e.g., precipitation, sorption processes, membrane technology, oxidative processes, among others (Angelakis et al., 2018; Seow et al., 2016; Vuorinen et al., 2007).

Since then, thousands of organic substances have been detected in different water bodies around the world, which reach these through various anthropogenic sources of pollution, e.g., industrial wastewater, agricultural processes, domestic sewage, among others (Fan et al., 2019; Wang et al., 2020a). These compounds, in conjunction with inorganic pollutants, were called “emerging pollutants” (EPs) or “contaminants of emerging concerns” and some important examples are listed in Table 1. In this point, it is important to highlight that the capacity to determine EPs in water has been linked directly to the advance in analytical techniques through which this kind of study has been possible until now (e.g., gas chromatography, liquid chromatography, mass spectrometry, UV spectroscopy, among others). This has had a rapid increase since the end of the last century (Martín-Pozo et al., 2019).

Table 1. Main types of EOPs and their characteristics.

Class	Description	Examples	References
Pharmaceuticals	Different synthetic or natural substances employed for the treatment of diseases and health problems in humans and/or animals. The most used pharmaceuticals are analgesics, antihypertensives, and antibiotics. Humans and animals can excrete them and they can reach water bodies through domestic wastewater, water treatment plants, hospital discharge, and even agricultural wastes.	Ibuprofen, diclofenac, carbamazepine, clofibrate, paracetamol, metformin, valproic acid, amoxicillin, acetylcysteine, mesalamine, iomeprol, allopurinol, tylosin, ivermectin, ciprofloxacin, metoprolol, fenofibric acid, among others.	Ahamad et al., (2020) Chander et al. (2016) Kümmerer (2010) Küster and Adler (2014)
Pesticides	A group of high stable and toxic substances employed to treat any pest. This "-cide" compound class includes herbicides, insecticides, nematocides, fungicides, bactericide, among others. These can reach surface water and groundwater through agricultural waste, industrial waste, air transportation, and wastewater.	Endosulfan, glyphosate, chlorpyrifos, atrazine, methyl parathion, heptachlor, carbofuran, pentachlorophenol, dichlorodiphenyltrichloroethane, dichlorodiphenyldichloroethylene, diflubenzuron, azoxystrobin, chlorothalonil, among others.	Jatoi et al. (2021) Sarker et al. (2021) Smital et al. (2004)
Illicit drugs	A class of substances that are produced, formulated, distributed, acquired, and consumed in an illicit way and which are used for non-medical purposes. It also includes some legal drugs used for recreational purposes.	Amphetamine, cocaine, morphine, benzoylcegonine, norcocaine; methamphetamine, heroin, cannabis, among others.	Davoli et al. (2019) Fabregat-Safont et al. (2021) Gil et al. (2012)
Personal care products	Substances used directly on the human body. Generally, they modified in some way the appearance, odor, and feel of the body. These compounds can reach water bodies through recreational waters, air, domestic sewage, etc.	Parabens, triclosan, methyltriclosan, triclocarban, <i>N,N</i> -diethyl- <i>m</i> -toluamide, celestolide, galaxolide, toxalide, phantolide, cashmeran, traseolide, methoxycinnamates, benzophenone, among others.	Montes-Grajales et al. (2017) Pemberthy et al. (2020) Yang et al. (2017a)
Industrial chemicals and additives	Chemical substances employed in different industrial processes or used as additives in various products: food, plastics, gasoline, emulsions, etc.	Tributyltin, methyl <i>tert</i> -butyl ether, 1,2,3-benzotriazole, benzothiazol-2-sulfonic acid, bisphenol A, di-2-ethylhexyl phosphate, octylphenol, naphthalene, benzo[a]pirene, benzene, 2,6-di- <i>tert</i> -butylphenol, 2,4-dinitrophenol, pentachlorophenol, etc.	Esposito et al. (2020) Kantiani et al. (2010) Salthammer (2020)
Water disinfection by-products	Chemical substances derived from the treatment of polluted water with disinfectant agents (Cl ₂ , O ₃ , NH ₂ Cl, UV radiation). Mainly, the raw material for these by-products is organic matter: humic acids, fulvic acids, amino acids, lipids, carbohydrates, organic acids, etc.	Chloroform, bromodichloromethane, pentachloropropene, trichloroacetic acid, dichloro-hydroxy-benzoic acid, 1,1,1-trichloropropanone, chloroisobutanol, 2,2-dichloroacetamide, trichloronitromethane, tetrachlorothiophene, etc.	Li et al. (2022) Park et al. (2016) Yang et al. (2021a)
Waterborne pathogens	Microorganisms (bacteria, virus, protozoa, and helminths) that are propagated through water and can cause any disease in the human body. Some pollution sources are fecal matter, food, and other polluted products.	<i>Campylobacter</i> spp., <i>Escherichia coli</i> , <i>Legionella pneumophila</i> , <i>Salmonella enterica</i> , <i>Helicobacter pylori</i> , rotavirus, sapoviruses, hepatitis A virus, adenovirus, <i>Acanthamoeba</i> spp., <i>Cryptosporidium parvum</i> , <i>Toxoplasma gondii</i> , <i>Dracunculus medinensis</i> , among others.	Baig et al. (2021) Ramírez-Castillo et al. (2015)
Biological toxins	Different biological molecules synthesized by living organisms, e.g., bacteria, fungi, plants, and animals, with defensive functions. They can cause any disease in contact with some particular enzymes or cellular receptors of the human body.	Aflatoxin, ochratoxin A, sterigmatocystin, rubratoxin, patulin, byssochlamine acid, microcystin, anatoxin, saxitoxin, prymnesin, palytoxin, vomitoxin, azaspiracids, okadaic acid, etc.	Alves et al. (2019) Hudnell (2005) Joseph (2017)

Table 1. (Continuation)

Nanomaterials	Materials with nanometric dimensions that are highly used in many applications, e.g., cosmetics, textiles, drug delivery systems, composites, etc, due to their unique chemical, biological, and physical properties. They can reach water bodies through wastewater systems, including sewers, water treatment plants, and sludge-incineration plants.	Fullerenes and derivatives, carbon nanotubes, quantum dots, natural organic particles, plastic and other polymers nanoparticles.	Chaturvedi and Dave (2021) Graca et al. (2018) Klaine et al. (2008) Malakar et al. (2021)
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In this sense, it has been possible to detect, monitor, and quantify EPs in different water bodies, which have allowed the establishment of some important features about EPs. For example, the relationship between the degree of development (including, social and technological aspects) of a society and the organic pollution degree of water, as well as geographical distributions of EPs around the world have been established, which is of utmost importance to facilitate the design of strategies to solve problems associated to their pollution (Wen et al., 2017). Also, one of the important aspects of EPs that has been studied in detail is their toxicity to biota, from aquatic species, like fishes, to human beings. Through this class of study, it has been possible to determine different harmful effects of EPs, e.g., histopathological effects, sexual disorders, oxidative stress, acute toxicity, respiratory inhibition, cancerous diseases, among others, even at concentrations since ng/L, depending on the type of pollutant and the time of exposure (La Farré et al., 2008; Pablos et al., 2015). Likewise, current research trends are focused on determining and understanding the fate, environmental interactions and metabolic pathways of EPs and their by-products, which is important to establish treatments for diseases associated with EPs, among other things (Vasilachi et al., 2021).

As can be seen above, water pollution by EPs is a multidisciplinary issue, of not only a scientific nature but also concerning politics and society. Today, this problem is still approached from different points of view: analytical chemistry, metabolomics, environmental sciences, separation sciences, engineering, and so on. For this, the research on EPs is one of the most important environmental research fields and it will continue for a long time while it is possible to give some substantial solutions to this problem.

3. Emerging organic pollutants (EOPs) in water

Although there is no unique and definitive definition about EPs, there are important characteristics that are mentioned in any definition of them: different nature, different origin, and potential harmful effects to biota (Geissen et al., 2015; Peña-Guzman et al., 2019; Vasilachi et al., 2021). Firstly, the nature and characteristics of EPs are diverse, for example, they can be organic or inorganic, polar or nonpolar, dissolved or undissolved, metabolizable or non-metabolizable compounds. Also, they can have different types of functional groups (i.e., carboxylic acid, alcohols, amines, thiols, etc.) or be able to be present in different states (Calvo-Flores et al., 2018; Cvetnić et al., 2019). Secondly, sources of EPs are diverse and can be classified as natural (e.g., organic matter, mineralization, volcanic activity) or anthropogenic (e.g., agriculture, industry, daily

household activities). In this way, pollutants can reach water bodies resulting of various processes, such as discharge processes, surface runoff, atmosphere precipitation, or subsurface infiltration (Ahamad et al., 2020). Thirdly, depending on their concentration, EPs can cause many harmful effects on biota and especially on humans, who may suffer gastrointestinal, kidney, liver, nervous system and immune system disorders (Pablos et al., 2015; Sanchez and Egea, 2018).

Therefore, various similar definitions have been mentioned through the development of this environmental issue. In particular, Sauvé and Desroires (2014) reviewed on the concept of EPs and established the following: “EPs are naturally occurring, manufactured or man-made chemicals or materials which have now been discovered or are suspected present in various environmental compartments and whose toxicity or persistence are likely to significantly alter the metabolism of a living being”. Thus, and in a general way, EPs are understood as a class of chemical compounds present in the environment by natural and/or anthropogenic sources with a perceived or veridical threat to living beings, and in almost all cases, with a lack of published health criteria (Lei et al., 2015; Peña-Guzman et al., 2019). It is important to be highlight that the term “emerging pollutant or contaminant” is not only applied to new substances, i.e., newly introduced chemicals into the environment, but also applies to known compounds with previously unrecognized harmful effects on ecosystems (Petrovic and Barceló, 2006). It has also been recommended to use the concept of “contaminants of emerging concern” (Geissen et al., 2015; Sauvé and Desroires, 2014). Likewise, some authors use this concept to refer exclusively to synthetic organic pollutants, however, it is a limited definition knowing the vast quantity of chemicals that arrive in water bodies, their different nature and characteristics, not only limited to organic compounds. One reason for this may be the relatively high quantity of organic emerging pollutants with respect to those inorganic pollutants, and their use in daily life (Ahamad et al., 2020). In this review, the term “emerging organic pollutants”, or simply EOPs, will be used to refer to EPs but only to those of organic nature.

Nowadays, a high quantity of EOPs have been detected and reported. For example, the European Union reported in 2016 more than 1000 substances as EOPs through its NORMAN network, a special platform designed for monitoring and tracking these pollutants (Dulio et al., 2018; NORMAN, 2021). The situation is not different for North America, Asia and Latin America, where the reports about EOPs have had a consistent increment since 2000 and it can be calculated thousands of EOPs in their environment (Bunke et al., 2019; Ramírez-Malule et al., 2020).

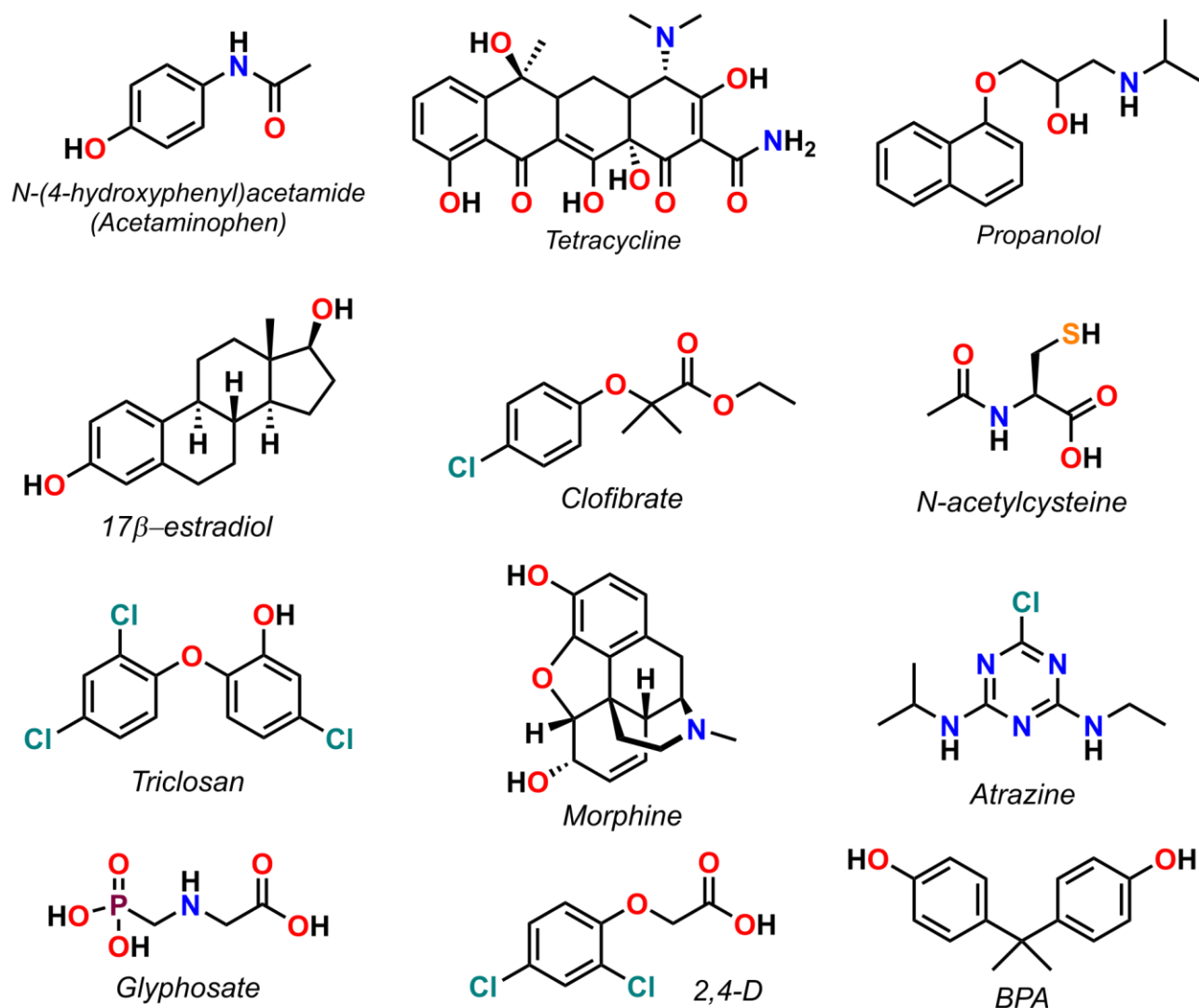
3.1. Types of EOPs

Due to the diversity of reported EOPs, they are usually classified in almost 20 different classes related to their use or function. The main ones are: pharmaceuticals, pesticides, illicit drugs, personal care products, industrial chemicals and additives, water disinfection by-products, waterborne pathogens, biological toxins, and nanomaterials (Abdulrazaq et al., 2020). Table 1 shows a summarized description and some examples of each one, while Figure 1 shows the most representative examples of EOPs.

Among them, pharmaceuticals are one of the best known, with an increasing production and use due to the improvement in health care and rising living standards. It has been reported that the worldwide consumption of pharmaceutical compounds is about 100,000 tons per year and more than 10,000 pharmaceuticals are approved for

humans, many of which have been found in aquatic environments at concentrations of ng/L to low µg/L range (Derksen et al., 2004; Kümmerer, 2010; Parra-Saldivar et al., 2020). These EOPs are subdivided into different classes of compounds according to their characteristics and functions, such as antibiotics, analgesics, hormones, β-blockers, and blood lipid regulators (Ahamad et al., 2020). Some representative examples of pharmaceuticals as EOPs are analgesics and nonsteroidal anti-inflammatories such as ibuprofen, acetaminophen (or paracetamol), and diclofenac, which are used to relieve pain, reduce inflammation and some symptoms like fever. The common dosage of these drugs is a few hundreds of mg per day, part of which is completely transformed into the body, generating a series of derived metabolites, and the rest is excreted as an unchanged molecule (Marchlewicz et al., 2015; Murdoch and Hay, 2015).

Figure 1. Chemical structures of some representative EOPs.



Other representative examples are antibiotics, such as tetracycline, oxytetracycline, erythromycin, and ciprofloxacin, employed to kill or inhibit the growth of microorganisms (Godoy and Sánchez, 2020); β -blockers, such as propranolol and metoprolol, which have a direct effect on the autonomic nervous system, playing a role in blood pressure control (Godoy et al., 2017; McBean et al., 2018; Wiysonge et al., 2017); hormones like estrone, 17 β -estradiol, and 17 β -ethinylestradiol (Houtman et al., 2018; Méndez et al., 2017); and lipid regulators like clofibrate, clofibric acid, and bezafibrate (Calvo-Flores et al., 2018; Hernando et al., 2007; Zhang et al., 2020a). Likewise, a few pharmaceutical compounds used for particular purposes can be included in this category of EOPs due to the increment of their usage. For example, biological thiols (i.e., RSH, where *R* is an organic group) such as L-cysteine and N-acetylcysteine are used in various pharmaceutical formulations due to their quelating power against different metal ions and their redox behavior (Mokhtari et al., 2017; Sales et al., 2003). Also, different pharmaceutical excipients, e.g., tartrazine, aspartame, benzalkonium chloride, propyl gallate, etc., have been found in water environments and it has been determined that they could represent a serious environmental risk (Carlsson et al., 2006; Haywood and Glass, 2011). In this sense, there is a high variety of pharmaceutical compounds from a chemical point of view, i.e., presence of different functional groups and different ways to carry out a chemical reaction, which in almost all cases are not metabolized before being excreted. For this, one important aspect of study is the determination of metabolites or by-products of pharmaceutical compounds in the environment and the metabolic pathways by which they can interact with natural structures and microorganisms; as well as their ecotoxicological effects (Gogoi et al., 2018; Rivera-Utrilla et al., 2013).

In some cases, personal care compounds and illicit drugs are linked to pharmaceutical compounds, however, there is an important difference according to their usage (Gil et al., 2012). Firstly, personal care compounds include disinfectants, soaps, insect repellents, preservatives, fragrances, and sunscreen ultraviolet filters, which are employed to satisfy any particular aspect of the care of the human body (Pemberthy et al., 2020). These compounds are highly used in daily life, even in higher quantities than those recommended. Additionally, personal care compounds can reach water bodies through other important routes like recreational activities, bathing and washing clothes (Lei et al., 2015). Thus, it has been reported that personal care compounds such as triclosan, triclocarban, and galaxolide, may be present in water bodies at concentrations of a few nanograms per liter to a few milligrams per liter (Chaturvedi et al., 2021; Montes-Grajales et al., 2017).

On the other hand, illicit drugs correspond to legal (e.g., morphine) or illegal drugs (cocaine, methamphetamine) used for recreational purposes, i.e. in order to generate a psychostimulant effect on the human body (Davoli et al., 2019). They can be divided into opiates, central nervous system depressants, central nervous system stimulants, and hallucinogens (Calvo-Flores et al., 2018). There are different sources by which these substances and their metabolites can reach water bodies: human excretion, sweat, saliva, accidental spillage or wastes from clandestine laboratories (Binelli et al.,

2012). Actually, hundreds of millions of people use illicit drugs. For example, cannabis is used by about 130-190 million people worldwide, followed by cocaine and opiates (Binelli et al., 2012). These facts are the reason why these substances began to be found in water bodies, even at levels of $\mu\text{g/L}$ (Archer et al., 2017; Castiglioni et al., 2006; Fantuzzi et al., 2018).

Pesticides are another important class of EPs in water environments. According to EPA (2021), this term is used to refer to a broad class of substances employed for preventing, destroying, repelling, or mitigating any pest. This includes different groups of “-cide” compounds used to treat particular pests, e.g. herbicides (i.e., weeds), fungicides (i.e., fungus), insecticides (i.e., insects), and so on (Calvo-Flores et al., 2018). It has been estimated that the global production of pesticides exceed 4 millions tonnes by 2018 (FAOSTAT, 2021), out of which approximately 48 % are herbicides, 30 % are insecticides, 18 % are fungicides, and 4 % are other pesticides (Sharma et al., 2019). These chemical compounds or mixtures are highly used to increase agricultural productivity, mainly; however, it is estimated that only 0.1 % of the applied pesticides reach the targeted pests, while 99.9 % remain long in the environment and can be bioaccumulated due to their characteristics such as long half-life and high lipophilicity (Sarker et al., 2021). Thus, they reach surface waters and groundwater through different sources, e.g. by chemical runoff during improper storage, loading, and disposal, as well as their misapplication; through their use in urban areas, air transport, runoff and erosion events, which facilitate the gradual leaching of these compounds into soil and water bodies (Peña et al., 2020; Vryzas, 2018). Among the most representative pesticides reported in water environments are herbicides such as atrazine, glyphosate, and 2,4-dichlorophenoxyacetic acid or simply 2,4-D (Chandra and Usha, 2021; Charles et al., 2019; Li et al., 2021a); insecticides such as endosulfan, chlorpyrifos, and carbofuran (Campbell et al., 2004; Jacob et al., 2020; Sathishkumar et al., 2021); and fungicides such as azoxystrobin and tebuconazole (Manjarres-López et al., 2021; Rodrigues et al., 2017; Syafrudin et al., 2021; Zubrod et al., 2019).

Another kind of EOPs are industrial chemicals and additives, which has emerged with the industrial development and has been detected recently in surface waters and groundwater, in comparison with pesticides and pharmaceuticals (Calvo-Flores et al., 2018). These compounds, as their name suggests, correspond to different chemicals used in any part of an industrial process or added to any product to provide it with particular properties. Thus, they are classified based on their function or some particular characteristic: antioxidants, flame retardants, plasticizers, dyes, polyaromatic hydrocarbons, surfactants, antifouling compounds, volatile organic compounds, gasoline additives, among others (Liu et al., 2019; Mukhopadhyay and Chakraborty, 2021; Pandey et al., 2021). An important example of these pollutants is bisphenol A or simply BPA, a compound used as raw material in the production of polycarbonates and epoxy resins plastics, as well as additive in different plastics due to its antioxidant and flame retardant properties (Plattard et al., 2021). Due to the high usage of BPA, its levels have increased in water environments around the world, reporting concentrations at ng/L level (Dupuis et al., 2012; Wang et

al., 2020b). Also, there are chlorinated BPA derivatives, which are products of the reaction between BPA and disinfectant products such as sodium hypochlorite (NaOCl) and chlorine, detected and reported in levels higher than 50 ng/L in industrial wastewater, sewage sludge, sediments, distribution pipes, and drinking water (Andra et al., 2015; Cantoni et al., 2021; Fukazawa et al., 2001). This is an important example of how interactions between a pollutant and the environment can result in even more harmful substances. Other industrial chemicals of emerging concerns are flame retardants, used as additives to avoid or diminish the further development of ignition, such as 3,3',5,5'-tetrabromobisphenol A, tris(2-chloroethyl phosphate), and some polychlorinated alkanes (Cristale et al., 2013; Liu et al., 2021; Yang et al., 2019); dyes, used widely in textile industry to provide colors to clothing, like methylene blue, acid blue 25, orange II, crystal violet, acid orange 7, and alizarin yellow (Hanafi and Sapawe, 2020; Zhou et al., 2021); and plasticizers, employed to impart flexibility and workability to plastics, such as di-(2-ethylhexyl) phthalate, diethyl phthalate, di-n-butyl phthalate, and benzyl butyl phthalate (Teuten et al., 2009; Zhang et al., 2018). On the other hand, some industrial chemicals such as surfactants, e.g., nonylphenol and octylphenol ethoxylates, and antifouling compounds, e.g., tributyltin and irgarol, have emerged as monitored pollutants in water environments due to the current increment of detergent industry and membrane technology, respectively (Kamei et al., 2020; Kortner et al., 2009; Vargas-Berrones et al., 2020).

Even in the process of treatment, water can be polluted. This is the case of water disinfection by-products as EOPs, which are formed in the treatment of polluted water by disinfectant agents like chlorine (Cl_2), ozone (O_3), chloramine (NH_2Cl), and UV radiation (Park et al., 2016). Among these, chlorine is known as one of the main disinfectant agents for water treatment since the identification of waterborne pathogens, being inexpensive and relatively easy to produce and use (Mazhar et al., 2020). Therefore, it is highly used for disinfection nowadays. However, it has been reported that chlorine interacts with different components of dissolved organic matter in polluted water, leading to the generation of various halogenated by-products with potential harmful to living organisms (Dong et al., 2021a). More specifically, when chlorine gas is put in contact with water, hypochlorous acid is formed ($\text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{H}^+ + \text{Cl}^-$), which is a powerful oxidant agent that kills pathogenic microbes present in the medium. In addition to this, and because of its non-specific action, hypochlorous acid also reacts, e.g., through oxidation, addition, and substitution reactions, with dissolved organic matter (fatty acids, humic acids, fulvic acids, amino acids, lipids, etc.), producing a high quantity of chlorinated by-products, e.g., chloroform, trichloroacetic acid, dichloroacetamide, etc (Alexandrou et al., 2018; Gilca et al., 2020). The same applies to other kinds of disinfectants, which through different action pathways lead to many EOPs such as carbonyl compounds, brominated compounds, and nitro compounds, from natural organic matter (Ding et al., 2019). Today, more than 600 water disinfection by-products have been reported, many of which pose an environmental hazard (Srivastav et al., 2020).

As mentioned early, microorganisms have been recognized as important water pollutants since the last century. Until now, they constitute a special class of EOPs because of their ability to generate resistance against disinfection treatments, their ability to mutate and adapt to changing conditions of the environment, their different pollution sources, and the lack of a definitive and completely effective treatment methodology for their removal and/or elimination from water media (Channa et al., 2021; Espinosa et al., 2020). Different pathogens are included in this category: bacteria, viruses, protozoa, and helminths. Some important examples are bacteria such as *Escherichia coli*, which can cause acute diarrhea, bloody diarrhea, and gastroenteritis; *Pseudomonas aeruginosa*, which can cause infections on lungs, urinary tract, and kidney; and *Helicobacter pylori*, which is precursor of chronic gastritis, ulcer disease, and gastric cancer (Deng et al., 2019). Also, some important waterborne viruses are hepatitis A and B viruses, as well as rotavirus capable of causing gastroenteritis. Likewise, protozoa like *Acanthamoeba spp.*, precursors of amoebic, meningoencephalitis, keratitis, and encephalitis, and helminths like *Dracunculus medinensis*, which can cause ulcerating skin infection, are of emerging concern (Bridle, 2014; Ramírez-Castillo et al., 2015). Although illness caused by waterborne pathogens have declined since the last century, it is estimated that more than 750 million people are directly affected by waterborne pathogens worldwide due to the lack of efficient water disinfection treatments (Pruden, 2014). Another type of natural-occurring water pollutant is biological toxins or simply biotoxins. These are biological molecules or metabolites produced by some living organisms as a defensive strategy, mainly. However, they can reach the water environment and, through it, enter the human body, causing different diseases and, in some cases, death (Alves et al., 2019; Joseph, 2017). For example, fungi can produce ribosome-inactivating proteins, which are capable of inhibiting RNA translation and cause cellular apoptosis (Dosio et al., 2011; Ng, 2004). Fungi, in conjunction with bacteria and other microorganisms, are present in different environments and materials, colonizing and producing biofilms (Espinosa et al., 2020), through which their toxins reach surface water and tap water. Likewise, seawater and freshwater are also polluted by biotoxins produced from algal bloom, i.e., cyanobacteria, dinoflagellates, and diatoms, which have potent neurotoxic and hepatotoxic effects, e.g., microcystins, endotoxins, nodularins, anatoxins, and saxitoxins (Liu et al., 2021b; Tran et al., 2020).

In addition, several pufferfish species (*Tetraodontidae* family) produce a potent neurotoxin called tetrodotoxin, which is capable of blocking voltage-gated sodium channels of nerve fibers, leading to weakness, paralysis and even death (Chen et al. 2016; Ramba-Alegre et al., 2017). In this way, a major reason for the increment of biotoxins in water media is nutrient discharges in many environments, which lead to an increased development of algal blooms and other toxin-producing organisms (Calvo-Flores et al., 2018).

For its part, nanotechnology has grown rapidly since the beginning of this century, being widely applied in many fields and products,

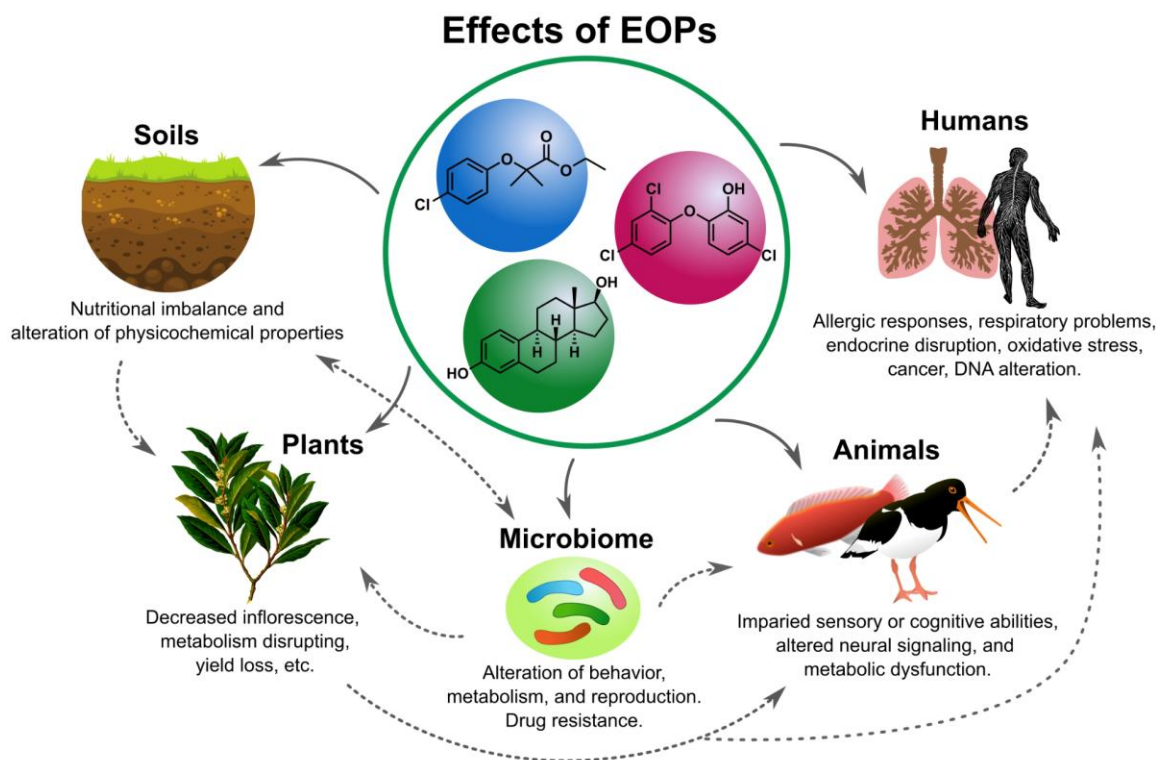
e.g., cosmetics, textiles, medicines, paintings, electronics, etc. In this category, fullerenes, carbon nanotubes, graphene, polymer nanoparticles (including plastics), composite materials, and natural organic particles (i.e., clays, organic matter, metal oxides, etc.) are included, which, depending on the case, have unique properties such as high surface area, high thermal and electrical conductivity, high surface reactivity, and photocatalytic properties (García-Quintero and Palencia, 2021; Turan et al., 2019). Due to their high usage, nanomaterials have become a water pollutant of emerging concern, reaching water bodies through different sources: wastewater effluents, direct discharges, accidental spillages, air transportation, and rainwater runoff (Klaine et al., 2008). An important example of this is plastic nanoparticles. They can be pre-formed and reach aquatic environments through several pathways, but also, they can be formed by the interaction of plastic materials with the environment, i.e., degradation of plastics by abiotic or biotic process (Canesi et al., 2015). In this way, these plastic nanoparticles interact with different aquatic organisms and cause adverse effects, e.g., oxidative stress, metabolism modification, affection of cellular function, and even cellular apoptosis (Peng et al., 2020). On the other hand, carbon nanomaterials such as fullerenes, carbon nanofibers, carbon nanotubes, graphene nanomaterials, and even their chlorinated derivatives, have been reported on water environments at ng/L levels (Alpatova et al., 2013; Sanchís et al.,

2018). Some harmful effects to living organisms like metabolic disturbance, respiratory system affections, and neurotoxic effects, have been observed for these materials (da Rocha et al., 2019; Shvedova and Kisin, 2008). Also, natural organic nanoparticles play an important role in nanomaterial pollution and represent a potential hazard for living beings. These have anthropogenic and natural pollution sources, e.g., organic matter degradation, volcanic activity, incineration, etc., and have been detected at mg/L level in groundwater and aquifers (Ermolin et al., 2018; Malakar et al., 2021).

3.2. Effects on environment and human health

The diverse structural characteristics and behavior of EOPs lead to different modes of action and effects in the environment and in the human body, which is an aspect of great interest in the current environmental research. More specifically, EOPs are distributed around the globe through many pollution sources, reaching different ecosystems and environments, i.e., rivers, oceans, soils, air, forest, etc., and interacting directly or indirectly with many species present there, e.g., plants, microorganisms, animals, and humans. These interactions result in different effects, which in most cases, if not all, are harmful or represent a danger to the development of life (see Figure 2) (Tang et al., 2019).

Figure 2. Effects of EOPs on environment and human health. It is important to realize the connection between different living organisms and ecosystems, indicated by dotted arrows, which results in indirect harmful effects caused by EOPs.



Firstly, it has been discussed that EOPs can contribute to climate change, altering temperature, salinity of water bodies, and natural phenomena like precipitation; which led to an environmental disturbance that can affect different ecosystems (Noyes et al., 2009). Secondly, it has been reported that EOPs can affect the physicochemical characteristics of soils such as pH, soil aggregation, bulk density, and water holding capacity (de Souza Machado, 2018; Wan et al., 2019). Also, they can cause imbalance in the nutrition of soils and, in this way, affect the growth of plants (Lozano and Rillig, 2020; McGinnis et al., 2019). Plants can uptake EOPs from the soil through their roots, allowing them to enter in important metabolic pathways and interact with key enzymes and metabolites, which results in harmful effects such as decreased inflorescence, yield loss, reduced production of secondary metabolites, vitamin deficiencies, among others (Bouaicha and Corbel, 2016; Haq and Kalamdhad, 2021). Once EOPs reach soils and water bodies, they can interact with their microbiome, affecting them at different degrees. For example, EOPs can alter the behavior of microorganisms, their metabolism, and their reproduction. Furthermore, an important effect on microorganisms is the increase in drug resistance by the continuous exposure to EOPs, which indirectly affects humans in the treatment of different microbial diseases (Cerqueira et al., 2020; Gomes et al., 2020). In animals, EOPs generate notable changes in behavior, e.g., reproduction, animal movement, predation-avoidance, aggression and grouping, mainly due to physiological alterations such as impaired sensory or cognitive abilities, altered endocrine/neural signaling, and metabolic dysfunction (Saaristo et al., 2018). From a biochemical point of view, EOPs can increase or diminish the hormonal activity in animals, or in the worst case, they block the hormonal action by competing with the hormone receptor, mimicking or impersonating the endogenous hormones. This results in behavior alterations since hormones participate in the control of reproduction, sexual differentiation, organ coordination, brain organization, among others (Arguello-Pérez et al., 2019; Jacquín et al., 2020). Likewise, the alteration of the behavior of an animal can indirectly affect other animals linked to the same food chain or within the same ecosystem (Brodin et al., 2014; Fleege et al., 2003). In addition, serious conditions such as cancer and teratogenicity in animals due to the exposure to EOPs have been reported (Amoatey and Baawain, 2019; Pesavento et al., 2018). Finally, EOPs reach humans by the already mentioned pollution routes, i.e., water, air, and soils, but also by bioaccumulation of these pollutants in the food chain (Amutova et al., 2021). In the human body, EOPs can cause a diversity of affections depending on the degree and time of exposure. For example, they produce a series of allergic responses, e.g., skin and

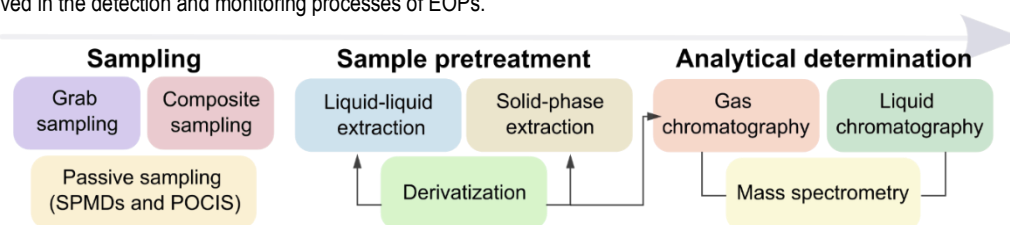
ocular irritation, and acute effects with direct actions in the central nervous system (Enyoh et al., 2020). Some symptoms associated with EOPs toxicity are vomiting, dizziness, breathing difficulties, anxiety, depression, muscle ache, and headache (Bhavaya et al., 2021). More critical effects caused by EOPs are cardiovascular problems, diabetes, endocrine disruption, infertility, oxidative stress, immune suppression, liver dysfunction, and respiratory problems (Alharbi et al., 2018; Baderna et al., 2013). Moreover, long exposure to EOPs lead to cancer and even DNA alterations (Lei et al., 2015; Liu et al., 2021c).

Ecotoxicological analysis of EOPs and their effects on human health remains an important research topic due to the limited knowledge obtained until now and the difficulties associated with measuring defects caused by EOPs. Some aspects such as metabolic pathways, interaction sites, toxicity levels, and synergistic effects are of emerging interest. Likewise, according to the available literature and the reported effects of EOPs it is important to realize that it is necessary to focus on the development of removal methodologies and the regulation of the use and disposal of these organic substances.

4. Detection and monitoring methodologies

Detecting and monitoring EOPs in the environment involves a series of carefully-prepared steps through which it is possible to ensure a correct determination of the pollutant level in the medium, avoiding and/or eliminating possible bias or errors in the analysis due to the presence of interferences, matrix effects, or instrumental failures. Ideally, these procedures should be as environmentally friendly as possible and at a low economic and energetic cost, however, without compromising reliable results. In this point, it is important to realize that because of the diversity of characteristics and properties of EOPs, i.e., chemical state, acidity, polarity, reactivity, solubility, etc., and the complex mixtures of these pollutants in different water environments, it is almost impossible to develop an analytical method that ensures the detection of all well-known organic pollutants. For this, different methodological processes and techniques for the determination of each class of EOPs are needed. In a general sense, each detection methodology focused on EOPs in water samples requires, after sampling, a sample pretreatment, which involves extracting, concentrating, and cleaning up the sample with the targeted EOPs. After that, it is necessary to select a proper analytical method by which the pollutants are separated, detected, and quantified (see Figure 3) (Hartmann et al., 2020; Tang et al., 2019).

Figure 3. Main steps involved in the detection and monitoring processes of EOPs.



4.1. Sampling

Before EOPs detection, it is necessary to define the best way to take the sample, in this case, from water environments; including the consideration of aspects such as geographic distribution, sampling frequency, weather, matrix, and sample quantity, which are selected based on the type of analysis and the pollutants to be measured (Geissen et al., 2015). In this way, sampling is one of the primary steps of every monitoring program focused on EOPs and it could be achieved through different methods and plans. Firstly, direct sampling using the hand is the most commonly used and easiest method. It is based on taking the sample directly from the water stream in a particular container using the hand. It has been suggested that sample containers or sampling equipment should be made from aluminum, stainless steel, fluorocarbon polymers, and glass, since other materials like polyethylene, rubber, or other plastics, can absorb or desorb targeted EOPs from/into the sample (Alvares and Jones-Lepp, 2010). This sampling method applies not only to take grab or single samples, but also to take composite samples, which are representative of a defined profile of the water stream (Metcalf et al., 2014). When it is necessary to take samples from deeper waters, submersible bailers or thief samplers are commonly used. These are based on a bottle or tube that serves as a water collector through caps or check valves (Llamas et al., 2020). Moreover, sampling from groundwater requires the utilization of portable peristaltic pumps to draw the water sample from deeper monitoring or supply wells and collect it into a particular vessel. Also, automated sampler devices are used to take samples from surface waters and groundwater when the sampling is required at predetermined time intervals or the presence of water may be intermittent. Some sophisticated automated sampling devices have the possibility of taking a sample or multiple samples, collecting them into refrigerated container vessels, and being programmed via land-line or cellular phone connections (Erikson et al., 2013; Mucciarone and Dunbar, 2020).

On the other hand, passive sampling devices (PSDs), or also time-integrated sampling devices, are an alternative to grab sampling techniques for surface waters and groundwaters when it is necessary to detect and quantify organic pollutants at very low concentrations, since grab sampling usually require large sample volumes for this and the results are generally not satisfactory (Hawthorne et al., 2009). PSDs are used to collect a pollutant sample at a predetermined interval of time by means of pollutant retention processes, which means that the pollutant concentration results to be integrated over the sampling time. Thus, PSDs are very useful to determine levels of exposure of aquatic organisms to EOPs in a certain period of time (Garces et al., 2018; Silvani et al., 2017). In general, PSDs are constructed similarly and their operation is the same: they have a phase, called the receiving phase, where the analytes are retained through a diffusion process induced by the pollutant concentration gradient established between the external phase (i.e., water environment) and the receiving phase. The rate of chemical uptake and the selectivity of the transport process of the analytes are regulated by a membrane, called the limiting phase (Godlewska et al., 2020). In this sense, different PSDs have been

designed and highly implemented to detect EOPs, among which semipermeable membrane devices (SPMDs) and polar organic chemical integrative samplers (POCIS) have been extensively explored (Harman et al., 2008). SPMDs are based on a lay-flat low density polyethylene membrane tube containing a small volume of a neutral lipid such as triolein, which serves as a receiving phase for hydrophobic EOPs (i.e., chemicals with moderate to high octanol-to-water partition coefficients, $\log K_{ow} > 3$) (Kim et al., 2014). For example, SPMDs have been utilized to measure low concentrations of alkylphenols, polychlorinated biphenyls, polybrominated diphenyl ethers, and other polycyclic aromatic hydrocarbons (Lima et al., 2019; Miège et al., 2012; Smedes, 2019). For its part, POCIS are fabricated using a sorbent or mixture of sorbents, which serve as the receiving phase, sandwiched between two sheets of a microporous polyethersulfone membrane. Initially, POCIS were fabricated using poly(divinylbenzene)-co-*N*-vinylpyrrolidone as the receiving phase, which allowed the retention of polar EOPs, or in other words, those pollutants with $\log K_{ow} < 3$ (Harman et al., 2012). However, new receiving phases have been designed through time to expand the possibility of POCIS to measure more EOPs, even those with hydrophobic nature. Some examples of these sorbents are Strata-X, Oasis MAX, Chromabond HRX, Strata XAW, and Strata X-CW, which are finely divided hydrophobic or hydrophilic beads made from different functionalized polymers (Godlewska et al., 2021a). Also, molecularly imprinted polymers, ionic liquids, and carbon nanotubes have been used as the receiving phase (Feng et al., 2019; Godlewska et al., 2021b; Wang et al., 2017). POCIS have been used to measure levels of a multitude of EOPs in water environments, including mainly pharmaceuticals, pesticides, and hormones (Criquet et al., 2017; Cristóvão et al., 2021; Gong et al., 2018). Importantly, the parts of these samplers can be modified, e.g., the type of membrane, receiving organic phase, or sorbent, and be used in conjunction with the aim of retaining and measuring a greater quantity of EOPs at the same time, obtaining higher efficiencies, better cost-benefit relationships, and simplified analysis (Esteve-Turrillas et al., 2007; Godlewska et al., 2020). However, these present some disadvantages such as complicated calibrations and the difficulty of assessing whether equilibrium has been reached or not due to variability of parameters such as temperature, water flow, and biofouling (Harman et al., 2012). Additionally, other types of PSDs such as Chemcatchers, polyethylene strips, polymers on glass, and solid-phase microextraction devices, have also been developed and used in water sampling (Alvarez and Jones-Lepp, 2010).

4.2. Sample pretreatment

After sampling, it is necessary to carry out a sample pretreatment focused on preparing the sample for further analysis, i.e., the analytical determination of EOPs in the sample. This pretreatment typically involves clean-up, extraction, and pre-concentration processes of the sample to be analyzed. First, cleaning-up the sample requires the elimination of possible interferences or unwanted material in the sample matrix such as undissolved organic matter and/or other microscopic materials, metal ions, oxygen, and

other chemical interferences, through methodologies like filtration, centrifugation, flocculation/coagulation, pH-adjustments, among others (Cerutti et al., 2019). After this, extraction and preconcentration methodologies are required to generate an analytically-detectable concentration of EOPs in the sample; however, this is not necessary in the cases where PSDs have been used as sampling techniques, since through their operation, these devices already extract and concentrate EOPs from the water environment and these can be released later in a relatively easy way (Castro et al., 2021). In this sense, extraction and preconcentration techniques like solid-phase extraction and liquid-liquid extraction are the most commonly used when dealing with EOPs in water environments (Tsai et al., 2021). However, in recent years innovative sample pretreatment techniques such as microwave assisted extraction, pressurized liquid extraction, stir bar sorptive extraction method, among others, have been developed and utilized for sample pretreatment in the monitoring of EOPs (Sanchez-Prado et al., 2015; Subedi et al., 2015). Next, some of these extraction techniques will be discussed.

4.2.1. Liquid-liquid extraction (LLE) techniques

As its name suggests, LLE is based on the partitioning of a compound between two liquid phases, in this case, an aqueous phase (the water sample) and an immiscible organic phase (or the extraction phase). Usually, the employed organic phase is ethyl acetate, hexane, isooctane, toluene, chloroform, or methylcyclohexane (Matamoros et al., 2012). However, supercritical fluids and superheated liquids have also been used as the extraction phase (Leazer et al., 2009; Maciel et al., 2018). This technique has been used for decades for the extraction of apolar organic pollutants from water samples, such that the extraction process is well-known and overstudied. The physicochemical characteristic of the compound(s) to be extracted, e.g., polarity, pKa, log K_{ow}s, etc.; the type of organic solvent, water:organic solvent ratio, the volume of each phase, and the number of extractions directly affect extraction efficiency and yield, as well as parameters like pH and ionic strength. These last two can be useful to enhance the extraction efficiency through the displacement of the partition equilibrium. It means that, at a certain pH and a generally greater ionic strength value, neutral and apolar molecules prefer to be in the apolar organic phase (Gezahegn et al., 2019).

At first instance, the use of a hydrophobic extraction phase seems to be a limitation of LLE associated with the extraction restricted to apolar compounds only; however, different chemical derivatization methodologies have been successfully proposed to overcome this, not just for enabling the extraction of polar organic pollutants, but also as a necessary treatment prior to their analytical determination, e.g., by gas chromatography (Manca et al., 2017). In these methodologies, different derivatization reagents react in some way (for example, through alkylation, acylation, or silylation reactions) with the targeted compounds with the aim to modify their properties, in this case, to convert them into more apolar (and volatile) compounds. Thus, the new derivative compound can be extracted and quantified properly (Baghdady and Schug, 2016).

Some particular examples of derivatization reagents include *N*-methyl-*N*-(tert-butyldimethylsilyl), trifluoroacetamide, bis(trimethylsilyl) trifluoroacetamide, *N*-methyl-*N*-(trimethylsilyl) trifluoroacetamide, trifluoroacetic acid, diazomethane, phenazine methosulfate, dansyl chloride, *N,N*-diethyldithiocarbamate, isobutyl chloroformate, and acetic anhydride (Alvares and Jones-Lepp, 2010; Carro et al., 2013; Jain and Verma, 2018; Wang et al., 2019a). Also, derivatization agents which provide some particular properties, like fluorescence, to the analyte have been used, e.g., fluorescamine. This is particularly useful when the employed analytical technique poses a fluorescence detector (El-Yazbi et al., 2019). Other important methodologies focused on enhancing the extraction efficiency and capability are ion-pairing, complexation, phase transfer catalysis, and nanoparticle-assisted extraction (Basheer et al., 2019).

According to the above, classic LLE has been applied for decades to the extraction of a high variety of EOPs such as pharmaceuticals, including antibiotics, analgesics, anti-inflammatories, cardiovascular agents, and anxiolytics; hormones, e.g., 17- β -estradiol, 17- α -ethinylestradiol, and estrone; pesticides such as chlorpyrifos and atrazine; and polyhalogenated aromatic hydrocarbons (Borrull et al., 2020; Miossec et al., 2020; Wang et al., 2013). Nevertheless, due to the main drawbacks of LLE like the high consumption of time and the use of large volumes of toxic organic solvents, which results in a laborious procedure and, in many cases, an expensive and contaminating extraction technique, some derivative LLE techniques have been developed. Among these, liquid phase microextraction techniques such as dispersive liquid-liquid microextraction (DLLME), single drop microextraction (SDME), and hollow-fiber liquid-phase microextraction (HF-LPME) are included (Carasek et al., 2018).

4.2.1.1. Liquid-phase microextraction

First, DLLME is a type of LLE technique at a miniaturized level (microliter level) based on the dispersion of the extraction phase into the aqueous phase, where the targeted EOPs are found. Additionally, it may require a dispenser solvent, which is mixed with the extraction solvent and facilitates the dispersion process; and the extraction phase needs to have a high-density and apolar nature, which allows its dispersion in water and its recovery easily by centrifugation at the end of the process (Quigley et al., 2016). This arrangement provides certain advantages over conventional LLE such as fast extraction and low costs, due to the high mass transfer of apolar EOPs from the aqueous phase to the finely dispersed organic phase and the low quantity of solvent volumes employed in the process, respectively (Li and Ding, 2021). On the other hand, SDME is based on the introduction of a drop of extraction phase into the aqueous phase employing a microsyringe. The utilization of an organic solvent drop enhances the mass transfer process and, consequently, the extraction rate due to a greater surface area-to-volume ratio of the extraction phase, resulting in "a minimalist technique capable of providing excellent analytical data" as Tang and co-workers (2018a) mentioned. Some advantages of SDME are low instrumental requirements, simplified operation,

non-complex setup, fast extraction, and the employment of low quantities of organic solvents. However, it has an inherent and important drawback associated with drop instability, which led to the option of considering other microextraction techniques (Jain and Verma, 2011; Ye et al., 2007). For its part, in HF-LPME a hydrophobic hollow fiber is used as the container of a small amount of the extraction phase. More specifically, the extraction phase is injected into the lumen and pores of the hollow fiber, which is in contact with the aqueous phase or the sample, such that the targeted EOPs can diffuse through the pores by means of a partition process between the two phases (Kraševac and Prosen, 2021). With HF-LPME it is possible to obtain high enrichment and selectivity factors, ensuring excellent pollutant concentration and sample clean-up (Khan et al., 2020). Also, simplicity and low costs are linked to this technique (Savatierra-stamp et al., 2018). However, some drawbacks are generally slow extraction, the hollow fiber needs to be replaced after each extraction, and there is a lack of commercially available equipment (Abdel-Rehim et al., 2020; Gjstad and Pedersen-Bjergaard, 2013). Although the above-mentioned inconvenients and/or disadvantages, these microextraction techniques have represented an important tool in EOPs monitoring, thus, through them a high variety of EOPs have been extracted even at pg/mL level, for example, industrial additives such as BPA and its derivatives; polybrominated diphenyl ethers, phthalate esters, hormones, preservatives, alkylphenols, pesticides, and pharmaceutical compounds (Li et al., 2021b; Primel et al., 2017; Sereshti et al., 2021; Wang et al., 2019b). In addition, some important advances and innovative alternatives in the design and use of these liquid-phase microlevel extraction technologies have been reported, e.g., the implementation of ionic liquids as the extraction phase, which provides a reliable, environmentally-friendly and efficient approach of the extraction process (Rivera-Vera et al., 2019); the possibility of automation by implementation of syringe-type extraction containers (Senovieski et al., 2020); and the employment of ultrasonic radiation to significantly accelerate the mass transfer process, which has allowed the establishment of techniques such as ultrasound-assisted DLLME and ultrasound-assisted emulsification microextraction (Regueiro et al., 2008; Pérez-Outeiral et al., 2016). For more details, some reviews focused only on liquid-phase microextraction techniques have been published in recent years (Carasek et al., 2018; Hashemi et al., 2017; Rutkowska et al., 2019).

4.2.2. Solid-phase extraction (SPE) techniques

SPE is the most used extraction and concentration technique applied to thousands of inorganic and organic compounds, including EOPs monitoring (Hashemi et al., 2018). In contrast to LLE and, as its name suggests, SPE techniques are based on an extraction process mediated by a solid phase, also called the sorbent. The usage of solid sorbents for EOPs extraction and clean-up provides some important advantages such as higher enrichment factors, fast procedures, and lower consumption of organic solvents, in comparison with LLE techniques. In terms of its general operation, SPE techniques work

in three main steps: (i) conditioning the solid sorbent, which is found inside a cartridge, by passing a particular solvent through it to increase the effective surface area and to reduce possible interferences; (ii) loading the liquid sample through the sorbent by either gravity, vacuum-induced, or syringe-push flow, to promote pollutant retention by direct physical or chemical interactions between the targeted EOPs and the sorbent; and finally, (iii) washing the sorbent with a proper solvent or mixture of solvents to recover the retained EOPs (Ca et al., 2009; Filik et al., 2012). This principle of operation and the employed setup in SPE techniques have allowed the design and establishment of automated devices, which is a relevant advantage over LLE methodologies (Calderilla et al., 2018; Domínguez et al., 2016). In this way, SPE has been studied and explored extensively, such that different solid sorbents are already commercially available and they can be found in different configurations: thin flat discs, small cylindrical cartridges, and multi-well plates (Kyle, 2017). Additionally, sorbents are usually made from alkyl-modified silica, e.g., octadecyl-bonded silica, octyl-bonded silica, butyl-dimethyl bonded silica, phenyl-bonded silica, etc.; or cross-linked copolymers, which comprise polystyrene divinylbenzene-based copolymers and functionalized hydrophobic polymers with polar and/or ionizable groups (Andrade-Eiroa et al., 2016). Alkyl-modified silica sorbents are employed as reverse phase solid-phase extractants, and through them, it has been possible to extract and concentrate a multitude of hydrophobic EOPs (Martín et al., 2017). However, these silica-based sorbents have some important disadvantages, such as instability at extreme pH, low recovery when dealing with polar EOPs, and the presence of residual silanol groups capable of interacting or reacting with the analytes, adsorbing them irreversibly (Telepchak et al., 2004). For this reason, relevant alternatives such as polymeric sorbents have emerged and been explored extensively. Polymeric SPE sorbents, as Fontanals and co-workers (2019) established, “combine outstanding morphological properties that promote capacity and retention with tuned chemical properties that allow suitable interactions with many types of compounds and show enhanced stability under several SPE conditions”. In this way, pollutant-polymer interactions include Van der Waals forces, π - π interactions, dipole-dipole interactions, electrostatic forces, hydrogen bonds, and, more recently, dynamic covalent bonds (Drzymala et al., 2015; Faraji et al., 2019; Yang and Zhang, 2012). Among polymeric SPE sorbents, commercially available hydrophilic-lipophilic balanced polymeric phases, also known as HLB polymeric phases, and in-house polymeric phases are highlighted. These can be made either from the copolymerization of polystyrene divinylbenzene with another class of polymers, e.g., polyvinyl pyrrolidone, poly(4-vinylpyridine), poly(methacrylic acid), etc., or the chemical modification of a hydrophobic polymer matrix with polar or ionic groups such as amine, nitro, sulfonic, acetyl, and carboxyl groups (Andrade-Eiroa et al., 2016; Bratkowska et al., 2010; Buszewski and Szultka, 2012). Due to their chemical diversity, high stability, wide range of supported working conditions, high efficiency and capability, polymeric sorbents have been used extensively for the extraction of a large number of EOPs with different physicochemical properties, as a pretreatment step in

monitoring programs focused on pharmaceuticals (Huang et al., 2017; Wang et al., 2020c), flame retardants and industrial additives (Kaziur-Cegla et al., 2020; Zhong et al., 2019), polyaromatic hydrocarbons (Thomsen et al., 2007), pesticides (Corcellas et al., 2013), among others (Płotka-Wasyłka et al., 2017).

In this same sense, molecularly imprinted polymers, or MIPs, have arisen in recent years as an important alternative in SPE through which high extraction selectivity can be achieved, keeping the above-mentioned advantages provided by polymeric structures (Beltran et al., 2010a). The high selectivity of MIPs sorbents is provided by the specific cavities in their structures, which allows the retention of molecules with a particular shape and the capability of interaction with the chemical groups of the sorbent's surface, whereas the other compounds can pass through them without any significant retention. Besides, as many other types of polymers, MIPs can be obtained in a relatively simple way and processed in different arrangements, obtaining suitable devices for the extraction and monitoring of specific pollutants from/in water samples (Bhagal et al., 2021; Dong et al., 2021b). For example, MIPs sorbents have been used for the extraction of emerging aminoglycosides antibiotics (Zhang et al., 2020b), bisphenol A (Karrat and Amine, 2021), diclofenac (Cantarella et al., 2019), ketoprofen (Zunngu et al., 2017), parabens (Beltran et al., 2010b), clofibric acid (Duan et al., 2013), atrazine (Zarejousheghani et al., 2014), among other EOPs (Liu et al., 2021; Wang et al., 2019c).

Currently, there is a need for advancing in the development of SPE sorbents with more efficiency, capability, and even versatility, such that, it has turned the attention to the consideration of other types of promising materials. For example, carbon-based (nano)materials such as carbon nanotubes, fullerenes, graphene, and graphene oxide, which have excellent properties like high specific surface areas, high mechanical strength, and thermal conductivity (Arcoleo et al., 2020; Muñoz et al., 2004; Ruiz et al., 2019). In addition, they can participate actively in redox processes, which allows them to be highly used in electrochemical SPE techniques (Hatamluyi and Es'haghi, 2017; Shamsayei et al., 2016). Likewise, there is a tendency for using organic-inorganic hybrid materials, such as metal-organic frameworks, which have a nanostructured network useful to pollutant removal, high specific surface area, and good thermal and chemical stability (Composite materials, e.g., polymers-graphene oxide, polymers-organic frameworks, among others, are also of emerging interest (Gao et al., 2021; Liu et al., 2018). Undoubtedly, bio-derived materials are a current research topic in materials science and analytical chemistry, since they represent an environmentally-friendly solution to the employment of petroleum-derived materials. Thus, for example, some researchers have employed chitosan, agarose, alginate, and cellulose as SPE sorbents, obtaining positive results in terms of extraction efficiency (Pacheco-Fernández et al., 2020).

Through time, different SPE techniques and modifications of the classic SPE have been proposed with the aim to satisfy or accomplish various requirements or drawbacks in terms of extraction methodologies like sample matrix, equipment, recovery, selectivity, efficiency, etc. Among the most relevant are dispersive solid-phase extraction (DSPE) and stir-bar sorptive extraction

(SBSE) (Faraji et al., 2019). For its part, DSPE involves the dispersion of a SPE sorbent into a liquid sample, without the employment of any sophisticated setup. After the extraction process is completed, the sorbent is recovered from the solution using centrifugation and/or filtration. The main advantage of DSPE over other extraction techniques is the reduction of extraction time that allows more samples to be analyzed in shorter periods of time. Also, DSPE results to be cheap, simple, rugged, and safe (Islas et al., 2017). However, its main drawback is the impossibility to change solvent between the extraction and preconcentration steps and, as in other SPE methodologies, it is necessary to select a proper sorbent to extract the targeted compounds (Chisvert et al., 2019; Walorczyk et al., 2015). Although this, DSPE have been used for extracting different EOPs, such as estrogens, steroids, β -blockers, industrial additives, acidic pharmaceuticals, antidepressant drugs, and antibiotics (Celano et al., 2014; Gao et al., 2019; Sajid et al., 2021; Wang et al., 2016).

On the other hand, SBSE employs a glass magnetic stirrer (typically, 1.5 cm long) coated with polydimethylsiloxane for extracting apolar EOPs from aqueous samples by their stirring for a determined period of time. The extraction process is mediated by a partition equilibrium of the pollutants between the aqueous phase and the polydimethylsiloxane coating. After completing extraction, processes like thermal desorption or liquid desorption can be utilized for the recovery of the pollutants (Zheng et al., 2020). SBSE has important characteristics such as the possibility of employing low volumes of sample, simplicity, fast extraction, and high efficiency (Camino-Sánchez et al., 2014). Thus, it has been used for extracting polycyclic aromatic hydrocarbons, polychlorinated biphenyls, pesticides, hormones, pharmaceuticals, and personal care products (Bratkowska et al., 2011; Murrell and Dorman, 2021; Pintado-Herrera et al., 2014). However, the utilization of a polydimethylsiloxane coating as the extraction phase limits the recovery of EOPs to apolar compounds. In this sense, some alternative polar extraction phases have been postulated, but they have not been sufficiently accepted because they are mostly not compatible with pollutant recovery methodologies and/or have inferior performance characteristics compared to polydimethylsiloxane (Gilart et al., 2014; Ochiai et al., 2018). Until now, SPE techniques are an active research topic, such that innovative setups, configurations, modifications or derived techniques are constantly postulated (Benedé et al., 2018; Gamonchuang and Burakham, 2021).

4.2.2.1. Solid-phase microextraction (SPME) techniques

As in the case of LLE techniques, SPE has also been developed at microlevel, which involves the usage of low volumes of sample and extracting solid phase (in the order of μL), and results in a simple, cheap, versatile, and easy-to-automate extraction technique (Spitelun et al., 2013). As in SPE, SPME techniques utilize a solid sorbent to extract pollutants, usually made from fused silica fibers coated with a polymer layer. This polymer layer serves as an adsorbent and, depending on its properties, it is used to extract analytes with particular physicochemical properties (Riboni et al.,

2021). Additionally, SPME techniques are varied in the shape of support, configurations and operation modes. For example, SPME can be classified as static or dynamic. Static SPME techniques, such as thin-film SPME, fiber SPME, and rotating disk sorptive microextraction, carry out the extraction process using a constant stirring until it is completed. On the other hand, in dynamic SPME techniques, such as in-tube SPME, in-tip SPME, in-needle SPME, and capillary SPME, the extraction process is carried out by the dynamic elution of the sample through the solid sorbent (Portillo-Castillo et al., 2018; Vas and Vékey, 2004). Also, depending on their modes of extraction, SPME techniques can be divided into headspace SPME, direct-immersion SPME, and membrane-protected SPME (Zacharis and Tzanavaras, 2020). Likewise, techniques like DSPE and SBSE have also been developed to work at low volumes of sample and sorbents (Benedé et al., 2018; Senovieski et al., 2020). Thus, SPME has been established as an effective and reliable extraction methodology, satisfying important characteristics like eco-friendly operation, low costs, and high efficiencies, which result in essential advantages over other pollutant extraction methods, such that, SPME is one of the most used extraction techniques currently. Among its applications, the extraction of pharmaceuticals, pesticides, personal care products, among other EOPs, from water samples are highlighted (da Silva Sousa et al., 2021; González-Hernández et al., 2021; Kraševac and Prosen, 2021; Naccarato et al., 2021; Zhang et al., 2017). Some drawbacks of SPME include the limited effectiveness of already-developed sorbents and the facility to desorb the targeted compounds once their extraction has been completed (Dimpe and Nomngongo, 2016; Spietelun et al., 2013). However, great progress has been achieved in this topic through the employment of microwaves and ultrasound to increase the extraction and desorption rates/effectivities (Albero et al., 2018; Naccarato et al.,

2021; Zhang et al., 2017), as well as the development of more effective adsorbents, including versatile and functional nanomaterials (Feng et al., 2021; Jagirani and Soyak, 2020; Riboni et al., 2021).

4.3. Analytical techniques for EOPs detection and monitoring in water samples

After carrying out a proper sampling and the respective sample pretreatment, it is time to detect and quantify the targeted EOPs. As mentioned earlier, it is not possible to develop a single analytical method that ensures the detection and quantification of all reported EOPs in a single sample. In this sense, it is necessary to utilize different analytical and spectrometric techniques for, first, separating the EOPs in the sample and, then, detecting and quantifying them. For this, chromatography techniques, such as high-performance liquid chromatography (HPLC) or its variant ultra-high performance liquid chromatography (UHPLC), and gas chromatography (GC), are implemented (Borrull et al., 2020; Riboni et al., 2020). These can have ultraviolet-visible (UV-Vis), fluorescence, or diode-array detectors, but also, they can be coupled to mass spectrometry (MS), which has the capability (and the advantage over other detectors) to measure EOPs even at trace concentrations (Comtois-Marotte et al., 2017). However, a few alternatives, but less implemented, techniques have been utilized recently to detect and quantify EOPs, e.g., Raman spectroscopy and Fourier transform infrared (FTIR) spectroscopy. Table 2 illustrates summarized protocols and techniques employed in some relevant and recent works focused on EOPs detection and monitoring in water environments. Next, each methodology will be discussed in a deeper way.

Table 2. Some relevant detection and monitoring protocols of EOPs in water environments.

Type of sampling	Extraction and preconcentration technique	Instrumental analysis	Measured EOPs	Type of water sample	Reference
GS	-	UHPLC/MS-MS	Illicit drugs (cocaine, amphetamine, methamphetamine, etc.), pharmaceuticals (lincomycin, trimethoprim, sulfamethoxazole, ketoprofen, etc.), and caffeine	Wastewater	Di Marcantonio et al. (2021)
GS	SPE	HPLC/MS-MS	Pharmaceuticals (sulphonamides, tetracyclines, fluoroquinolones, β -blockers, anti-inflammatory drugs, and amphenicols)	Surface water	Kazakova et al. (2021)
GS	SPE	UHPLC/MS-MS	Chiral pharmaceuticals (atenolol, propranolol, metoprolol, venlafaxine, fluoxetine, O-desmethylvenlafaxine, among others)	Surface water	Ma et al. (2020a)
PS	-	UHPLC/MS (Q-ToF)	Pesticides (atrazine, diuron, carbendazim, hexazinone, dichlorobenzamide, among others) and pharmaceuticals (diclofenac, carbamazepine, sulfamethoxazole, lidocaine,	Groundwater and stormwater runoff	Pinasseau et al. (2019)

			among others)		
GS	SPE	LC/MS	Pharmaceuticals and illicit drugs (cocaine, amphetamine, fluoxetine, citalopram, mephedrone, ketamine, methylone, etc.)	Drinking water	Peng et al. (2019)
GS	SPE	GS/MS and HPLC/MS	A wide array of EOPs: pharmaceutical compounds (carbamazepine, ibuprofen, diclofenac, hydrochlorothiazide, trimethoprim, etc.), personal care compounds (triclosan, triclocarban, celestolide, galaxolide, tonalide, etc.), and organophosphate flame retardants and plasticizers (tri(<i>n</i> -butyl) phosphate and triphenyl phosphate)	Groundwater and surface water	Llamas-Dios et al. (2021)
GS	SPE	UHPLC/MS	Different EOPs, including pesticides, pharmaceuticals, natural substances, personal care products, industrial chemicals and additives, hormones, etc.	Wastewater	Qian et al. (2021)
GS + CS	SPE	UHPLC/MS-MS	More than 50 EOPs, including antibiotics, biocides, anti-inflammatory/analgesic drugs, antiepileptic drugs, lipid regulators, and caffeine.	Wastewater	Yang et al. (2017b)
GS	SPME	UHPLC/MS-MS and GS/MS	Biocides (dibenzofuran, diazinone, metolachlor, otcizer, etc.), personal care compounds (oxybenzone, galaxolide, avobenzene, octinoxate, etc.), pharmaceuticals (benzyl benzoate, doconexent, phenylephrine, etc.), industrial additives and chemicals (diethylene glycol dibenzoate, diisooctyl phthalate, 4-tert-octylphenol, styrene, caprolactam, etc.) among others.	Surface water	Wooding et al. (2017)
GS	SPME	GC/MS	Some UV-filters and polycyclic musk compounds (octocrylene, caffeine, benzophenone-3, ethylhexyl methoxycinnamate, among others)	Wastewater and surface water	Moeder et al. (2010)
GS	SPE	HPLC/MS	A few pharmaceutical and personal care compounds (acetaminophen, cotinine, fluoxetine, norgestrel, diethylstilbestrol, progesterone, etc.)	Wastewater	Hedgespeth et al. (2012)
PS	-	UHPLC/MS	Pesticides (acetamiprid, atrazine, ancymidol, azoxystrobin, bromacil, carbofuran, diazinon, fipronil, etc.)	Surface water	Taylor et al. (2021)
GS	-	UHPLC/MS	Pesticides and their transformation products (atrazine, metolachlor oxanilic acid, metolachlor sulfonic acid, chloridazon, desphenyl chloridazon, among others)	Surface water	Warner et al. (2021)
CS	SPE	UHPLC/MS-MS	Illicit drugs and their metabolites (methamphetamine, amphetamine, morphine, codeine, 6-monoacetylmorphine, benzoylecgonine, cocaine, ketamine, etc.)	Wastewater	Wang, et al. (2021)
CS	SPE	LC/MS-MS	Illicit drugs (amphetamine, methamphetamine, 3,4-methylenedioxymethamphetamine, cocaine, and cannabis)	Wastewater	Mercan et al. (2019)

CS	SPE	LC/MS-MS	Illicit drugs and their metabolites (cocaine, amphetamine, ephedrine, diazepam, alprazolam, LSD, morphine, etc.)	Wastewater	Mastroianni et al. (2017)
CS	SPE	HPLC	Industrial additives (di-tert-butylphenol, irganox 1010, irganox 1076, ethanox 330 and cyanox 1790)	Wastewater	Hernández-Fernandez and Rodríguez (2019)
GS	SPE	HPLC/MS-MS	Biocides and their metabolites (carbendazim, diuron, diuron-desmonomethyl iodocarb, dichloro-isothiazolinone, etc.)	Run-off water	Bester and Lamani (2010)
GS	SPE	LC/MS	A high quantity of EOPs, including biocides, pharmaceuticals, personal care compounds, industrial additives and chemicals, and even unregistered EOPs.	Groundwater	Ter Laak et al. (2012)
GS	-	HPLC and FTIR	Some representative pharmaceuticals and pesticides (desloratadine, paracetamol, ibuprofen, β -estradiol, ethynylestradiol, carbamazepine, sulfamethoxazole and atrazine)	Wastewater	Quintelas et al. (2020)
GS	SPE	GC/MS-MS	Bisphenol A, bisphenol F, and their respective glycidyl ethers	Tap water, surface water, and snow water	Jiao et al. (2012)
PS	-	GC/MS	Alkylphenols (phenol, 2-ethylphenol, 2-isopropylphenol, 2-phenylphenol, octylphenol, nonylphenol, among others)	Produced water*	Silvani et al. (2017)
GS	LLE	GC/MS	Water disinfection by-products, including trihalomethanes, haloacetic acids, halogenated aromatic hydrocarbons, among others.	Drinking water and surface water	Vozhdaeva et al. (2021)
GS	SPE	GC and HPLC/MS	Water disinfection by-products (chlorinated hydrocarbons, brominated hydrocarbons, halogenated aromatic hydrocarbons and their derivatives)	Surface water	Li et al. (2022)
GS	LLE and SPE	GCxGC/MS	A multitude of volatile EOPs (halogenated hydrocarbons, aromatic hydrocarbons, polychlorinated biphenyls, among others)	Wastewater	Murrell and Dorman (2021)
GS	LLE	UHPLC/MS-MS	Different EOPs, including pesticides, pharmaceuticals, personal care compounds, and industrial chemicals.	Wastewater and surface water	Salvatierra-stamp et al. (2018)
GS	-	HPLC	Biological toxins (saxitoxins, gonyautoxins, C-toxins, among others)	Surface water	Clemente et al. (2010)
GS	-	UHPLC/MS-MS	Cyanotoxins (anatoxin-A, homo-anatoxin, cylindrospermopsin, nodularin, and microcystins)	Drinking water and surface water	Pekar et al. (2016)
GS	-	Raman spectroscopy	Carbon nanomaterials (graphene oxide)	Surface water	Yang et al. (2020)
CS	LLE	LC/MS	Carbon nanomaterials (fullerenes: C ₆₀ and C ₇₀)	Wastewater	Farré et al. (2010)

GS: Grab sampling; PS: Passive sampling; CS: Composite sampling; LC: Liquid chromatography. *It refers to the water utilized in oil exploration and gas industry, including both formation water and injected water ([Silvani et al., 2017](#)).

4.3.1. Gas chromatography

GC is one of the most used analytical methods to separate and detect EOPs in water environments (see [Table 2](#)). In this technique, 30 m- and 60 m-length capillary columns with a diameter between 0.1 to 0.5 μm and solid sorbents made from alumina, fused silica, or molecular sieves, are employed. Depending on their configuration, GC columns can be classified as porous layer open tubular (PLOT), support coated open tubular (SCOT), and wall coated open tubular column (WCOT), being this the most used due to its efficiency, speed, and higher resolution ([Llamas-Dios et al., 2021](#); [Rahman et al., 2015](#); [Van Gansbeke et al., 2015](#)). According to its operation, GC is limited to the separation and quantification of non-polar, volatile and thermally stable EOPs, e.g., aromatic hydrocarbons, aliphatic hydrocarbons, and their halogenated derivatives. However, different derivatization methodologies have been designed to enable the quantification of polar EOPs, such as pharmaceuticals, pesticides, personal care products, among others, through GC. In this sense and, as mentioned in [section 4.2.1](#), these processes involve chemical reactions using various derivatizing agents to obtain compounds with a more apolar and volatile nature. Although this improves separation, sensitivity, selectivity, and the capability of the technique, it also represents a disadvantage, since derivatization can be complicated or tedious, time-consuming, uncompleted, or cause degradation of some analytes. Additionally, it is usually a non-eco-friendly process ([Bowden et al., 2009](#); [Ji et al., 2020](#)). Finally, different detectors are used in GC analysis, for example, flame ionization detector (FID), electron capture detector (ECD), and, of course, MS, which is the most used due to its sensitivity and low detection limits ([Benedé et al., 2014](#); [Ratola et al., 2006](#)).

In this sense, a multitude of works focused on EOPs monitoring in water environments using GC have been published through time. Some of them utilized GC with FID or ECD as a simple, inexpensive, fast, and efficient analytical methodology for EOPs determination in aqueous samples, e.g., pesticides and water disinfection by-products, reporting detection limits at $\mu\text{g/L}$ level ([Li et al., 2022](#); [Ratola et al., 2006](#); [Zhang et al., 2008](#)). However, when lower detection limits are required, MS is the proper detector, as will be discussed in a next section. For this reason, almost all of the published works focused on analytical determination of EOPs employing GC are based on GC/MS and GC/MS-MS couplings. Through these, industrial additives, pesticides, alkylphenols, polycyclic aromatic hydrocarbons, pharmaceuticals, and water disinfection by-products have been determined in water samples at ng/L and pg/L level ([Arcoletto et al., 2021](#); [Glineur et al., 2021](#); [Jiao et al., 2012](#); [Silvani et al., 2017](#); [Vozhdaeva et al., 2021](#)). Also, two-dimensional GC (GCxGC) coupled with MS have utilized to determine EOPs in water environments. This technique utilizes two orthogonally aligned capillary columns, both with different stationary phases, which enhance notably the chromatographic resolution and, in this sense, it results to be a suitable analytical technique when dealing with complex mixtures and matrices ([Castillo Meza et al., 2020](#); [Murrell and Dorman, 2021](#)). Using GCxGC/MS, it has been possible to determine different EOPs in

aqueous samples, such as polycyclic aromatic hydrocarbons, phenols, phthalate esters, parabens, hormones, anti-inflammatory drugs, pesticides and phenolic industrial additives ([Arismendi et al., 2019](#); [Murrell and Dorman, 2021](#); [Prebihalo et al., 2015](#)).

Currently, GC is positioned as a good alternative to quantify EOPs in a diversity of aqueous environments. However, due to the already-mentioned drawbacks of this technique, it is less implemented than LC for EOPs determination in water samples, also, taking into account that the majority of water EOPs are polar and non-volatile compounds ([Borrull et al., 2020](#); [Pinos Vélez et al., 2019](#)). This was easily corroborated by the proportion of the articles, using GC and LC for determining EOPs in water, indexed in Scopus (from 2000 to present) ([Scopus, 2021](#)). Approximately, for each article that implemented GC as the analytical technique for EOPs quantification in water samples, there are three articles that implemented LC as the analytical technique, i.e., GC is implemented in 25 % of the cases.

4.3.2. Liquid chromatography

Among LC techniques, HPLC and UHPLC are the most implemented for EOPs determination in water environments (see [Table 2](#)). UHPLC, in comparison with HPLC, provides a higher efficiency and peak capacity through the reduction in particle size (less than 2 μm) ([Celano et al., 2014](#)). Usually, both LC techniques utilized C_8 and C_{18} hydrocarbons bind to a solid fused silica support as the stationary phase. Among these, C_{18} column offers more robustness, since it allows in a higher degree the determination of both polar and non-polar EOPs in the same aqueous sample ([Pekar et al., 2016](#); [Salvatierra-Stamp et al., 2015](#)). In terms of mobile phase, mixtures of water with an organic solvent, e.g., methanol or acetonitrile, are generally used. Also, some additives can be used, such as organic acids (formic or acetic acid) or organic salts (ammonium acetate or ammonium formate), which improve in some way the separation process or the chromatographic signals. In LC, the polarity of the mobile phase can be changed throughout the analysis by programming a solvent gradient, i.e., changing the ratio of the employed solvents in a controlled way, such that the separation of polar and non-polar EOPs are enhanced ([Mirasole et al., 2016](#)). Additionally, due to its versatile and efficient operation, LC generally do not require derivatization methods, resulting to be a simpler separation technique with high capability for the determination of EOPs with different physicochemical characteristics, e.g., polarity, acidity, molecular weight, etc., and functional groups, e.g., hydroxyl, amine, carboxylic acid, thiol, among others ([Peng et al., 2019](#)).

According to the above, LC has been implemented extensively for detecting and quantifying EOPs in environmental aqueous matrices. For example, [Qian and co-workers](#) recently ([2021](#)) implemented SPE and UHPLC coupled with MS for investigating the presence of different EOPs in water samples of various Chinese full-scale wastewater treatment plants. The chromatography technique comprised the utilization of a C_{18} column, a mobile-phase gradient elution implementing 2 mM ammonium acetate and methanol as the solvents, and a high-resolution orbitrap mass spectrometer as the

detector. This allowed them to determine and screen out 568 organic substances present in those samples, among which 167 pharmaceuticals, 85 pesticides, 86 endogenous substances, 64 industrial chemicals, 14 personal care products, 17 industrial additives, and 6 hormones were identified at low $\mu\text{g/L}$ level. Between samples, pesticides and pharmaceutical compounds were highlighted as the most prevalent EOPs and, in addition, a correlation between the levels of anti-hypertension drugs (metoprolol and irbesartan) and hypertension prevalence in China was found. In a similar way, Wang et al. (2021a) implemented an analytical methodology based on a novel and fast on-line SPE-UHPLC-MS/MS for simultaneously quantifying 12 illicit drugs and their metabolites in wastewater, including methamphetamine, amphetamine, morphine, codeine, cocaine, ketamine, norketamine, and methcathinone at ng/L level. As in the previous work, the authors implemented a C_{18} column and a mobile-phase gradient elution with methanol, acetonitrile, and an aqueous acetic acid solution as the solvents. Other works focused on the determination of illicit drugs in water samples using UHPLC have also been published recently (Di Marcantonio et al., 2021; Mercan et al., 2019).

Although pharmaceuticals, pesticides, and illicit drugs can be considered as important pollutants extensively determined in water environments (Quintelas et al., 2020), other kinds of EOPs have also been the focus of many monitoring works, for example, personal care compounds, industrial additives, biological toxins, and even nanomaterials (Clemente et al., 2010; Farré et al., 2010; Pekar et al., 2016; Salvatierra-stamp et al., 2018; Wang et al., 2010). This has not only been carried out using LC/MS couplings, but also utilizing LC with diode array detector (Hernández-Fernandez and Rodríguez, 2019), fluorescence detector (Clemente et al., 2010; Speltini et al., 2015), and UV-vis detector (Fatoki et al., 2018). These detectors provide a good analytical response and sensitivity at $\mu\text{g/L}$ and even ng/L concentrations. However, they are limited in sensitivity when it is necessary to measure lower levels of EOPs (pg/L and below) and, as in the case of fluorescence and UV-vis detectors, to the requirement of a light absorption process by the targeted organic pollutant(s), which is not possible in many cases (García-Alonso and Pérez-Pastor, 2019).

4.3.3. Mass spectrometry

As mentioned in previous sections, MS is the most implemented detector in EOPs-determining protocols because of its sensitivity, simplicity, capability to identify both known and unknown EOPs in

complex matrices, and its easy, well developed, and effective coupling with LC and GC (Li et al., 2022). In its operation, first it is necessary to generate gaseous ions from the sample molecules. To do this, an ionization source that works properly on chromatographic couplings is utilized, for example, electrospray ionization or ESI. Most of the works for the analysis of EOPs in water samples are carried out implementing ESI in either positive or negative ionization modes (Jones-Lepp et al., 2012). Furthermore, other ionization techniques, such as atmospheric pressure chemical ionization, have also been used for EOPs analysis in water (Niu et al., 2020; Portolés et al., 2014).

Once gaseous ions are formed in mass spectrometers, they need to be separated and analyzed according to their mass-to-charge (m/z) ratio. For this, different analyzer with different separation power (or resolution) are implemented: magnetic sector, electrostatic sector, single quadrupole, time-of-flight (TOF), ion trap, Orbitrap, and Fourier transform ion cyclotron resonance (FTICR). Table 3 shows the main characteristics of different MS analyzers. Among these, FTICR provides the highest resolution, but with a higher cost associated with its operation (Menger et al., 2020). For this, quadrupole, ToF, and Orbitrap analyzers are also well implemented (Hedgespeth et al., 2012; Murrell and Dorman, 2021; Silvani et al., 2017). Another alternative is the coupling of two or more analyzer to improve notably the separation power, e.g., triple quadrupole and quadrupole-TOF (with single or multiple quadrupole), resulting in the so-called tandem MS (MS/MS). By this methodology, it is possible to analyze a specific group of EOPs with a specific range of m/z values through either, the entrapment of primary gaseous ions and its further fragmentation(s), or the time-dependent modification of the analyzed m/z values. This allows for a more detailed and accurate analysis even in complex matrices (Jiao et al., 2012; Salvatierra-stamp et al., 2018).

4.3.4. Other analytical techniques

Currently, most of the works focused on the determination and screening of EOPs use chromatographic techniques as the separation methodology and MS as the detector by excellence. However, in order to overcome some drawbacks associated with those techniques, such as expensive instruments, sample destruction, employment of toxic organic solvents, and non-portability, alternative techniques have been utilized for the same purpose, for example, Raman spectroscopy, FTIR spectroscopy, and emission photoinduced fluorescence.

Table 3. The characteristic of the most implemented MS analyzers in EOPs determination protocols (adapted from Liu et al., 2014).

Mass analyzer	Separation by	Typical resolution*	Mass accuracy
Quadrupole	Trajectory stability	2,000	100 ppm
Ion trap	Frequency	4,000	100 ppm
TOF	Flight time	10,000	10 ppm (using reflection)
Orbitrap	Frequency	100,000	< 5 ppm
FTICR	Frequency	10^5 - 10^6	< 5 ppm

*It is commonly defined as the ability to separate ions according to their m/z value (generally, z is one). It is calculated as $m/\Delta m$, where m is the nominal mass (actually m/z) for a particular peak in the mass spectrum, and Δm is peak width at 10 or 50 % of the peak height.

As a first example, [Quintelas and co-workers \(2020\)](#) recently reported the development of a FTIR transmission spectroscopy based chemometric methodology for the determination of eight EOPs (paracetamol, desloratadine, ibuprofen, β -estradiol, ethynylestradiol, carbamazepine, sulfamethoxazole, and atrazine) in wastewater. The methodology involved a sample scanning by FTIR and the recording of the obtained spectra. Then, a chemometric analysis was performed using sequentially a k-nearest neighbor (kNN) analysis to identify each sample pollutant; a preprocessing of the obtained spectra information by means of standard normal variate (SNV), multiplicative scatter correction (MSC), and first and second order derivatization; and, finally, a partial least squares (PLS) analysis in order to obtain suitable prediction models. This way, it was possible to obtain good results regarding the estimation of the targeted EOPs in wastewater at low mg/L level, resulting in a simple and eco-friendly protocol to measure EOPs. Importantly, some advances have been achieved in the pollutant's detection by FTIR through the implementation of glass optical fibers, which allow on-site, real-time monitoring of EOPs ([Michel et al., 2004](#)). More sophisticated systems have been developed using a fiber-type design coated with a polymeric membrane for simultaneously extracting and quantifying EOPs in water. This type of sensors exhibited high sensitivities and provided rapid analysis for the detection of chlorinated aliphatic hydrocarbons even at concentrations from 5 ppb; thus, it was proposed as a promising alternative technique for water contamination monitoring ([Lu et al., 2013](#)).

A relatively similar technique to FTIR, Raman spectroscopy, has also been implemented for the monitoring of EOPs in environment. For example, due to the large-scale production of graphene oxide (GO) and its reported toxicity to living organisms, [Yang et al. \(2020\)](#) were interested in the utilization of Raman spectroscopy for GO determination in water samples, since other techniques do not properly achieve this. In this approach, they performed a derivatization methodology using hydrazine (N_2H_4) with the aim of diminishing fluorescence interference by GO, a common problem when dealing with GO determination. Then, they quantified, in a simple and fast way, GO present in aqueous samples by means of the GO's G and D bands observed in their Raman spectra, obtaining a good linearity in a concentration range of 0.001 to 0.6 mg/L and achieving the distinction of GO from other carbon nanomaterials in the same samples. In a similar way, Raman spectroscopy has been used successfully for microplastic monitoring in water environments ([Lê et al., 2021](#)).

On the other hand, an eco-friendly strategy based on photochemically induced fluorescence for the determination of some pharmaceuticals (carbamazepine, ofloxacin, and piroxicam) in water samples was proposed in 2015 by [Hurtado-Sánchez and collaborators](#). In this methodology, only a sample pretreatment by means of SPE, without any further chromatographic analysis, was done. After that, the preconcentrated aqueous samples were analyzed by induced-fluorescence measurements. Through this technique, it was possible to detect the targeted pharmaceuticals even at a concentration of 0.04 ng/L, resulting to be a simpler and greener technique than LC/MS for the determination of this kind of

EOPs. However, limitations regarding to the structure of the pollutants, i.e., it need to have at least a chromophore fragment still remain.

5. Removal techniques of emerging water pollutants

After considering detection and monitoring methodologies for EOPs in water environments, it is turn to review the different technologies implemented on EOPs removal from this medium. Generally, these are divided into conventional methodologies, which comprise well established techniques implemented at low and large scale for removing both dissolved and non-dissolved pollutants, e.g., sedimentation, coagulation-flocculation, activated sludge, filtration, etc.; and non-conventional methodologies, which comprise recently developed, more sophisticated and highly efficient technologies implemented as an alternative for EOPs removal.

5.1. Conventional water treatment

Conventional water treatment is focused on the removal of solids, organic matter, microorganisms, and some dissolved compounds (e.g., salts, nutrients, etc.) from polluted water. Generally, this is performed at low, medium and large scales in wastewater treatment plants (WWTPs) by means of the implementation of different decontamination techniques in various well-designed steps, which are classified as primary (or mechanical), secondary (or biological), and tertiary (or chemical). Primary treatment steps involve the removal of suspended solids and colloidal particles through sedimentation, coagulation-flocculation, and/or air flotation processes, producing high quantities of sludge. In this step, grease and oils can also be skimmed off. Subsequently, in secondary treatment steps, microorganisms and dissolved organic and inorganic compounds are intended to be removed using trickling filters and/or activated sludge processes. These processes utilize the action of microorganisms to degrade the soluble organic pollutants. Finally, tertiary treatment steps aim to increase the quality of the treated water by the killing/removal, in a higher degree, of microorganisms and organic dissolved pollutants employing disinfection/oxidation methodologies, e.g., ozonation, chlorination/bromination, and UV irradiation ([Kerasi et al., 2021](#); [Turan et al., 2021](#)).

In this sense, EOPs are subjected to different physicochemical processes, such as sorption, dispersion, dilution, photodegradation, and biodegradation, throughout a completed conventional water treatment. However, due to the complexity on structure and behavior of EOPs and their relatively low concentrations in water environments, in conjunction with the particular operating mode of each employed technique, the resulting removal efficiencies varies greatly for each treatment step and each organic pollutant ([Ferreiro et al., 2020](#)). For example, it has been demonstrated that the removal efficiency of dissolved EOPs in primary treatment steps varies in the range of 20 to 50 %, whereas secondary treatment steps can be achieved efficiencies between 30 to 70 % ([Cristaldi et al., 2020](#); [Khasawneh and Palaniandy, 2021](#); [Quach-Cu et al., 2018](#)). In

addition, the inclusion of a tertiary treatment step, which is not always implemented in conventional water treatments, enhances the total removal efficiency towards some dissolved EOPs (80 % or more), but a great quantity of them usually remains in effluents (Di Marcantonio et al., 2020; Mailler et al., 2016a). In the case of undissolved EOPs, such as microplastics, these treatments can be more effective, reaching removal efficiencies higher than 90 %, even utilizing only primary and secondary treatment steps. Some particular examples of the aforementioned are reported in Table 4. Interestingly, negative efficiencies have also been reported for some EOPs. These have been explained in terms of the variation of environmental temperature, the partition equilibria of each pollutant, and the degradation of parent chemicals and conjugates excreted by human bodies, for example, which led to the release of more pollutant molecules during the treatment (Golovko et al., 2021). This way, different pharmaceutical, pesticides, personal care compounds, and industrial chemicals have been found in effluent waters and sludge even at concentrations of mg/L and mg/g, respectively (Bijlsma et al., 2021; Firouzsalari et al., 2019; Khasawneh and Palaniandy, 2021; Wang et al., 2020d).

Based on many studies focused on the efficiency of different WWTPs for removing EOPs, authors generally agree that

conventional wastewater treatment systems do not achieve it properly and, until now, there is the need for developing more advanced and versatile technologies that ensure higher removal capabilities. Among these emerging techniques, membrane-based separation systems, effective sorbents, advanced oxidation reactions, and sophisticated biological treatment will be discussed.

5.2. Non-conventional methodologies

Currently, non-conventional methodologies are an important research topic on water treatment systems, such that they continue to be developed and investigated, achieving a few advances in their establishment at medium and large scale until now. Here, it is important to take into account that these techniques are not a full replacement for conventional water treatment methodologies. First, the majority, if not all, of non-conventional treatment techniques need to be coupled with preliminary water treatment steps in order to eliminate either potential physical and chemical interferences or treatment-inactivating species. Second, they seek to improve the effectiveness on EOPs removal by means of different physico-chemical or biological mechanisms after the implementation of conventional treatment in almost all cases (Tak and Vellanki, 2019).

Table 4. Some relevant studies of EOPs removal by conventional water treatment employed in different WWTPs.

Location	Number of evaluated WWTPs	Treatment steps*	Targeted EOPs (Average efficiency [%])	Reference
China	16	Primary + Secondary + Tertiary	Hormones (~40), personal care products (~45), food additives (~30), pharmaceuticals (~25), pesticides (~10), natural substances (~45), others (~30).	Qian et al. (2021)
Italy	76	Primary + Secondary + Tertiary	Benzoylcegonine (~100), 11-nor-carboxy- Δ^9 -tetrahydrocannabinol (~75), ketoprofen (~90), sulfamethoxazole (~40), carbamazepine (10 or less), trimethoprim (~60), lincomycin (~0), estrone and a few related hormones (more than 50).	Di Marcantonio et al. (2020)
Sweden	15	Primary + Secondary + Tertiary	164 EOPs, including pharmaceuticals, personal care products, industrial chemicals, per- and polyfluoroalkyl substances, and pesticides (Positive removal efficiencies were obtained for less than half of them).	Golovko et al. (2021)
Spain	2	Primary + Secondary	Microplastics (91–97)	Franco et al. (2021)
China	2	Primary + Secondary + Tertiary	Endocrine disrupting compounds: paroxetine (36–49), fluoxetine (32–45), sertraline (56), citalopram (38–55), venlafaxine (4–6.5), amitriptyline (23–69), bisphenol A (96–99), and estriol (100).	Cao et al. (2020)
Peru	4	Primary + Secondary	27 pharmaceutical compounds. For 13 of them, the reported removal efficiency was positive (20–100), whereas for the others it was negative (-250 – -10).	Nieto-Juarez et al. (2021)
Colombia	2	Primary + Secondary	Acetaminophen (23–95), azithromycin (35–37), ciprofloxacin (35–65), clarithromycin (15–35), clindamycin (35–40), diclofenac (10–35), doxycycline (23–48), irbesartan (8–20), losartan (7–38), naproxen (20–37), norfloxacin (37–68), valsartan (25–45), venlafaxine (35–60), carbamazepine (-5–10), erythromycin (-3–12), metronidazole (-75 – -10), sulfamethoxazole (-64 – -3), and trimethoprim (-33 – -5).	Botero-Coy et al. (2018)
Mexico	1	Primary + Secondary + Tertiary	35 pharmaceuticals, including naproxen, acetaminophen, and diclofenac. The most abundant detected pharmaceuticals presented positive removal efficiencies (> 97 %).	Rivera-Jaimes et al. (2018)

*The combination of treatment steps can vary depending on each evaluated WWTP.

5.2.1. Adsorption-based methodologies

Adsorption is a well-known physicochemical process implemented for EOPs removal from water environments. More specifically, adsorption is a surface phenomenon in which a particular molecule (the adsorbate) is transferred from a liquid or gaseous phase to the surface of a solid material (the adsorbent) or, in some cases, a liquid phase. This mass transfer is controlled by means of thermodynamic equilibria, but also, it is described qualitatively by the favoring of certain interactions between adsorbate and adsorbent (Agboola and Benson, 2021). Among these, physical molecular interactions like van der Waals forces, polar interactions, hydrogen bonding, and electrostatic interactions, are highlighted. Chemical interactions, in which a new chemical bond is formed, e.g., coordination bond, purely covalent bond, and dynamic covalent bonds, are also considered. For the first case (physisorption), the process is a reversible nature, while, in the second case (chemisorption), the process is irreversible and at least one subsequent methodology is required to regenerate the adsorbent (Ünveren et al., 2017). The type and extent of adsorption are influenced by the concentration of adsorbate and adsorbent, the nature of adsorbate and adsorbent, surface area, pH, temperature, and interfering substances. All of

these are common parameters taken into account when studying adsorption kinetics and equilibria, since they affect the maximum removal capacity (Aarab et al., 2020; Delgado et al., 2019).

Experimentally, the removal of EOPs from an aqueous medium by an adsorption process can be summarized as: (i) adding the adsorbent to the liquid solution where the targeted pollutant is present, (ii) mixing properly and waiting until the equilibrium is reached, (iii) removing the adsorbent from the liquid solution, usually, by a simple filtration, and (iv) desorbing the adsorbed pollutants by another liquid solution with a proper set of conditions to regenerate the adsorbent (Martín et al., 2018). In this way, as a non-conventional water treatment technique, adsorption has important characteristics like efficiency, cost-effectivity, and simple design, as well as easy operation and regeneration (Bhatnagar and Anastopoulos, 2017; Perez et al., 2020). As shown in Table 4, different types of adsorbents and, thus, different types of interactions have been evaluated for EOPs removal from water. They are mainly classified according to their nature as natural adsorbents and synthetic adsorbents.

For their part, natural adsorbents comprise both organic and inorganic raw renewable materials utilized for pollutant removal from aqueous environments. Among natural organic adsorbents,

Table 4. Some examples of adsorbents developed for EOPs removal.

Adsorbent	Type of treated water	Evaluated EOPs	Removal capacity or efficiency*	Reference
Flax shives and oat hulls	Artificial polluted water	Carbamazepine	40 – 100 mg/g (24 – 72 h)	Aghababaei et al. (2021)
Wood, coal, peat, and coconut	Wastewater	15 EOPs, including atrazine, carbamazepine, diclofenac, ketoprofen, sulfamethoxazole, among others.	Up to 81 % (10 mg/L adsorbent; 45 min)	Maillet et al. (2016b)
Rice husk	Artificial polluted water	Acid Orange 7	~ 4 mg/g (3 h)	Swarnalakshmi et al. (2018)
Wood, rice husk, olive stones, and herbaceous waste	Wastewater	Amoxicillin	80 – 95 % (1 – 400 g/L; 5 – 30 min)	Saldarriaga et al. (2021)
Activated carbon	Artificial polluted water and wastewater	Carbamazepine and sildenafil citrate	> 90 % (100 mg/L; 10 h)	Delgado et al. (2016)
Activated carbon	Industrial wastewater	2-nitrophenol and ketoprofen	84 – 179 mg/g (300 min)	Sellaoui et al. (2021)
Bentonite clay	Artificial polluted water	Carbendazim	1.5 mg/g (15 min)	Rizzi et al. (2020)
Montmorillonite	Artificial polluted water	Amitriptyline	276 mg/g (4 h)	Chang et al. (2021)
Microporous triazine polymer	Artificial polluted water	Sulfamethoxazole	483 mg/g (1 h)	Akpe et al. (2020)
Hyper-crosslinked porous polymers based on spirobifluorene, triptycene, and 2,5-dibromopyrazine	Artificial polluted water	Bisphenol A, 2,4-dichlorophenol, 2-naphtol, and bisphenol S	455 – 562 mg/g (10 min)	Jia et al. (2018)
Hydroxypropyl- β -cyclodextrin	Artificial polluted water	Bisphenols (A, S, and F)	80 – 96 % (2 g/L; 60 min)	Cai et al. (2020)
Poly(styrene-co-divinylbenzene) and carbon nanotubes	Artificial polluted water	Simazine, prometon, and prometryn	~ 26 μ g/g (10 min)	Jiang et al. (2021)
Wood-based cellulose nanocrystals	Artificial polluted water	Auramine O	20 mg/g (5 – 10 min)	Pinto et al. (2020)
Chitosan-hydroxyapatite nanocomposites	Artificial polluted water	Norfloracin	92 % (0.5 g/L; 120 min)	Nayak et al. (2021)
Graphene oxide	Artificial polluted water	Valsartan, oxybenzone, caffeine, sulfamethoxazole, bisphenol S, metolachlor, carbamazepine, and sucralose.	> 90 % (0.1 – 0.6 mg/L; 15 min)	Fu et al. (2021)

*Additional information, such as the adsorbent's concentration and contact time, is provided.

plant-derived materials, such as rice husk, sugarcane bagasse, almond shell, oil palm shell, cotton waste, cashew nutshell, garlic peel, etc. (Ighalo and Adeniyi, 2020; Jain et al., 2016), and other bioderived materials like chitin, fungi, peat, biomass, yeast, and eggshell, are included (Vahabisani and An, 2021; Younas et al., 2021). The utilization of this kind of adsorbents has important advantages: (i) abundant availability, (ii) cheap materials, (iii) minimal pretreatment requirements, and (iv) high removal efficiencies and capabilities. In addition, they are in accordance with the actual trend associated with the implementation of renewable resources, e.g., agriculture and animal waste, for the design of new functional materials and the establishment of sustainable economics (Kyzas and Kostoglou, 2014). Through them, it has been possible to remove efficiently a high quantity of EOPs from water environments, including pharmaceuticals, endocrine disrupting compounds, pesticides, caffeine, dyes, industrial additives, among others (Al-Yousef et al., 2021; Lv et al., 2021; Tang et al., 2021; Yong et al., 2018). Stability and by-products release are important drawbacks of biomass as adsorbents, such that, chemical and physical modifications or pretreatments have been proposed to overcome it, e.g., through bases, metal salts, acids, extractions, oxidation, etc. (Adewuyi et al., 2020).

On the other hand, natural inorganic adsorbents are one of the most utilized, commercially available adsorbents for EOPs removal, since they are well-studied, efficient, and stable adsorbents. In this case, activated carbon, silica gel, activated alumina, zeolites, resins, and clays are included (Chua et al., 2021; Diagboya and Dikio, 2018). Among these, activated carbon are of great interest and implementation due to its high surface area and removal efficiency towards organic molecules, such as dyes (Delgado et al., 2019). However, the employment of clays (e.g., zeolites) as adsorbents have also increase recently, especially, for removing charged molecules and ions from aqueous environments. These natural materials have exceptional properties like cation exchangeability, high pore-volume, and large specific surface area (Cardona et al., 2020). Clays, in their natural form or chemically modified, have been applied as adsorbents for many EOPs in waters (Najafi et al., 2021; Rizzi et al., 2020). In contrast with bioadsorbent, inorganic adsorbents can have less removal capability due to the limited type and points of interactions with pollutants, i.e., biomass-derived adsorbent generally possess a greater quantity of functional groups than natural inorganic adsorbents. Thus, some surface chemical modifications have been employed to enhance their removal efficiency by the inclusion of different functional groups (Lerma et al., 2018; Ullah et al., 2020).

Finally, synthetic adsorbent comprise mainly well-designed polymeric materials obtained through different chemical methods at laboratory and industrial scale. The main advantages of a synthetic approach to obtain new adsorbents is the possibility to enhance notably removal efficiencies by tuning the chemical and physical structure of the adsorbent. They can have high porosity and surface area as activated carbon (Waheed et al., 2021). Thus, a diversity of polymeric structures, e.g., polyethyleneimine, polymeric dextrans, polyvinyl chloride, polyacrylic acid, polystyrene resins, etc., have been applied as adsorbents for pollutant removal from water media

(Moradi and Sharma, 2021; Romita et al., 2019). Likewise, molecular imprinted polymers have also been used as adsorbents for EOPs since, as discussed early, these present a higher selectivity in their removal performance (Bhagal et al., 2021). Although their important characteristics as adsorbents, synthetic polymers deal with some relevant disadvantages like the utilization of petroleum chemicals in the synthetic process and the low degradation rates of the final materials, which direct the attention and preferences for bio-based adsorbents (Flores-Céspedes et al., 2020). Other type of synthetic materials can be included in this category, for example, carbon-based nanomaterials, polymeric nanomaterials, metal nanoparticles, metal-organic frameworks, and organic/inorganic hybrid materials, which present enhanced removal efficiencies (Adegoke et al., 2020; Gil et al., 2021; Kyzas and Matis, 2015). Particularly, nanomaterials, e.g., graphene, graphene oxide, carbon nanotubes, fullerenes, TiO₂ nanoparticles, Fe₂O₃ nanoparticles, silica nanoparticles, etc., are of emerging interest as EOPs adsorbents due to their unique properties such as high mechanical strength and thermal conductivity, magnetic response, high specific surface areas, useful optical properties, and controlled chemical behavior (Kazemi et al., 2018; Peralta et al., 2021).

5.2.2. Membrane separation systems (MSSs)

MSSs have been utilized since the last century for the treatment of simple and complex mixtures, including polluted water, and they have proven to be a good complement to conventional water treatment methodologies. In terms of operation, MSSs use a semipermeable barrier (a liquid or solid membrane) to separate the components of a mixture according to two main aspects: molecular weight (or size) and solubility/diffusion in the membrane. In addition, the separation process is mediated by a driving force, which can be a hydrostatic pressure gradient, temperature gradient, electrical field, or a chemical potential gradient (Palencia et al., 2016). Currently, pressure-driven MSSs are highly implemented for different purposes, including the removal of organic pollutants from water environments. Depending on the type of compound or particle that can be removed, pressure-driven MSSs are classified as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO). For example, MF and UF are employed to separate colloidal particles, microorganisms, cells, and high molecular weight polymers, whereas NF and RO are useful to separate small organic molecules, salts, and ions (Kárászová et al., 2020). As a non-conventional water treatment technique, MSSs have important properties such as high efficiency, simple operation, lower energy consumption, continuous separation, the ease of industrial scale-up, and the possibility of coupling with important analytical techniques or other kind of processes (Lerma et al., 2016). However, a few drawbacks of MSSs are membrane fouling, which results in higher costs and energy consumption, and selectivity, which limits their applicability in many cases (Chang et al., 2019; Park et al., 2017).

As can be seen in Table 5, different approaches have been utilized for EOPs removal from water samples. In this sense, works focused on the type of membrane (polymeric, metal, or ceramic), its physical

Table 5. Some recent membrane-based methodologies for EOPs removal from water.

Membrane	Type of water	Targeted EOPs	Removal efficiency or capacity (%)	Reference
Aluminosilicate-based MF membrane	Artificial polluted water	Benzophenone-4	> 97	Sun et al., 2021
Polyvinylidene fluoride MF membrane with supported activated carbon	Artificial polluted water	Carbamazepine, tetracycline, norfloxacin, and sulfamethoxazole	> 90	Chen et al. 2021
Polyvinylidene fluoride hollow fiber UF membrane	Raw water	Humic acid	> 90	Ma et al. (2020b)
Polysulfone, polyethersulfone, polyamide, and regenerated cellulose UF membrane	Artificial polluted water	Alkylbenzene sulfonates	55 – 91	Kowalska (2008)
Polyamide RO membrane	Wastewater	Caffeine, theobromine, theophylline, amoxicillin and penicillin G	Up to 99	Lopera et al. (2019)
MXene clay-cellulose acetate NF membrane	Artificial polluted water	Rhodamine B	99	Xu et al. (2020)
Poly(diallyl dimethylammonium chloride) and poly(sodium styrenesulfonate) NF membranes	Artificial polluted water	Perfluorooctanoic acid, perfluorooctanesulfonic, amoxicillin, and tetracycline	Up to 90	Wang et al. (2021b)
β -cyclodextrin modified cellulose NF membrane	Artificial polluted water	Bisphenol A	Up to 100	Lv et al. (2021)
Carbon impregnated chitosan-based loose NF membrane	Wastewater	Eriochrome black T, methylene blue, rhodamine B, red brown dye, congo red, and humic acid.	> 80	Halakami et al. (2021)
Forward osmosis MF membrane bioreactor	Wastewater	Enrofloxacin, sulfamethazine, cephalexin, amoxicillin, lomefloxacin, and ampicillin	58.9 – 100	Qiu et al. (2021)
Lacasse-coated poly(vinylidene fluoride) membrane	Artificial polluted water	Congo red	> 90	Zhu et al. (2020)
Membrane bioreactor combined with UV/H ₂ O ₂	Artificial polluted water	17 α -ethinyloestradiol	> 99	Da Costa Fonseca et al. (2021)
Metal (cerium) organic framework – polyether sulfone UF composite membrane	Artificial polluted water	Humic acid	99	Mansor et al. (2021)
Ceramic (α -Al ₂ O ₃) membrane decorated with CoFe ₂ O ₄ nanocatalyst	Artificial polluted water	Methylene blue and ibuprofen	> 95	Wang et al. (2020e)
Ultrasound, adsorption, and polysulfone UF membrane	Wastewater	Diclofenac, carbamazepine, and amoxicillin	99	Naddeo et al. (2020)

structure (e.g., pore size), the arrangement of the separation system (e.g., coupled membranes, recirculation systems, etc.), and the development of membrane-based hybrid methods for EOPs removal have been published in recent years (Hilal and Wright, 2018; Tang et al., 2018b). For example, polymeric and ceramic MF and UL membranes have been utilized for EOPs removal by means of adsorption processes (Qalyoubi et al., 2021). Through them, relatively high efficiencies (> 90 %) have been obtained for particular EOPs, e.g., benzophenone-4, estrone, 17- β -estradiol, 17 α -ethynyl estradiol, antibiotic resistance genes, carbamazepine, tetracycline, among others (Chen et al., 2021; Li et al., 2019; Ma et al., 2020b; Sun et al., 2021). One of the main advantages of MF and UL membranes is the low required hydraulic pressure (i.e., lower energy consumption) and higher fluxes (i.e., faster separations); however, their removal efficiencies are more limited for small pollutants in comparison with NF and RO. Usually, NF and RO reach higher efficiencies in EOPs removal due to their low pore size (0.0001 – 0.001 μ m), but with higher energetic costs and low fluxes.

In addition, NF and RO have a higher susceptibility to fouling (Lopera et al., 2019; Xu et al., 2020).

To overcome these drawbacks, the coupling of various membrane modules, for example, MF-NF, MF-OR, UL-NF, UL-RO, etc., as well as the chemical or physical modifications of membrane surface or matrix have been proposed (Ezugbe and Rathilal, 2020). The latter is of great interest in polymeric membranes due to their structural versatility and functionality, e.g., superficial inclusion of functional polymer chains for diminishing fouling susceptibility and/or enhancing removal selectivity (Oshiba et al., 2021; Zhu et al., 2020). Specially, membrane based on polymers are of the most implemented since they result to be cheap, easily obtained, versatile, and have good mechanical and chemical stability. Thus, a high quantity of polymeric membrane has been used with a focus on EOPs removal from water. Common employed polymeric membranes are made from polypropylene, polyvinylidene fluoride, polyethersulfone, or polysulfone. However, new polymeric membranes are developed constantly from different polymer

structures. For example, Pagno and co-workers reported recently the preparation of novel polymeric membranes made from polyesters like poly(butylene adipate-co-terephthalate) and poly(ϵ -caprolactone) for the effective removal (> 60 %) of tetracycline in aqueous samples (Pagno et al., 2020). Polyelectrolytes, such as poly(diallyl dimethylammonium chloride) and poly(sodium styrenesulfonate), have also useful for obtaining multilayer membranes applied in the removal of EOPs in saline wastewaters, as reported by Wang et al. (2021b). On the other hand, biopolymer have gained relevance for preparing membranes focused on EOPs removal, e.g., cellulose and chitosan. Employing them, removal efficiencies have exceeded 80 % (Lv et al., 2021; Mansoori et al., 2020).

Finally, membrane-based hybrid methods have arisen recently as an efficient non-conventional water treatment. Among these, advanced oxidation, ozonation, coagulation, photocatalytic processes, adsorption, and biological treatment in conjunction with membrane filtration have been proposed (Stylianou et al., 2015; Zainith et al., 2021). Particularly, MSSs in conjunction with biological treatment, also called “membrane bioreactors”, have successfully applied for EOPs removal and they are positioned as a prominent water treatment technique. The combination of the EOPs rejection capacity of MF and UF membranes with the biological degradation provided by microorganisms results to be a highly effective technique for wastewater treatment (Saidulu et al., 2021). Personal care products (> 80 %), antibiotics (> 70 %), pesticides (> 90 %), among others EOPs have been removed from water in high efficiency using this hybrid technique (Qiu et al., 2021; Ren et al., 2021; Santos de Almeida Lopes et al., 2020). Other novel, effective hybrid technique is polymer-enhanced ultrafiltration or liquid-phase polymer-based retention, which combines the rejection of macromolecules by a UF membrane with the capability of a soluble polymer to interact with particular species, resulting in an ultrafiltration process capable of retaining much smaller molecules. This technique has been well-studied and applied for the removal of different EOPs from water, for example, dyes and antibiotics, in high efficiencies (Palacio et al., 2020; Palencia et al., 2017). That is

how membrane-based hybrid methods have gained great acceptance in scientific community and, actually, they can be considered as one of most promising non-conventional water treatments.

5.2.3. Advanced oxidation processes (AOPs)

AOPs are a type of non-conventional water treatment that have been studied and applied since the end of the last century. Until now, some of them have been used at medium and large scale for water treatment, while the majority of the studies focused on these processes remain at laboratory scale. Here, a brief introduction to AOPs will be carried out, however, a deeper discussion of this topic can be found in specialized reviews (Bartolomeu et al., 2018; Ricardo et al., 2021). In a general sense, AOPs are based on particular chemical reactions by which EOPs can be fully oxidized and mineralized, producing carbon dioxide, water, and inorganic ions. These processes can be divided in homogeneous AOPs, which include photolysis (i.e., UV light), sonolysis (i.e., ultrasound), ozonolysis, hydrogen peroxide, Fenton processes, electrochemical methods, and their possible combinations; and heterogeneous AOPs, which comprise the employment of solid semiconductors catalysts, such as TiO₂, ZnO, MoS₂, among others, in conjunction with light (Rodriguez et al., 2011). Through them, large amounts of highly oxidizing species (radicals), such as OH[•], O₂^{•-}, or SO₄^{•-}, are generated *in situ* and, in this sense, a series of cascade-type reactions are initiated with the participation of the pollutant's molecules. It has been reported that the radical species can degrade organic pollutants by hydrogen abstraction, electron transfer, or addition to double bonds and aromatic systems (Chávez et al., 2020). Here, organic radicals are produced, but they rapidly undergo additional reactions that produce the final most-oxidized products (Stanbury et al., 2020). In this way, harmful EOPs are converted to innocuous chemical species.

Table 6 provides representative examples of the application of different types of AOPs to EOPs removal from water.

Table 6. Some relevant examples about the utilization of AOPs for EOPs removal from water.

Method	Type of water	Targeted EOPs	Removal efficiency or capacity (%)	Reference
Sonolysis	Artificial polluted water	Acetaminophen, cloxacillin, diclofenac, naproxen, piroxicam, sulfacetamide, and cefadroxil	Up to 100	Camargo-Perea et al. (2021)
Sonolysis (activated by O ₂ or Fe(III))	Artificial polluted water	Metazachlor	Up to 97	Kask et al. (2019)
Sonolysis + Photocatalysis (TiO ₂)	Artificial polluted water	Sulfamethoxazole, bisphenol A, and atrazine	>98	Ryu et al. (2021)
UV irradiation	Artificial polluted water	N-chlorourea	92	Yang et al. (2021b)
UV irradiation	Artificial polluted water	Tetracycline	75	Huang et al. (2019)
UV irradiation	Wastewater	Ampicillin, tetracycline, ofloxacin, florfenicol, cephalexin, and amoxicillin	>90	Ding et al. (2020)
O ₃ /UV irradiation	Surface water	More than 20 EOPS, including alachlor, atrazine, azithromycin, carbamazepine, clarithromycin, clindamycin, clothianidin, diclofenac, diphenhydramine, fluoxetine, isoproturon, among others.	>85	Gorito et al. (2021)

O ₃ /UV irradiation	Wastewater	Carbamazepine, fluoxetine, gemfibrozil, primidone, sulfamethoxazole, and trimethoprim	~20 – 100	Sgroi et al. (2021)
H ₂ O ₂ / UV irradiation	Artificial polluted water	Methylene blue, basic blue 41 and acid orange 7	Up to 100	Dhawle et al. (2021)
H ₂ O ₂ / UV irradiation	Artificial polluted water	Nitrosamines	>75	Zhou et al. (2012)
Peroxymonosulfate (activated by Co ²⁺)	Artificial polluted water	1,4-dioxane	100	Feng et al. (2020)
Peroxydisulfate (activated by TiO ₂)	Artificial polluted water	Bisphenol A, 4-chlorophenol, sulfamethoxazole, and carbamazepine	>80	Son et al. (2021)
Photo-Fenton process (using sun light)	Artificial polluted water	Simazine	>90	Lojo-López et al. (2021)
Fenton process	Wastewater	Diuron, gabapentin, sulfamethoxazole, terbutryn, and terbuthylazine	70 – 100	Goswami et al. (2021)
Photo-Fenton process	Artificial polluted water	2,4-D, diazepam, nicotine, and paracetamol	Up to 85	Nippes et al. (2021)
TiO ₂ -based photocatalysis	Artificial polluted water	Quinalphos and 2-chlorophenol	>70	Sharotri et al. (2019)
TiO ₂ -based photocatalysis	Artificial polluted water	Rhodamine B and Methyl Orange	>90	Narzary et al. (2020)
TiO ₂ /hydroxyapatite-based photocatalysis	Artificial polluted water	Ciprofloxacin and ofloxacin	Up to 100	Bouyarmane et al. (2021)
Nano-hybrid photocatalyst based on TiO ₂ and MoS ₂	Artificial polluted water	Methylene blue	97.5	Karpuraranjith et al. (2022)
Polyacrylonitrile/TiO ₂ /polyaniline photocatalytic and adsorptive membranes	Artificial polluted water	Congo red	>80	Xu et al. (2020)

Homogeneous, single AOPs, such as sonolysis and photolysis, employ an energy supply (ultrasound and UV light, respectively) to promote the decomposition of EOPs either by the production of hydroxyl radicals or by the direct fragmentation of the pollutant structure. For its part, the application of an ultrasound frequency (300-1000 kHz) to a liquid sample with EOPs generates a cavitation phenomenon inside it. During this, different reactive radical species, mainly produced by the homolytic fragmentation of water (i.e., $H_2O \rightarrow H^\bullet + OH^\bullet$) are produced at three zones delimited by cavitation bubbles: a gaseous zone (inside the bubbles), a liquid-gas interface, and a bulk liquid zone. This way, the produced radical species interact with the pollutants and degrade them (Camargo-Perea et al., 2020). Ultrasound is a green, simple, and efficient technique that has been employed for the removal of different organic pollutants, such as pharmaceuticals, industrial additives, pesticides, and other kind of EOPs (Camargo-Perea et al., 2021; Kask et al., 2019; Ziyilan-Yavas et al., 2021). The efficiency of sonolysis can be modulated by the applied ultrasound frequency, temperature, pH, and the presence of certain species that enhanced the degradation process (Navarro et al., 2011). However, this technique presents limitations on the degradation of particular type of pollutants, i.e., pollutants with different polarity can experience different degradation, and the rate of that degradation (Ryu et al., 2021). On the other hand, some particular compounds that present the property of absorbing UV light (e.g., halogenated aromatics, nitro compounds, phenols, conjugated hydrocarbons, among others), resulting in an excited more energetic state, can be degraded by light irradiation. In addition, the excited molecules of the pollutant can interact with oxygen to undergo different reactions capable of degrading them in a higher degree (Huang et al., 2019).

Some authors also suggest the formation of oxidizing hydroxyl radicals in solution by UV irradiation (Yang et al., 2021b). For this process, only UV lamps (or, in some cases, sun light was also used) are generally required to irradiate the sample through an established interval of time, which results to be a highly accepted, simple process. UV light has been employed for the efficient degradation of bisphenol A, polycyclic aromatic hydrocarbons, disinfection by-products, antibiotics, among others (Chu et al., 2021; Lei et al., 2021; Rosińska, 2021). However, some disadvantages have been highlighted, such as limitations in the compounds that can be treated by this method, interference by turbidity and chemical species, and the generation of some harmful by-products (i.e., volatile halogenated hydrocarbons) (Fast et al., 2017).

To overcome the drawbacks associated to ultrasonics and UV irradiation and, at the same time, to enhance the degradation/removal efficiency through AOPs, some reagents and/or catalysts have been included in these processes. For example, ozone (O₃) and hydrogen peroxide (H₂O₂) has widely implemented for this purpose (Gorito et al., 2021). As light sensitive species, both of them result to be fragmented when they are irradiated with UV light, generating reactive OH[•] and HO₂[•] radicals capable to degrade the organic molecules present in the aqueous medium. In addition, ozone and hydrogen peroxide can directly oxidize them due to their intrinsic reactive nature (Abrile et al., 2021). The utilization of these combined AOPs leads to high efficiencies for the removal of pharmaceuticals compounds even up to 100 % of their initial concentrations in wastewater, as reported by Sgroi and co-workers (2021). Complex mixtures of EOPs in WWTPs have also been treated by means of O₃/UV, obtaining > 80 % of removal efficiency (Mathon et al., 2021). Other kind of EOPs, such as dyes, pesticides,

and disinfection by-products have been successfully removed from water employing $\text{H}_2\text{O}_2/\text{UV}$ combined methods (Dhawle et al., 2021; Lojo-López et al., 2021; Zhou et al., 2012). However, some drawbacks of these oxidation systems lies in the utilization of relatively high concentration of oxidizing agents for degrading pollutants at very low concentrations, which results not only in high costs, but also in indirect environmental effects; as well as, ozone has limited solubility in water and the process presents great susceptibility towards temperature and pH variations (Brienza and Katsoyiannis, 2017).

More sophisticated AOPs involves the utilization of other type of reactive species for degrading EOPs, by instance, sulfate radical ($\text{SO}_4^{\bullet-}$). In the first case, the process involves the in-situ production of $\text{SO}_4^{\bullet-}$ by precursor oxidants, like persulfate ($\text{S}_2\text{O}_8^{2-}$), also called or peroxydisulfate, and peroxymonosulfate (HSO_5^-). The produced sulfate radicals have a good oxidizing character ($E^0 = 2.5 - 3.1 \text{ V}$) and, thus, they are capable of degrading organic pollutants, producing CO_2 and H_2O (Uzunboy et al., 2021). Some advantages of employing sulfate radicals as oxidizing agents include cheap and very stable precursors, as well as, different ways for activating them (e.g. heat, bases, transition metals, and UV light) (Divyapriya and Nidheesh, 2021; Gong et al., 2020). Employing sulfate radical-based AOPs, toxic organic solvents, industrial additives, pharmaceuticals, and dyes have been removed from water in high efficiencies (Ding et al., 2020b; Feng et al., 2020; Son et al., 2021). On the other hand, AOPs can be enhanced by the employment of various promoters and catalyst; being relevant examples iron (Fe) ions and some semiconductors, as mentioned above. More specifically, Fe(II) and Fe(III) can catalyze the degradation of organic pollutants by means of the production of OH^{\bullet} radicals in presence of H_2O_2 . This methodology is called Fenton processes (Zhang et al., 2019). Although they can be carried out by heat, UV light is most preferred as the promoter agent due to the high reached quantum and removal efficiencies. In terms of chemical reactions, photo-Fenton processes can be summarized as the direct reaction between Fe(II) and H_2O_2 to produce Fe(III) and hydroxyl radicals ($\text{Fe}^{2+} + \text{H}_2\text{O}_2 \rightarrow \text{Fe}^{3+} + \text{OH}^- + \text{OH}^{\bullet}$), followed by the regeneration of Fe(II) from Fe(III) by UV light ($\text{Fe}^{3+} + \text{H}_2\text{O} + h\nu \rightarrow \text{Fe}^{2+} + \text{H}^+ + \text{OH}^{\bullet}$) (Wang and Tang, 2021). Strictly, these processes must be conducted at acidic pH (~ 3), since iron ions can be precipitated as iron hydroxides at higher pH (Scaria et al., 2021). In addition, some complexes of iron with organic acids, which usually absorb light in a larger range of wavelengths, have been employed to enhance the efficiency of Fenton processes (Miller et al., 2016). As a non-conventional water treatment, (photo-)Fenton reactions result to be simple, flexible, low or null energy requirement (the employment of sun light is also possible), and easy-to-handle. This way, they have been widely used for treating different effluents and degrading organic pollutants, for example, herbicides, antibiotics, dyes, and illicit drugs (Da Silveira Salla et al., 2020; Goswami et al., 2021; Lojo-López et al., 2021; Nippes et al., 2021). However, some drawbacks of this methodology have been highlighted, such as mandatory acidification, the removal of iron after the treatment, and the employment of relatively large

quantities of iron to reach a high efficiency in some cases (Bartolomeu et al. 2018).

Finally, various heterogeneous photocatalysts based on semiconductors have been developed as an efficient alternative to degrade and remove organic pollutants in water. Among these, titanium dioxide (TiO_2) has been widely explored and applied due to its relevant properties, such as high efficiency, low toxicity, higher resistance against photo-corrosion, availability, and good cost-benefits (Gupta et al., 2021; Paumo et al., 2021). In general, TiO_2 (and other kind of semiconductor materials employed for this purpose), has the ability to absorb light and promote one electron in its valence band to its conduction band, while a hole is also generated in the valence band. Then, the produced hole in the semiconductor materials can interact with water molecules or hydroxyl anions to generate OH^{\bullet} radicals (capable to degrade organic pollutants) in the medium; or with pollutant molecules by electron transfer, degrading them. On the other hand, electrons are transferred to oxygen (O_2), producing $\text{O}_2^{\bullet-}$ radicals, which are also able to react with pollutant molecules. In this way, a catalytic cycle is established based on purely electron transfers from the molecules in the medium to the semiconductor material mediated by light, as shown in Figure 4 (Schneider et al., 2014). Various TiO_2 -based photocatalyst systems have been explored for the removal of dyes, pesticides, pharmaceuticals, and even nanoplastics (Domínguez-Jaimes et al., 2021; Narzary et al., 2020; Sharotri et al., 2019). Likewise, composite TiO_2 -based materials with enhanced physicochemical properties, e.g., adsorption of pollutant molecules, as well as hybrid oxidative processes based on TiO_2 photocatalysis were also reported as catalysts with high efficiency for photodegradation of organic pollutants (Bouyarmane et al., 2021; Karpuraranjith et al., 2022).

As alternatives, other types of semiconductors have been employed for photocatalyzed degradation of EOPs in waters. Important examples are cadmium sulfide (CdS), zinc oxide (ZnO), tungsten trioxide (WO_3), tin dioxide (SnO_2), among others (Zhu and Zhou, 2019). However, these have not outweighed the inherent advantages of using TiO_2 .

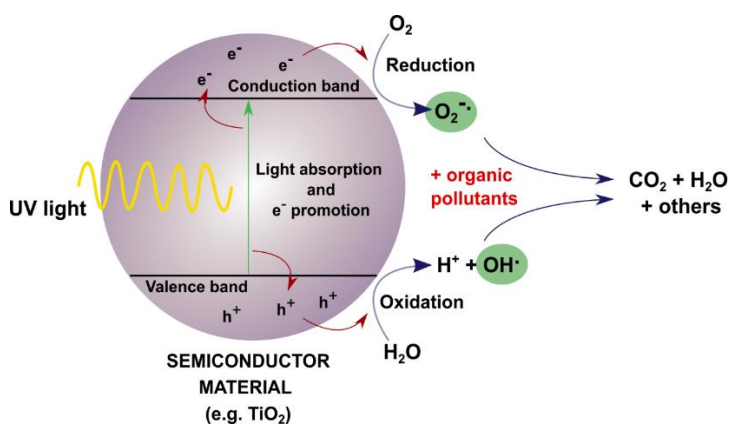


Figure 4. General mechanism of the degradation of organic pollutants by TiO_2 -based photocatalysis.

Until now, semiconductor-based photocatalysis represent a very promising method for the treatment of polluted water. However, various aspects need to be solved for a proper implementation of these processes at large scale, e.g., light absorption efficiency by the semiconductors and the recovery of the catalyst after treatment.

6. Conclusions and perspectives

EOPs in water bodies at nano-, micro- and even milligrams per liter level represent a huge risk for the availability and usage (human and industrial) of this important natural resource, as well as, for the life of different living beings, including aquatic organisms and humans. According to the above, there is a current need for the detection and monitoring of EOPs in water, such that, it is possible to determine their concentrations, fate, and environmental behavior. Likewise, the establishment of regulations on the common utilization of EOPs and the development of proper solutions towards their removal or elimination require knowing about their presence in water environments. Thus, different analytical protocols have been well-studied and explored for determining EOPs. Generally, they involves three main steps: (i) taking a sample from the targeted water body, (ii) if required, treating the sample by means of preconcentrating and cleaning up techniques, e.g. SPE and LLE, and (iii) analyzing the treated sample using mainly a chromatographic technique (LC or GC) coupled with MS. In this way, EOPs have been detected even at trace concentrations in various water environments, including pharmaceuticals, pesticides, personal care products, industrial chemicals, cyanotoxins, and others. Once they are detected, it is needed to think about their removal from the aqueous medium. For this, usually employed or conventional methodologies like primary and secondary treatment steps that

commonly utilize filtration, coagulation/flocculation, and biological treatment, can contribute to it, but with limited removal efficiencies. Thus, a tertiary treatment step, or a non-conventional water treatment, is required to accomplish it completely. Among these, adsorption-based techniques, membrane-based separation systems, and AOPs are included, which have been explored extensively for EOPs removal from water. Each methodology has its own advantages and disadvantages and, thus, some of them can be more suitable for the removal of a specific class of EOPs than others. In addition, there is the possibility to use in conjunction different removal techniques, such that the removal efficiency can be enhanced notably (>90 %).

Currently, the determination of EOPs in water and their removal from it remain as important topics in different research fields, such as environmental sciences, chemistry, engineering, physics, among others. Future research need to be focused on some particular aspects that have to be addressed from many points of views to advance in the establishment of proper solutions to this problematic: (i) designing and unifying accurate analytical protocols to determine/monitor EOPs with the aim to obtain comparable results and not to incur in bias, (ii) optimizing the parameters (e.g. type of adsorbent, membrane, or oxidizing agents, temperature, pH, interference elimination, etc.) associated with each removal methodology for ensuring the highest efficiencies, (iii) studying in a higher degree the coupling of different removal techniques for treating polluted waters, and (iv) advancing in the utilization of these techniques at higher scales. All the above should be approached from an ecofriendly point of view, taking into account the implementation of renewable raw materials, the importance of recycling, and the actual need to ensure circular economics.



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