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Phosphorus recovery from waters using nanofiltration

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ABSTRACT

Recovery of phosphorus has been gaining importance due to its natural scarcity and expensive price. In this work, nanofiltration (NF) has been tested as a technology to treat surface waters with phosphorus and simultaneously recover that phosphorus. Two membranes of same material, but different molecular size were tested (NF270 and NF90). Results showed that membrane fluxes were more affected by the presence of salts, natural organic matter, and especially microcystins than phosphorus. In addition, phosphorus removals were very high (>90%) and varied with the membrane type, feed conductivity, and dissolved organic carbon (DOC). The increase in feed conductivity and DOC originated higher phosphorus removals. Furthermore, the presence of microcystins did not affect the high-phosphorus removals. Results show that more than 96% of the total phosphorus mass can be recovered using NF membranes. NF is therefore a technology that should be considered for phosphorus recovery from surface waters.

Keywords: Phosphorus; Recovery; Removal; Nanofiltration; Surface waters

1. Introduction

Element phosphorus (P) is essential to all life (e.g. plants, animals, and bacteria) and is a key ingredient in fertilizers to sustain high-crop yields. Phosphorus has no substitute in food production and in a world of nine billion people by 2050, securing sufficient phosphorus will be critical for future growing food needs [1]. Phosphorus is also used in products such as detergents, matches, grenades, and flares. Phosphorus is not widely circulated on the globe, because the flux from land to water is substantial but the reverse flux is extremely limited [2,3]. Specifically, the latter flux occurs only via fishery and seabirds' droppings, unlike nitrogen, which is released into the atmosphere through denitrification [4].

Today, the world main source of phosphorus—mined rock phosphate—is non-renewable, and is becoming increasingly scarce and expensive [5]. Peak phosphorus was estimated to occur by 2035, after which demand would outstrip supply [1]. In addition, phosphorus is a key limiting nutrient in eutrophication of lowland rivers and lakes [6,7], the warmer temperatures and the excessive amount of P in water can contribute to eutrophication and is sometimes responsible for the lack of clean water resources [4]. The major sources of phosphorus to rivers and lakes

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are sewage/industrial effluents (point sources) and agricultural run-off (diffuse sources). In addition, the rising temperatures lower the water quality in lakes through increased thermal stability and altered mixing patterns, resulting in reduced oxygen concentrations and increased release of phosphorus from sediments [8]. Thus, there is an international concern about phosphorus in water, and the European Union (EU) Water Framework Directive requires widespread control of phosphorus inputs to rivers to sustain/improve river and lakes ecology [9]. Phosphorus is targeted under water directive because of its contribution to eutrophication of surface waters, proliferating phytoplankton, and epiphytic and benthic algae [6,10]. The excessive growth of algae may cause water quality problems, including odor, taste and cyantoxins, and therefore, unsafe drinking water [11].

Development of technologies for phosphorus removal started in 1950 [12], in response to the issue of eutrophication and the need to reduce the levels of phosphorus entering surface waters. Removal was initially achieved by chemical precipitation, which remains the leading technology today. More recently however, biological phosphorus removal has become firmly established, crystallization technology has also completed its progress towards commercialization and technologies extending chemical precipitation to assist nutrient removal are beyond the pilot stage. Other relevant technologies are at various stages of development or have been investigated as stated by Morse [12].

To increase the phosphorus recovery, advanced water treatment process like membrane processes can be used. Nanofiltration (NF) is one attractive process for selective phosphate recovery. It removes multivalent ions, small hazardous organic compounds (e.g. pesticides, endocrine disruptors), microcystins, and natural organic matter (NOM) between other compounds. The membrane acts as a physical barrier, allowing water to pass while retaining the suspended solids and even dissolved materials [13], depending on the type of the membrane and operation conditions. In this context, this study pretends to evaluate the use of NF to treat and recover phosphorus from surface water, contributing to the requirements of EU Water Directive and recover phosphorus.

2. Materials and methods

2.1. Membranes

In this study, two different commercial NF membranes of polyamide, NF90 and NF270 kindly supplied by Dow Filmtec, USA, were used. Membranes were first compacted and stabilized with pure water, and then were characterized for pure water permeability and molecular weight cut-off (MWCO). MWCO was obtained by permeation of neutral solutes of different molecular weights between 46.1 and 594.5 g/mol, and determined using the method proposed by Rosa and de Pinho [14]. The characteristics of the membranes used in experiments are presented in Table 1. NF270 was considered a loose NF membrane, while NF90 a tight NF membrane [15,16].

2.2. Water samples

Two models and three surface water samples were studied. Model waters were constituted of deionized water (DI) supplemented with KCl (1 mM) and KH_2PO_4 and with KCl (1 mM), $CaCl_2$ (1 mM), and KH_2PO_4 (reagents of analytic grade). The surface waters were from Funcho (in Algarve, Portugal) and Amoreiras (in Alentejo, Portugal) reservoirs and were also supplemented with KH_2PO_4 and microcystins-LR (MC) (10 μg/L). Another sample of Funcho surface water was also studied, which was supplemented with KH_2PO_4 but without MC. Microcystins were extracted from a culture of *Microcystis aeruginosa* supplied by Pasteur Culture Collection (PCC7820) and maintained in laboratory. Stock solutions of microcystins were prepared as already described elsewhere [20]. The characteristics of these waters are presented in Table 2.

2.3. Nanofiltration experiments

Laboratory NF experiments were performed in a commercial bench-scale plate and frame unit (M20 unit), using four membranes corresponding to an area of 720 cm^2. Experiments were performed at a constant pressure of 10 bar and temperature of 21.0 ± 0.1 °C.

Two sets of experiments were made. In the first set, experiments with DI as feed water were performed with both types of membranes. The objective of these experiments was to evaluate the efficiency of the selected membranes to remove phosphorus. For the more efficient type of membrane, the recovery of phosphorus was evaluated using surface waters, which constituted the second set of experiments. Experiments took between 1 and 1.5 h for DI and between 1.5 and 3 h for surface waters. Experimental details are presented in Table 3.

NF experiments at different water recovery rates (WRR) were performed using the method described previously [20]. All samples from the feed, concentrate, and permeate solutions, taken at different recovery rates, were analyzed for microcystins,

Table 1
Characteristics of the membranes used in experiments

Membrane	Pure water permeability (21°C) l/(h m^2 bar)	MWCO (Da)	Zeta potential at pH 7 (KCl solution at 30 μS/cm) (mV)	pH of isoelectric point	Observations
NF270	10.66	200	−21.6 [17]	3.5 [18]	Hydrophilic, high-salt rejection [19]
NF90	7.63	110	−24.9 [17]	4 [18]	Hydrophobic, almost complete salt rejection [18]

According with Plakas and Karabelas [17], Nghiem et al. [18], and Mänttäri et al. [19].

Table 2
Characteristics of the waters used in the experiments

Water types		pH	Conductivity (μS/cm)	Phosphorus (mg P/L)	DOC (mg C/L)	TOC (mg C/L)	UV_{254} (1/cm)	MC (μg/L)
Model	DI + KCl + P	5.6 ± 0.0	119.7 ± 0.1	0.66 ± 0.5	–	–	–	–
NF270	DI + KCl + $CaCl_2$ + P	5.6 ± 0.0	408.3 ± 0.6	0.66 ± 0.5	–	–	–	–
Model	DI + KCl + P	5.7 ± 0.0	141.5 ± 0.1	0.66 ± 0.5	–	–	–	–
NF90	DI + KCl + $CaCl_2$ + P	5.8 ± 0.0	390 ± 20	0.66 ± 0.5	–	–	–	–
Surface	FW + P	7.5 ± 0.0	417 ± 1	2.2 ± 0.5	2.06 ± 0.02	2.41 ± 0.08	0.08 ± 0.00	–
	FW + MC + P	7.6 ± 0.0	371 ± 0	2.2 ± 0.5	2.50 ± 0.05	3.28 ± 0.02	0.02 ± 0.00	8.59
	AW + MC + P	8.3 ± 0.0	624 ± 0	2.2 ± 0.5	–	–	0.1 ± 0.00	8.32

DI: deionized water; FW: Funcho water; AW: Amoreira water; P: phosphorus; DOC: dissolved organic carbon; TOC: total organic carbon; UV_{254}: UV absorbance at 254 nm; MC: microcystins-LR.

Table 3
Experimental details

Membrane	Water types	Experiment details (WRR, 10 bar, 21 ± 0.1°C)	Main parameter examined	Other parameters examined
NF270	DI + KCl + P	58 min	P	Conductivity, pH, permeate flux
	DI + KCl + $CaCl_2$ + P	62 min		
NF90	DI + KCl + P	83 min	P	Conductivity, pH, permeate flux
	DI + KCl + $CaCl_2$ + P	85 min		
NF90	FW + P	91 min	P	DOC, conductivity, pH, permeate flux
	FW + MC + P	212 min	P, MC	DOC, conductivity, pH, permeate flux
	AW + MC + P	188 min	P, MC	DOC, conductivity, pH, permeate flux

DI: deionized water; FW: Funcho water; AW: Amoreira water; P: phosphorus; MC: microcystins-LR; DOC: dissolved organic carbon.

dissolved organic carbon (DOC), pH, and conductivity. Flux was continuously measured during the experiments. At the final of experiments and between each NF run, membranes were washed until the pure water flux reached 98% of the initial value measured after compaction and the bulk conductivity was similar to that of DI.

2.4. Analytical methods

Samples were analyzed for pH (at 25°C, using a Whatman WTW pH340 meter), conductivity (Crison GLP32 conductimeter), DOC, respectively (Shimadzu TOC 5000A analyzer, 50 ppb to 4,000 ppm), phosphorus (Spectronic Unicam UV300 UV/VIS spectrophotometer) using standard methods of analysis [21]. Triplicate samples of all NF streams were taken to minimize statistical variance of the results. When the standard deviations of the samples were higher than the mean, the measurement was rejected and a new determination was made. All analyses were made within 24 h, and blank samples were used as control.

Microcystins were analyzed by HPLC-PDA using a Dionex Summit system, which includes a high-pressure gradient pump Dionex Summit, an autosampler Dionex ASI-100, a column oven Dionex STH-585, and a photodiode array detector Dionex PDA-100. A C18 column was used (Merck Purospher STAR RP-18 endcapped, 3 μm particles, LiChroCART 55 × 4 mm). The mobile phase used a gradient of milli-Q water and acetonitrile, both with 0.05% (*v*/*v*) of trifluoracetic acid. Chromatograms were analyzed between 180 and 900 nm, with a main detection at 238 nm for the typical microcystins spectra [22,23].

3. Results and discussion

3.1. Phosphorus removal

Phosphorus removals for model and surface waters are presented in Fig. 1. In this figure, the impact of feed conductivity and organic matter (by DOC) in the phosphorus removal is shown.

For the loose NF270, phosphorus removal ranges between 69 and 75% for the model water with KCl, and between 60 and 97% for the model water with $CaCl_2$ (Fig. 1(a)). An increase in phosphorus removal is observed with salt concentration, more evident when $CaCl_2$ is present, due to the ionic strength increase in bulk feed water. Increasing solution ionic strength leads to the compression of the membrane surface double layer, resulting in a reduced electrostatic interaction between both [15].

For the tight NF90 membrane, high removals of phosphorus are presented in Figs. 1(a) and (b), for model and surface waters, respectively. Results show that MC do not influenced the removal of phosphorus (Fig. 1(b)). Removals vary between 94 and 97% with KCl, 93 and 94% with $CaCl_2$ (Fig. 1(a)), and between 85 and 98% for surface waters (Fig. 1(b)). Results clearly show that removal of phosphorus is influenced by feed water conductivity and DOC, with a correlation between them. For model waters (without DOC, Fig. 1(a)), there is a clear increase of phosphorus removal with salt concentration for both membranes, except for NF90 with calcium due to the high-feed conductivity and consequently high-ionic strength. For surface waters (waters with DOC, Fig. 1(b)), phosphorus removal also increases with the increase of both feed conductivity and DOC. According to Niewersch et al. [24], the feed solution composition influences the retention of each species due to the required electroneutrality that includes all the ions in solution and the building of electric double layers on the membrane surface. Multivalent cations will be retained by membrane and indirectly will increase the total concentration of the anions in feed water (Fig. 2), to maintain the electroneutrality of the solution—the existence of a cation always implicates the existence of its counter-ion in solution, the anion. If different species of anions are in solution they complete each other for the cationic counter-ion. This mechanism increases the removal of the ions as the concentration of the salt ions increase in feed water (Figs. 1 and 2). In addition,

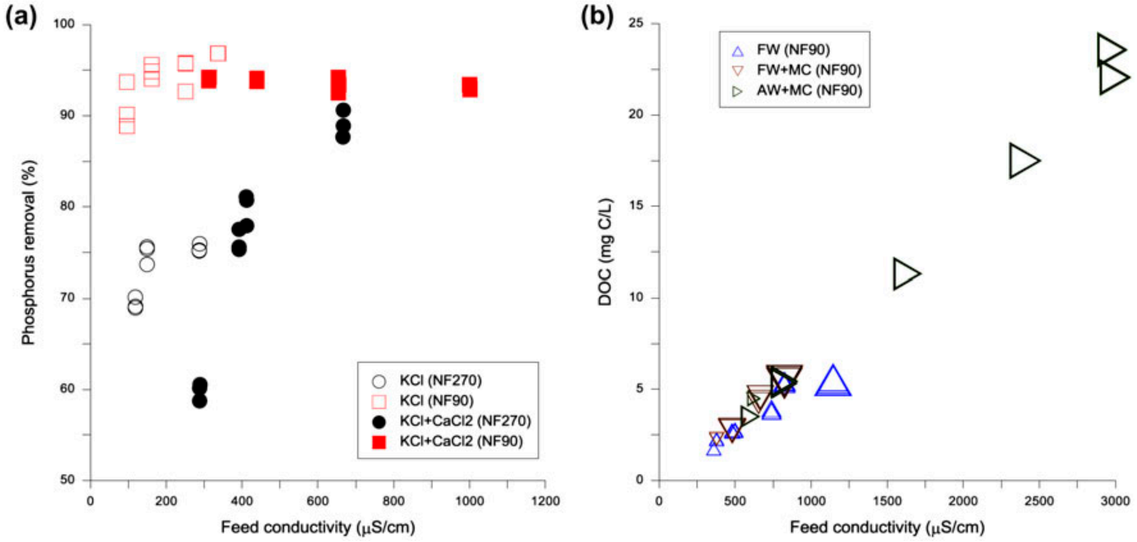

Fig. 1. Variation of phosphorus removal with feed conductivity and DOC (a) model and (b) surface waters (the smallest symbols represent phosphorus removals of 85% and the largest symbols removals of 98%).

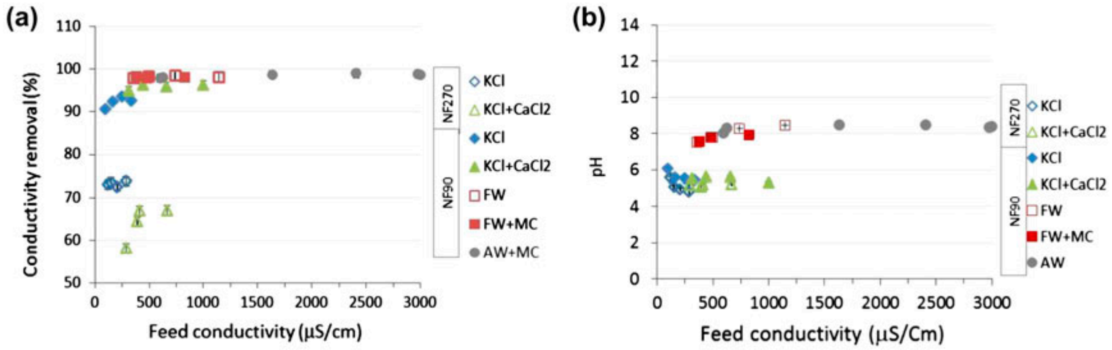

Fig. 2. (a) Conductivity removal with feed conductivity for model and surface waters; and (b) variation of pH with feed conductivity.

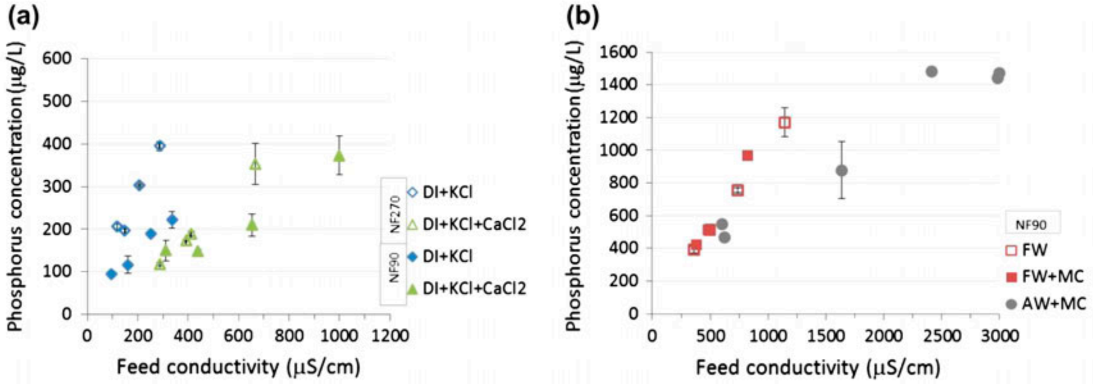

Fig. 3. Phosphorus recovery with feed conductivity for (a) model waters and (b) surface waters (Funcho water (FW) and Amoreiras water (AW) using NF90).

the phosphorus species in water vary with pH (Fig. 2(c)) as reported by Hong et al. [25]. At pH ≤6 the phosphate dominant specie present in water is $H_2PO_4^-$, with a molecular weight of 97 g/mol and a Stokes radius of 0.256 nm, while at pH ≥7 the phosphate dominant specie is HPO_4^{2-} with a molecular weight of 96.0 g/mol and a Stokes radius of 0.323 nm [25]. As surface waters present a higher pH than model waters (Fig. 2(c)), bigger phosphate species are present in surface waters and removal is higher. NOM may also contribute to phosphorus removals due to the increase in repulsions forces between the membrane surface and NOM or phosphorus.

Fig. 2 shows that salt removal is higher for NF90 membrane than for NF270, and for the latter membrane, water with $CaCl_2$ shows the lowest removals. The loose NF270 membrane presents a salt removal sequence that follows the Donnan exclusion theory ($KCl > CaCl_2$), because an increase in the order of cation charge density, resulted in low removals (attraction forces on the cations became stronger) [26]. In this case, the predominant mechanism of salt removal is charge exclusion. The tight NF90 membrane presents some Donnan theory discrepancy ($CaCl_2 > KCl$). This removal sequence is mostly associated to the ions dimensions, since Ca^{2+} hydrated radius is larger than K^+ [27] and therefore, in this case, the mechanism of size exclusion is the most applicable. López-Munoz et al. [16] obtained similar results

Table 4
Mass of phosphorus recovered in concentrate for NF90 membranes

Water type	Phosphorus recovered (%)
KCl	97.1
$KCl + CaCl_2$	96.0
FW	96.7
FW + MC	99.6
AW + MC	98.6

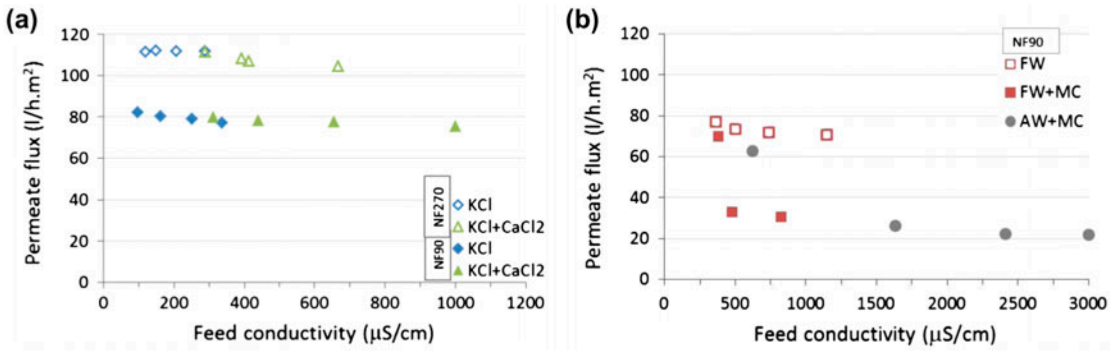

Fig. 4. Variation of permeate flux with feed conductivity for (a) model and (b) surface waters.

using NF270 and NF90 membranes for removal of NaCl, $CaCl_2$, and Na_2SO_4 salts.

3.2. Phosphorus recovery

The high removals of phosphorus (Fig. 1) indicate high-phosphorous concentration in the NF concentrate stream and thus high-phosphorus recovery. Fig. 3 shows the variation of phosphorus concentration in concentrate stream with feed conductivity and Table 4 the mass of phosphorus recovered at 86% of water recovery rate, determined by mass balance calculations. As conductivity increase, the concentration of phosphorus in concentrate stream also increases for all studied waters (Fig. 3). However, the increase in recovered phosphorus is much more pronounced for surface waters, because the initial concentration of phosphorus in surface waters was higher, as well as the ionic strength and the DOC, as explained in last section. Results also show that using tight NF90 membranes, it is possible to recover more than 96% of phosphorus present in surface water (Table 4), which shows the importance of this process in phosphorus recover.

3.3. Permeate flux

Fig. 4 presents the variation of the permeate flux with feed conductivity for the model waters using both membranes, and for surface waters using NF90. NF270 membrane has a higher permeate flux than NF90, related with its higher permeability. Results indicate that permeate fluxes decrease with feed conductivity increase, more important for waters with calcium (Fig. 2(a)). The decrease is more pronounced in NF270 membrane, due to the narrowing effect of pores as a result of calcium adsorption and increase in membrane charge (become less negative). Also, the osmotic pressure near membrane surface is high as a result of the high-salts removal, especially for NF90 (Fig. 2(a)), which reduces the salts transfer across the membrane and leads to a flux decrease [28]. Jarusutthirak et al. [29] obtained similar results for solutions with divalent (Ca^{2+}) and monovalent (Na^+) ions.

For surface waters, results show a higher flux decrease with feed conductivity than for model waters, due to the presence of NOM and its fouling character [5–7]. In addition, this decrease is much more pronounced when MC are present (Fig. 4(b)), since MC are much bigger than the membrane size (~1,000 g/mol to 110 Da [30]). However, the decrease in flux observed for waters with MC is much more pronounced than obtained in previous studies [19,31], explained by the tight characteristics of the NF90 membranes used in this study.

4. Conclusions

This study shows that NF can be used for both treat and recover phosphorus from surface water reservoirs. The main conclusions are:

(1) Phosphorus can be recovered from surface water using tight NF membranes, with recovers higher than 96%.
(2) Removals of phosphorus were higher than 90%, and microcystins did not affected the phosphorus removal; removal increased with feed conductivity and DOC due to electroneutrality effects, pH, and the presence of NOM.
(3) The presence of phosphorus did not affect significantly the membrane permeate flux in model waters or surface waters–calcium, NOM, and microcystins are the major cause of membrane fluxes decrease.
(4) NF is a technology that can be used to recover phosphorus from surface waters and simultaneously produce water with good quality.

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