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Advances in membrane separation of urban wastewater effluents for (pre) concentration of microcontaminants and nutrient recovery: A mini review

Dennis Deemter [a], Isabel Oller [a], Ana M. Amat [b], Sixto Malato [a,*]

[a] Plataforma Solar de Almería-CIEMAT, Carretera de Senés Km 4, Tabernas, Almería, Spain
[b] Grupo Procesos de Oxidación Avanzada, Universitat Politècnica de València, Campus de Alcoy, Spain

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ABSTRACT

This revision work focuses on the recent advances in the separation of microcontaminants from urban wastewaters, using ultrafiltration and Nanofiltration membranes. Conventional systems show advantages such as low pressure and fouling, competitive energetic- and maintenance costs compared to reverse osmosis, and higher rejection rates of organic microcontaminants compared to membrane distillation. However, these rejection rates strongly depend on temperature, flow, and pressure, as well as surface charge and concentration, challenging the adequate treatment of more complex matrices. Recent advances in material science strongly improved the implementation possibilities of different membrane types. In conventional industrial processes and especially in wastewater treatment, offering not only cost reducing solutions for urban wastewaters, but also more efficiency for the remediation of a high variety of industrial wastewaters. Moreover, membrane separation systems show great potential and applicability for added value substance recovery from wastewaters for the agricultural, chemical and consumer industry, for more sustainable natural resources use. Finally, perspectives on promising technologies for the implementation and combination of different membrane separation methods in treatment trains, such as advanced oxidation processes, are given, also aiming for zero-liquid discharge, to prevent microcontaminants and valuable resources from passing through conventional methods and focusing on closing the water cycle.

Abbreviations

AnMBR	anaerobic membrane bioreactor
ACS	activated chlorine species
AO	anodic oxidation
AOPs	advanced oxidation processes
BBM	biomimetic and bioinspired membrane
BDD	boron doped diamond
CEC	contaminants of emerging concern
CNT	carbon Nanotubes
COD	chemical oxygen demand
CPC	compound parabolic collector
CVD	chemical vapor deposition
EAOP	electrochemical advanced oxidation process
EC	electro coagulation
EDDS	ethylenediamine-N, N'-disuccinic acid
EF	electro-Fenton
EO	electrooxidation
EPS	extracellular polymeric substances
FO	forward osmosis
GAC	granulated activated carbon
IC	inorganic carbon
MABR	membrane aerated biofilm reactor
MBBR	moving bed bioreactor
MBMBR	moving bed Membrane bioreactor
MBR	membrane bioreactor
MCs	micro contaminants
MD	membrane distillation
MF	microfiltration
MFC	microbial fuel cell
MLD	minimum-liquid discharge
MNB	micro nano-bubble
MWCO	molecular weight cut-off
NF	nanofiltration
PA	polyamide
PAC	powdered activated carbon

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* Corresponding author.
E-mail address: sixto.malato@psa.es (S. Malato).

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PhF	Photo-Fenton
RO	reverse osmosis
ROC	reverse osmosis concentrate
SAAO	solar assisted anodic oxidation
SGM	sol-gel method
SMBR	submerged membrane bioreactor
TMP	trans-membrane pressure
TOC	total organic carbon
UF	ultrafiltration
UWW	urban wastewater
UWWTP	urban wastewater treatment plant
ZLD	zero-liquid discharge

Introduction

Water can be considered as a commodity nowadays as it is the most important requisite to organic life on Earth. The decrease in water availability provoked by the Climate Change, specifically sweet water, jointly with the significant increase in water consumption and the pollution of available sources due to the increasing world population and wealth, require a continuous development of water technologies [1,2].

These water technologies are not only necessary for the treatment of used water, but even more for the creation of alternative water sources, such as desalination systems. One of the main usages of such reclaimed water would be irrigation in agriculture, which accounts for around 70% of the global water consumption, as well as for human consumption and energy production [3].

One of the most efficient ways to meet these challenges is the direct reuse of treated (waste)water to close the water cycle guaranteeing, at the same time, a full control of the quality of the water in every step of the cycle. The practice of water reuse is not new, as it is known to be applied in irrigation as early as the Bronze Age (3200 – 1100 BC), by ancient civilizations such as the Cretans, the Egyptians, and the Mesopotamians. Greek and Roman civilizations (1000 BC – 330 AD) are known to have applied wastewater for both irrigation and fertilization around their major cities. This dangerous unplanned use of raw wastewater is something that is still nowadays being done at small scale in arid regions, for example in Algeria, Morocco and Egypt [4,5]. Urban wastewater (UWW) consists out of effluents coming from domestic, industrial, and agricultural areas. Their composition may vary as well as the concentrations of the present ions, pollutants, and physicochemical parameters, [6] having substantial influence on soil composition, including its fauna and flora, such as earthworms [4].

In order to be able to safely reuse the wastewaters coming from agricultural and industrial activities, as well as UWW treatment plant (UWWTP) effluents, they need to be treated to prevent elevated concentrations of organic compounds, salts, and microbiological matter. Especially in UWWTP effluents, a cocktail of different organic compounds at low concentrations can be found which are identified as ‘Contaminants of Emerging Concern’ (CEC) [7,8]. These microcontaminants (MCs) coming from the use of pharmaceuticals, pesticides, cosmetics, and other organic compounds show concentrations ranging from μg/L till ng/L. [9] As conventional UWWTPs are not efficient enough for their removal, they often end up in the environment after discharge, resulting in bioaccumulation, chronic toxicity, endocrine disruption and irreversible soil pollution, calling for the rapid application and integration of novel treatment technologies [10,11].

Many different UWW treatment methods and processes are available, although the actual implementation of these applications is staying behind, mainly for economic reasons due to the extra-cost of the treatment itself [12–15]. Another highly important reason is the lack of legislation to regulate the safe reuse of these effluents containing CECs. In Europe, only Switzerland has specific legislation about this matter, which enforces, since 2016, the removal of 80% of MCs in UWWTPs. This is mainly performed by applying ozonation combined with adsorption techniques with powdered activated carbon (PAC) and granular activated carbon (GAC), additionally to the traditional biological and physical UWW treatment systems. Furthermore, the footprint of both activated carbon forms is intended to be reduced by producing biochar regionally from wood, biogenic waste and sewage sludge [16,17].

At the European level, the recently published EU Regulation 2020/741 regarding the minimum requirements for the reuse of water (EC Water Reuse, 2020), establishes provisions on comprehensive water management in order to guarantee the safe use of reclaimed water, promoting the circular economy and supporting adaptation to climate change [18].

Therefore, combination and integration of different novel and already available technologies is considered a challenge to tackle, not only to raise the economic efficiency, but also the practical one. This can be found in different pretreatment methods to raise process efficiencies, such as the pre-concentration of microcontaminants at the same time valuable nutrients are directly recovered in treated streams [19]. A clear example can be found in the combination of different membrane systems with different Advanced Oxidation Processes (AOPs) as polishing treatments, as well as nutrient recovery systems, or also called membrane-based hybrid technologies [20,21].

In this sense, the principles of minimum-liquid discharge (MLD) and zero-liquid discharge (ZLD) show increased research interest for the full crystallization of, for example, reverse osmosis concentrates (ROC), saline wastewaters, and industrial wastewaters by applying electrodialysis (ED), electrodialysis reversal (EDR) and membrane distillation (MD) [22–25]. The crystals on their turn, can be refined and further processed into sustainable acids and bases, metals, minerals, nutrients, and salt compounds as a recovery mechanism of added value substances from wastewater [23,26].

This revision work describes the main technologies applied in UWW treatment, based on membranes, AOPs and the recovery of valuable nutrients from wastewater. Starting with the advances made in the development of different membrane materials, as also the improvement of their working principle and selectivity, including the prevention of fouling mechanisms to improve their operation time by surface functionalization. Following with the implementation of different AOPs in the treatment processes using membranes and novel methods for valuable nutrients recovery, at the different stages of the treatment.

Membrane separation

The principle of membrane separation is based on the different physicochemical parameters. Further mechanisms are the Donnan effect, molecular charge, surface charge, the trans-membrane pressure (TMP), and the crossflow velocity. Combination of membrane separation with pretreatments such as acidification, coagulation, flotation, and physical adsorption are very common, as they greatly extend membrane operation and lifetime.

The subject of specific rejection of compounds by membranes is important, as it is depending on several physico-chemical parameters. The importance of mapping and reporting the efficiency of different membranes and membrane systems is very high. It makes possible to effectively combine two or more different membrane types or systems, which would solely be inefficient for the treatment of UWW, greatly increasing their synergy. It would also make possible to develop custom-fit systems to treat wastewaters directly on-site, thinking about several industrial and agricultural areas, or for example effluents coming from hospitals. Both on a large or small scale, preventing the relatively concentrated wastewater streams to dilute with other wastewaters on its way to the UWWTP must be considered an important issue [27]. In Table 1 an overview of main membrane types, their materials, and drawbacks described in this work can be found.

Table 1
Overview of main membrane types, their materials, and their drawbacks.

Membrane type	Membrane material	Drawback
Size exclusion	Polymeric	Chemical sensitivity
	Ceramic	Expensive
Membrane distillation	Polymeric/ceramic	High energy consumption
Membrane bioreactor	Polymeric/ceramic	Fouling High energy consumption Microbiological stability

Membrane systems

Microfiltration (MF) is one of the oldest pressure-driven membrane applications and has the largest pore size of membrane separation technologies, ranging from 10 – 0.1 μm, with TMP between 0.2 and 5 bar. These inexpensive membranes are commonly used to filter out large particles, to reduce the total organic carbon (TOC) and the chemical oxygen demand (COD). Both ceramic and polymeric MF membranes are commonly used for wastewater filtration, and significantly decrease fouling in the following smaller pore size membranes [28,29].

Ultrafiltration (UF) has better selectivity than MF, but lower selectivity compared to NF membranes, as their pore size range between 0.1 – 0.01 μm, with a TMP between 1 and 10 bar. They operate commonly in the dairy, beverage processing and pharmaceutical industry, as well as in UWW treatment. It is able to retain suspended solids, colloids, emulsions, bacteria, and viruses [30].

Nanofiltration (NF) is a relatively simple and inexpensive technology that operates under lower pressures compared to RO, showing still excellent retention capacity towards MCs and a selection of ions. The pore size lies between 0.010 and 0.001 μm, with a TMP 5 – 10 bar. Commercially, polymeric polyamide (PA) NF membranes are mainly used as they offer excellent results regarding the retention till particles as small as monovalent ions [29].

In Reverse osmosis (RO) everything is retained except water molecules. The TMP when operating RO is between 15 and 27 bar for brackish water, and 50–80 bar for seawater desalination, with a pore size of 0.001–0.0001 μm. Although RO obtains excellent results, and is therefore the most used process in desalination, it operates at very high pressures which come along with high energy consumption. In addition, when considered as alternative water source for irrigation activities, remineralization of the obtained distilled water (or mix with freshwater) is required beforehand. Another downfall is the fact that RO membranes are highly susceptible to fouling, such as scaling, which makes pre-filtration of the matrix highly necessary [31,32].

Forward osmosis (FO) is a process where the osmotic pressure difference is used to draw water to the feed solution through a selectively or semi-permeable membrane, and so, rejecting molecules and ions. FO offers in this way lower and reversible fouling, with lower operation and equipment costs, as well as higher water recovery rates as compared to before mentioned pressure driven categories [33].

In membrane distillation (MD), heat is used to let vapor permeate through a hydrophobic membrane based on the vapor pressure difference between its surfaces. The process is performed at low pressures and has low fouling rates compared to high pressure membrane processes, such as the previously described RO. The main challenges to overcome in this type of membrane separation system are the membrane wetting and membrane fouling, latter being both organic and inorganic [34,35]. A new development is the hybridization of MD with the membrane bioreactor (MBR), into a system where the dewatered concentrate coming from the anaerobic digester is depleted of water, resulting in a precipitation product that can be applied as a liquid biofertilizer, of which also struvite can be recovered [34].

Membrane bioreactor systems

The membrane bioreactor (MBR) is a hybrid system that exist out of a membrane unit for physical filtration, as well as a bioreactor for biodegradation which can be used as treatment of wastewaters. MBRs have a small ecological footprint, high effluent quality, and less sludge production than conventional biotreatment. There are two general types of MBRs, the aerobic and the anaerobic MBR, though in both the high consumption of energy is considered an important drawback. New advances in this membrane-based technology show that they can be combined with microbial fuel cells (MFC), to efficiently treat wastewater, while also recover energy [36,37]. A variety on the MBR, where the biomass is suspended in the matrix, is the moving bed bioreactor (MBBR), where the biomass is grown as a biofilm on carriers made out of plastic, spongelike, or other materials. Lately, there is increased interest in combining conventional MBRs with MBBR into hybrid systems, also called moving bed membrane bioreactor (MBMBR), which further extends the advantages of high specific biomass, higher efficiency in nutrient removal, and flexible operation [38,39].

Aerobic MBR systems utilize the metabolism of microbes to breakdown matter in UWW treatment, mainly existing out of C, N, P, and S, while using oxygen. The efficiency is strongly depending on the bioactivity and the biodiversity of the microorganisms, and physicochemical factors such as temperature and salinity [40].

Opposite to this, the anaerobic MBR (AnMBR**)** operates without oxygen, produces like aerobic MBR high quality permeate, but due to the anaerobic environment, it produces less sludge and is also able to produce methane gas, which can be used as a combustible for the production of energy, in order to balance the energy consumption of the overall process, like with an MFC [36,41].

Submerged MBR (SMBR) are also highly employed and available at commercial level. The SMBR offers one of the most efficient solutions to limit fouling in MBRs. This is performed by enhancing the mass transfer by applying a gas/liquid two-phase flow. The permeate stream is taken from the matrix by vacuum while air washing is applied to prevent cake layer formation, whereas fouling is reversed by sequenced aeration and filtration-relaxation, backwashing [42].

Other novel variations within the MBR principles include the membrane-aerated biofilm reactor (MABR). In this type of MBR, gaseous electron acceptor, such as O_2, or electron donors, such as H_2 and CH_4, are fed inside of a usually hydrophobic membrane on which a biofilm grows, significantly reducing the necessary aeration, as the gas is directly delivered to the organism, instead of dispersed into the aqueous medium. The feeding of the gas can be either dead-end or flow-through, and in the case of CH_4 could be delivered from a coupled AnMBR [43]. Ren et al. recently developed an electrochemical MABR for the removal of antibiotics. The system enhanced the degradation of both sulfamethoxazole and trimethoprim, as also enriched the genus of *Xanthobacter*, which is able to degrade intermediate degradation products of the two compounds [44].

One of the drawbacks of MBRs is their low ability to reduce or eliminate unwanted microorganisms within its microbiological community, as compared to conventional biological treatment. This thereby reduces the ability of the system to biodegrade highly persistent MCs and the reduction of COD. A solution to this problem is the addition of Nano-Fe_3O_4 particles, resulting in decreased membrane fouling, lowering the *Bacteroidetes* and increasing the *Proteobacteria* growth in the microbial community, which results in a decrease of COD in the effluent [45]. Another solution to this problem can be found in the addition of PAC to the MBR. Asif et al. found that the PAC addition promotes the growth of 24 out of 31 genera of bacteria for a more diverse microbial community, especially for denitrifying bacteria, as well as for nitrifying and denitrifying functional genes, and MC biodegrading bacteria and genes. High concentrations of PAC in the MBR did not compromise the MC removal, nor the microbial community evolution [46].

Further challenges in the application of MBR techniques lie in the treatment of high saline wastewaters (10–100 g/L), as well as in concentration shocks, sudden changes in compound concentration such as antibiotics. These extreme conditions for bacteria, sudden or constant, cause them to increase their defense mechanism in the form of extracellular polymeric substances (EPS) production, one of the main fouling mechanisms in MBR systems and membrane systems in general. Therefore, specific microbial communities have to be found, formed, and conditioned for the treatment of such wastewaters, as well as studying of the effects of these conditions on the microorganisms. Halophilic microorganisms are such microbes that can successfully be applied till salinities of 150 g/L in wastewater treatment by MBR system [40]s.

Membrane materials

One of the main classes of membranes applied in wastewater treatment are ceramic membranes. Relatively expensive, ceramic membranes offer excellent properties regarding pH and temperature operation range, and mechanical strength. Ceramic membranes can be produced from many different materials, but mainly oxides of aluminum, titanium, silicon and zirconium [47].

Ceramic membrane supports are generally produced by the compounding of different ceramic oxide materials, (polymeric) binders, and stabilizers. Mixtures of these materials are then pre-formed by extrusion or slip casting in the desired forms and air dried into so-called ‘greens’ before further thermal treatment. The different thermal treatments, e.g., sintering, determine the integral strength of the membranes, the pore size, pore morphology and distribution, and layer thicknesses. Mainly based on the different times and temperatures maintained. The main build-up of ceramic membranes consists out of the support material, (multiple) intermediate layers, and the final membrane layer [48].

The different pore sizes and pore morphologies of ceramic membranes are mainly based on their prime material, which is most often particles. There are numerous limitations to produce smaller ceramic particles, that, on their turn, behave differently in the production process as smaller they get, based on the volume to surface ratio. Therefore, another approach to produce membranes in the Nano range is the application of the sol-gel method (SGM). This method uses different precursor compounds to produce a gel-like substance, which is then applied on the support and sintered. Something performed by Qin et al. who prepared yttria-stabilized ZrO_2 NF membranes from size-controlled spherical ZrO_2 Nanoparticles produced by a reversed micelles-mediated SGM, for the treatment of wastewaters containing pesticides. The successfully prepared NF membranes with a MWCO of 800 ± 50 Da were able to obtain a carbofuran removal of over 80% and fouled membranes were easily cleaned by an alkali washing treatment and low-temperature calcination [49,50]. Another commonly used method for the production of ceramic membranes is the chemical vapor deposition (CVD). Here a precursor vapor material is applied on the support to obtain small particles that are then sintered, obtaining small pore sizes. Pore sizes and morphologies are on their turn also strongly dependent on sintering times, temperatures, and atmosphere [51,52]. The geometries of the different ceramic membranes are mainly the disk, flat-sheet, and the tubular and hollow fiber membranes, latter being able to have multiple channels. The produced ceramic membranes are most often combined in modular set-ups and applications can often be found in industrial wastewaters for their stability, which is necessary due to the often-harsh conditions that come in these industries, such as extreme pH values, high temperatures, and abrasive chemicals [48].

Efforts to lower the price of ceramic membranes are found in methods to lower the sintering-energy consumption, as it is good for 60% of the membrane price, where material cost and fabrication procedure are good for another 20% each. To obtain this, research is mainly performed to decrease the sintering temperature by applying materials with low melting point such as kaolin and fly ash, to accelerate the sintering speed by applying spark plasma sintering or microwave heating and decreasing the sintering time by applying co-sintering or membrane structure optimization. Another recent development is the development of no-sintering processes such as with geopolymer and Portland cement-based membranes for industrial applications [53,54]. Enhanced fabrication procedures for the production of ceramic membranes for wastewater treatment and desalination in the future is mainly expected to be found in 3D printing as a decline in costs of 50–75% is expected in the next decade. 3D printing significantly increases customization, low-cost to prototype and test designs, has sustainability benefits, and reduces production time. However, its current difficulties are the lack of resolution, appropriate materials and build volume scale [55,56].

New developments in the production of ceramic membranes can be found in the application of materials coming from other industries such as solid municipal waste treatment, or the cement and concrete industry. Materials recovered from different waste streams within these industries are used following the principle of Circular Economy and with the aim to produce significantly cheaper ceramic membranes. Examples of the use of these materials can be found in the work of Mouratib et al. who developed low-cost ceramic MF membranes made from alumina- and silica-rich water treatment sludge, to filter wastewater [57]. Khadijah et al., developed low cost, green silica based ceramic hollow fiber membranes from waste rice husk for water filtration [58]. Lorente-Ayza et al. compared the extrusion and the pressing method for the successful production of low-cost ceramic support material for MF membranes. They used as raw material chamotte coming from the Spanish tile industry, a local Spanish clay mixture, and low-cost potato starch as an organic pore former. It was found that extruded material resulted in less porous material and smaller pore sizes, than material formed by dry pressing. Their work will be utilized in the near future to produce multilayer ceramic membranes for UF and NF as well. Planning to do so, by developing thinner and selective layers [59].

Waste materials are not only used to produce the ceramics, but recycling wastes from the food, agricultural and industry are also used as pore-forming agents to produce porous ceramics [60].

Another main class of membranes in water treatment are polymeric membranes. Although significantly cheaper than ceramic membranes, polymeric membranes are more sensitive to chemicals, and are more prone to fouling. They can be categorized in two process classes, non-pressure driven, such as pervaporation, dialysis, and membrane distillation, and pressure driven such as MF, UF, NF, RO, and FO [61,62].

All of these processes are widely applied in successful technologies ranging from the removal of natural organics, microbes, MCs, and heavy metals from UWWs, dyes and oily substances from industrial wastewaters, biomedical applications such as kidney dialysis, due to their low energy use and simplicity as compared to thermal separation processes. Their many forms come as hollow fiber membranes, (rolled) flat sheet membranes, tubular, and electro-spun nanofiber. All coming with their unique material properties and pore design, based on their polymerization method [63].

In order to make these commonly used polymeric membranes more sustainable, new ways are investigated to produce them from biodegradable polymers instead of oil based polymers. Main examples regarding biodegradable polymeric membranes in wastewater treatment are the application of cellulose acetate, chitosan, and chitin membranes. Disadvantages, however, are their oftentimes even more limited pH range, particle affinity, and the biodegradation, and so, selectivity rate, environmental pollution by its non-biodegradable counterpart in the case of blends and composites, and inferior mechanical properties in comparison to conventional materials [64,65].

There are different movements in the development of composite membranes, such as with the combination of polymers, or the application of e.g., a polymer layer on ceramics. The most common type of polymeric composite membranes are thin film composite (TFC) membranes, existing out of three layers, a woven or non-woven fabric, to support the intermediate layer with micro sized pores, and finally the PA selective layer. The pore size of each layer is around 60–80 μm, 50–100 μm, and 100–200 nm, respectively [66]. Saini et al. writes about the tremendous potential of new polymer membrane technologies that make use of polymer nanocomposite membranes for applications as small as of synthetically produced hydrogen gas, filtering out CO_2, N_2, and CH_4. Something that can be useful when applying novel EO reactors, that produces this gas [67]. Another example of highly advanced membranes are the biomimetic and bioinspired membranes (BBM). These BBMs consist of membranes with integrated biological functional molecules or bioinspired functional elements that are able to separate compounds in the sub-Nanometer scale. Examples for these molecules are artificial water channels, membrane protein channels, and carbon Nanotubes (CNT). Their application can currently be found in water purification, but finds significant potential in the specific separation of antibiotics, homogeneous catalyst retention, organic acid and gas separation as well [68].

Another way to effectively apply membrane separation is to pre-concentrate water contaminated streams for a subsequent AOP application to rejection streams, mainly for lowering the volume to be treated and to reduce the consumption of reagents as the concentration of contaminants would be higher than in the raw wastewater. This is a key topic for the elimination of MCs [69].

Oftentimes, permeate volumes still contain very low concentrations of (certain) MCs, as the membrane selectivity is determined by factors such as system parameters, physicochemical properties, and concentration factor. The permeate volumes can then be polished by different selected AOPs that are to be matched with the MCs that are insufficiently retained by the different membrane types and systems, in order to obtain significantly higher removal of MC [70].

Membrane fouling

Fouling is the process in which a variety of undesired organic and inorganic matter is deposited onto the membrane, eventually leading to the blockade of the membrane pores, after increasingly reducing the flux and the selectivity of the membrane. Organic membrane fouling is induced by the collection of organic material on the membrane surface, as well as in the pores, by proteins, humic acids, polymers, and polysaccharides. Inorganic fouling is the crystallization and precipitation of salts in the matrix on the membrane surface, mainly due to supersaturation. In the case of metallic deposition it is called scaling. Common salts in wastewater are carbonates, phosphates, sulfates, and sodium chloride [71].

The term biofouling is used for the formation of biological material, such as algae, bacteria, fungi, and plankton on the membrane surface. Bacterial EPS can be formed helping bacteria to resist flow and turbulence from the surrounding matrix. In MD for example, the thermal stress can elevate the formation of EPS. Over time, the biofouling is initiated by the formation of the conditioning film through scaling and organic fouling, followed by their reversible attachment by the formation of the protobiofilm. The bacteria in this protobiofilm then form the EPS, due to which an irreversible attachment of the biofilm on the membrane surface starts, maturing over time. Once matured, parts of the biofilm start to detach itself into the matrix, contributing to the protobiofilm formation [71]. The communication by the bacteria to produce EPS under stress conditions, is done via quorum sensing, performed by the secretion and detection of diffusible molecules named autoinducer, or signal molecules. A relatively new solution to this problem can be found in the deployment of functional bacteria, fungi, or enzymes to suppress this communication, what is called quorum quenching [45,72,73].

Fouling prevention

In order to guarantee the viability of membranes, the prevention and reversal of fouling is of the utmost importance. The main fouling reversal mechanism is the application of 'back-wash' cycles, whereby the flow direction is reversed for a certain period of time. Other treatments can be found in chemical cleaning, which involves the use of an acidic and/or basic medium, often limited, especially in the case of polymeric membranes as extreme pH values are detrimental to their structural and chemical integrity [74,75]. Acid-base treatment is thereby also a temporary solution to remove reversible fouling layers. However, operation must be put on-hold and the chemicals bring extra cost with them. In the case of ceramic membranes, fouling can also be reversed by the incineration of the fouling layer. An effective solution, but replacement of the membranes or a full stop of the system during the incineration treatment is necessary [76].

Other antifouling mechanisms are also emerging. This can be found in the form of super-hydrophilic or super-hydrophobic membrane surfaces, the development and design of the pores, the pore-density, and morphology within these materials. The Janus membrane, named after the two-faced Roman god Janus, is based on the principle of applying different membrane layers on each other, with opposite properties, such as hydrophilic and hydrophobic, wettability, pore size or structure, thermal/electrical conductivity, and chemical activity. It offers a good option towards the preselection of compounds directly on the membrane surface and can prevent fouling to a great extent [77].

MBR system aeration, which is essential to keep the desired dissolved oxygen concentration for the biomass metabolism, can be used as antifouling mechanism. However, the agitation that aeration creates towards the bacteria can also increase the formation of EPS and foam. As different aeration patterns are continuously being researched for the different MBR principles, as a way to decrease energy costs as well, it was found that coarser bubbles are preferred over finer bubbles, as they remove more cake on the membrane [39,78,79].

The application of AOPs against fouling

To prevent fouling of membranes, different AOPs can be utilized. Ozonation, commonly used in UWW treatment, can be deployed as pre- or post-treatment, as well as *in-situ* cases. Special care should be taken when considering ozonation in hybrid systems using polymeric membranes, as only few commercial polymer membranes do not degrade by the generated radicals [80].

Photocatalytic membranes directly combine the application of AOPs with the membrane material, by producing them from photoactive materials such as TiO_2 and ZrO_2. The efficiency of these novel membranes is lower for the degradation of MCs, as the immobilized photocatalytic material has significantly less active site as compared to suspended photocatalytic particles. However, the application of photocatalytic materials in membranes resulted to require extended operation times by other means of fouling prevention, such as with photocatalytic TiO_2. This material possesses the property of photo-induced super-hydrophilicity (PSH) when irradiated by light [81].

Brillas et al. observed that current research regarding these materials do not consider the light transport as fundamental aspect of light-matter interaction. They suggest applying biomimicry from phototroph organisms to improve the quantum yield of such surfaces by taking nature as an alternative guide in the development of novel materials. They identified micro- and nanostructures present in nature to prepare new bio-inspired photocatalytic and photo electrocatalytic material and reactor design, rather than focusing on the development of the composition of the semiconductor photo(electro)catalyst [82].

Gupta et al. reported a complex membrane system that exists out of the combination of many anti-fouling and membrane-based-AOP technologies. They developed a submerged photocatalytic oscillatory membrane reactor, with membrane aeration, for the removal of MCs from UWW as a tertiary treatment. The reactor uses suspended nano-sized TiO_2, which is irradiated with UV light to produce hydroxyl radicals for the removal of MCs, while the aeration and oscillation is utilized to prevent the TiO_2 from fouling the membrane. They reached 90% MC removal of diclofenac, sulfamethoxazole, and hydrochlorothiazide, in

Milli-Q water. Practical applications of this kind of membrane systems must be tested in actual wastewater treatment integration systems [83].

Combination of membranes and AOPs

Advanced oxidation processes are characterized by the production of hydroxyl radicals that can degrade MCs in UWWTP effluents. AOPs are an important solution for a sustainable tertiary treatment of UWWTP effluents, as only simple chemicals are necessary, and the irradiation can be provided in a renewable form, with solar radiation, using reactors such as the compound parabolic collectors (CPC) [84]. An example of these AOPs, which uses the Sun and CPC reactors, is solar photo-Fenton and is based on catalytic iron cycles, using H_2O_2 as an oxidant, and UV–vis light from the Sun to produce the hydroxyl radicals [85].

Although UV-C on its own is not an AOP [86], its application is essential to many AOPs. UV-C is UV light applied in the range of 254 nm and its source varies from the Sun, as also different kinds of lamps. UV radiation is commonly applied with oxidants such as H_2O_2, O_3, and Cl_2, catalyzing them or the production of related radical species. The application of UV lamps is still very expensive and uses electricity that is often not generated from sustainable sources. Great advantage of UV lamps, however, is the fact that they can be applied throughout the day and any day of the year, by disregarding the weather conditions. New types of lamps, such as UV-C LEDs and the lamp design rapidly raises the effectiveness of this promising technology. An example of this is the development of UV-C LED and light fabric combinations [87].

Photo-Fenton

Photo-Fenton (PhF) is based on the catalytic cycle of Fe species (Fe^{2+}/Fe^{3+}) and is promoted by the presence of H_2O_2 and UV–vis light for the production of hydroxyl radicals. Hydroxyl radicals are a highly reactive and non-selective radical species which can be generated by different processes. These fast reaction rates are ideal in effluents coming from membrane-based treatments such as UF and NF, which, in the case of the permeate volume contains minimal amounts natural organic matter (NOM) and other radical scavenging compounds, competing with the MCs for the generated radicals. The same goes for concentrate volumes containing high concentrations of MCs. Significantly, increasing the change for the hydroxyl radicals to encounter MCs for degradation [88]. One of the main disadvantages of classic PhF is that has to be applied at pH 3 to prevent the Fe to precipitate, making it necessary to acidify the UWW with costly chemicals, that come with danger during transport and storage. A solution to this problem is the application of Fe complexing agents, such as Ethylenediamine-N, N'-disuccinic acid (EDDS), which is an environmentally friendly solution as it is nontoxic and biodegradable [89,90]. When utilizing EDDS to keep the Fe in solution, the PhF process can be applied up to pH 9 [91].

Another oxidant that can be used in this AOP is persulfate. Reaction rates of persulfate radicals are generally slower and more selective as compared to hydroxyl radicals. Nevertheless, high efficiencies of MC removal in UWWS can still be obtained with this oxidant during (solar) PhF, being, in many cases, even a better choice, depending on the consistency of the matrix [92].

In areas with less sun hours and intensity, other AOPs are preferred. Electrooxidation (EO) is one of them, an AOP needing electricity instead of sunlight which can be generated by renewable energy, such as wind energy and modern biomass energy technologies. A further advantage of this technology is that it can also be deployed when there is very-low till no sun irradiation, such as during cloudy weather and at night [93]; or for wastewater treatment applications at a small scale in remote areas where no energy grid is available [94].

Another non-selective, relatively expensive, but highly effective AOP is ozonation. It can thereby not only be used as a MC degradation treatment, but also in different ways as a pretreatment of UWWTP effluents. Ozone is able to break down organic macromolecules, such as microbial cell walls, into shorter chain intermediate products, making it possible to enter other cells for biodegradation. This process generates the highly reactive hydroxyl radicals on the way. Thereby, ozonation can be applied at room temperature and ambient pressure and does not produce sludge, while residual ozone decomposes in water and oxygen [95].

Electrooxidation

EO and electrochemical AOPs (EAOP) have drawn a lot of attention lately, as they offer a high efficiency, cost effectiveness and environmental compatibility [96]. EO creates the possibility to directly electrooxidize absorbed MCs on the electrode surface, as well as the significantly larger production of different radicals into the matrix for MC degradation, such as $^{\bullet}OH$, ClO^{-} and $SO_4^{-\bullet}$. As the system needs wastewaters with high conductivity, and therefore high ionic loads, to easier generate an electron flow, EO is especially eligible for the treatment of concentrate volumes coming from membrane treated wastewaters. The higher the conductivity is, the lower the ohmic resistance, and so the required energy consumption. The production of the different radical species on the anode surface is directly related to the presence and concentration of the different ions. A common ion specie in wastewater is Cl^{-}, from which active chlorine can be generated, by the adsorption of free dissolved ions on the anode surface in the form of direct active chlorine species (ACS) formation. Indirect ACS formation takes place in the matrix, where the resulting compounds degrade the MCs. The higher the current density is, the higher the production of ACS [97].

One of the challenges within EO is the stability of the electrodes. It is therefore one of the most important subjects, and studies are mainly focused on the further development and application of the widely implemented boron doped diamond (BDD) electrodes. BDD's success is mainly due to its large potential window (2.4 – 2.6 V against a standard hydrogen electrode (SHE)), allowing high oxygen evolution overvoltage, and so permitting the oxidation of water into physisorbed hydroxyl radicals [94,98]. When a cathode is applied it is even possible to produce onsite H_2O_2, which contributes to the PhF process in the presence of Fe through the reduction of produced O_2 gas [93].

There are several processes through which EO can be applied. Anodic oxidation (AO) is the technique of direct oxidation of MCs on the anode surface by electron transfer, or by the oxidation of hydroxyl radicals, H_2O_2, O_3, ACS, and peroxynitrate produced on the anode surface [99].

Electro-Fenton (EF) is also an EO process, here chemicals such as Fe and H_2O_2 are used to produce hydroxyl radicals, by H_2O_2 through the Fenton process. In this case, H_2O_2 consumption as a consumable to be added, can be reduced by applying a cathode as mentioned before [100]. Variations on this AOP can be found by the combination of before mentioned EO processes with sunlight, known as solar assisted AO and (solar) photoelectro-Fenton. These combined processes have the advantage of the degradation by the sun light, or photolysis, as well as the activation of the oxidants and generation of the radical species by UVC. Thereby, the self-quenching effect of the free radicals is restrained, increasing the number of radicals, and so, the system oxidation capacity [101].

Ozonation

Ozonation is an efficient technology for the treatment of UWWTP effluents, where ozone is directly oxidizing the MCs in UWW, or indirectly through other processes by means of hydroxyl radicals. Ozone has a relatively slow dissolution rate and rapid decomposition in the aqueous phase. A solution to this problem can be found by producing ozone micro- and Nano-bubbles (MNB), significantly increasing the lifespan as they have higher mass transfer efficiency, less rising velocity, higher persistence time and bursting energy, and so, the reactivity in the aqueous phase. Another advantage is the much higher volume to surface ratio at nano scale, making the production of hydroxyl radicals more likely and easier [102]. Ozonation is often combined with other reagents such as H_2O_2 and UV irradiation, to obtain synergetic performances and catalytic effects towards the production of hydroxyl radicals. However, the H_2O_2/O_3 ratio and the pH should be carefully monitored, to avoid the over-production of hydroxyl radicals, inducing scavenging and their recombination. Furthermore, ozonation can also be combined with electro coagulation (EC) where the metal ions catalyze the ozone production to produce more hydroxyl radicals [70,95]. Switzerland, the first country in the world to enforce legislation related the treatment of UWW to a minimum of 80% MC removal, is widely applying ozonation, with or without GAC filters, proving the effectiveness of this technology [16].

A recent advance with this process in combination with membrane separation in a hybrid system is presented by Khalifa et al., where ozonation is used as an EC enhancer, facilitating the size-exclusion of the emerged SiC ceramic flat sheet microfiltration membrane for the treatment of oily wastewaters [47].

Nutrient recovery

Treated UWWs are characterized by containing a wide variety of different ions and other compounds. These ions and compounds can be recovered, which significantly contribute to the self-proficiency and environmental gains that could be obtained. Furthermore, other than with desalinized waters from RO, they can be utilized directly for crop irrigation, where even positive effects have been reported thanks to the consequent reduction on fertilizer use. It significantly reduces the needed quantities, and so, the crop production price. Water reuse for irrigation of crops by nutrient rich wastewaters is also called 'fertigation', and its application aims to significantly lower or even eliminate the use of mineral fertilizers [6,103,104]. The practice of fertigation has beneficial effects on the growth of crops and is expected to be increasing, as 35% of rainfed and 60% of irrigated crops is located within a 20 km proximity of a UWWTP, its practical implementation is thereby relatively accessible as well [105,106].

An example of this nutrient recovery is the recovery of ammonia from wastewaters. Nitrogen, along with phosphor and potassium, is one of the major components in fertilizers, as its availability as a macronutrient is essential to crop growth and development [107]. Ammonia can be recovered from UWWs by applying membrane-based systems such as NF to concentrate such wastewaters, and so creating ammonia enriched permeate streams [108,109].

Phosphate recovery from UWW

Phosphates account for another major part of used artificial fertilizers and can be recovered from UWW, such as from the dairy industry, winery and olive mill wastewater, and domestic and livestock wastewaters [110,111]. The recovery of phosphates from UWW decreases the risk of environmental disruption after their discharge, something which can result in the eutrophication of different surface waters [112]. The availability of mineral phosphates in the world is relatively low and rapidly decreasing, and so, contributes to geopolitical tensions, while its refinery process is an energy intensive one, strongly contributing to greenhouse gas emissions [113]. Phosphate species in UWW can be organic and inorganic and can be found in both solid and dissolved phase. Its removal can be performed through biological, chemical, and physical processes. Phosphate solids or particles in UWW can be removed by different clarification steps. One of the main technologies for the removal of dissolved phosphate in UWW is through the formation of struvite. The struvite is produced by the precipitation of dissolved phosphoric compounds coming from the before mentioned industries and urine present in UWW. Membrane technologies can offer different solutions to the production of struvite: Firstly, from the separation of urine from any possible unwanted solids coming from concentrated sources, such as urinary and mobile toilets, and collection basins used in intensive animal husbandry, as well as the separation of phosphate compounds coming from industrial sources; Secondly, in the pre-concentration of more diluted sources such as UWW or contaminated surface waters. A positive side effect is the prevention of undesired struvite formation on surfaces in the equipment and treatment processes of UWWTPs [114,115]. Recently, an advance in the recovery of phosphate has been found in the application of rare earth elements (REE) for its adsorption [116].

Both FO and MD can be applied to extract water from dewatered sludge centrate coming from anaerobic digesters used to digest sludge coming from the primary settler in UWWTPs or rest products coming from algae production from UWWTPs. The extracted water can directly be used for the irrigation of crops, whereas the retentate can be used as a liquid biofertilizer, from which struvite and nitrogen fertilizers can be produced by precipitation [117,118]. Simoni et al. compared two forms of MD, vacuum MD, and direct contact MD, for the application of simultaneous recovery of phosphorous and ammonia from UWW. Finding that vacuum MD showed better results at low pH values, and direct contact MD at high pH values [119]. Further novel applications of MD can be found in membrane crystallization through MD. Where traditionally MD is used to concentrate water and crystallization is performed by utilizing crystallizers and evaporators. Membrane crystallization through MD utilizes a membrane contactor, making it possible to use residual heat at temperatures as low as room temperature. This significantly reduces the energy consumption and thereby offers well-controlled nucleation and growth kinetics, fast crystallization rates, the promotion of heterogeneous nucleation or good control of the supersaturation by the membrane surface [120]. MD-crystallization already showed that it can be economically profitable, although is strongly depending on market value of the crystalized salts. Other factors contributing to the viability of this system is the membrane price, the overall mass transfer coefficient, and the membrane area, which depends strongly on the concentration of the osmotic agent [121].

Another recent advance in the production of struvite from UWW is the biomineralization by microorganisms to produce biological struvite. This principle could potentially be integrated in existing UWWTPs, by using one of the previously described MBR systems [122–124].

Toxicity of resources used for fertigation

One of the main concerns of the application of these water and nutrient sources is their toxicity induced by residual MCs and their degradation products, which could obstruct or prohibit the germination of seeds, as well as the root and shoot development [104,105]. Other concerns are changes in the soil microbiome and the accumulation of salts and heavy metals [125,126]. Therefore, multiple authors performed different toxicity tests to map the acute and chronic toxicities of created waters for crop irrigation, as well as the potential applicability based on soil and crop types [127–130].

When looking at the presence of MCs in precipitation products such as struvite, it has been found that MCs do not sorb themselves on the surface of these precipitation products. However, MCs can be included when struvite nucleates on colloidal particles containing them. MC presence has only been registered in significant lower concentrations as compared to land applied biosolids [131].

Lipid extraction from UWW

Another application of nutrient recovery can be found in the extraction of lipids from UWWs. Where, otherwise, these lipids are produced from different crops and livestock sources, even more increasing the pressure on available agricultural lands, water, and food products. Therefore, instead of producing them from raw food resources, these lipids can be recovered from UWWs as large amounts of them are originating from domestic use. Estimations of the total amount of lipids available in European UWWs depend on the specific treatment step and their yield but could be good for up to 24% of the EU diesel demand. Significantly contributing to the creation and closing its circular economy [132].

Main remarks and future outlook

Membrane separation has already shown its potential and wide array of applications for water and wastewater restoration, including combination with biotreatment for UWW effluents remediation or direct UWWTP effluent polishing. However, further treatment of rejection streams containing different contaminants at higher concentration than in the initial source is needed. The combination of different classes of membranes within an UWWTP, as well as the combination with AOPs and other wastewater treatment methods into hybrid systems is essential to obtain their maximum efficiency and synergy.

Another essential topic is fouling of all membrane materials. The solution to prevent and control fouling can be found in the hybridization of materials and composite membranes, with the use of stronger (but more expensive) ceramic membranes also being a choice. Ceramic membranes are better adapted to be combined with AOPs, including being part of the AOP treatment through the use of photocatalytic membranes. Otherwise, the efficiency of these novel membranes should be enhanced before being a consistent alternative.

Hydroxyl radicals generated by AOPs produce fast degradation reaction rates favored by higher concentration of contaminants. Therefore, they are ideal to be applied to effluents coming from rejection streams of membrane-based UWWTP effluent treatments, such as UF and NF.

More research focused on the economic viability of membranes in UWW treatment is desired, especially on large scale applications already installed in running UWWTPs. This will help to convince the short-term, as well as the long-term advantages of the already existing technologies, freeing subsidies and helping in the further development of membrane systems used for UWW treatment.

Membrane technologies must be considered as part of the UWW reclamation strategy, even more when recovery of nutrients and added value substances are to be tackled. Membranes significantly increase process efficiency, by offering economically and sustainable competing alternatives to the use of artificial fertilizers coming from energy extensive production in the case of nitrogen and phosphorus compounds.

Declaration of Competing Interest

The authors declare that they have no known competing financial interest or personal relationship that could have appeared to influence the work reported in this paper.

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