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Review

Challenges and Advances in Tertiary Waste Water Treatment for Municipal Treatment Plants

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Abstract: Municipal waste water treatment plants have a fundamental task, which is to rid waste water of toxic and health-threatening organic and inorganic substances, including unwanted microorganisms and other pollutants, with the highest possible efficiency so that the discharged water does not contaminate the natural environment, which happens in the case of imperfect cleaning. Current WWTPs usually have a preliminary, primary, and secondary stage, and in very few cases even a tertiary stage, which no longer guarantees the sufficient removal of pollutants from waste water. This research presents the current situation in different parts of the world concerning the possibility of solving the current situation regarding the tertiary and quaternary stages of this process, especially in small and rural WWTPs serving up to approx. 10,000 equivalent inhabitants, which could ensure the removal of so-called emerging pollutants, including microplastics, and would stop WWTPs being point sources of environmental contamination.

Keywords: waste water treatment; tertiary treatment; microplastics; micropollutants removal

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1. Introduction

Waste water treatment plants have a fundamental task, which is the removal of toxic and health-threatening organic and inorganic substances, including unwanted microorganisms and other pollutants, from waste water with the highest possible efficiency so that the discharged water does not contaminate the natural environment, which happens in cases of insufficient treatment [1]. This water is also often used for crop irrigation, and not only in semi-desert areas; thus, the sustainable use of treated municipal waste water is absolutely essential [2]. Waste water, whose value has not been appreciated until recently, is increasingly recognized as a potential "new" source of clean water. The process of using treated waste water to produce drinking water is called potable water reuse [3]. In several countries, such as Singapore, Australia, and Namibia, but also in the USA, e.g., California, Virginia, and New Mexico, recycled water from treatment plants is even consumed, demonstrating that even treated waste water can be safe and clean [4]. Thus, waste water and sewage water are becoming extremely valuable commodities.

One of the current tasks, on a global level, is to clean as much waste water as possible. According to the 2017 UN World Water Development Report titled "Waste water: The Untapped Resource" [5], better waste water management brings social, environmental, and economic benefits that are crucial for sustainable development and necessary for achieving set goals [6].

Waste water treatment plants play a crucial role in achieving this goal, with their basic design and operations being well known and used worldwide. Waste water treatment plants are located throughout the territories of countries around the world. However, in North America and Europe, about 86.5% of domestic waste water undergoes the treatment process, while in Sub-Saharan Africa only 20% does [7]. Moreover, the technical level of these municipal waste water treatment plants varies even in economically developed countries. Usually, the equipment of treatment plants, and thus the quality of their outgoing water, differs between larger cities and small treatment plants in rural areas. Decentralized waste water treatment, considered an effective way of controlling water pollution in rural areas, is rapidly developing in many countries. Simultaneously, deeper analyses and evaluations of the operation of smaller treatment plants are still lacking, especially concerning their effectiveness in degrading various pollutants, chemical oxygen demand, denitrification and dephosphorization, etc. [8,9]. In practice, the choice of waste water treatment technology often depends on costs and the possibility of adapting it to local conditions [10,11]. Decentralized waste water treatment systems designed for small-scale operation not only reduce the impacts of insufficiently treated waste water on the environment and public health but also ensure sufficient clean water even in less accessible areas.

Waste water, or sewage, is classified according to its origin as municipal, domestic, or industrial, which also influences the presence of pollutant types [12]. The general types of pollutants seen include pathogenic organisms, synthetic organic chemicals, inorganic chemicals, sediments, radioactive substances, oil, synthetic dyes, etc. In recent years, waste water monitoring has primarily focused on heavy metals, polycyclic aromatic hydrocarbons, polychlorinated biphenyls, pesticides, volatile organic compounds, particularly chlorinated substances, chlorobenzenes, phthalates, and alkylphenols [13]. Recently, new substances have begun to be monitored, such as pharmaceuticals, hormones, antibiotics, personal care products, perfumes, new pesticides, poly- and perfluoroalkyl compounds, microplastics, antibiotic-resistant genes, and thousands of other similar compounds that conventional treatment processes cannot fully remove [14].

The aim of this research is to show, using the typical examples of current decontamination methods, whether the application of them would be suitable for specific conditions. This manuscript describes the current state of effective technologies for tertiary waste water treatment and shows that it is necessary to focus on scaling them up and on their optimal combination in particular.

2. The Issue of the Tertiary Stage of Waste Water Treatment Plants as a Decisive Factor in the Removal of Polluting Toxic Substances

The Typical Organization of Individual Processes in WWTPs

Waste water treatment plants (WWTPs) are tasked with collecting waste water from residential areas or industrial sectors and removing pollutants. The goal of this process is to return this resource to the water cycle, either by discharging it into watercourses or reusing it in activities such as agriculture. The water entering a WWTP goes through a series of physical, chemical, and biological processes aimed at removing the pollutants it contains. However, even a brief description of WWTPs reveals different interpretations of the function of these so-called cleaning stages, particularly in terms of the definition of the tertiary stage.

The cleaning process is typically divided into four stages known as preliminary, primary, secondary, and tertiary treatments. A diagram of a typical WWTP is shown in Figure 1. The content of the individual stages is not entirely strict and it always differs slightly in various treatment plants. However, the following activities usually occur at each stage [15].

The preliminary stage removes coarse impurities using coarse filtration, separating large and medium-sized solid waste using various screens. Then, fat and sand particles are removed using separators and degreasers.

The primary stage separates suspended solid particles using gravitational forces, with the pollutants adhered to these particles being partially removed. During this process, chemical substances, such as coagulants and flocculants, may also be added to improve the sedimentation of solid substances and remove phosphorus. In some cases, alkaline and acidic substances are used to neutralize the water's pH. The waste water is temporarily held in a sedimentation tank, where heavier solid substances sink to the bottom while lighter solid substances float to the surface. After settling, these substances are retained while the remaining liquid is either discharged or moved to the more stringent secondary phase of waste water treatment. The coagulation/flocculation process is significant, removing large amounts of organic compounds and suspended particles, including inorganic sediments [16]. However, the water still contains organic pollutants (polyamines, polydiallyldimethylammonium chloride, and polytannate); pathogens, including viruses and bacteria; inorganic coagulants based on aluminum compounds (aluminum sulfate, aluminum chloride, and sodium aluminate); ferric sulfate; ferrous sulfate; ferric chloride; and ferric chloride sulfate [17,18]. Recently, natural coagulants have started to be used more frequently. Coagulation/flocculation also seems to be a very promising technology for removing antibiotic- and sulfonamide-resistant genes [19].

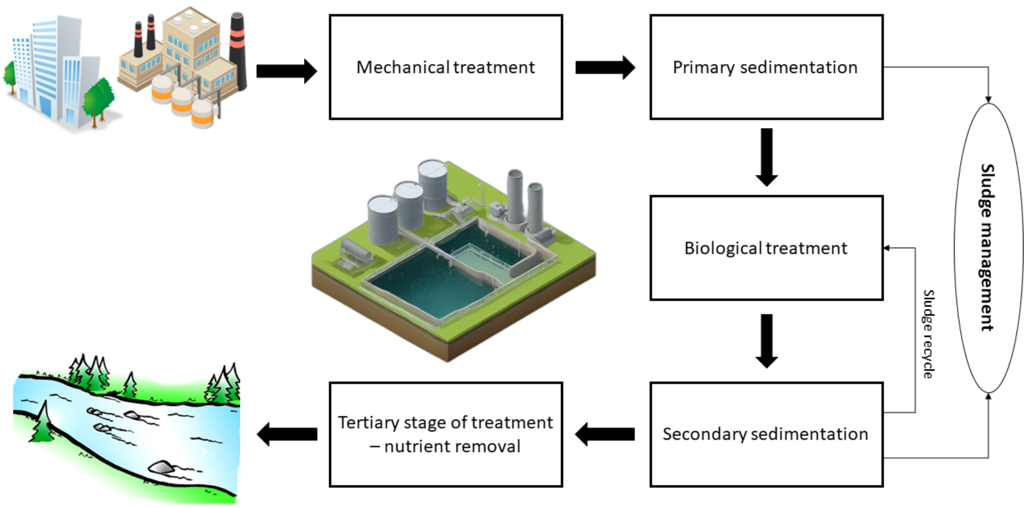

Figure 1. Diagram of a common waste water treatment plant.

The secondary stage cleans the water on a deeper level than the primary and involves the decomposition of its biological waste content primarily through aerobic biological processes. It reduces the amount of biodegradable organic matter present before the water is discharged for further treatment. During the secondary treatment, biological processes are used to remove dissolved and suspended organic substances typically measured as the biochemical oxygen demand (BOD) or chemical oxygen demand (COD). These processes are carried out by microorganisms in a controlled aerobic or anaerobic manner, depending on the treatment technology. Bacteria and protozoa consume biologically degradable soluble organic contaminants such as carbohydrates, hydrocarbons, fats, and short-chain organic carbon molecules from human and food waste, soaps, and detergents. At the same time, some of the so-called emerging pollutants, metal ions, and some antibiotic-resistant genes are captured in biological sludge [20,21]. Additionally, studies have shown that more than 80% of the microplastic particles entering WWTPs are retained in this sludge [22], leading to further MP release into the environment depending on the chosen sludge disposal method [23,24].

The tertiary stage oscillates between methods used for removing nutrients, especially nitrogen and phosphorus, and new techniques, so this subsequent stage is sometimes referred to as the quaternary one. Advanced oxidation processes are used to remove emerging organic micropollutants [25,26], such as the application of photo-Fenton reactions [27], photocatalysis [28,29], the use of ion exchangers to remove metal ions [30], ionic liquids [31], or membrane separations [32–34]. Both stages include everything above the secondary stage, with the quaternary stage emphasizing more advanced interventions such as photocatalysis, plasma technologies, electron beams, reverse osmosis, etc. The tertiary stage, however, is a technically established term. Each of the mentioned stages deals with different pollutants and their removal, with the water becoming cleaner as it progresses through these stages.

Regenerated water treated at the so-called tertiary level in waste water treatment plants mainly has the potential to be used in plant production, especially for growing fodder, irrigating urban landscapes, recreational and ecological activities, industry, and recharging aquifers to increase strategic water supplies in countries with water shortages. In this way, precious fresh water can be saved, given that the use of fresh water for industry and agriculture represents 20% to 67% of the use of fresh water resources [35,36]. Nevertheless, if the quality of regenerated water does not meet international standards, it can cause serious health risks for the population and the pollution of soil and groundwater with heavy metals [37], industrial organic substances [38,39], and particularly the newly monitored so-called emerging pollutants [3,40], which often have endocrine-disrupting properties [41]. Waste water, whose value has not been appreciated until recently, is increasingly recognized as a potential "new" source of clean water for drinking and non-drinking purposes, with social, environmental, and economic benefits; nevertheless, it must be stripped of undesirable substances [42]. A new trend of scientific and practical interest is currently focused on the removal of antibiotic-resistant genes [43]. Unfortunately, the removal of harmful microorganisms and parasites, along with newly monitored micropollutants, from water by conventional treatment plants is insufficient, and pollutants are only partially removed, e.g., even common pharmaceuticals are only removed at a rate of approximately 18–32% [35,44].

Regarding WWTPs in the future, a groundbreaking stance was taken by the European Parliament in 2024 [45], which considers the already-mentioned traditional removal of nitrogen and phosphorus as the tertiary stage and modern removal techniques focused on new pollutants as the quaternary stage. It also mandated that by the end of 2035, all urban waste water treatment plants serving 100,000 or more equivalent inhabitants must ensure quaternary waste water treatment. By the end of 2040, quaternary treatment would also be mandatory for all agglomerations with an equivalent population of over 10,000 inhabitants unless the member state can prove, based on risk analyses, the absence of risks to the environment or public health from water leaving its WWTPs. The focus is primarily on microplastics, pharmaceuticals, cosmetics, etc., with four high-risk substances (telmisartan, bisphenol A, beta-estradiol, and perfluorooctane sulfonic acid) serving for the control and comparison of quaternary water treatments. Additionally, according to the European Parliamentary Research Service (EPRS) [45], the recycling of treated water after quaternary processing will be required.

3. Micropollutants in the Waste Water and Effluents Discharged from Most Current WWTPs

Agriculture is the main cause of water degradation on Earth through its pollution of rivers and streams, wetlands, lakes, and groundwater. During each rainfall, fertilizers, pesticides, and animal wastes from farms and livestock operations are washed into our water bodies, as well as nutrients and pathogens like bacteria and viruses. However, the main share of water degradation in waste water is attributed to human activity, from our sinks, showers, and toilets, especially after taking pharmaceuticals or antibiotics, as well as from commercial and industrial activities and stormwater runoff, which carries road salts, oils, fats, chemicals, and other impurities from impermeable surfaces into our water bodies. According to the UN, more than 80% of the world's waste water is returned to the environment without being treated, and the rest is treated in WWTPs that currently lack sufficient efficiency, thus becoming point sources of effluents.

In waters at the outlet of WWTPs, a variety of micropollutants can be detected worldwide, with their composition varying not only by continent but also by local region. For example, in several Chinese treatment plants, monitoring results showed that phenolic estrogenic compounds (PEs), macrolides, and fluoroquinolones predominated at both the inlet and outlet. Tetracyclines, bezafibrate, caffeine, steroidal estrogens, and PEs exhibited a high and stable removal efficiency, while other micropollutants showed significantly variable removal efficiency, with the most effective removal being a combined process consisting of ultrafiltration, ozonation, and ClO_2 [46]. However, in the sludge, substances such as sulfamethoxazole, ofloxacin, ciprofloxacin, clarithromycin, erythromycin, estrone, and bisphenol A were found in the effluent, along with β-estradiol3-sulfate and many other substances, which thus return to the environment. The presence of micropollutants can also fluctuate seasonally. Di Marcantonio et al., 2020 [12], studied and monitored 76 treatment plants and detected 13 organic micropollutants, with the highest concentrations being of illicit drugs, i.e., benzoylecgonine, 11-nor-carboxy-Δ9-tetrahydrocannabinol, amphetamine (AM), methamphetamine (MET), and ketoprofen. In each of the treatment plants, modified treatment systems were applied, which removed 60–70% of the drugs. Nevertheless, these methods were ineffective against perfluorinated alkyl substances (PFASs). Nam et al., 2014 [47], focused particularly on pharmaceuticals, with a removal efficiency ranging from 6% to 100%. Their laboratory experiments showed that compounds with a log $K_{ow} > 2.5$ (particularly bisphenol-A, 2,4-D, carbamazepine, triclocarban, and 4-nonylphenol) were effectively removed by coagulation, and the adsorption effect increased proportionally with the hydrophobicity of the micropollutants. Photodegradation by sunlight also effectively removed sulfamethoxazole, sulfamethazine, caffeine, diclofenac, ibuprofen, and acetaminophen, which are photosensitizing. Chlorination was relatively ineffective at removing micropollutants due to the lower chlorine dose used (2 mg/L), its shorter contact time (1 h), and the already lower micropollutant content in the chlorination phase in the WWTP. The technology here mainly involved coagulation/flocculation at the water inlet, followed by sand filtration and subsequently disinfection with the aforementioned chlorine oxide combination. The results suggest that micropollutants, in the coagulation phase at WWTPs, can be removed not only by coagulation itself but also by adsorption onto clay particles, especially in turbid water, and by photodegradation from sunlight in open-air locations, which is a beneficial finding for the construction of, for example, rural treatment plants. In general, it appears that traditional treatment plants with a secondary activated sludge stage or biofiltration are only limited decontamination barriers for micropollutants like antibiotics, anti-inflammatory drugs, β-blockers, and *X*-ray contrast media, which are often present in these waters, because these substances are only partially degraded in conventional WWTPs. A detailed overview of selected micropollutants, and the concentrations of them entering and exiting treatment plants in various countries, is provided in the review in [13].

The most frequently detected micropollutants, among hundreds of others, are summarized in Table 1.

Recently, several studies have examined the impacts of PFAS exposures on health. PFASs have been detected in biota, drinking water, food, air, and human serum [14,48,49]. In response to new information about PFASs' toxicity, several countries, including Canada, the United Kingdom, Sweden, Norway, Germany, and Australia, have issued regulatory guidelines for PFASs in water. In the USA, additional regulatory measures are being considered. PFASs are absorbed from the soil by plants and grains and can easily enter animal organisms [50–52]. The problem is that the biological secondary stage of WWTPs' treatment process provides the microbial decomposition of the highly toxic precursors of these PFASs, which are then released into effluents [53]. In the tertiary stage, which is therefore essential, they should be separated (by adsorption, filtration, or electrochemically), possibly even by a biological stage of treatment with selected microorganisms.

Based on the analysis of articles on the topic of micropollutant removal in WWTPs, and according to a selection of publications listed in Scopus [54], it has been shown that this removal significantly depends on the primary sorption of micropollutants onto the sludge in waste water treatment plants. For this reason, it is clear that these articles' conclusions may also be somewhat inconsistent and that there is not just one cleaning option. A combination of methods is always necessary, with specific solutions needing to match the given situation. However, after biological treatment, filtration should be applied, followed by oxidation (ozonation, or more advanced forms), disinfection, and adsorption.

Table 1. The most frequently detected micropollutants in waste water.

Group of Pollutants	Micropollutants
Pharmaceuticals	Acetaminophen, Diclofenac, Ketoprofen, Mefenamic acid, Naproxen, Salicylic acid, Carbamazepine, Bezafibrate, Clofibric acid, Gemfibrozil
Antibiotics	Erythromycin, Sulfamethoxazole, Trimethoprim, Azithromycin, Amoxicillin, Sulfamethoxazole, Lincomycin, Metronidazole, Flumequine—all in hundreds of ng/L
β-blockers	Atenolol, Metoprolol
Nervous stimulants	Caffeine, Salbutamol, Diazepam, Primidone, Carbamazepine—in hundreds of ng/L, with carbamazepine generally considered difficult to degrade
Fragrances, personal care products	Tonalide, Galaxolide, Diethyltoluamide, 4-benzophenone, 1,4-dioxane, methyl-propyl parabens
Steroid hormones	17α-Ethynylestradiol, Estrone, Estradiol, Estriol
Surfactants	Nonylphenol, Octylphenol
Plasticizers and fire retardants	Dimethyl adipate and Tris (1-chloro-2-propyl) phosphate
Phosphates and surfactants	Tweens (Polysorbates) and sodium lauryl sulfate, Bis-(2-ethylhexyl) phthalate, Tris (2-chloroisopropyl) phosphate
Industrial chemicals	Plasticizers (Bisphenol A), Fire retardant, Pesticides (Atrazine, Diuron, Diazinon, Clotrimazole, Tebuconazole, Chlorpyrifos, Malathion, Simazine)
Per- and polyfluorinated compounds	Perfluorooctanoic acid and perfluorooctanesulfonate (PFAS). This pollutant, often called "eternal", is currently the most popular and possibly the most closely monitored waste water contaminant, usually in concentrations of ng/L

Authors [54,55], according to the geographical area and technical characteristics of their studied WWTPs, have declared the main micropollutants in waste water and suitable processes for each of them. They point out that the waste water situation in different WWTPs is unique and that pollutant removal is determined by the synergistic influence of all used technologies, including sewage sludge, and that this problem can only be solved comprehensively. Additionally, while it is not possible to determine the optimal arrangement of individual treatment stages solely from the analysis of the water entering the WWTP, known general conclusions from a number of specific solutions can be used. These include the tendency of individual micropollutants to undergo biological degradation, their ability to adsorb light radiation, the surface functional groups of micropollutants, their hydrophobicity, and other information [56,57]. Out of 1545 retrieved studies [57], 21 full articles were analyzed, which showed seven processing options concerning the removal of micropollutants from waste water, which were successfully and effectively removed using these advanced cleaning techniques. Advanced oxidation processes, membrane processes, and adsorption processes have proven to be optimal solutions for the removal of micropollutants in waste water treatment plants (WWTPs).

The obtained information showed that the use of advanced cleaning options is associated with two significant problems: high operating costs and the formation of hazardous by-products and concentrated secondary metabolites and residues. This is especially true in the case of small treatment plants with no access to scientific facilities, where finding tertiary solutions is necessary but also challenging, as these plats require trouble-free operation that is as economically feasible as possible. High-level analytical equipment is needed because thousands of pollutants are present simultaneously, hundreds of which must be safely detected with respect to their negative impact on the environment, including their ecotoxicity. These mostly include industrial chemicals (tetraethylene glycol, laureth-5, and di-(2-ethylhexyl) phosphoric acid), pharmaceuticals (salicylic acid, diclofenac, losartan, valsartan, venlafaxine, oxazepam, lamotrigine, carbamazepine, tramadol, hydrochlorothiazide, theophylline, furosemide, ranitidine, bicalutamide, and metformin), and stimulants such as caffeine and nicotine [58].

As early as 2011, Deblonde et al. [59] gathered data from 44 studies and selected 50 pharmaceuticals, 6 phthalates, and bisphenol A. The concentrations of these measured in the influent ranged from 0.007 to 56.63 μg per liter, and their removal rate ranged from 0 to 97%. The concentration of caffeine in the influent was the highest among the studied molecules (an average of 56.63 μg per liter), with a removal rate of around 97%. The concentrations of ofloxacin were the lowest, ranging between 0.007 and 2.275 μg per liter in the influent to the treatment plant and 0.007 and 0.816 μg per liter in the effluent. Regarding the removal rate, it was higher than 90% for most phthalates, around 50% for antibiotics, and 70% for bisphenol A. The most resistant to removal were analgesics, anti-inflammatory substances, and beta-blockers, with removal rates of 30–40%.

Similarly, in a review published in the last decade [60], the occurrence of these substances in the environment due to the insufficient performance of current WWTPs was described, which is mainly due to their still-imperfect analytical capabilities, where, for example, the detection of the secondary metabolites of emerging pollutants is lacking and their health effects are still unclear. The most studied pollutants are non-steroidal anti-inflammatory drugs, β-blockers, anti-depressants, and the antiepileptic carbamazepine. High occurrences of up to 7000 ng/L of tramadol are seen, while the trend in illicit drugs (cannabis, heroin, and cocaine, as well as other hallucinogens like 3,4-methylenedioxy-N-methylamphetamine (MDMA)) varies at the inlet of WWTPs in different countries (around a few tens of ng/L). Although [60] provides a table of emerging pollutant occurrences in England specifically, the situation may be similar in other countries in Europe as well. For example, in the Czech Republic, [61] reports that, in large cities, WWTPs remove amphetamine the best (84–100%) of all illicit drugs, which occurs at inlet concentrations of 44–170 g/L. WWTPs remove methamphetamine only partially, at around 40% (at inlet concentrations between 232 and 4700 ng/L), and approximately 50% of ecstasy (3–60 ng/L), which is similar to the removal rate of the main metabolite of cocaine, benzoylecgonine. Emerging pollutants are detected in all WWTPs worldwide; however, the composition of these pollutants varies by region. For example, in China, the main contaminants include caffeine, the artificial sweetener sucralose, the corrosion inhibitor benzotriazole, PCBs, antibiotics, and other pharmaceuticals and pesticides. A total of 943 compounds were monitored and polycyclic aromatic hydrocarbons (PAHs), phosphorus flame retardants, phthalates, benzothiazoles, and phenol were found to be potentially harmful micropollutants in treated waste water [62].

Current knowledge suggests that it is necessary to re-examine the environmental role of waste water treatment plants, which may also function as reservoirs and providers of antibiotic resistance and as hotspots for the spread of antibiotic resistance genes among different species of bacteria. Waste water contains antibiotics, disinfectants, and metals that can create selective pressure for antibiotic resistance even at low concentrations. Our knowledge of antibiotic resistance in waste water has expanded in recent years, particularly due to advances in the molecular methods available. However, it is unclear how active horizontal gene transfer is in waste water and what role waste water treatment plants play in the issue of pathogens' resistance to antibiotics. Currently, antibiotics are found worldwide, not only in waters from healthcare facilities or the pharmaceutical industry, but also in municipal waste water. The main routes by which pharmaceuticals from households enter municipal waste water are human excreta, flushing, and household waste. Some cases from Asia are reported in [63]. These antibiotics have potential genotoxic characteristics. A study conducted in India reports that 56% of household waste is released through domestic sewers. Amoxicillin, tetracycline, and dicloxacillin have been detected in drinking water in Japan. In many countries, antibiotics such as sulfamethoxazole, trimethoprim, norfloxacin, and metronidazole are present in waste water discharged from households, with significant variations in waste water across different countries.

Recently, large amounts of residual antibiotics have been found in the aquatic environment due to human activity and healthcare, which could affect the microecological balance of aquatic organisms and promote the emergence of antibiotic-resistant bacteria

(ARB) and genes (ARGs). In China, such genes (e.g., floR, sul1, sul2) have already been found in 139 strains of bacteria isolated from various aquaculture environments [64].

Wang and Chen, 2022 [65], analyzed the feasibility and effectiveness of removing antibiotic-resistant genes (ARGs) using various waste water treatment processes, including biological processes such as membrane bioreactors and constructed wetlands; chemical processes such as ozonation, chlorination, Fenton oxidation, and other advanced oxidation processes (AOPs); physicochemical processes such as UV radiation and ionizing radiation; and physical processes such as coagulation and membrane filtration.

Wetlands (constructed wetlands), for example, reduced the tetracycline-resistant genes in effluents from animal farming, which contained significant amounts of tetM, tetW, and tetO, by 90%. Wetlands were also effective in removing sulfonamide-resistant genes, ensuring large amounts of sul1, sul2, tetM, tetO, tetQ, tetW, and intI1 were reduced by 1–3 orders of magnitude. They were also effective in the elimination of bacteria carrying sulf1, which typical WWTPs cannot achieve [66]. Constructed wetlands are often mentioned as a suitable element for inclusion in the tertiary stages of waste water treatment plants, and it would certainly be worth considering their construction (known for decades, e.g., in [67], especially in rural treatment plants) more thoroughly.

Among the popular methods for removing antibiotics and ARGs are chlorination, ozonation, advanced oxidation processes, coagulation, membrane technologies, and adsorption. A recent review of the literature on these topics is provided by Du et al. [68]. They also suggest that the simplest method for removing these genes and antibiotics could be adsorption, such as on modified biochar, which also improves its textural properties. Adsorption efficiency can be significantly increased via hydrogen bonding, π-π interactions, electrostatic interactions, and functional group complexation [69]. Abd-El Monaem et al., 2022 [70], prefer chitosan as an adsorbent agent with its large number of hydroxyl and amino functional groups, which make it suitable for the adsorption of mostly negatively charged ARGs.

Most of the scientific community today leans towards the opinion that the current WWTP system, based only on a secondary biological treatment, is entirely insufficient for removing such substances, and additional technologies located "beyond the secondary stage" are being chosen. The most success has so far been achieved with the removal of nitrogen and phosphorus from effluents after the secondary stage, and perhaps also in disinfection interventions reducing pathogenic microorganisms, with ozonation commonly cited. Emerging pollutants, microplastics, and resistant genes are currently the focus of attention, and they are only partially removed in current treatment plants. Emission limits for these pollutants do not exist, but such data are available for most heavy metals. Information on health hazards can then be obtained through ecotoxicological tests, e.g., in the literature review by Tobey E., 2023 [71]. Their bibliography contains citations related to new-approach methodologies (NAMs) used in the field of ecotoxicology. NAMs are technologies and approaches that do not use animals for testing chemical hazards and exposures or risk assessments, instead using other methods such as in vitro tests, computational models, cell models, and organs-on-chips. Their bibliography includes peer-reviewed literature published from 2010 to July 2023.

The situation is somewhat better with metal removal, according to Vardhan et al., 2019 [72]. The suitable application of modified low-cost natural sorbents is probably also the solution for the removal of industrial metal from waters. However, an important step in this research is to predict the structure of the surface-modified adsorbent to improve its adsorption properties.

4. Waste Water Treatment Plants as Point Sources of Environmental Contamination, Especially by Newly Monitored Pollutants

The fact that waste water at both the inlet and outlet of WWTPs contains emerging pollutants has been known for several decades, and the findings are being refined with the development of analytical methods capable of detecting nanogram concentrations. These are complex detections of these substances, with around 700 substances identified, according to [73–75], as being present in waste water and effluents in very small concentrations, often in ppm to ppb. A systematic review of several studies on this topic conducted up to 2021 was published by Shehu et al. [76], focusing on pesticides, microplastics, pharmaceuticals, personal care products, and per- and polyfluoroalkyl substances. Their review highlights the significant variation in the quantity and composition of emerging pollutants across different countries, such as polyfluorinated compounds at rates of up to 1383 ng/L in Thailand and 48.6 ng/L in Denmark. Similarly, microplastics are most prevalent in waters in Asia, with up to 80 particles/L, while in the USA, their concentration is several orders of magnitude lower. Emerging pollutants were mostly detected in Africa, where they are more widespread due to various socioeconomic problems that have led to poor hygiene systems and a drastic lack of waste water treatment plants in many regions. A total of 290 emerging pollutants, mainly pharmaceuticals and pesticides, were found in waters of all categories, including drinking water [77]. In Southern California, the concentrations of various EPs were measured in effluents from several municipal treatment plants discharging directly into the sea, including naproxen, gemfibrozil, atenolol, and TCPP (tris (2-chloro-1-methylethyl) phosphate, a flame retardant), with the most frequent detections being in concentrations > 1 μg/L [78].

In recent decades, due to the enormous development of scientific knowledge, particularly in the field of sustainable living, with the aim of developing new drugs, antibiotics, hormones, personal care products, fire retardants, and a vast array of other substances, but also due to current agricultural practices using effective herbicides and pesticides and the new era of synthetic plastics, waste products from these essential societal products are entering the environment at an alarming rate. As a result, entirely new and still not fully understood risks to human health and aquatic ecosystems already exist and continue to emerge [79].

Therefore, given the widespread presence of new contaminants in waste water, justified concerns have arisen regarding public health and the impact of these pollutants on the environment [35,80]. These pollutants are unequivocally released into the environment and can have a range of harmful impacts on humans [81,82], including the development of resistant bacteria, the castration of marine animals, neurotoxic side effects, endocrine disorders, and tumor formation. It seems to be proven that prolonged exposure to these chemicals may exacerbate or even become the primary cause of diseases such as type 2 diabetes, obesity, cardiovascular diseases, and certain types of cancer [83,84].

5. Removal of Microplastics from the Waste Water Entering Treatment Plants

5.1. The Detection of Microplastics Entering Treatment Plants

Microplastics are defined as plastic particles smaller than 5 mm. They are formed by the degradation, abrasion, or disintegration of plastics and are found everywhere. They are synthetic polymers of various shapes, from 5 mm to the smallest nano- or micrometer particles. Essentially, they have contaminated the entire planet.

To study the possibility of removing microplastics from waste water, it is necessary first to quantify their concentration in a given volume of water, for example, through filtration. Microplastics, as heterogeneous particles, can be removed from waters through sieves, as the pore size of the sieve, typically ranging from 1 μm to 500 μm, has a decisive impact on the amount of microplastic removed. However, some particles that would fit through a given sieve opening may not pass through due to their irregular shape, thus skewing the size distribution of microplastics. According to statistics, the most commonly used plastics are polystyrene, polyethylene terephthalate, polyurethane, polyvinyl chloride, polyethylene, and polypropylene (PP), which together account for approximately 81% of the plastic demand in European countries [85].

Waste water samples containing microplastics (see Figures 2 and 3) also contain other natural organic (e.g., NOM, plankton) and inorganic particles, with organic particles being removable by catalytic peroxidation (30%) or, preferably, by Fenton's reaction. Inorganic particles can be removed by sedimentation, e.g., in variously dense salt solutions. Modern analytical methods, however, also allow for automatic searching for microplastics in samples used for Raman spectroscopy, FTIR, SEM, etc. There are reports [86] that the number of particles entering a treatment plant can be as high as 10,000/L, while in its effluent this can drop to a few hundred/L, with a part of the microplastics likely being retained in the primary and secondary treatment stages. However, retention can occur even in the tertiary stage, where tertiary treatment technologies are considered to be physical, such as membrane filtration, ultrafiltration, or reverse osmosis.

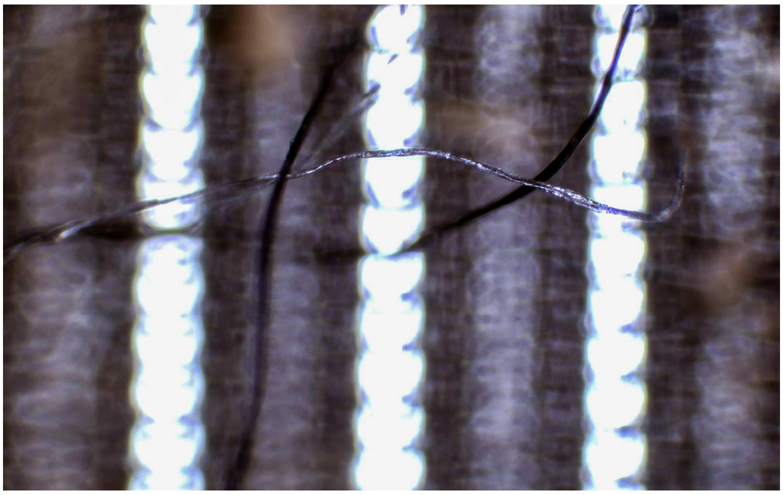

Figure 2. Synthetic fishing line, river Jizera, North Bohemia. The authors thanks to Eva Horakova and Lenka Wimmerova for the photo.

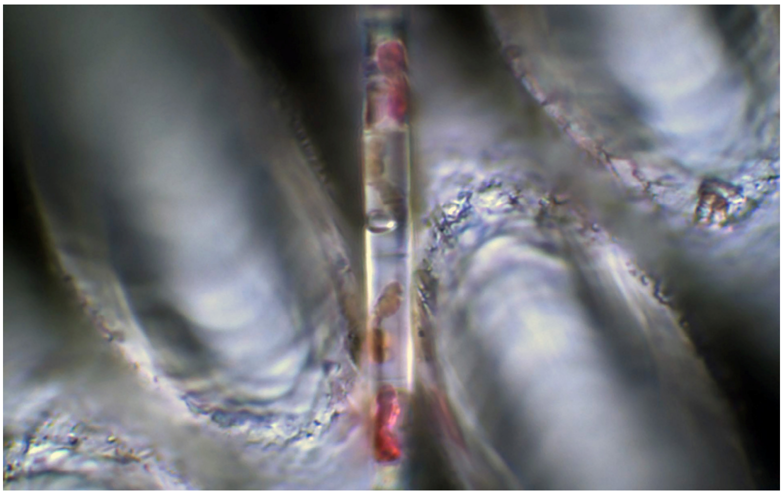

Figure 3. Silicon particle, river Becva, Moravia region. The authors thanks to Eva Horakova and Lenka Wimmerova for the photo.

As for the composition of microplastics, they are most commonly polyester (28–59%), followed by polyethylene, polyethylene terephthalate, polyamide, acrylate, polypropylene, polystyrene, polyurethane, polyvinyl alcohol, and polylactides, which also occur significantly, comprising around 5–27% of all microplastics. A significant source of microplastics in households is the washing and cleaning of clothes. It has been stated that most microplastics, about 70%, entering treatment plants are larger than 500 µm, while, in the effluent, 90% of the microplastics present are smaller than 500 µm, with a high percentage of those around 20 µm. However, these numbers are not accurate, and no one dares to specify the exact numbers of very small microplastics and possibly nanoplastics.

5.2. Possibilities for the Removal of Microplastics from Waste Water Treated in Small Waste Water Treatment Plants

Microplastics in the water we drink and use daily are becoming an increasingly concerning problem in developing, and possibly even more so in technically advanced, countries [87,88]. Currently, dozens of reports and reviews can be found on these substances, which could already form a separate monograph. Plastic particles are difficult to remove using standard water treatment methods, and their persistence in the environment and potential negative impacts on human health necessitate the development of effective removal techniques. Sun et al., 2019 [86], report that the removal of microplastics from treatment plant effluents leaving the secondary stage is over 88%, while that from the tertiary stage (including biologically aerated filters, dissolved air flotation, disk filters, rapid sand filtration, and granular filters) is over 97%. Their study summarizes how individual treatment plant stages gradually remove microplastics. However, these figures should be taken with great caution, as actual removal values can vary significantly between treatment plants. Flocs of bacterial extracellular polymers formed in the secondary stage can "encapsulate" microplastics. Furthermore, if coagulation/flocculation, e.g., with ferrous ions or aluminum sulfates, was also applied in the primary or secondary stages, it could contribute to the capture of microplastics. However, the mechanism of their capture by flocs is not entirely clear. It is likely that mainly large microplastics are encapsulated because the number of particles over 500 μm in size in the effluent from the secondary stage is negligible, according to some authors. However, others report that microplastics between 500 and 1000 μm account for about 43% of particles seen at this stage [87]. A biofilm can also form on the surface of the microplastics in the secondary stage, which completely changes their surface properties and "hides" their functional groups, making these particles, especially the extremely small ones, difficult to capture and non-sedimentable. It seems that the secondary stage captures large particles, which has perhaps been experimentally proven, as Sun et al., 2019, report [86].

In the tertiary stage, if membrane technologies (ultrafiltration, reverse osmosis), disk filters, or dissolved air flotation sand filters are applied, there is a further decrease in the content of microplastics in the effluent. The smallest size fractions detected (20–100 μm and 100–190 μm) were the most frequent [89].

These are the aforementioned discrepancies in our understanding of microplastics' removal from real waters and cannot be taken unreservedly, as the dilemma of defining the tertiary stage arises again, because many authors attribute the principle of coagulation/flocculation to the tertiary stage. For example, Sun et al., 2019 [86], expect the final content of micropollutants, from the original 100% of particles entering the treatment plant, to be reduced to approximately 1% after their removal using membrane technologies as a tertiary stage. Apparently, microplastics are best removed using a membrane bioreactor, although not many have been installed worldwide so far and the reason for this cannot be assessed. Otherwise, after such a defined tertiary stage, it is mainly small microplastics under 190 μm that have been mentioned as appearing in the effluents from treatment plants [89]. However, no specific effluent treatment process aimed at removing microplastics has been applied using any of the technology developed so far, which is still in its preliminary research stage.

Generally, it is assumed that the installation of a tertiary stage is necessary for the complete removal of microplastics from waters leaving treatment plants. Further advanced cleaning technologies in the tertiary stage, including membrane filtration (microfiltration and ultrafiltration), promisingly reduce the amount of microplastics in the final waste water. Other tertiary cleaning stages, such as advanced oxidation processes (AOPs) Fenton, electro-Fenton, photoelectron-Fenton, photo-Fenton, ozonation, and hydrogen peroxide (H_2O_2) photolysis, can be incorporated to target the remaining microplastics that persist after conventional treatments. Ahmed et al., 2024 [90], do not reject bioremediation as a possible method, which is essentially the adhesion of microplastics to microorganisms, including microalgae, nor do they reject the idea membrane bioreactors, whose development seems generally underestimated.

Electrocoagulation should also be tested on a larger scale, particularly for the removal of fibrous microplastics. Another innovative method is the sol–gel process, which involves microplastics' sorption into synthetic amorphous Si oxide. Finally, the well-known method of ozonation with filtration–adsorption on activated carbon [91] is also a method that is expected to be particularly suitable for capturing very small microplastics. Generally, it is assumed that microplastics could also adsorb well onto various sorbents. However, solid–solid adsorption is very tricky. The adsorption capabilities of microplastics are largely dependent on the oxidation state of their surface, their shape heterogeneity, and aggregation, which govern the density of their carboxyl sites, their distribution, and their subsequent availability for the sorbent's functional groups. These parameters change over time in a real environment, and this phenomenon is not yet fully understood [92]. Fresh microplastics will adsorb differently to aged ones, which not only oxidize but also adsorb other dissolved organic substances or metal ions with different charges and similar hydrophobicity or electrostatic attractions. These differences mainly show up in their adsorption rates in ultrapure or real water: in real water, the common presence of DOM (dissolved organic matter) or divalent metals can modify the typical negative charge of pure microplastics to positive, allowing for their adsorption onto a sorbent with a negative surface charge (of course, at a suitable pH and above the zero point of the charge of the adsorbent), which could be activated carbon or one of the clays, such as bentonite. The tendency of bentonite to form aggregates of clay particles with almost colloidal properties in an aqueous environment allows it to adhere to the surface of the microplastic and turn to sediment, thereby removing microplastics of suitable sizes from real water [93]. This was documented by Arenas et al., 2021 [91], in water from Lake Geneva, where the adsorption of polystyrene microplastics onto activated carbon was up to three times higher in lake water than in ultrapure water. This also works the other way around, where the activation of activated carbon (AC) by divalent metals, e.g., ZnCl2, making the carbon surface positive, allows for the adsorption of negatively charged microplastics via electrostatic forces, which favor the excellent texture of AC for its good adsorption [94]. Even the addition of a surfactant allows a microplastic's nonpolar end to bind to a nonpolar sorbent and the sorbent's polar end to bind to the polar surface of microplastic, thereby enabling the mutual binding of microplastics to zeolites or bentonites [95].

The adsorption of micro- or nanoplastics can also be achieved using synthetic sorbents, e.g., metal–organic frameworks: polystyrene was well adsorbed, for example, by CuNi@C, or by so-called magnetic microrobots, which are certainly interesting for further study; however, in the complicated real environment of micro- or nanoplastics, microrobots are still too sophisticated for tertiary water treatment. Nonetheless, it would be ideal to have "searchers" who could find dispersed nanoparticles of synthetic organic plastics and distinguish them from organic micro–nanoparticles and then adsorb and remove them from a volume of real water. Perhaps, for the time being, this role could be fulfilled by selected electro-methods, such as electrocoagulation, or biodegradable biopolymers.

At first glance, it would also be possible to capture microplastics by flotation, using air bubbles, but it would be necessary to modify the surface of the bubbles so that they have a positive charge and can electrostatically capture negatively charged microplastics. This is a beautiful theory, but it is necessary to consider the possibility that adsorbed micropollutants may change the charge of the microplastics' surface. Similarly, a heterogeneous particle–water system could be treated using micro- and ultrafiltration.

Technologies that merely transfer microplastics between phases create a problem because they do not address the subsequent similar issue of what to do with the microplastics in the next phase. Low-cost sorbents, such as biochars, also retain a number of other micropollutants and there is a simple solution to their disposal, which is energy recovery. Clays can be recycled into lightweight cements, for example, for outdoor use, where their spontaneous combustion, which could occur at high temperatures above 200 °C, does not pose a risk [96].

Bioremediation is interesting, specifically in terms of the adhesion of micro–nanoplastics to microalgae, which can easily locate these types of plastics in water [97]. They can then be easily coagulated (e.g., with chitosan) and used for energy, such as in biofuels or by using the lipids from microalgae for liquid fuels.

All these technologies have drawbacks, either of an environmental nature or in that they are expensive. Consideration is also being given to installing filters in pipes that carry water leaving washing machines, which could significantly ease the burden on tertiary treatment installations. A "green" approach is also being considered, i.e., coagulation with natural coagulants, such as starch, or adhesion to low-cost biochar. Among the advanced methods considered, the use of photocatalysis (preferably with a micro–nano catalyst) in combination with biodegradation is noteworthy. Another challenging vision being considered involves the definitive abandonment of synthetic plastics and their replacement with biodegradable ones [98].

The capture of microplastics on a sorbent layer could probably also be applied on a large scale, e.g., as part of the tertiary stage of small waste water treatment plants. Microplastics could also be effectively captured by coagulation/flocculation [99] and by recently proposed natural coagulants, such as chitosan–tannic acid (95%), protein amyloid fibrils (98%), and starch (>90%) [100]. The reaction of double-layered hydroxides with negatively charged microplastics is interesting, as a colloidal interaction with the coagulation/flocculation type occurs [101]. It is also possible to pre-treat microplastics with partial oxidation via photocatalysis followed by biotreatment [102]. In essence, no single technology can be used to treat all microplastics, but several probably can, as shown by studies such that of Nasir et al., 2024 [103], who strongly emphasize the conclusions of Sun et al., 2019 [86]. When recommending an appropriate technique, coagulation [104] or membrane filtration [105] are alternated, although microplastics' fouling of and retention on particles of various types of sorbents must be considered in the latter case.

No specific cleaning technology for the removal of MPs or NPs has yet been used in any waste water treatment plants in the world. Cleaning technologies targeting MPs and NPs are still at the laboratory scale or preliminary research stage. Several researchers have used various techniques, including adsorption, bioremediation, photocatalysis, filtration, coagulation/flocculation, electrosorption, electrooxidation, and ultrasound on a small scale, to explore the potential of different developed techniques for removing MPs and NPs [106]. However, one thing is clear: waste water treatment plants are a vector for the entry of microplastics into the environment.

6. Conclusions

The comprehensive cleaning of municipal waste water in small and large agglomerations is a socially extremely important problem. Currently, it is absolutely impossible to remove pathogenic microorganisms, emerging micropollutants, microplastics, and other substances from municipal waters, and their presence is constantly, and even newly, detected. These substances are dangerous for organisms of all levels and are now being intensively studied in terms of their health effects, which enables the development of new analytical methods for their detection and relevant ecotoxicological tests. Unfortunately, even new analytical methods are not yet capable of reproducibly detecting their presence in concentrations that can be environmentally and health-problematic, which are μg/L or ng/L. For the vast majority of these pollutants, there are still no realistic and scientifically proven standards for their permissible concentration in waters of all categories. This is true even for metals and metalloids, which have defined permitted concentrations in water of all categories; however, thanks to industrialization, they also appear in significant quantities in waste water, even from smaller treatment plants. Similarly, even in smaller local treatment plants, especially in agricultural areas, pesticides and herbicides, or antibiotics used in livestock breeding, together with common pollutants, are abundant in waste water.

Simultaneously, the issue of removing bacteria resistant to antibiotics and resistant genes is also very important in small and rural treatment plants. So far, none of the currently detectable pathogens or any antibiotic-resistant genes have been removed from waste water. The complex presence of various micropollutants, which can synergistically have a highly harmful effect on aquatic animals or enter into soils, agricultural crops, drinking water, and food, has not yet been studied in depth. Therefore, no standard technologies that are generally available and could bring about clear results based on pilot experiments have been selected for the tertiary or quaternary stages of treatments at local smaller waste water treatment plants.

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