

Quaternary Treatment of Urban Wastewater for Its Reuse

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Abstract: In today's ongoing rapid urban expansion, deforestation and climate changes can be observed mainly as unbalanced rain occurrence during the year, long seasons without any rain at all and unordinary high temperatures. These adverse changes affect underground water levels and the availability of surface water. In addition, quite a significant proportion of drinking water is used mainly for non-drinking purposes. With several EU countries increasingly suffering from droughts, reusing quaternary treated urban wastewater can help address water scarcity. At the European level, Regulation 2020/741 of the European Parliament and of the Council of 25 May 2020 on minimum requirements for water reuse was adopted. This regulation foresees the use of recycled wastewater mainly for agricultural irrigation. This article provides an overview of various processes, such as filtration, coagulation, adsorption, ozonation, advanced oxidation processes and disinfection, for quaternary treatment of urban wastewater in order to remove micropollutants and achieve the requirements for wastewater reuse. According to the literature, the most effective method with acceptable financial costs is a combination of coagulation, membrane filtration (UF or NF) and UV disinfection. These processes are relatively well known and commercially available. This article also helps researchers to identify key themes and concepts, evaluate the strengths and weaknesses of previous studies and determine areas where further research is needed.

Keywords: irrigation; micropollutants; wastewater reuse; water scarcity; quaternary treatment



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1. Introduction

Water has several unique functions. It is the source of ecosystem quality, ensures existence for all animals and humans on our planet and plays a key role in the production of food and energy. However, the long-term environmental degradation due to the development of the world economy and urbanization with climate and demographic changes is slowly depleting its quantity and thus water resources are being polluted and depleted [1]. In addition, quite a significant proportion of drinking water is used mainly for non-drinking purposes. It is clear that water scarcity and drought events are likely to be more severe and more frequent in the future. The so-called “water stress” is increasingly coming to the force and it is assumed that this issue will be one of the most significant challenges of the 21st century [2]. The water stress is intensified by deforestation which reduces the global forest cover where rainwater could be stored and then re-generated, thus reducing the global average temperature of the Earth. Due to the deforestation and the associated lack of precipitation on the planet, we are observing a gradual increase in dry periods, which prevail for a long time in the world. Generally, the dry season has increased by 29% since 2000 and for the European continent, the dry seasons during the last few years has been the worst in the last 500 years [3].

The number of people on our planet who live in a state of drinking water scarcity for a period of 1 month is estimated to be around 4 billion, and it is predicted that by 2050 it will reach about half of the world's population [4]. In terms of total water demand, it has increased threefold since 1950, to around 4 000 km³/year, and is projected to reach around

6000 km³/year by 2050. This is why water is also referred to as “the oil of the 21st century” by several economic and scientific journals [5].

The largest quantities of fresh water are used worldwide in the industrial and agricultural sectors. The growing human population is responsible for the global expansion of agricultural land that needs to be irrigated. Over the past half century, the global irrigated area has more than doubled, with agricultural irrigation accounting for more than 70% of freshwater consumption [6]. The World Bank warns that if a deep-water crisis occurs, regions such as the Middle East and North Africa could lose up to 6% of their GDP [7].

Based on this huge number of facts and predictions for the coming decades, the question of how to supplement and replace the missing water resources gradually arises. The answer is to look for non-traditional and new sources of water, represented by quaternary treated wastewater [8]. The European Commission defined quaternary treatment as the additional and advanced treatment of urban wastewater in order to eliminate the broadest possible spectrum of micropollutants.

This treatment reduces the risk of micropollutants uptake by plants and soils when the effluent is reused for crop irrigation [9].

The reuse of quaternary treated wastewater for agricultural irrigation is a market-driven activity based on the demands and needs of the agricultural sector, especially in countries facing a shortage of water resources. Irrigation with quaternary treated wastewater could reduce the “water stress” in the given location to a certain extent. In developed countries, it represents a strategy for improving the state of the environment and sustainable agricultural production. At the same time, it is assumed that the reuse of quaternary treated wastewater from urban wastewater treatment plants has less impact on the environment than other alternative methods of water supply, such as water transportation or seawater desalination [10]. The reuse of quaternary treated wastewater for agricultural irrigation can also contribute to the promotion of a circular economy by recovering nutrients (especially potassium, nitrogen and phosphorus) from recycled wastewater and applying them to crops through fertigation techniques. It could therefore potentially reduce the need for additional application of mineral fertilizer and thereby contribute to the recovery of nutrients. In this way, the environment would be less burdened by artificial fertilizers. For end users (farmers), the quaternary treated wastewater is a relatively cheap substitute for artificial fertilizers. For this reason, this type of water can be an interesting commodity in agriculture [11–13].

On the other hand, wastewater recycling poses concerns about microbial risk, the presence of resistant microorganisms (i.e., antibiotic resistant bacteria and genes (ARB&ARGs)) and the presence of micropollutants, such as pesticides, pharmaceuticals, illegal drugs, synthetic and natural hormones and personal care products [10]. The occurrence of these pollutants in the environment is related to various human and industrial activities and are associated with biologically adverse effects on living organisms, such as toxicity and endocrine disruption. The problem may be that some of them are persistent, bioaccumulative and toxic (PBT) or persistent, mobile and toxic (PMT) [14,15]. The current urban wastewater treatment plants are not designed to eliminate micropollutants and can remove many chemicals only to a limited extent, depending on the treatment conditions, but also depending on mobility and resistance to degradation. Then these micropollutants pass through wastewater treatment processes and they may end up in the aquatic environment, becoming threats to wildlife and problems for drinking water production and water reuse. In addition, in most urban wastewater treatment plants, monitoring actions for micropollutants have not been well established and the release of micropollutants and ARB&ARGs into the environment (except Switzerland) is not currently regulated, nor is their occurrence in wastewater for reuse in agriculture, so there are certain risks for public health. The main risk is related to the consumption of raw or undercooked vegetables contaminated with pathogenic microorganisms originating from the use of untreated or poorly treated wastewater for crop irrigation [16]. The monitoring of micropollutants in wastewater to

reuse for crop irrigation is one of the main areas of discussion among scientists, legislators and stakeholders at the European Union (EU) level [17].

Legislation in the field of using treated wastewater for irrigation develops differently in individual countries [18]. At the European level, Regulation 2020/741 of the European Parliament and of the Council of 25 May 2020 on minimum requirements for water reuse was adopted in 2020. This regulation foresees the use of recycled wastewater mainly for agricultural irrigation and sets minimum requirements for the quality of recycled wastewater, which it divides into four quality classes (A to D), from crops consumed raw to technical and energy crops. At the same time, this regulation establishes the permitted methods of irrigation. The purpose of this regulation is to facilitate the implementation of water reuse wherever appropriate and cost effective, thus creating a supportive framework for those Member States that want or need to reuse wastewater. This Regulation lays down minimum requirements for water quality and monitoring and risk management provisions for the safe use of recycled wastewater in the context of integrated water management. Water reuse is a promising option for many Member States, but currently only a small number of them implement this practice and have adopted national legislation or standards in this regard. In addition, Member States may use treated wastewater for other purposes, such as water reuse in industry [19].

Adherence to the minimum requirements for water reuse should be in line with the EU's water policy and should contribute to the achievement of the Sustainable Development Goals set out in the United Nations 2030 Agenda for Sustainable Development, in particular the goal of ensuring the availability and sustainable management of water and sanitation for all, as well as substantially increasing water recycling and safe water reuse globally. The aim of this regulation should also be to ensure the application of Article 37 of the Charter of Fundamental Rights of the EU, which concerns environmental protection [20].

Despite these general requirements, there are still risks that need to be addressed individually depending on the sources of wastewater pollution, the rate of removal of specific pollutants, and the methods and goals of using recycled wastewater. Despite the legislative framework adopted above, the rate of water reuse in the EU is low, which results in high investments needed to modernize urban wastewater treatment plants and a lack of financial incentives for quaternary treated wastewater reuse in agriculture. Regarding agricultural products, the reason for the low rate of water reuse is the possible health and environmental risks and possible obstacles to the free movement of such products that have been irrigated with recycled water. These doubts should be resolved by promoting innovative systems and economic incentives that take into account the costs and socio-economic and environmental benefits of quaternary treated wastewater reuse [21].

The aim of this review article is to provide systematic insights into quaternary treatment of urban wastewater by different processes, such as filtration, coagulation, adsorption, ozonation, advanced oxidation processes and disinfection. This article contributes to a better understanding of the described processes with their advantages and disadvantages and also builds and maintains trust in wastewater reuse and promotes it as a possible alternative to reduce the risk of water scarcity for irrigation in the EU.

2. Filtration

One of the oldest methods of water treatment still in use around the world is filtration, which physically removes (separates) particles from the water. For the removal of suspended materials, granular media filters or membrane filters are used. The filtration efficiency depends on the size of the gaps between the filter media, or the pore size of the membrane used [22].

In filtration, depth or cake filtration can be distinguished. In depth filtration, particles are caught in the pore system of the medium through the attachment, whereas in cake filtration, a "cake" is formed on the surface of the medium, with most of the solids being removed at the surface [23].

2.1. Sand Filtration

Historically, the first filters used in water treatment were slow sand filters, which are still in use today. The slow sand filtration process provides treatment through physical filtration of particles and biological degradation in the sand bed [24]. This is followed by a process of adsorption, mechanical filtration and degradation, with the important physical factors that determine pollutant removal being the use of slow filtration rates (0.1–0.3 m/h) and fine sand [25]. Slow sand filtration techniques are characterized by their low capital cost and effectiveness in reducing pathogen load, removing turbidity, suspended solids, and toxic metals in treated water [26].

Rapid sand filtration is characterized by higher filtration rates between 5 and 15 m/h and also differs from slow sand filtration in the quality of the sand used. As in rapid sand filtration, much coarser sand is used with an effective grain size in the range 0.35–0.60 mm. The use of coarser sand results in the pores of the filter bed being relatively large and the impurities contained in the raw water penetrating deep into the filter bed [27].

2.2. Membrane Filtration

Membranes function as selective barriers, allowing the passage of components and retaining other components based on pore size, shape and chemical/physical properties. These membrane filtration systems are commonly known as pressure-driven processes [28].

These filtration processes can be classified as microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO). Microfiltration allows the separation of particles with an average size greater than 0.1 μm and varies the working pressure between 1 and 3 atm. Ultrafiltration membranes have pore diameters from 0.01 μm to 0.1 μm with working pressures from 2 to 7 atm. Nanofiltration membranes have pore diameters between 1 nm and 10 nm and working pressures of 5–20 atm. Reverse osmosis requires higher pressures of 30 to 50 atm as the pore size is 0.1 to 0.6 nm [29].

The process of membrane separation combined with activated sludge is referred to in wastewater treatment as a membrane bioreactor (MBR), while low-pressure membrane filtration, either microfiltration (MF) or ultrafiltration (UF), is used to separate the wastewater from the activated sludge [30]. As a biological unit for wastewater treatment, MBR has been proven to be effective in removing organic and inorganic matters [31]. MBR systems have several advantages including high performance compared to a conventional activated sludge precipitator (CASP) and no need for a secondary clarification, low workspace requirements, removal of micropollutants down to the discharge limits, complete retention of bacterial flakes by the membrane, and faster removal of persistent micropollutants [32].

Membrane technology is the advanced method of separation and water treatment, which is characterized by high efficiency, low energy cost, ease for continuous operation, and low footprint [33]. But one of the most challenging problems in membrane filtration is fouling, which reduces permeate flux, interferes with membrane selectivity or permeate quality, and increases membrane maintenance and replacement costs [34].

2.3. Applications of Filtration for Micropollutant Removals

Filtration as an effective water treatment method is applicable in quaternary wastewater treatment to remove micropollutants that are significantly present in the waters. Its removal efficiency can be demonstrated on selected significant micropollutants:

- **Microplastics**

More and more studies are devoted to the removing of microplastics as a global water pollutant. In the work published by Sembiring et al. [35], the effectiveness of rapid sand filtration (RSF) for the removal of microplastics (MPs) was determined. The MPs samples were made of plastic bags and flakes from tires ranging in size from 10 μm to more than 500 μm , and bentonite was added to represent turbidity in water. The removal efficiency using RSF with an effective size filter media of 0.39 mm and 0.68 mm was 85% to 97% [35].

Wolff et al. [36] showed that the efficiency of sand filtration as the final stage of treatment in municipal wastewater treatment plants was 99% in removing MPs.

The work of Bayo et al. [37] deals with the removal of MPs from the final effluent of an urban wastewater treatment plant by two methods, namely membrane (MBR) and rapid sand filtration (RSF). At influent, the mean MPs concentration was $4.40 \pm 1.01 \text{ MP L}^{-1}$ and the major isolated forms of microplastics were fibers with a concentration of $1.34 \pm 0.23 \text{ items L}^{-1}$. Both methods were found to be effective for MPs removal, with MBR efficiency and RSF efficiency being 79% and 76%, respectively. Selective particle removal was confirmed here, as the removal efficiency of microplastic particulate forms was much higher (99% for MBR and 96% for RSF), as compared to microfiber removal (58% for MBR and 54% for RSF) [37]. Filtration has been shown to be effective in removing MPs, however, significant differences in removal efficiency were observed for smaller plastics that are not removed efficiently. It has also been shown that the filtration process can break down the particles, creating larger amounts of very small microplastics [38].

- Microbial pollution

The protozoa, bacteria and viruses that make up microbial water pollution vary greatly in size but can be removed using different types of filtrations. The following filtration methods are required for the complete physical removal of certain groups of microbial pathogens: rapid and slow sand filters with coagulation with a filter size of $4 \text{ }\mu\text{m}$ for protozoa such as *Cryptosporidium* and *Giardia*; microfiltration with a filter size of $0.2 \text{ }\mu\text{m}$ for bacteria such as *E. coli*, *Salmonella* and *Cholera*; and ultrafiltration with a filter size of $0.01 \text{ }\mu\text{m}$ for viruses such as *Rotavirus*, *Norovirus*, and *Hepatitis-A* [22].

To successfully meet the drinking water specifications submitted by the respective countries, membrane filtration is effective in the removal of pathogens from water, either as a stand-alone unit or as a hybrid unit [39]. In Marsono et al.'s research [40], the overall immersed membrane microfiltration with a pore size of $0.05 \text{ }\mu\text{m}$ had a removal efficiency of 100% for *E. coli* and with a pore size of $0.07 \text{ }\mu\text{m}$, the removal of *E. coli* was 99.8% [40].

Incorporation of antimicrobial nanoparticles into membranes can improve removal efficiency, as confirmed in the work of Kacprzyńska-Gołacka et al. [41], in which they achieved complete inhibition of cultured colonies of Gram-negative (*Escherichia coli*) and Gram-positive bacteria (*Bacillus subtilis*) when silver oxide (AgO) was coated on the surface of polyamide microfiltration membranes.

- Pharmaceuticals and endocrine disruptors

Numerous studies have also looked at the use of filtration methods to remove pharmaceuticals and endocrine-disrupting chemicals. In a study by Maryam et al. [42] a loose nanofiltration membrane was used to filter pharmaceutically active substances namely diclofenac, ibuprofen and paracetamol at different pH values as these drugs are the most common type of non-steroidal anti-inflammatory drugs recorded in drinking and treated waters. The highest removal efficiencies of the selected drugs were obtained as follows: 99.74% removal of diclofenac at pH 3, 80.54% removal of ibuprofen at neutral pH and 36.16% removal of paracetamol at pH 12 [42].

The study by Couto et al. [43] investigated the rejection of betamethasone and flucanazole as pharmaceutically active compounds by nanofiltration and reverse osmosis. The ability of nanofiltration and reverse osmosis membranes to reject pharmaceuticals decreased with increasing permeate recovery rate, with the first appearance of pharmaceuticals in the permeate occurring at 40% and 60% permeate recovery rates for nanofiltration and reverse osmosis, respectively [43].

Membrane processes have shown their applicability for the removal of the most common endocrine disruptors found in water and wastewater, especially high-pressure and denser membranes such as nanofiltration and reverse osmosis [44]. More than 90% removal efficiency of a steroid hormone micropollutant 17β -estradiol using an activated carbon fiber-ultrafiltration composite membrane is reported by Zhang et al. [45].

Nakada et al. [46] achieved more than 80% removal efficiency of various pharmaceutically active compounds such as phenolic antiseptics, acid analgesics, anti-inflammatory

agents, antibiotics, endocrine disrupting phenolic chemicals, and natural estrogens using a combination of ozonation and sand filtration with activated sludge treatment.

- Pesticides

Membrane technologies can also be used as effective methods to remove pesticides from water [47]. The study by Mukherjee et al. [48] evaluated the removal efficiency of 43 pesticides from water using a thin-film composite polyamide membrane prepared by interfacial polymerization of 1,3-phenylenediamine and 1,3,5 trimesoyl chloride coated on asymmetric polysulfone support. Of the 43 selected pesticides, 33 were removed at more than 80%. The highest removal efficiencies were achieved for the persistent organochlorine insecticides, namely 100% endosulfan, 95% dichlorodiphenyltrichloroethane and 92% hexachlorocyclohexane [48].

Wang et al. [49] used macroporous membranes doped with micro-mesoporous β -cyclodextrin polymers for the separation of organic micropollutants including the pesticide 2,4-dichlorophenol and achieved removal efficiencies of more than 99.9%.

- Heavy metals

Technological advances in membrane development have promoted the increased use of membranes for the filtration and extraction of heavy metal ions from wastewater [50]. In the work of Qi et al. [51], using a positively charged NF membrane prepared by using 2-chloro-1-methyliodopyridine as an active agent to graft polyimide polymer onto the membrane surface achieved removal efficiencies of toxic heavy metal ions of 96% for CuCl_2 , 95.8% for NiCl_2 , and 98% for CrCl_3 .

High efficiency removal of heavy metal ions by nanofiltration membrane was achieved by Morandi et al. [52] using a membrane prepared by incorporating boehmite nanoparticles functionalized with curcumin into a polyethersulfone membrane; the ejection of Fe^{2+} , Cu^{2+} , Pb^{2+} , Mn^{2+} , Zn^{2+} and Ni^{2+} was measured at 99.88, 98.72, 99.61, 99.31, 99.11 and 99.51%, respectively.

In a study by Rezaee et al. [53], they evaluated the use of polysulfone (PSF)/graphene oxide (GO) nanocomposite membranes in terms of arsenate rejection from water, and the maximum rejection was obtained for PSF/GO-2 type at 83.65% at 4 bar pressure. It has been confirmed by many other works that membrane filtration methods have promising potential for practical applications for heavy metal removal [54,55].

3. Coagulation

A physical-chemical method that could be used for the quaternary treatment of wastewater is coagulation. This treatment technology is effective for the removal of pollutants [56,57] as well as the reduction of colloidal turbidity and suspended solids [58]. Coagulation is useful in protecting the environment and human health, while being simple, efficient and low in energy consumption [59].

3.1. Mechanisms of Coagulation

Coagulation is generally encountered as an intact process with flocculation. While the basis of coagulation is the addition of coagulants with rapid mixing, which cause destabilization and neutralization of suspended particles [60], during flocculation, destabilized particles are aggregated through gentle agitation that leads to the formation of larger particles called flocs [61]. The formed flocs are subsequently separated by filtration or sedimentation [62].

The coagulation can be categorized into four mechanisms: simple charge neutralization, charge patching, bridging and sweeping [63].

- Simple charge neutralization

Since most of the colloidal particles dispersed in the water are negatively charged, the process of charge neutralization occurs by adsorption of positively charged cations or polymers. When a metal salt coagulant or cationic organic polymers are added to the water,

they can be rapidly hydrolyzed to form various cations that interact with the negatively charged surface of the particles and neutralize them [61,64].

- Charge patching

The adsorption of polymers onto local sites of a particle causes patches of local charge reversal, resulting in a positive–negative attraction between the particles [65].

- Bridging

By adding non-ionic polymers or long-chain low-surface charge polymers alone or together with metal salts, they dissociate and form larger molecules. A particle attaches to the chain of one polymer, and other available active surface sites of other particles can attach to the remainder of the polymers since these polymers can have a linear or branched structure with high surface reactivity. A bridge is formed between the particles, as the polymer-colloidal groups can form an enmeshment, and thus the particles become heavier and that settles down the flocs [61].

- Sweeping

Adding a high concentration of metal salts to water causes the precipitate of amorphous metal hydroxides and forms heavier amorphous gelatin flocs [66].

3.2. Influencing Factors of Coagulation

The coagulation and flocculation processes are influenced by several factors, but in particular, they are influenced by speed and time of mixing, raw water properties, temperature, pH, type and dose of coagulant.

- Mixing

Mixing is an important factor in these processes as rapid mixing is required to promote the interaction of coagulants with suspended particles and the formation of microflocs, while slow mixing is required to promote the aggregation of microflocs into large flocs. The rate of floc formation may be reduced if the mixing speed is too low and the mixing time is too short. To achieve high flocks settling efficiency, it is necessary to form larger flocs, which may be limited if the mixing speed is too high and the mixing time too long [67].

- Coagulant type

Coagulants effective for pollutant removal can be classified into two main groups, namely chemical and natural coagulants. Chemical coagulants include hydrolyzing metallic salts (ferric chloride, ferric sulphate, magnesium chloride, and alum), pre-hydrolyzing metallic salts (poly aluminum chloride (PAC), poly ferric chloride (PFC), poly ferrous sulphate (PFS), poly aluminum ferric chloride) and synthetic cationic polymers (aminomethyl polyacrylamide, polyalkylene, polyethylenimine, polyamine). The most used coagulants in water and wastewater treatment worldwide are alum salts and PACs because they are considered to be widely available and have high treatment efficiency [68].

Due to their biodegradability and availability, natural coagulants represent an opportunity to improve nature and the ecosystem. The advantage of natural coagulants is that they are an environmentally friendly product, and their use does not have a negative impact on human health and the environment [69]. Natural coagulants include microorganisms (bacterial, microalgae, fungal), substances, seeds and plant extracts, starch, fruit waste, chitosan and isinglass [68,70,71].

- Coagulant dosage

For the effective removal of pollutants, a sufficient dose of coagulant is required to destabilize all the colloidal particles. However, with an excessive dose of coagulant, stabilization of suspended particles can occur, which reduces the efficiency of the process [72].

- pH

In the case of inorganic coagulants, pH directly influences the formation of existing hydrolysis product species because pH affects the hydrolysis and polymerization reaction of aluminum, iron ions, and charge density [63].

One important factor in assessing the suitability of using coagulation is the production of the voluminous sludge that is generated as a result of coagulation. The use of inorganic coagulants results in the production of large quantities of metal hydroxide toxic sludge, making it difficult to dispose of and causing an increase in the metal (e.g., aluminum) concentration in the treated water, which may have implications for human health [73].

Several studies have focused on regeneration of the coagulants from water treatment sludge and further reuse in water and wastewater treatment. Other sludge disposal alternatives are incineration, land application and landfilling [74].

3.3. Applications of Coagulation for Micropollutant Removals

- Microplastics

Coagulation has proven effective in removing microplastics from wastewater [75] and drinking water [76]. Ziembowicz et al. [77] investigated the removal efficiency of six different types of microplastics (three types of PE and three types of PVC) from tap water using $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ as coagulants. The microplastic removal effect reached its highest value at the initial neutral pH of tap water (pH 7) and when $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ was used at the coagulant dose of 0.05 g/L, i.e., 28–44% for PE types and 89–100% for PVC types. Similarly, they investigated the removal efficiency of MP via detergent-assisted coagulation and found that the addition of SDBS (sodium dodecyl benzenesulfonate) surfactant to tap water prior to the coagulation process resulted in removal efficiencies greater than 95% (Al-coagulant) and 80% (Fe-coagulant) for each of the microplastics tested [77].

Similarly, high PE removal efficiency, 84.9%, was achieved by Li et al. [78] using magnesium hydroxide formed under alkaline conditions with anionic polyacrylamide (PAMAM) as a dual coagulant. Hu et al. [79] achieved high removal efficiency of 100 nm 5.0 μm polystyrene microplastics by using poly-aluminum silicate sulphate (PSiAS) and poly-titanium silicate sulphate (PSiTS).

According to Gong et al. [80], coagulation was shown to be effective for the removal of nanoplastics when the removal efficiency was 96.6% from 50 mg/L of nanoplastics of carboxyl-modified polystyrene using 10 mg/L of aluminum chloride as the coagulant.

- Disinfection by-products

Coagulation can also be used to remove disinfection byproducts (DBP) resulting from reactions between disinfectants and natural organic matter [81]. Lin and Ika [82] observed superior DBP formation potential reduction of PACl. Wang et al. [83] compared the coagulation efficiency of three Al-based coagulants, aluminum sulfate, poly-aluminum chloride and a novel type of covalently bonded hybrid coagulant (synthesized using AlCl_3) for controlling DBP formation and DBP-associated toxicity. The results showed that the highest removal (by 50%) of the aggregated DBP concentration was obtained by using polyaluminum chloride at a dose of 1.5 [Al]/[DOC] [83].

- Turbidity

In the work of Dahasahastra et al. [84], a regenerated coagulant from a byproduct of the coagulation–flocculation–sedimentation process of drinking water treatment was used as a coagulant to remove turbidity, as it contains a significant concentration of aluminum. A 74% turbidity removal was achieved when 1 mL/l of regenerated coagulant was added to synthetic turbid water with effluent turbidity concentration < 20 NTU [84].

Coagulation is also very effective when combined with other methods such as adsorption. For example, Zahmatkesh et al. [57] used FeCl_3 as a coagulant in combination with adsorption on activated carbon, and 99% elimination of NTU was achieved.

The use of plant-based coagulants has also been shown to be effective for turbidity removal [85]. The results of Zedan et al. [86] showed that the removal efficiency was 84.6, 95.2, and 97.8% at initial turbidities of 13, 54, and 194 NTU using walnut seed extract doses of 3.0 mL/L. Ahmad et al. [87] achieved turbidity removal of 24.2% by using the plant *P. sarmentosum* at an optimum dosage of 5 g/L.

- Pharmaceuticals

Numerous studies have also looked at the use of coagulation to remove pharmaceuticals. In Tahraoui et al.'s work [88], the coagulants copper sulfate, ferric chloride and a combination of cupric sulfate and ferric chloride in a ratio of 1:1 were used. The best results using these coagulants on pharmaceutical plant effluent were obtained using a mixed coagulant ($\text{CuSO}_4 + \text{FeCl}_3$) at pH = 5 and dose = 600 mg/L with a DOC reduction of 97.3%.

The use of natural coagulants for the removal of pharmaceuticals is a promising method for wastewater treatment [89]. According to Nonfodji et al. [90], the use of a natural coagulant from *Moringa oleifera* seeds in the treatment of hospital wastewater resulted in turbidity removal efficiencies of 64% and COD of 38%. In the work of Iloamaeke and Julius [91], *Phoenix dactylifera* was shown to be an effective natural coagulant in the treatment of pharmaceutical wastewater.

- Heavy metals

The removal of heavy metals from water has received a great deal of attention due to their adverse effects on aquatic organisms and even human health. Coagulation is one of the available technologies for the removal of heavy metals from waters [92]. In Johnson et al.'s work [93], dosing 40 mg/L ferric chloride and 0.5 mg/L polymer achieved metal removal efficiencies of 95% lead, 92% chromium, 79% copper, 57% zinc, and 17% nickel.

The use of a natural coagulant to remove heavy metals has also been investigated. In Skotta et al.'s work [94], the use of *Lepidium sativum* mucilage as a bioflocculent in water treatment yielded Cu^{2+} and Zn^{2+} removal at 87% and 71%, respectively.

4. Adsorption

As we have seen in the previous chapters of this review, advanced water treatment methods come in different forms. They can bear chemical, physical or even biological resemblance, while alternatively it is possible to be a combination of all, thus creating hybrid systems. This availability of various processes is necessary if we are to remove pollution, and harmful compounds released into waters by anthropogenic activities [95]. Pollution does not have to be physical only; it is defined as a release of unwanted amounts of any particles (chemicals) such as organics and inorganics, but even as a form of energy (such as noise and light) into the environment. Certain amounts of these pollutants in nature can cause destructive or long-term negative effects on the ecosystem and living organisms [96].

There are diverse ways of purifying treated wastewater to acceptable values, after which, the water will not impact the processes it would be used for. In specific cases, it can improve the recipient (be it water bodies, processes or soil etc.). The principle of purifying process can be destructive or separative, and adsorption is part of the latter. Adsorption can be also used as an emergency water treatment process in case of polluted effluents during breakdowns. It will also serve as filtration media in that case [97]. The adsorption process is widely known as an efficient method for the removal of pollutants from wastewaters thanks to its adaptability, simplicity, ability to use adsorbent material again, and low cost and quite importantly, adsorbents are safe and do not pose a threat to the environment. Moreover, other advantages include their origin, regenerative ability, economic aspect and they are easily accessible [98].

It is important to note the difference between absorption and adsorption. While they have the common word "sorption", they are inherently different. The former happens in the whole volume of another material (typically liquid or gas), while the latter takes action only on the surface of the solid media. The removed substance is called an adsorbate, while the solid material is an adsorbent [99].

Adsorbents should generally have many pores with a large specific surface area. The solids can have a uniform or diverse particle size, containing homogenous or heterogeneous pores. Adsorbents should be selective to certain substances contained in the purified fluid, while being inert to the bulk of the fluid [100]. According to IUPAC classi-

fication [101]; adsorbents as porous materials are separated according to the nature and structure of their pores in classes as follows [102]:

- macroporous materials with pores structure > 50 nm and $d > 50$ nm,
- mesoporous materials with pores structure 2–50 nm and $d \approx 2$ –50 nm,
- microporous materials with pores structure < 2 nm and $d < 2$ nm.

The shape or structure of pores can be understood better with nitrogen (N_2) adsorption, which creates isotherms and hysteresis as shown in Figure 1 [103]. Corresponding pore shapes are included in the figure.

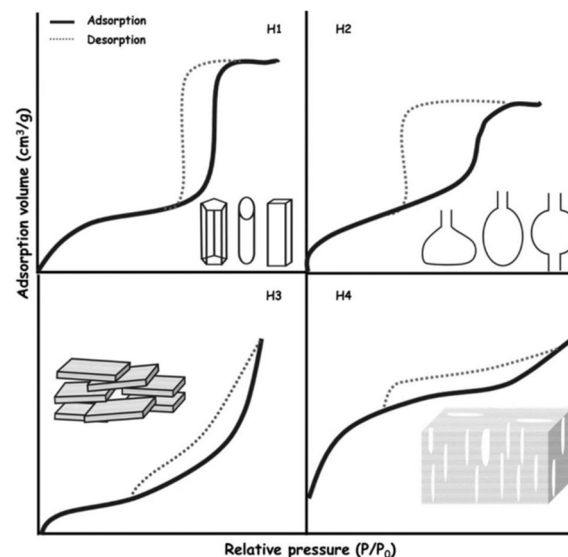


Figure 1. Adsorption according to pore shape [103].

We must remember that even in adsorption there is energy involved. Figure 2 [104] shows the possible interactions of adsorbate and adsorbent. In this regard, two possible options of adsorbate binding to the adsorbent surface are available. The nature of adhesion is reflected also in their names: physisorption and chemisorption.

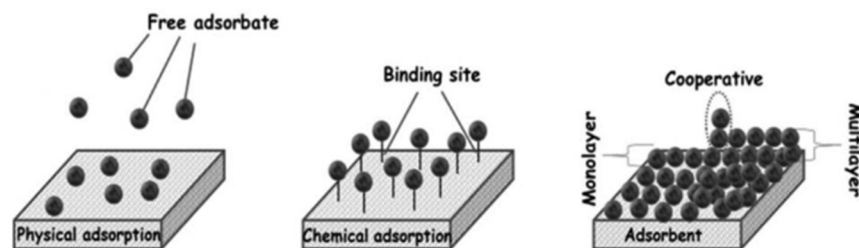


Figure 2. Difference between physisorption, chemisorption and mono/multilayer adsorption [104].

4.1. Physisorption and Chemisorption

As the name for physisorption suggests, the physical bonding is created by minimizing surface energy. Forces responsible for binding the adsorbate to the adsorbent could be Van der Waals, hydrogen bonding, electrostatic forces, and hydrophobic interactions with the first one being the most common [105]. Regarding the actual energy of adsorption, when we assume that these forces are similar to the ones of vapor condensation of the adsorbate, the released energy could be compared to adsorbate condensation energy at the given conditions. The overall nature of physisorption makes it possible for it to be a reversible process. The strength of a bond between the adsorbent and adsorbate is relatively weak, and the bond is easily disrupted by changing conditions, which leads to a release of a pollutant into the surrounding medium, generally labeled as desorption. Energy of adsorption in physisorption ranges usually between 8 and 40 kJ/mol, while

in chemisorption it goes over, reaching as high as 800 kJ/mol. This fact gives away the nature of the bond in chemisorption. A covalent bond is created between the adsorbent and adsorbate, altering the structure of the latter and simultaneously limiting sorption to the monolayer. While the overall sorption capacity of the adsorbent is not maximized by filling the pores, the chemical reaction is more selective, thus making chemisorption valuable. Desorption becomes ineffective and unpractical due to chemical change in the structure of the adsorbate [106,107].

4.2. Characterization of Adsorbents and the Adsorbent Process

There are multiple mathematical ways to characterize the process of adsorption: adsorption isotherms, kinetics and thermodynamics. Parameters which are used in adsorbent characterization designing are in Table 1 [108–110]. Parameters and graphical interpretation of previous characterization depends on multiple specifications of adsorbent.

Table 1. Parameters used in adsorbent characterization and designing of technologies [108–110].

Parameter	Symbol	Unit
Iodine number	IN	mg/g
Ash		%
Porosity	ε_p	–
Skeletal density	ρ_{He}	kg/m ³
Geometrical density	ρ_{Hg}	kg/m ³
Bulk density	ρ_s	kg/m ³
Specific surface	S_{BET}	m ² /g
Particle size	d	mm
Pore size	d_p	Å
Moisture	–	%

Adsorption isotherm, as the name implies, is a quantitative measurement of equilibrium between free adsorbate in the fluid and bonded on the adsorbent. It is also useful in designing an adsorption system and helps to describe the interaction between the adsorbent and adsorbate [111]. The adsorption process itself must abide hydrodynamics in the solution, which means that adsorption happens as shown in Figure 3 [112].

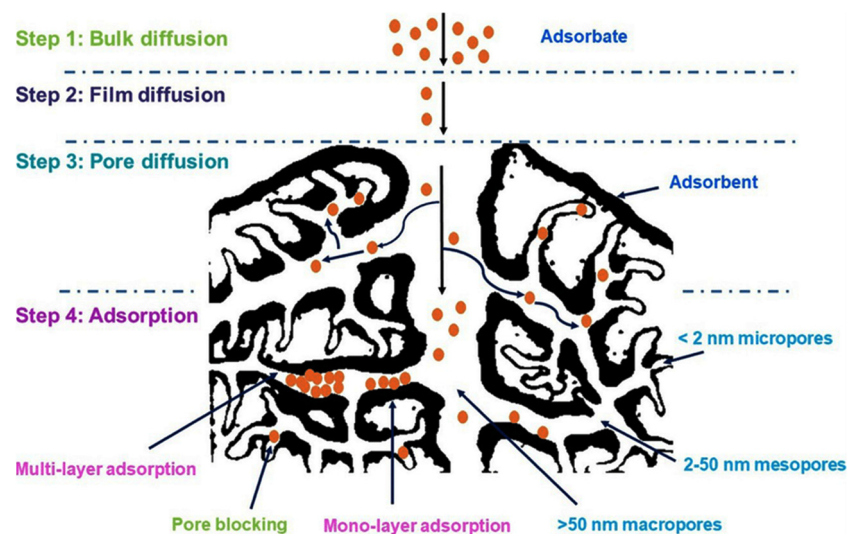


Figure 3. Adsorption mechanism of pollutants [112].

In simplicity, for adsorption to work, there needs to be 1. interparticle diffusion, 2. intraparticle diffusion, and 3. surface sorption [106,112]. As for the adsorption kinetics, commonly applied models are pseudo-first order (PFO), pseudo-second order (PSO),

Avrami and Elovich models [106]. An important note about calculation of kinetic equation parameters is inclusion of equilibrium data or close ones. Determining a better model for the process is biased, which is caused by a used method for the calculation. Because of this, PSO was leading as the most fitting model in numerous studies. First, the diffusion-controlled process is described by PSO more precisely than with PFO, although the former model cannot describe the steep rise in adsorption in a short time [113]. Moreover, the parameters in the PFO and PSO kinetic model do not have physical meaning, they are derived from multiple processes happening during the adsorption (for example combination of diffusion and adsorption). On the other hand, the parameters in the Elovich equation do have physical meaning [106,114].

As we have discussed in the previous text, adsorption seems like a simple process, but it has many applications in water treatment. We must always keep in mind that purifying water is needed and wanted, and as a result, we produce toxic or non-toxic solid waste. As a general consensus, materials used for adsorbents are cheap. Most of the carbon-based adsorbents can be prepared from waste materials such as tomato seeds, plum stones, coconut shell, and rice straw shell etc. Waste materials are not overlooked as before, environmental thinking affects research to look for alternative raw sorbent material sources. Activated alumina, zeolites, silica gel and activated carbon (from coal) are still commonly used for their specific uses, but their counterparts receive a lot of attention too [115,116]. One should always bear in mind the rules for a good adsorbent, which are the following [117]:

- (a). high selectivity for specific pollutants,
- (b). possibility of regeneration,
- (c). non-toxicity to humans and the environment,
- (d). non-corrosiveness to construction materials of the system,
- (e). low cost,
- (f). mechanical stability,
- (g). market availability.

4.3. Adsorption as a Method for Removal of Organic Micropollutants

As concerns about water availability become more prevalent, so has monitoring expanded to a more specific pollutant. They also can be referred as contaminants of emerging concern (CECs) or organic micropollutants (OMPs), which are mostly unregulated chemicals in the environment and their biological effects are not yet studied in detail, but they definitely pose threat for the aquatic environment and human health [118,119]. Stopping the rise of an antibiotic presence in an environment is essential for the protection of aquatic life and limiting the ability of the microbial population to gain resistance [120,121]. Regular wastewater treatment plants (WWTP) are not designed to remove CECs, but they are still able to reach a removal efficiency ranging from 30% to 65%. In municipal wastewaters, CECs are also present, which consist of pharmaceuticals and personal care products, endocrine disrupting compounds, disinfection byproducts, and fluorinated organic chemical compounds such as perfluorooctanoic acid and perfluorooctane sulfonate [122–124]. The presence of these chemicals and their concentration levels are achieved by overconsumption of medications and their irresponsible disposal. It is possible to remove these compounds from wastewaters by adsorption. For example, from 18 monitored CECs, only 1,4-Dioxane, Sucralose, and Iohexol showed no concentration decline in a 1.5 m bed depth of granulated coconut shell-based media. Flow through filter bed was 54–65 m³/d, which resulted in 45 min of contact time. A possible reason for the low removal efficiency of 1,4-Dioxane could be the low carbon partitioning coefficient, which in turn affects sorption [125]. For example, caffeine, carbamazepine, gemfibrozil, and sulfamethoxazole are documented to be removed to levels below detection limits [119,125].

Alternatively, modified adsorbent materials can be applied to treat wastewater containing pharmaceuticals. Sand filtration and a subsequent graphene-based nano-adsorbent reactor was studied. The reactor was patented under the name RECAM[®]. Used graphene

had a bulk density of 1.05–1.80 g/cm³ at 25 °C, mesoporous nanostructure with a surface area of 890 m²/g and a carbon content of 97.5%. The operation pressure was in a range of 0.4–0.6 bar with a flowrate of 4.3–5.5 mL/min resulting in a contact time of 75 min. The process was in use for days, resulting in a measured breakthrough curve with a breakthrough point at the 45–55 days mark for carbamazepine, diclofenac and ibuprofen. Interestingly, it was not observed for caffeine. At the start-up phase, the process was different for each chemical, with diclofenac being the shortest (14 days) and caffeine the longest (50 days). Over the duration of the process, removal efficiencies reached higher than 95% [126].

Adsorption Mechanism of Organic Pollutants

Adsorption for organic pollutants follows mechanisms such as partitioning, pore filling, electrostatic interaction, electron donor and acceptor interaction, and hydrophobic interaction. Additionally, adsorption of pharmaceuticals on nanotubes is believed to follow physisorption as their main mechanism [127].

Partitioning happens when the adsorbate diffuses into the pores of the biochar that has not yet been carbonized. After this step, adsorption of the organic adsorbate can proceed by adhering to the adsorbent surface. Generally speaking, adsorbents (or biochar) have higher volatile content and organic contaminants [128].

Pore filling occurs when mesopores and micropores are present in the adsorbent, as well as polarity of organic contaminants. Prerequisites for a good pore filling mechanism is little volatile matter content in the biochar and a lower concentration of organic contaminants in the liquid [128].

Electrostatic interaction occurs with ionizable organic compounds to the positively charged surface of the adsorbent. It is dependent on the pH and ionic strength of the aqueous solution. Lower pH favors this mechanism, while higher pH hinders the efficiency of the process. It was also experimented that adding NaCl to a solution with methylene blue resulted in lowered adsorption for the dye from 4.5 to 3 mg/g [128].

An electron donor and acceptor interaction is common with aromatic compounds as is adsorbents having a graphene-like structure. For biochar to have electron-acceptor/donor capabilities is dependent on the pyrolysis temperature. Below 500 °C, biochar acts as an acceptor and above this temperature it behaves as a donor. Sulfamethoxazole adsorption was studied on π -electron graphene-enriched biochar and great adsorption capabilities were noted, especially with adsorbate aniline-protonated rings. Another interesting point regarding this mechanism is atrazine adsorption enhancement due to chlorine and aromatic carbon interaction on the biochar surface [128].

Hydrophobic interaction occurs during adsorption, when neutral compounds are present. Reported organic pollutants such as benzoic acid, o-chlorobenzene acid and p-chlorobenzene acid obey this type of adsorption mechanism. Higher pyrolysis temperature improves the sorption processes connected to this mechanism [128].

4.4. Heavy Metal Removal from Wastewaters by Adsorption

The presence of heavy metals in wastewater can also be natural, but in the last few centuries, it has been to a substantial extent achieved by anthropogenic activities. Mining, battery, nuclear, textile, and tannery are all industries which produce important materials or products we cannot live without, but their side effects are affecting human health and the environment [129]. Generally, activated carbons are used as adsorbents to treat water containing heavy metals. The ability to remove heavy metals is dependent on a microporous structure, large surface area and chemical complexity. For adsorption of heavy metals, regular activated carbon can be used, but alternative cheaper variants are studied. This includes biochar, byproducts, agricultural waste, seafood waste, food waste and soil particles. There is also the possibility to use natural zeolites, natural diatomite, and natural clay, etc. since they are economically viable [130–132].

4.4.1. Effect of pH

The effect of pH plays a key role in many chemical processes and the adsorption of heavy metals is not any different. It affects the ionization and solubility of metal ions and surface functional groups. For Cr^{6+} ion, a pH as low as 2 is preferred, but the exact process conditions depend on the metal ion speciation and functional groups situated on the adsorbent surface. A possible explanation is dichromate species being present at such low pHs [133,134]. Cr^{6+} can be also reduced to Cr^{3+} by electron donor groups on the adsorbent. Regarding Cr^{4+} , 95% adsorption potential was achieved with rice husk carbon. Adsorbent amount, temperature, time and optimal pH at 12 were observed. By applying coconut jute carbon for adsorption, 99.8% removal efficiency was reached. Optimal conditions were low Cr^{4+} concentration and low pH. Low pH also favors the adsorption of As^{3+} . Specifically, for hydrochars that were modified by KOH, cadmium adsorption efficiency was recorded best for pHs ranging from 4.0 to 8.0. Metallic ions like Cu^{2+} , Zn^{2+} , and Pb^{2+} have an optimal pH for adsorption ranging from 4.0 to 6.0. As for the adsorption capacity of metal ions, it increases in the following order: Zn^{2+} , Cd^{2+} , Cu^{2+} , Fe^{2+} , Pb^{2+} [133–135].

4.4.2. Effect of Temperature

The temperature effect on heavy metal adsorption is supported by multiple research papers to be positive at higher temperatures. This fact is probably linked with possible chemisorption and creation of multiple active sites, subsequently rising adsorption capacity. Keeping the previous information in mind, rising temperatures way too high would make the adsorption process worse. Heavy metal adsorption could also be an endothermic process, which would explain this behavior [136–138].

4.4.3. Effect of Contact Time

Rising contact time for heavy metal adsorption processes achieved better results for different adsorbents. It was noted that adsorption is quick at the start and reaches equilibrium gradually [138].

4.4.4. Adsorption Mechanisms of Heavy Metals

There are various variables such as specific areas of the adsorbent and surface-active functional groups, and the mechanism can vary from metal to metal. The most prominent adsorption mechanisms for heavy metals are physical adsorption, electrostatic adsorption, precipitation, ion exchange, complexation, and reduction [128,138].

It was demonstrated that physical adsorption is most common with heavy metals such as As, Cd, Zn and U, which are immobilized on the surface. Higher temperatures seem to provide better removal by altering the biochar structure [128,138].

Ion exchange is connected to negatively charged surface groups on the adsorbent and positively charged one on heavy metal ions. Contrary to physical adsorption, cation exchange capacity decreases with pyrolysis temperatures higher than 350 °C. The force that is responsible for the binding is called Coulombic. It was reported that heavy metal ions such as Cd^{2+} and K^{+} in water solution abide this form of the mechanism. K^{+} occurred on the deprotonated functional groups, while Cd^{2+} was affected by two different cation- π bonding mechanisms. Also, the adsorption between Hg^{2+} and Zn^{2+} was measured and compared and the former adsorbed much higher the latter. This type of adsorption mechanism has low adsorption capacity and is very pH dependent. Reports state that the higher iron oxide content in the adsorbent material, the better the cation exchange capacity [128,138].

The electrostatic adsorption mechanism is keen to take place in adsorbents with plenty of negatively charged active sites on them. The strength of this bond is related to the pH of the solution, ionic radius, valence state of the heavy metal, and zero potential of the biochar. It was reported multiple times that Hg followed this mechanism of adsorption on the biochar. The described mechanism is also connected with redox reactions, which can be seen with Cr^{6+} adsorbing to the surface thanks to electrostatic forces and then reduced to Cr^{3+} by elemental carbon [128,138].

There is also a possibility of the precipitation effect taking place during adsorption, which is caused by the presence of functional groups such as PO_4^{3-} and CO_3^{2-} . These groups are contained in larger numbers in animal-based biochar, compared to plant-based ones. Heavy metal ions such as Cd^{2+} , Pb^{2+} and others can form stable elements, for example, CdCO_3 and PbCO_3 [128,138].

Complexation was reported with adsorbents (biochar) made at lower pyrolysis temperatures, containing phenolic, lactonic and carboxyl functional groups. Oxidation of the adsorbent surface makes it easier for heavy metals to form complexes. Plant-based raw material is mentioned as being a better starting material for biochar production, due to greater content of the previously mentioned functional groups. Heavy metals which were observed to abide this mechanism are Cu, Cd, Ni, and Pb [128,138].

4.5. Control of Disinfection Byproducts Contained in Wastewaters with Adsorption

If we want to achieve highly purified water, disinfection is often needed. Generally, the water would be used as drinking water, but with recent environmental problems, water is used for different applications, which also demand disinfection. There are several ways to disinfect water and using chemicals is one of them.

The combination of ultraviolet (UV) light and chlorine or chloramine generates highly reactive molecules such as hydroxyl radicals, reactive chlorine species, chlorine radicals, dichlorine radical anions and chlorine monoxide. These species reliably destroy the ability of microorganisms to reproduce or outright kill them [139]. Albeit, using such methods can lead to the formation of halogenated disinfection byproducts (DBP) [140].

A study that applied ozonation and subsequent treatment of the sample (wastewater from municipal water reclamation plant, Singapore) to an adsorption column showed positive removal efficiency for possible halogenated precursors. The BAC column consisted of granulated activated carbon that had an effective particle size of 0.86–1.00 mm, apparent density of 0.5 g/cm^3 and uniformity coefficient of 1.7. Rinsed GAC was inoculated with activated sludge obtained from a water reclamation facility, with a mixed liquor suspended solids concentration of 3500 mg/L. After BAC treatment, wastewater parameters TOC, UVA_{254} , and SUV_A were significantly lowered from 8.74 mg/L, 0.062 mg/L, and 0.71 mg/L to 3.85 mg/L, 0.014 mg/L, and 0.36 mg/L. Overall O_3 -BAC treatment had great removal efficiency for aromatic compounds, nitrile organic matter and disinfection byproducts formation potential (DBPFP). Removal of DBPs achieved 46.0% efficiency in the raw wastewater. DBPFP, trihalomethanes (THMs) and haloacetic acids (HAAs) reached removal efficiencies of 81.5–97.5%. The process lowered the formation potential of THM by 87.6% and more than 99% for trichloronitromethane (TCNM), halo ketones (HKs), and haloacetoneitrils (HANs), while HAAs achieved only 29.7% reduction [141].

4.6. Removal of Micro/Nanoplastics by Adsorption

Activated carbon and graphene materials are used as adsorbents for microplastics and bioplastics removal to a significant extent; this is because of their availability and price [142,143]. Biochar was studied as an adsorbent material to remove polystyrene microbeads. Biochar was prepared from corn straw and hardwood feedstock and was directly applied into sand filtration to increase microplastics removal. The sand filtration removal efficiency increased from 60–80% to 95% by using the modified system. The mechanism showed that larger microplastics were stuck between particles and smaller ones were trapped in biochar pores. During the process it was reported that biochar formed larger particles (described as colloidal) and trapped even more microplastics, which made them immobile [144].

Modified biochar materials are also viable for microplastic removals, as it was presented in [145,146]. Biochar was modified by impregnating iron nanoparticles into the structure, which granted intensified magnetic and surface qualities to the material. Then it was tested for nanoplastics adsorption removal and the researchers' results showed that the process achieved 100% removal efficiency, which in comparison to 75% of raw biochar is a

great difference. Moreover, they concluded the effect of solution pH on the process, which seemed to have almost none. Characteristics of the adsorption principle was discussed to be surface complexation and electrostatic interactions between the nanoplastics and nanoparticles. Regeneration was successful due to retainment of adsorption capacity.

Adsorption of microplastics with different shapes was experimentally tested by pyrolyzing pine and spruce bark at a temperature of 475 °C. Its morphology was subsequently modified through steam activation at 800 °C. The monitored microplastics species were spherical polyethylene microbeads (10 µm), cylindrical polyethylene pieces (2–3 mm), and fibers of a fleece shirt. The results show a more promising removal of larger particles, almost 100% retention for fleece shirt fibers and a complete one for cylindrical polyethylene particles. The negative outcome of the study is low removal efficiency for polyethylene microbeads [147].

5. Advanced Oxidation Processes (AOPs)

Advanced oxidation processes (AOPs) are new, highly effective technologies to eliminate persistent micropollutants by utilizing the oxidizing potential of reactive oxygen species (ROS) generated in situ via chemical or physical pathways. ROS are a group of radicals, ions or molecules that have at least one unpaired electron. There are two types of ROS—free oxygen radicals and non-radical ROS (Table 2) [148–152]. The hydroxyl radical ($\bullet\text{OH}$) is the most commonly used in AOPs technology due to its advantages such as non-selectivity, high reactivity and ease of formation. It is also often considered a green harmless oxidant because non-toxic byproducts are formed when pollutants are oxidized [151]. The chemical AOPs method in the aqueous phase can initiate the formation of $\bullet\text{OH}$ radicals in the presence of chemical precursors such as O_3 or H_2O_2 , with or without catalysts or promoters. Some physical methods of AOPs can directly initiate $\bullet\text{OH}$ radical formation in the aqueous phase without the presence of promoters. Such methods include photolysis, sonolysis, radiolysis or supercritical water oxidation [150].

Table 2. Example of ROS [148–152].

ROS as Free Oxygen Radicals	Non-Radical ROS
Hydroxyl radical $\bullet\text{OH}$	Hydrogen peroxide H_2O_2
Superoxide anion radical $\text{O}_2^{\bullet-}$	Singlet oxygen $^1\text{O}_2$
Alkoxy radical $\text{RO}\bullet$	Ozone/trioxygen O_3
Peroxy radical $\text{ROO}\bullet$	Organic hydroperoxides ROOH
Hydroperoxide radical $\bullet\text{OOH}$	Hypochloride HOCl
Nitric oxide $\text{NO}\bullet$	Peroxynitrite ONOO^-
Thiyl radicals $\text{RS}\bullet$	Nitrocarbonate anion $\text{O}_2\text{NOCO}_2^-$
Sulphonyl radicals $\text{ROS}\bullet$	Dinitrogen dioxide N_2O_2
Thiyl peroxy $\text{RSOO}\bullet$	Nitronium NO_2^+
Sulphate radicals $\text{SO}_4^{\bullet-}$	Highly reactive lipid- or carbohydrate-derived carbonyl compounds

In general, AOPs occur in two phases. In the first phase is the production of radicals and the second phase is the reaction of these radicals with micropollutants present in the wastewater [149,153]. Based on the in situ radical production, AOPs can be categorized into four groups (Figure 4). Chemical AOPs use a chemical reagent and a catalyst; photochemical ones are based on the use of a solar energy or a UV source. An electrical source is used to generate radicals in electrochemical methods of AOPs. Sonochemical AOPs use an ultrasound in the first phase to generate radicals [151,154].

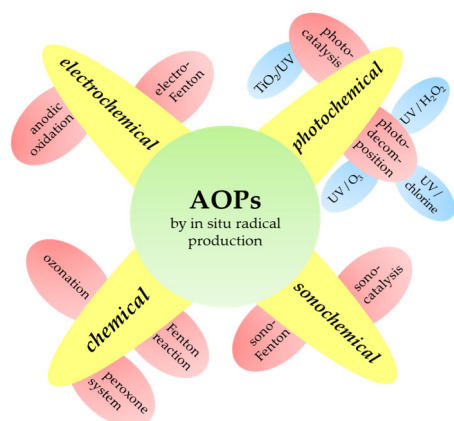
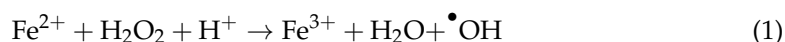


Figure 4. Some types of AOPs by in situ radical production.

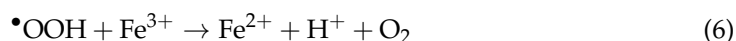
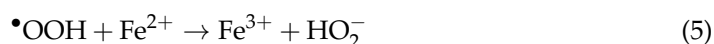
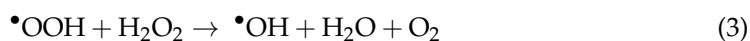
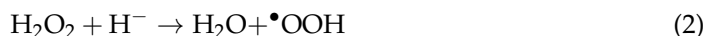
5.1. Chemical Types of AOPs

5.1.1. Fenton's Reaction Technique

The Fenton reaction is a type of chemical AOP. The principle is the use of hydrogen peroxide and ferrous ions to generate hydroxyl radicals at an acidic pH. The mechanism of formation of hydroxyl radicals in the Fenton reaction involves approximately 20 reactions. However, in general, the equation can be written according to Equation (1) [155].



In the reaction mechanism, in addition to hydroxyl radicals, hydroperoxide radicals are also formed as intermediates (Equation (2)), which can subsequently react with hydrogen peroxide (Equation (3)), hydroxyl radicals (Equation (4)) or iron ions (Equations (5) and (6)) [156,157].



The efficiency of organic pollutant degradation in Fenton's reaction technique depends on several parameters such as pH, concentration of chemical reagent and initial organic pollutant content. pH is a key parameter for the Fenton reaction. The optimal pH range is 2–4 and is connected to the nature of the reagents. If the pH was neutral or alkaline, there would be risk of iron precipitating in a form of insoluble compounds and additionally, losing its catalytic abilities. At a lower pH than the previously mentioned range, the scavenging effect of $\bullet\text{OH}$ by H^+ would be too prominent, which would result in inhibition of the Fenton degradation effect. However, most wastewaters have a pH value higher than the optimal range. For this reason, it is necessary to adjust the pH value of the wastewater before the Fenton reaction itself, but at the same time, it is necessary to adjust the pH value even after the treatment process in order to achieve suitable outlet pH of the treated wastewater. From this point of view, the Fenton reaction is an expensive process because large amounts of chemicals are required [158].

Zhao et al. [159] applied the Fenton reaction to remove recalcitrant dissolved organic matter. Their results show 77.7% removal efficiency at the optimal dosage of 11.5 g H_2O_2 /L and Fe^{2+} / H_2O_2 ratio of 1:20.

In a review by Wang et al. [160], the removal of microplastics (MPs) and nanoplastics (NPs) by the Fenton reaction is mentioned. The Fenton reaction could change the chemical structures of MPs, reduce the particle size of MPs or mineralize MPs. This is mostly

caused by $\bullet\text{OH}$ attacking the C–H bond to form a C–O bond and destabilizing the micro/nanoplastic structure. If the C–C bond is attacked, the chains become separated and the larger particles crumble into smaller ones, generating a greater reaction area for the radicals to attack [161].

According to [162], Fenton reactions are effective for the inactivation of pathogenic microorganisms in various types of wastewaters.

The electro-Fenton reaction is in situ generation of H_2O_2 in the medium on the electrode by a process called oxygen reduction reaction (ORR). Dissolved oxygen is the main part of H_2O_2 generation. As for the cations, Fe^{2+} is added to the system from external sources. Due to the electrochemical nature of the process, iron ions are regenerated during the reaction [163]. The electro-Fenton reaction, just as the classical homogeneous Fenton reaction, suffers from pH prerequisites, resulting in high chemical usage and creation of unwanted sludge [164].

Electrochemical peroxidation (EP) is based on in situ Fe^{2+} generation thanks to the iron (steel) anode while H_2O_2 is externally added to the reaction mixture. In a study there was direct comparison of a substance named Reactive Black 5 (RB5) with classical Fenton for dye removal. It was shown that in simulated wastewater, classical Fenton was faster and gave more satisfactory results compared to EP. This partially happened with coagulation, but effective azo bond destruction was still observed, which caused discoloration of the sample. In real wastewater fast discoloration happened too but this was probably caused by the conditions. Forty percent color removal was noted in the real wastewater. During the EP process, H_2O_2 was introduced in the sample from the beginning but the $\bullet\text{OH}$ creation lasted during the whole reaction time and Fe^{2+} needed for the reaction in both simulated and real wastewater, was available for the reaction thanks to slow release from the anode. The dye was gradually removed from the samples by degradation and not by coagulation [163].

The heterogeneous Fenton reaction consists of either iron minerals (for example magnetite– Fe_3O_4), iron-based composite catalysts, iron-based semiconductors, iron-modified porous materials and externally added H_2O_2 . The huge advantage of this process is independence from pH, and it consumes less H_2O_2 than the classical Fenton reaction. The availability of iron minerals is high, and their cost is rather quite low. To some extent, the heterogeneous Fenton reaction is present even in soil because of iron minerals and H_2O_2 can be generated by photochemical reactions using iron oxides [165].

The heterogeneous electro-Fenton is a mix of the electro-Fenton and heterogeneous Fenton reaction, albeit without electro-Fenton disadvantages. Used iron carriers such as Fe_3O_4 , support the mixture with Fe^{2+} and Fe^{3+} cations available for oxidation or reduction. It is not dependent on pH and its use of H_2O_2 is less intense due to lower iron abundance in the mixture. There is also a higher ability of the iron to regenerate due to the electrochemical nature of the process. Studies proved that modified iron carried by metal ions such as Ni can assist with H_2O_2 activation [164].

5.1.2. Ozone-Based Processes

Ozone-based processes are oxidative technologies that use a strong oxidizing agent, specifically ozone. Ozone reacts nonspecifically and easily with pollutants due to its high reduction potential (2.07 V vs. SHE) and reactivity. Ozone-based processes can be used to color and odor COD, nutrients or micropollutants removal. Ozone can react with pollutants through two reaction pathways—direct and indirect reaction. In the direct reaction, ozone molecules react directly with the pollutant. This reaction is also called ozonolysis [166–169].

Ozonolysis is a direct reaction of ozone with pollutants. In general, it is a selective reaction with a slow reaction rate. The unsaturated bond in pollutants is split by ozone according to the Criegee mechanism. Ozonolysis is technically and economically a feasible treatment process to eliminate micropollutants in wastewaters. For example, the removal efficiency of particular cytostatic compounds, antibiotics and other dissolved organics (like phenols, aniline, thioethers, aromatics, amines, pharmaceuticals) is more than 90% [169]. Ozonolysis is also effective in the elimination of organochlorine pesticides [170]. Tripathi

and Hussain [168] indicated that ozonolysis is an attractive disinfectant for inactivation of both virus and bacteria. The elimination efficiency of different viruses and bacteria is between 78 and 99.9% by ozonolysis.

In the indirect reaction, reactive oxygen species are generated from ozone, which react with the pollutant. These two reaction pathways lead to different oxidation products. Indirect reaction belongs to AOPs and it is usually catalyzed by ozonolysis. For example, hydrogen peroxide, UV radiation, iron ions or an alkaline condition can be used as a catalyst [166–169].

Due to the fact that ozone is an unstable gas, it must be generated in situ. In situ generation of ozone can be from air, pure oxygen or water using some form of energy (Table 3).

Table 3. Different methods of ozone generation [166–169].

Method of Ozone Generation	Principle	Ozone Source
Electrical	Electrical discharge	Air or O ₂
Electrochemical	Electrolysis	Water (highly purified)
Photochemical	Irradiation ($\lambda < 185$ nm)	Air, O ₂ , water
Radiation chemistry	X-rays, radioactive γ -rays	Water (highly purified)
Thermal	Light arc ionization	Water

The most widely used method of ozone generation is the electrical method via dielectric barrier discharge (DBD). In dielectric barrier discharge ozone generators, ozone is produced using energy from electrons in an electric field between two electrodes. At least one of the electrodes must be covered with a dielectric material. The electrodes are separated by a gas-containing space or gap. When a high voltage alternating current is created between two electrodes, the gas is ionized. The resulting ions act as charge carriers for the second electrode, and when they collide with an oxygen molecule, ozone molecules are formed. The formation of ozone molecules can be described by Figure 5 [169–172].

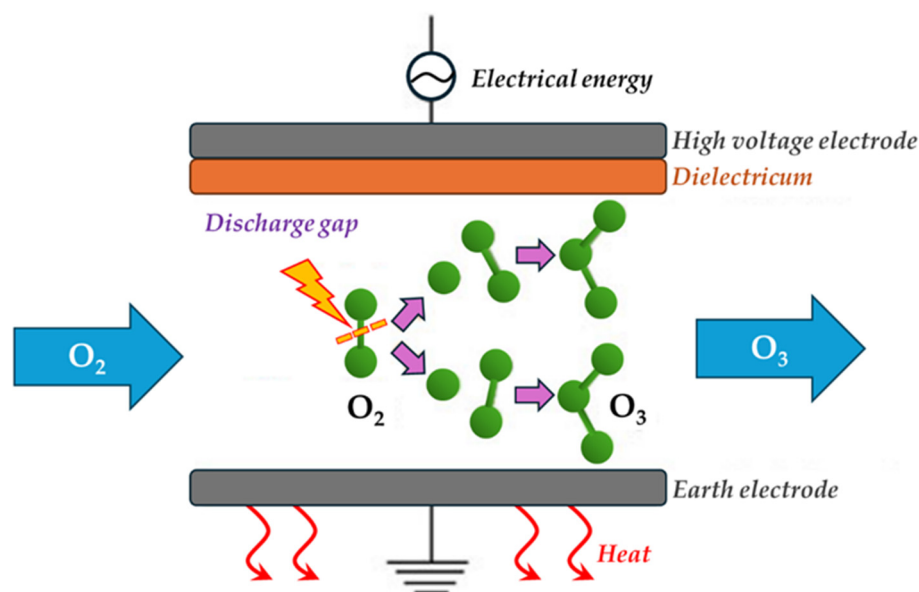
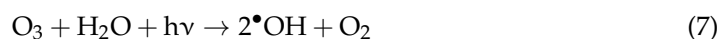


Figure 5. Principle of dielectric barrier discharge [171].

The combination of ozone and catalyst such as UV, H₂O₂ or iron ions belongs also to chemical AOPs due to the generation of reactive oxygen species (ROS).

The combination of ozone and UV is also defined as photochemical AOPs. The principle is using UV for the photodecomposition of ozone in aqueous media. Photolysis of ozone leads to hydroxyl radical formation (Equation (7)). Hydroxyl radicals can react not only with pollutants, but conversion to peroxy radical occurs with the reaction of hydroxyl radicals with ozone molecules (Equation (8)) [151,173].



The treatment process O_3/UV has benefits such as disinfection and toxicity reduction. O_3/UV can be used to abate ozone-resistant trace organic contaminants more efficiently and lead to less toxic byproducts formation in comparison with ozonation alone. The removal efficiency of some antibiotics such as carbamazepine, ciprofloxacin, clarithromycin, diclofenac, and sulfamethoxazole reach around 80–100% with the O_3/UV combination [166].

Presumido et al. [174] investigated a membrane ozone contactor for wastewater treatment. The application of a small dose of UV radiation had no effect on the oxidation of contaminants of emerging concern but had a positive effect on the reduction of microbial contamination. Despite the high reduction of potentially harmful bacteria, the reduction effect was transient. Microorganisms that survived the O_3/UV treatment process were able to grow back to their original concentrations. However, this process requires high energy to power the UV lamps and the ozone generator [173].

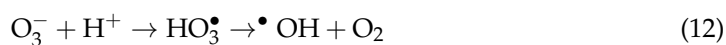
The peroxone technique combines ozone with hydrogen peroxide (H_2O_2). Supposedly, it is the most studied and implemented AOP based on ozonation [156,164]. The H_2O_2 molecule in aqueous media is dissociated into hydrogen cations and hydroperoxide anions (Equation (9)). Ozone reacts with hydroperoxide anions to form hydroperoxide radical and ozonide ions (Equation (10)) [151,169].



Ozone can also directly react with H_2O_2 to produce hydroxyl radical and ozonide ions (Equation (11)).



In both reaction mechanisms of the peroxone technique, ozonide ions are formed. The reaction between the ozonide ions and the hydrogen cation leads to the production of more hydroxyl radicals (Equations (12) and (13)) [151,169].



The reaction rate and efficiency of the peroxone technique is higher than ozonation alone. However, an excessive amount of H_2O_2 acts as a radical scavenger, so it is necessary to find the optimal dose of H_2O_2 [173]. In [175], the removal of 70 organic micropollutants and microbial contamination by ozonation and peroxone technique in sewage effluent was studied. The peroxone technique significantly decreased bacteria/virus inactivation compared to ozonation and the impact of adding H_2O_2 was insignificant to organic micropollutants removal.

5.2. Photochemical Types of AOPs

5.2.1. Photodecomposition Technique

As the name suggests, photodecomposition techniques are classified as a photochemical AOP. The generation of ROS is usually based on the decomposition of precursors such as ozone, hydrogen peroxide, chlorine or peroxodisulphates via ultraviolet radiation.

The combination of UV (200–300 nm) and H_2O_2 is the most frequently used UV-AOPs at full-scale [176,177]. The presence of UV breaks the O-O bond, resulting in two hydroxyl radicals according to Equation (14). Subsequently, the hydroxyl radical can participate in the degradation of harmful substances or further react with hydrogen peroxide to form a hydroperoxide radical (Equation (15)) [151].



Photodecomposition AOPs based on the UV/ H_2O_2 are typically affected by pollutant type and concentration, light transmittance of solution, pH, temperature, and H_2O_2 concentration. H_2O_2 concentration affects the process most significantly. If there is an excess of H_2O_2 , there is a decrease in the efficiency of pollutant degradation, because the H_2O_2 also acts as a radical scavenger. If the H_2O_2 concentration is too low, a small number of radicals is formed. For each type of wastewater, a sample is required to find the optimal dose of H_2O_2 [151,178].

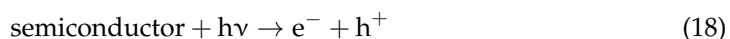
Another photodecomposition AOPs technique is used the decomposed of free chlorine by UV (UV/chlorine). In water treatment, free chlorine consists of HOCl and OCl^- and it is usually used as an oxidant and disinfectant. Photolysis of these precursors produces not only ROS but also reactive chlorine species (Equations (16) and (17)).



Miklos et al. [176] compared different UV-AOPs processes in municipal wastewater treatment. They focused on the removal of trace organic compounds and demonstrated that the combination UV/chlorine is more effective than UV/ H_2O_2 . Also, UV/chlorine has higher compound selectivity. However, negative oxidation byproducts might be produced by the UV/chloride technique, while negative byproducts have not yet resulted from the UV/ H_2O_2 technique [166]. Similar results for total organic carbon (TOC) removal were presented in [179]. TOC removal efficiency was higher by UV/chlorine [179]. According to [180], the UV/chlorine combination is a more cost-effective process than UV/ H_2O_2 . The benefits of UV/chlorine are higher removal efficiency, lower energy consumption and easy construction [180].

5.2.2. Photocatalysis Technique

In general, photocatalysis is considered as the acceleration of a photoreaction by the presence of a semiconductor catalyst. Photocatalysis is based on the absorption of the photons by the semiconductor. Results of absorption is excitation of electrons (e^-) from the valence band to the conduction band and formed holes (h^+) in the valence band (Equation (18)). The formed holes scavenge molecules of water (Equation (19)) or hydroxide anions (Equation (20)) to generate hydroxyl radicals and the excited electron react with dissolved oxygen in water (Equation (21)) to generate a superoxide radical anion ($\text{O}_2^{\bullet-}$) [151,181].



Wide-band gap semiconductors based on metal oxide such as titanium dioxide (TiO_2), zinc oxide (ZnO) or bismuth-based oxides are usually used in wastewater treatment technologies. The preference of ZnO semiconductors is due to its chemical stability, high electrochemical coupling coefficient and high photostability. TiO_2 is the most preferred

due to its stability, high activity, non-toxicity, low cost and chemical/biological inertness [151,170,182].

In a study by Martínez-Escudero et al. [183], the degradation of three antibiotics (erythromycin, larithromycin, sulfadiazine) was compared in a wastewater treatment plant effluent by photocatalysis using UV/TiO₂ and UV/ZnO. Results show that using UV/TiO₂ is more effective for the removal of these three antibiotics.

It is possible to use photocatalysis for micro/nanoplastics removal. About 25% removal efficiency was achieved using UV/TiO₂ [184,185].

The photocatalysis technique also includes methods such as the Photo-Fenton process, which is a combination of UV/Fe²⁺/H₂O₂. In the basic Fenton reaction, sludge containing Fe³⁺ is formed, while in Photo-Fenton, Fe³⁺ is photolytically reduced to Fe²⁺. Ultimately, this reaction regenerates the Fe²⁺ catalyst [151]. For example, Beyazit and Karaca [186] report that the combination of UV and the Fenton process causes an increase in COD removal efficiency compared to the Fenton process alone.

5.3. Electrochemical Types of AOPs

5.3.1. Anodic Oxidation Technique

The anodic oxidation technique (Figure 6) is the simplest and most popular electrochemical advanced oxidation process [187]. This is a surface-controlled process. Anodic oxidation is characterized by the generation of hydroxyl radicals (•OH) on the anode surface (M) by oxidation with water without using chemical reagents [151]. Based on the oxygen evolution potential, the anodes used in anodic oxidation are divided into active and inactive. The active anodes have low oxygen evolution potential and may interact strongly with the generated free radical and its oxidation to chemisorbed oxygen or superoxide is increased [188]. These anodes are constructed from mixed metal oxides (e.g., oxides of ruthenium, iridium and platinum) [189]. The inactive anode has a higher oxygen release potential and weak interactions with the radicals are formed, thus allowing them to react with pollutants [188]. Due to this fact, inactive anodes are considered as ideal anodes for the mineralization of organic pollutants [190].

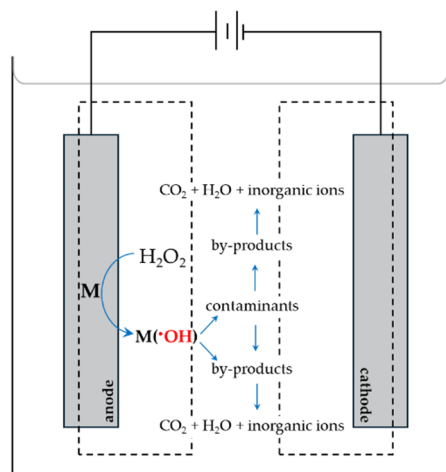


Figure 6. Schematic representation of the mechanism in anodic oxidation [189].

The boron-doped diamond electrode (BDD) is the strongest known inactive anode. It also has good stability and strong corrosion resistance. PbO₂ and SnO₂ are the two most common inactive metal oxide anodes for anoxic oxidation. Their advantages include strong oxidation ability, high oxygen evolution potential, excellent electrical conductivity and low cost [187,190]. Despite many advantages, the negatives probably outweigh the positives for active commercial use. There have been reports about optimization issues of the process and high manufacturing costs connected with the processing of gases and substrate materials which have not yet been solved. Another disadvantage is the worse

measurement of samples due to mistakes made by permeate getting between the electrode and epoxy resin, which is often applied to protect the measurement surface [191].

The authors of [192] summarize dye, pharmaceuticals, and pesticide degradation by electro-AOPs. It is shown that anodic oxidation has more than 90% efficiency removal of these pollutants.

5.3.2. Electro-Fenton Technique

The Fenton reaction can be improved by using an electrical source (electrode). The electro-Fenton technique (Figure 7) is based on the continuous electrogeneration of hydrogen peroxide (H_2O_2) via an oxygen cathodic reduction acidic pH value (2.8–3.5). Injected oxygen is from air or pure gas [193]. There is also a reduction of Fe^{3+} on the surface of the cathode, which means that the Fe^{2+} used in the Fenton reaction is continuously regenerated. This maintains the activity of the $\text{Fe}^{3+}/\text{Fe}^{2+}$ cycle to form homogeneous $\bullet\text{OH}$ with minimum iron hydroxide sludge precipitation [194,195]

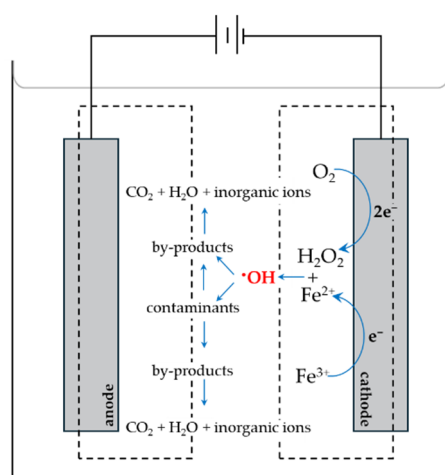


Figure 7. Schematic representation of the mechanism in the electro-Fenton technique [189].

Electro-Fenton is undeniably an efficient method for the degradation of persistent organic contaminants in various types of wastewaters [196].

5.4. Sonochemical Types of AOPs

Sonocatalysis

Sonolysis is considered to be a safe, clean and versatile technique. The principle of sonolysis is using ultrasound waves without the presence of catalysts, to produce radicals in aqueous media (Equations (22) and (23)) [197]. The propagation of ultrasound wave induces the acoustic cavitation, which involve the formation, growth, and violent implosion of micro-bubbles in a liquid [198].



Sonolysis alone does not produce a sufficient amount of hydroxyl radical. Therefore, sonolysis is often combined with other catalysts (e.g., UV, TiO_2), which increases the efficiency of the process. This process is called sonocatalysis or sonophotocatalysis depending on the used catalyst [151]

In a study by Hayati et al. [199], the combination of LED visible light and ultrasound waves to remove pharmaceutical (specifically sulfathiazole) from wastewater was studied. They confirmed the complete removal of sulfathiazole optimal conditions of sonophotocatalysis. The high efficiency of antibiotics removal was also reported by Calcio Gaufio et al. [200] when they applied ultrasound to conventional treatment processes.

The sono-Fenton technique is a Fenton reaction combined with ultrasound, where cavitation creates more $\bullet\text{OH}$ radicals and also accelerates Fe^{2+} regeneration [201]. $\bullet\text{OH}$ radicals are formed by sonolysis of water and hydrogen peroxide. The regeneration of Fe^{2+} occurs through the reaction of Fe^{3+} and $\text{H}\bullet$ (Equations (24)–(26)) [202].



The benefits of the ultrasound and the Fenton reagents are aggregated, allowing a higher generation of $\text{HO}\bullet$ and more effective degradation of organic pollutants [202].

6. Disinfection

To overcome water shortages, reused treated wastewater effluent for crop irrigation is counted as one of the most optimal solutions [203]. Aside from physical and chemical pollution of the wastewaters, biological pollution is not to be taken lightly. Therefore, treated wastewater without additional quaternary treatment, might serve as a water-borne pathogenic microorganisms spawning ground. Due to this fact, microbial contamination has become a problem endangering public health. Disinfection should be the last step in wastewater treatment to ensure the effectiveness of the applied methods. Regardless of the disinfection nature, whether being physical or chemical, present organic and inorganic substances can react and consume applied disinfectants or physically affect the applied processes [204,205].

6.1. Chemical Disinfection

6.1.1. Sodium Hypochlorite

Use of sodium hypochlorite (NaClO) is widely spread in water treatment works. It is not technologically demanding; its disinfection properties last for a long period of time and it has a low dosing cost [206,207]. Compared to gaseous chlorine and calcium hypochlorite, NaClO is more stable. The mechanism of chlorine disinfection lies in breaking chemical bonds such as proteins and highly active enzymes on the outer layer of the bacteria. The reaction happens with the addition of chlorine gas in water and it undergoes hydrolysis as shown in Equations (27) and (28):



Due to the neutral nature of HClO , it attacks negatively charged parts of bacteria and enters inside bacteria easily, while negatively charged ions of ClO^- serve as the main disinfectant at pHs higher than 7.5 [208]. The used phrase “free available chlorine” accounts for HClO and ClO^- . To study the disinfection ability, a solution of NaClO was prepared from stock solution (Sigma-Aldrich Saint-Louis, Missouri, USA) with an active chlorine ppm concentration range of 40,000–50,000. Solutions were standardized with diethylphenylenediamine (DPD). Studies about NaClO disinfection of secondary effluent concluded that acidic conditions of $\text{pH} = 4$ made chlorination most efficient. They monitored a real wastewater treatment plant and temperature fluctuations during the year had quite a significant effect on water pH. Rising temperatures during the summer months affected pH with the opposite trend and knowing that the correlation between pH and temperature was disproportionate, we can conclude an effect of low temperatures on pH. The experiment noted more than 3-log removal of coliforms at doses as low as 1.5 ppm of NaClO . Removal of total coliforms varied with dose and time. Log reduction of 0.45-log with a dose of 0.5 mg/L and a contact time of 15 min was the worst result, while a dose of 2.5–3 ppm over 15 min achieved 7.71-log reduction. Bacteria growth was noted to be

asymptotical and at the concentration with the highest removal of bacteria, their growth followed a declining trend which confirmed their removal [209].

6.1.2. Peracetic Acid

Peracetic acid (PAA) is generally sold as a mixture of PAA, acetic acid (PA), hydrogen peroxide (H_2O_2) and water. At standard conditions, standard reduction potential of PAA is equal to 1.96 V, which is higher than regular disinfectants like Cl_2 , HClO , ClO_2 and H_2O_2 . The first use of PAA was noted in the early 1980s. PAA has a generally low risk of creating harmful substances and has high pathogen inactivation across species [210,211].

Studies proved that a concentration time of 30–60 $\text{mg}\cdot\text{min}/\text{L}$ seems to remove most of the enteric bacteria such as *E. coli*, total coliforms, and *E. faecium*, etc. On the other hand, Gram-positive intestinal bacterium *Enterococcus* spp., had inactivation efficiency 1–2 log lower compared to *Enterococci* or *E. coli*. For multidrug-resistant *E. coli*, 25.1 $\text{mg}\cdot\text{min}/\text{L}$ of PAA was needed to reach 4-log reduction. Removal efficiency regarding spores is concerning and unacceptable and should have attention for more studies. Reaction species generated during disinfection are hydroxyl radicals, peracetyl radical and also methyl radical which could be part of the degradation process [205,212]. The disinfection mechanism of PAA relies on reacting with sulfur and sulphydryl bonds.

6.1.3. Performic Acid

Compared to PAA, performic acid (PFA) shows better inactivation abilities for *Escherichia coli*, fecal coliform, and *Enterococci*. In a bioreactor, inactivation efficiency of bacteria descended in the order of PFA, chlorine, and PAA [213].

For example, to achieve inactivation of *E. coli* and *Enterococci* in a sewer overflow, a PFA dose of 2–4 mg/L and 20 min of time was needed for a 3-log inactivation. The PAA time was 18 times longer for similar result. Regarding disinfection of the municipal secondary effluent, the concentration time for PFA was recorded to be 1.5 and 3.5 $\text{mg}\cdot\text{min}/\text{L}$ to achieve 1-log inactivation of *E. coli* and *Enterococci*. Disinfection of the tertiary effluent with PFA at a dose of 1.5 mg/L , which was already treated by filtration, showed inactivation of *E. coli* and *Enterococci* by 3-log and 2.8-log within 2 and 10 min, respectively. Additionally, degradation products of PFA are relatively harmless and do not pose a serious threat to water bodies [205].

6.2. Physical Means of Disinfection

6.2.1. UV Irradiation

Its disinfection ability affects a large spectrum of microorganisms with great efficiency, while not having the disadvantage of creating harmful byproducts which could greatly affect the receiving body. The wavelength range of UV irradiation lies between 10 and 400 nm. The principle of UV disinfection is to destroy the ability of microorganisms to reproduce by altering or degrading genetical material such as DNA and RNA (Figure 8) [214,215].

Negative aspects of UV disinfection are photoreactions of microorganisms, which may cause in certain cases a rise of active *E. coli* cells. This was observed at radiation conditions of 5 mJ/cm^2 . Another important concern is the presence of other pollutants such as chroma, turbidity and organic matter. Reported results inform us that turbidity less than 4 NTU does not affect UV disinfection to a large extent. The problem arises when turbidity becomes higher than 4 NTU [214]. Additionally, the latest studies confirm that microplastics affect disinfection with UV in a negative way. The first research paper experimented with polyethylene and polyvinyl chloride microplastics, while the tested bacteria were multidrug-resistant *E. coli* and *Enterococci* in pure water with a pH around 6.0. A microplastics-free sample showed the expected inactivation trend of bacteria while for water containing microplastics, the inactivation was slower or completely halted. It is speculated that microplastics can either absorb UV light or bacteria grow on the particles which protect them. While microplastic-free samples achieved max log removal with UV fluence of 10 mJ/cm^2 and 15 mJ/cm^2 for *E. coli* and *Enterococci*, respectively, it was shown

that there was almost no log removal for all microplastic types after application of the mentioned UV fluences [216]. The second research paper tested polyethylene affecting UV/H₂O₂, also in real secondary treated urban wastewater. The used equipment was a UVC 80 W lamp that emitted light with a wavelength of 254 nm. The dose of H₂O₂ was 30 mg/L and the concentration of microplastics were 0.25 g/L, 0.5 g/L, and 1.0 g/L. Log reduction according to time of disinfection is shown in Figure 9a,b. Note the efficiency of UV disinfection without microplastics, in this case one colony forming unit per 100 mL was achieved [204].

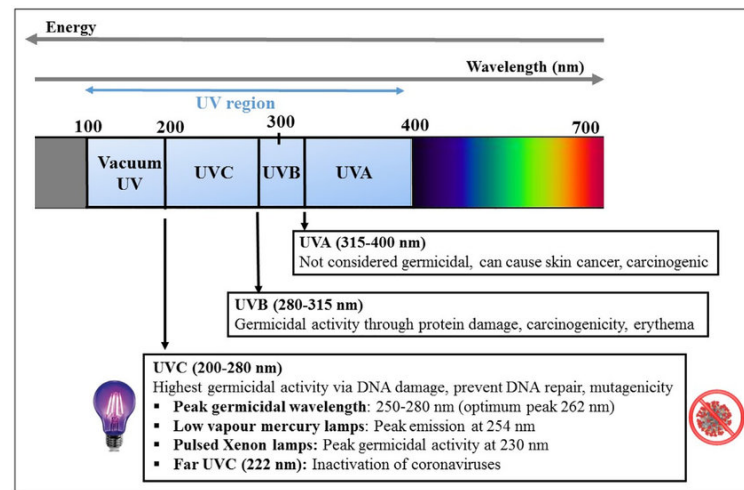


Figure 8. Spectrum of light and its disinfection capacities [214].

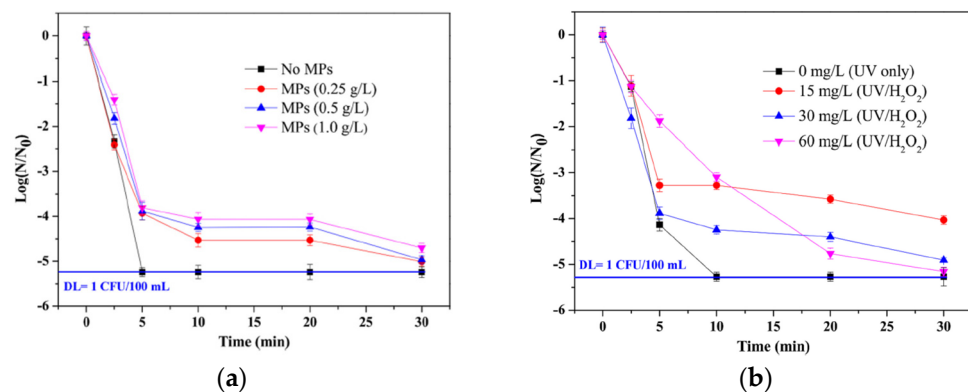


Figure 9. In the two graphs, there is shown dependence of log reduction of microorganisms from time. In graph (a) we see microplastics affecting ozonation and in graph (b), UV/H₂O₂ [204].

6.2.2. Sonolysis

The use of ultrasound (US) has been used for a long time in wastewater pollutant removal. Ultrasound is safe to use, has good distribution along water media and does not produce unwanted products. By applying a frequency of 16 kHz–100 MHz, we are able to inactivate microorganisms. In the wastewater, microbubbles are created in a process called cavitation, which triggers chemical reactions. The principle of disinfection by sonolysis depends on the type of radicals [217–219].

6.3. Use of AOPs as a Disinfection Method

Hydroxyl radicals (HO•) have been observed to destroy enzymes such as lysozyme and ribonuclease. Additionally, alteration of essential compounds takes place, amino acids are oxidized, modifications happen to sulfur groups, and crosslinking occurs which has the ability to make microorganisms to survive. Useful auto-reactions happen when unsaturated fatty acids are oxidized, which transform into lipid-peroxyl radicals [219].

Sulfate radicals ($\text{SO}_4^{\bullet-}$) mostly react with organics, which can offer many electrons. The negative charge present in $\text{SO}_4^{\bullet-}$ creates repulsion with the microorganism surface. Reported results of microorganism inactivation informs us about the generation of $\text{SO}_4^{\bullet-}$ from the peroxydisulfate system by natural magnetic pyrrhotite. Dissolution of cell casing is followed by degradation of intracellular substrates and the genome [219].

Superoxide radicals ($\text{O}_2^{\bullet-}$) are selective and have long-distance diffusion. $\text{O}_2^{\bullet-}$ radicals marginally affect the growth of *E. coli* while being at a low steady-state concentration of 1.5 nM. Compared to other ROS, it has higher efficiency. Again, the negative charge on the active substance generates repulsion with the cellular membrane. Moreover, $\text{O}_2^{\bullet-}$ could not react with peptides, carbohydrates, lipids and nucleic acids directly. Radicals were generated intracellularly and seemed to block biosynthetic enzymes with electrostatic interactions and acts selectively [219].

Singlet oxygen affects chains of amino acids, peptides, and proteins. $^1\text{O}_2$ attacks and destroys parts of tyrosine, tryptophan, and cysteine. The ability to disrupt enzymes makes it useful, even though it has quite a low redox capacity [219].

6.4. Electrochemical Disinfection

Electrochemical disinfection (ED) uses an external power supply to inactivate microorganisms present in the water. From a technical standpoint, it works based on direct oxidation, indirect oxidation and electric fields. Technologically, the effluent flows across electrochemical cell where the disinfection happens. Bacteria inactivation for different types of wastewaters or even tap waters is possible to a large extent completely. We need to remember that various conditions are applied at the studied inactivation potentials [220,221].

Direct oxidation happens when a multi-electron reaction is causing degradation of the proteins and functional groups inside a cell membrane. For this to happen, microorganisms need to get directly on the anode's surface. Applying low voltages causes dehydration and depression of the cells, while in a case of higher voltages, lipid peroxidation happens. Chain reactions take place, degrading microorganism function [220].

Indirect oxidation works by mediating compounds by reacting with an anode to produce intermediates which have oxidizing potential. Application of a milliampere (mA) electrical current produces reactive agents, which include reactive oxygen and chlorine [220,222].

The electric field can alone affect microorganism functions and in the end, completely inactivate bacteria. The mechanism for electrical field disinfection lies in membrane destruction and oxidative stress. Triggered reactions in lipids and modifications in membrane proteins affect its permeability [220].

7. Assessment of Reviewed Treatment Methods

The advantages and disadvantages of reviewed treatment methods are summarized in Table 4. The provided information is based on the recent literature and may be helpful to select suitable methods for wastewater quaternary treatment. The table mainly gives the qualitative assessment of reviewed methods and also gives information on which method is or is not commercially available. From an economic point of view, the reviewed methods were assessed as relatively low or high financial costs.

Table 4. Assessment of reviewed treatment methods [24–204].

Method	Advantages	Disadvantages
Sand filtration	Chemicals free, simple in operation, no harmful byproducts, relatively low financial costs, relatively well known and commercially available.	Very low removal efficiency for micropollutants and other contaminants, backwash is needed, and disposal of used sand.

Table 4. Cont.

Method	Advantages	Disadvantages
Membrane filtration	Well-defined and high removal efficiency of micropollutants, capable of removal of other contaminants and microorganisms, no toxic solid waste, chemicals free, no harmful byproducts and commercially available.	High energy demand, membrane fouling, disposal of concentrate, high water rejection, corrosive nature of the produced water, high-tech operation and maintenance, and relatively high financial costs.
Coagulation	Simple in operation, no harmful byproducts, relatively low financial costs, relatively well known and common chemicals are available.	Low removal efficiency for micropollutants, large amount of chemical sludge, introduction of coagulant salts in the aqueous phase, and sedimentation and filtration is needed.
Adsorption	High removal efficiency of micropollutants and other contaminants, simple in operation, chemical and sludge free, no harmful byproducts, relatively well known and commercially available.	Lower efficiency removal in the presence of NOMs, regeneration is needed, disposal of used carbon, production of toxic solid waste, desorption of sorbed contaminants, and relatively high financial costs.
Ozonation and other AOPs	Novel and promising technique, high removal efficiency of micropollutants, other contaminants and microorganisms, sludge free, ozonation is well known and commercially available.	High energy consumption, formation of harmful byproducts, interference of radical scavengers, strong developing is needed, focus on effective design and operation parameters is needed, other AOPs are not commercially available, and relatively high financial costs.
Chemical disinfection	Simple in operation, high removal efficiency of microorganisms, no toxic solid waste is produced, relatively low financial costs, relatively well known and commercially available.	Formation of harmful byproducts, not chemicals free, corrosive effects, requires understanding of principles of chemical disinfection, And does not prevent stored water from recontamination.
Physical disinfection (UV)	Simple in operation, high removal efficiency of microorganisms, no toxic solid waste, no harmful byproducts, chemicals free, relatively well known and commercially available.	Requires clear water, does not prevent stored water from recontamination, and relatively high financial costs.

8. Conclusions

- The existing literature is dominated by water scarcity and water stress, which are caused by rapid urban expansion, development of the world economy, demographic changes, deforestation and climate change.
- According to the predictions, the water scarcity and drought events are likely to be more frequent in the future. Due to these facts, the missing water resources must be replaced by a suitable water source.
- Quaternary treated urban wastewater has been proposed as an alternative water source for irrigation in Europe. For quaternary treatment, various additional processes

can be used, such as filtration, coagulation, adsorption, ozonation, advanced oxidation processes and disinfection. The choice of the specific process depends on various factors, including wastewater characteristics and treatment goals. According to the existing literature, we recommend for quaternary urban wastewater treatment, a combination of coagulation, membrane filtration (UF or NF) and UV disinfection. These processes are relatively well known and commercially available with high removal efficiencies of micropollutants and microorganisms.

- The quaternary treated wastewater reuse has the following innovativeness in the field of water management: an efficient use of water resources by citizens, industry and agriculture; promoting water saving and reuse; water-efficient technologies in all sectors; fitting in the context of the 2020 Circular Economy Action Plan; development of the huge potential for safe wastewater reuse in line with the new EU Regulation on water reuse; contribution to reduce greenhouse gas emissions; reduction the use of additional fertilizers resulting in savings for the environment, farmers and wastewater treatment; and the creation of green jobs in the water-related industry.
- Barriers to the reuse of quaternary treated urban wastewater are well characterized, and they mainly include concerns about microbial risk and presence of micropollutants; high investments for modernization of urban wastewater treatment plants; and a lack of financial incentives for quaternary treated wastewater reuse in agriculture.
- This review article serves as a basis for knowledge development, provides a comprehensive understanding of the current state of quaternary treatment of urban wastewater for its reuse, creates guidelines for practice, has the capacity to engender new ideas and serve as the grounds for future research directions. It helps researchers to identify key themes and concepts, evaluate the strengths and weaknesses of previous studies and determine areas where further research is needed.

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

•OH	hydroxyl radical
•OOH	hydroperoxide radical
AOPs	advanced oxidation processes
ARB&ARGs	antibiotic resistant bacteria and genes
BAC	biological activated carbon
BDD	boron-doped diamond electrode
CEC	contaminants of emerging concern
COD	chemical oxygen demand
DBD	dielectric barrier discharge
DBP	disinfection byproducts
DBPFP	disinfection byproducts formation potential
DNA	deoxyribonucleic acid

DOC	dissolved organic carbon
DPD	diethylphenylenediamine
e^-	electrons
ED	electrochemical disinfection
EU	European Union
GAC	granular activated charcoal
GDP	gross domestic product
GO	graphene oxide
h^+	holes
HAA	haloacetic acid
HAN	haloacetonitril
HK	haloketone
HOCl	hypochloride
IUPAC	international union of pure and applied chemistry
LED	light-emitting diode
MBR	membrane bioreactor
MF	microfiltration
MP	microplastic
NF	nanofiltration
$NO^\bullet$	nitric oxide
NOMs	natural organic matters
NTU	nephelometric turbidity unit
$O_2^{\bullet-}$	superoxide anion radical
OMP	organic micropollutant
PAA	peracetic acid
PAC	poly aluminum chloride
PBT	persistent, bioaccumulative and toxic
PE	polyethylene
PFA	performic acid
PFC	poly ferric chloride
PFO	pseudo-first order
PFS	poly ferrous sulphate
PMT	persistent, mobile and toxic
PSF	polysulfone
PSiAS	poly-aluminum silicate sulphate
PSiTS	poly-titanium silicate sulphate
PSO	pseudo-second order
PVC	polyvinyl chloride
RNA	ribonucleic acid
RO	reverse osmosis
$RO^\bullet$	alkoxyl radical
$ROO^\bullet$	peroxyl radical
ROOH	organic hydroperoxides
ROS	reactive oxygen species
$ROS^\bullet$	sulphonyl radicals
$RS^\bullet$	thiyl radicals
RSF	rapid sand filtration
$RSOO^\bullet$	thiyl peroxy
SDBS	sodium dodecyl benzenesulfonate
SHE	standard hydrogen electrode
$SO_4^{\bullet-}$	sulphate radicals
SUV_A	specific ultraviolet absorbance
TCNM	trichloronitromethane
THM	trihalomethane
TOC	total organic carbon
UF	ultrafiltration
US	ultrasound
UV	ultraviolet

UVA₂₅₄ absorbance of light in the UV part of the light spectrum at 254 nm
 WWTP wastewater treatment plant

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