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Research article

Removal of ciprofloxacin from seawater by reverse osmosis

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A B S T R A C T

Much of the deterioration of water resources is anthropogenically caused as a consequence of the incessant production of chemical compounds to obtain the quality of life that society demands today. This constant presence and harmful accumulation of these pollutants in different ecosystems have seen them emerge as a major concern both for human health and for environmental safety. Scientific advances have succeeded in legislating against, reducing and even eliminating priority pollutants, while new technologies are being constantly developed to identify and treat newly emerging pollutants. The objective of this work is the evaluation of the seawater reverse osmosis membrane as a method for the removal of an antibiotic present in seawater. The novelty of the study is that the tests were undertaken using water of high ionic strength. A critical selection of the antibiotic to be used in the study was carried out. The experiments were performed under constant pressure conditions, employing synthetic seawater in a pilot-scale unit with a commercial spiral-wound reverse osmosis membrane. Results are shown in terms of selectivity of the reverse osmosis process for antibiotic removal. The RO membrane element successfully reject most of the ciprofloxacin (removal rate >90%), with maximum rejection value of 99.96%.

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1. Introduction

Emerging pollutants (EPs) reach the environment from a variety of anthropogenic sources (Cámpos Gómez, 2003) and are distributed throughout all environmental matrices. Population growth and economic development (Barceló and López de Alda, 2008) contribute every day to the increased presence of these compounds. As a result of their high production levels (Bolong et al., 2008) and continuous emission into the environment, they have a negative impact on the environment irrespective of their degree of persistence (Petrovic et al., 2003). These types of pollutant are unregulated compounds (Noguera-Oviedo and Aga, 2016), characterized by appearing at mostly low concentrations (in the order of μg/l) (Noguera-Oviedo and Aga, 2016; Laprowth et al., 2012). They comprise a wide variety of chemical compounds, with the greatest concern centered on flame retardants, pesticide metabolites, drugs and their products of degradation through human waste and hormones (particularly estriol, 17-β-estradiol and 17-α-estradiol), pharmaceutical and personal care products and "life-style" compounds (Barceló and López de Alda, 2008; Gil et al., 2012; Kimura et al., 2003).

The main route of intrusion into the environment of these pollutants comes from wastewater effluents that are discharged into the sea in most cases. For this reason, technological applications tend to focus on improving treatments of residual waters. As these treatments are designed to generally eliminate solids and fecal contamination, most do not specifically address the removal of EPs (Bolong et al., 2008; Petrovic et al., 2003; Andreozzi et al., 2003; Fischer et al., 2017). In fact, numerous scientific studies conducted in purified wastewater confirm EPs presence at trace levels (μg/L) or even higher (Sultanaet al., 2017). On the other hand, the regeneration of purified wastewater allows completion of the entire water cycle, of particular importance in areas with scarce natural water resources, using different technologies including disinfection, reduction of salinity, etc.

Membrane-based processes have been gradually introduced in the regeneration process of purified wastewater in both secondary and tertiary treatments (Martinez et al., 2015), and are one of the main technologies which are also used in brackish water and seawater desalination. This widely implemented technology has proven to be a promising alternative (Bolong et al., 2008; Oller et al., 2011; Kimura et al., 2003), but there remains a need to evaluate it in terms of removal of these new micro-pollutants, which could

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double its effectiveness as a wastewater treatment process (WWTP).

In some cases, especially in coastal areas, the water intakes of seawater desalination treatment plants are located near the submarine outfalls of wastewater treatment plants. Consequently, if EPs are present in the discharge water they could potentially be reintroduced in the water cycle via the desalination plant water intake. As a result, research has tended to focus on the response evaluation of spiral wound reverse osmosis membranes as an EPs removal technology.

It should be noted that in Spanish legislation, and in particular Royal Decree 140/2003 of 7 February (BOE, 2003) which determines health criteria for the quality of drinking water, EPs substances are non-regulated. Many EPs compounds are new, and existing legislation may well need to be adapted if they are proven to affect human health.

1.1. Membrane technologies as pollutant treatment and removal method

Membrane technologies involve processes in which semi-permeable membranes acting as selective physical barriers are used to divide the feed stream into a permeate and a concentrate stream, thereby separating contaminants present in the wastewater (De la Macorra García and González, 2014). The main advantages of using these technologies include the removal of contaminants that are at low concentrations and/or dissolved in colloidal form. In addition, they allow continuous operation at moderate temperatures and with high selectivity control. These systems have a simple flow diagram, in addition to allowing modular and compact designs which do not require a lot of space, combine easily with other treatments and have no need for the use of additive products (Mulder, 1996; Rodríguez et al., 2006).

Among membrane technologies, reverse osmosis (RO), also known as hyper-filtration, is a membrane separation process which allows the removal of very small particles such as monovalent ions with diameters in the order of 10^{-3}-10^{-4} μm. As mentioned above, there is a wide variety of emerging pollutants. Similarly, there are a large number of commercial RO membranes. Both membranes and contaminants have different physicochemical properties (Nghiem and Fujioka, 2016; Van der Bruggen and Vandecasteele, 2002). In the investigations of Dan Libotean et al. (2008), the relationship (both qualitative and quantitative) between the chemical structure of the pollutants and the RO membranes is demonstrated. Attending to the aforementioned work and the references cited therein, the rejection rate of EPs is governed by the physicochemical properties of the EPs (e.g. molecular size, charge, hydrophobicity, etc.) and the membrane element characteristics (e.g. configuration, pore size, permeability, etc.) and the operating conditions of the process (e.g. pressure, flux, ionic strength of the feed water, temperature, etc.). Therefore, the present study aims to investigate the evaluation of a spiral-wound reverse osmosis membrane on the removal rate of an antibiotic present in seawater. In order to study the possible interaction between de RO membrane element and the antibiotic a standard polyamide thin-film composite membrane element and a common antibiotic was selected.

1.2. Pharmaceutical products as pollutants

Although, at global scale, caffeine is one of the compounds most consumed by humans, studies such as those by Rosal et al. and Teijón et al. show that at national level (in Spain), the presence of pharmaceutical compounds in WWTP exceeds that of this psychoactive drug (1.589 μg/l), with recorded values of 5.714 μg/l in the case of gemfibrozil and values higher than 2 μg/l for the antiepileptic carbamazepine (Rosal et al., 2010; Teijon et al., 2010; Matamoros and Salvadó, 2013). A European study that includes the analysis of 90 WWTP again confirmed higher values of pharmaceutical products compared to the 3 μg/l maximum concentration of caffeine (Loos et al., 2013). In view of the above, it was decided to concentrate on the pharmaceutical sector for the development of the present research study.

The global pharmaceutical market currently represents revenues for pharmaceutical companies of over one trillion dollars, and continues to grow due to an ever-growing access to an increasingly comprehensive supply of pharmaceutical drugs, both in industrialized and developing countries (European Environment Agency, 2010; Arzneimittelhersteller, 2015).

From an environmental point of view, pharmaceuticals have distinctive characteristics compared to conventional chemical contaminants (Cortacans Torre et al., 2006). In fact, these products are made up of large and chemically complex molecules, with very diverse structure, molecular weight and shape. Generally, the molecules are polar and have more than one ionizable group. The pH of the solution in which the molecules are found will affect both their properties and the degree of ionization. This type of compound is quite persistent in the environment, with a permanence from more than one year to several years, as is the case of clofibric acid. Drugs appear in the environment both in their initial form and through their metabolites, which are more polar and water-soluble.

Pharmaceutical products are developed to be biologically active, and there is a strong possibility that both the pharmaceutical residues and their metabolites or degradation products may have a difficult-to-predict eco-toxicological impact on the environment (la Farré et al., 2008). One of the biggest concerns in recent years has been the presence of antibiotics in the environment because they may favor the development of resistant bacterial strains, which would render ineffective this type of medication (Noguera-Oviedo and Aga, 2016). An Australian study analyzed the resistance of two bacterial strains (*Escherichia coli* and Xanthomonas maltophilia) to six antibiotics detected in WWTP effluent (ciprofloxacin, tetracycline, ampicillin, trimethoprim, erythromycin and sulfamethoxazole) confirming that bacterial strains showed resistance to all six antibiotics (Costanzo et al., 2005).

1.2.1. Antibiotics

The term antibiotic can be defined as a substance obtained by chemical modification of natural compounds or through the synthesis of chemical compounds (Kümmerer, 2009) that inhibits or stops the growth of microorganisms such as bacteria, protozoa and fungi. The recognition of antibiotics as agents for the prevention and treatment of infectious diseases has ensured continuous growth of the market (Gothwal and Shashidhar, 2015), generating a total dependence and intensive use which have caused the parallel appearance of microorganisms resistant to them. This is reflected in the increased difficulty in treating some diseases (Davies and Davies, 2010), and results in a doubly negative impact affecting both people and the environment.

After an intensive bibliographic review of the long list of antibiotics, the type selected for this study belongs to the fluoroquinolone (FQs) class. This is due to their high rate of detection and influx in treated effluents worldwide. In China, a study undertaken by Dong et al. of 19 antibiotics reported that these compounds had one of the highest concentrations detected (5.411 μg/l) (Dong et al., 2016).

In another study in Australia, Watkinson et al. evaluated the presence of 28 antibiotics, where again this class, and more precisely ciprofloxacin fluoroquinolone, had one of the highest concentrations (4.6 μg/l) (Watkinson et al., 2007). In Spain, very high maximum concentrations of antibiotics have been recorded. Most

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