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Micropollutants removal from secondary-treated municipal wastewater using weak polyelectrolyte multilayer based nanofiltration membranes

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ABSTRACT

Nanofiltration (NF) is seen as a very promising technology to remove micropollutants (MPs) from wastewater. Unfortunately this process tends to produce a highly saline concentrate stream, as commercial NF membranes retain both the MPs and most of the ions. The high salinity makes subsequent degradation of the MPs in a bioreactor very difficult. The main goal of this study is to prepare and study a NF membrane that combines a low salt rejection with a high MPs rejection for the treatment of secondary-treated municipal wastewater. This membrane was prepared using layer by layer (LbL) deposition of the weak polycation poly(allylamine hydrochloride) (PAH), and the weak polyanion poly(acrylic acid) (PAA), on the surface of a hollow fiber dense ultrafiltration (UF) membrane. The ionic strength of the coating solutions was varied and properties of the formed polyelectrolyte multilayers (PEMs), such as hydration, hydrophilicity, hydraulic resistance and ions retention were studied. Subsequently we tested the apparent and steady state rejection of MPs from synthetic wastewater under cross-flow conditions. The synthetic wastewater contained the MPs Diclofenac, Naproxen, Ibuprofen and 4n-Nonylphenol, all under relevant concentrations (0.5–40 μg/L, depending on the MP). PEMs prepared at lower ionic strength showed a lower hydration and consequently a better retention of MPs than PEMs prepared at higher ionic strengths. A strong relationship between the apparent rejection of MPs and their hydrophobicity was observed, likely due to adsorption of the more hydrophobic MPs to the membrane surface. Once saturated (steady state), the molecular size of the MPs showed the best correlation with their rejection, indicating rejection on the basis of size exclusion. In contrast to available commercial NF membranes with both high salt and MP rejection, we have prepared an unique membrane with a very low NaCl retention (around 17%) combined with a very promising removal of MPs, with Diclofenac, Naproxen, Ibuprofen and 4n-Nonylphenol being removed up to 77%, 56%, 44% and 70% respectively. This membrane would allow the treatment of secondary treated municipal wastewater, strongly reducing the load of MPs, without producing a highly saline concentrate stream.

1. Introduction

Over the last few years, a great concern has been highlighted regarding the occurrence of micropollutants (MPs) in aquatic resources and the subsequent effects on humans and the environment [1]. In addition to the 45 priority substances on the European Watch List (Directive, 2013) [2], an additional watch list of 10 priority substances that should be monitored within the European Union was recently included in Decision 495/2015/EU [3] indicating the growing attention to this issue. In this regard, effluents of wastewater treatment plants have been recognized as the main entry point of these compounds into the aquatic environment [4]. Conventional treatment methods do not lead to sufficient removal of MPs, and adding additional steps during wastewater treatment is seen as the most promising way to reduce the release of these compounds into surface waters [5]. To date, identification of technically and economically feasible advanced wastewater treatment options for the elimination of MPs from secondary-treated effluent is ongoing. Adsorption processes, advanced oxidation processes (AOPs) and membrane filtration are important examples of such technologies. Among these options, listed in Table 1, membrane technologies such as nanofiltration (NF) and reverse osmosis (RO) have attracted a great interest because of high removal rates (> 90%) of low molecular weight MPs, excellent quality of treated effluent, modularity and the ability to integrate with other systems. On the other hand,

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Table 1
The most-frequently used treatment technologies for removal of MPs from secondary-treated municipal wastewater.

Category of tertiary treatment	Subcategory	Advantages	Disadvantages/limitations	References
Advanced oxidation processes (AOPs)	Ozonation	Remarkable capability for removing most of the pharmaceuticals and industrial chemicals	As O_3 is a highly selective oxidant, ozonation often cannot ensure the effective removal of ozone-refractory compounds such as Ibuprofen.	[36]
		It has been successfully applied in many full-scale applications in reasonable ozone dosages.	Ozonation produces carcinogenic bromate from bromide that exists in secondary-treated effluents.	[36,37]
	Fenton oxidation	This kind of system is attractive because it uses low-cost reagents, iron is abundant and a non toxic element and hydrogen peroxide is easy to handle and environmentally safe.	In this process, the low pH value often required in order to avoid iron precipitation that takes place at higher pH values. This process is not convenient for high volumes of wastewater in full-scale applications.	[2,38]
	Heterogeneous photocatalysis with TiO_2	The principle of this methodology involves the activation of a semiconductor (typically TiO_2 due to its high stability, good performance and low cost) by artificial or sunlight.	The need of post-separation and recovery of the catalyst particles from the reaction mixture in aqueous slurry systems can be problematic. The relatively narrow light-response range of TiO_2 is one of the challenges in this process. This process is not convenient for high volumes of wastewater in full-scale applications.	[38]
	Photolysis under ultraviolet (UV) irradiation	Photo-sensitive compounds can be easily degraded with this method.	UV irradiation is a high-efficient process just for effluents containing photo-sensitive compounds. This process is not convenient for high volumes of wastewater in full-scale applications. The addition of H_2O_2 to UV is more efficient in removing MPs than UV alone, but UV/H_2O_2 is a viable solution for the transformation of organic MPs with low O_3 and ·OH reactivity.	[38]
	Ultrasound irradiation (Sonolysis)	It is a relatively new process and therefore, has unsurprisingly received less attention than other AOPs. But it seems that this process is economically more cost-effective.	There are very few studies and consequently rare experience about sonolysis of the effluent MPs.	[39]
Adsorption processes	Adsorption processes with activated carbon	It has been identified as powerful and easily adjustable technology to remove MPs.	This process should be followed by a final polishing step (sand filtration or UF membranes) to retain adsorbed contaminants and spent activated carbon. So higher energy requirements of UF membrane and the relatively high carbon dosage (up to 20 mg/L) necessary to achieve the required MPs removal.	[5]
		Large-scale trials have not only demonstrated excellent removal (> 80%) of a broad range of micropollutants, but also contributed to reducing the effluent toxicity.	In the case of “granular activated carbon”, a regeneration process of the spent carbon is required, while spent "powdered activated carbon" must be incinerated or dumped after filtration process.	
Membrane filtration	RO and NF membranes	These processes have attracted a great interest because of higher removal rate of low molecular weight PSs, excellent quality of effluent, modularity and ability to integrate with other systems despite their fouling problems.	High quantities of cations, anions, sulfate, MPs, etc. in the concentrate produced in NF and RO processes compel wastewater managers and decision makers to treat it with complicated processes specially in the case of full-scale applications. High energy consumption (about 4.7 and 3.4 kW h/m^3), high capital (334.3 and 338.2 \$/m^3/d) and operational costs (0.72 and 0.57 \$/m^3) of RO and NF membranes, respectively, and their problematic fouling issues may preclude membrane treatment as an option.	[6,40,41]

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