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Application of reverse osmosis process associated with EDTA complexation for nickel and copper removal from wastewater

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ABSTRACT

In this study, low-pressure reverse osmosis was analyzed to remove heavy metal ions (nickel and copper) from highly diluted feed flows. Given the ability of these ions to form complexes with EDTA, the effectiveness of a preliminary stage of complexation was also evaluated. The experimental system consisted of a reactor with commercial flat membrane of polyamide and cross flow. Studies employing solutions of single ions and also mixture of ions in different concentration ranges reached removals of 99%. The complexing agent forms a larger complex than the single ion in aqueous phase, thus increasing ion removal in terms of final concentration in permeate. Experiments made at different pressures showed the increase in pressure has an effect on increasing rejection and permeate flow, but the applied pressure of 0.5 MPa is sufficient to achieve 98.5% removal of metal ions and a flow of about 13 L/h m^2.

Keywords: Reverse osmosis; Heavy metals; Complexation with EDTA; Membranes

1. Introduction

Pollution of water is a chronic problem for societies, and heavy metals are among the main contaminants associated with it. They are a class of compounds of high toxicity that bioconcentrate in organisms [1]. Because they are not biodegradable, biological treatment of wastewater (activated sludge or digester) is not effective in their removal; in fact, heavy metals can slow or preclude it. The technologies available for removal of heavy metals from wastewater include chemical precipitation, carbon adsorption, ion exchange, and reverse osmosis (RO). Of these, chemical precipitation is most commonly employed, but they have disadvantages; one of them is it increases the total dissolved solids in wastewater that is being treated [2].

Membranes have gained an important place in chemical technology and are used in a broad range of applications. They show very satisfactory results in the treatment of heavy metals from wastewater, and are promising for reducing operating costs

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and increasing the efficiency of removal of pollutants according to Qdais and Moussa [3]. Another advantage of membrane processes is the possibility of combination with other types of processes, forming hybrid processes.

In the removal of heavy metals, a common type of hybrid process is the precipitation, adsorption, or complexation of metal ions to form an aggregate or larger ions, which facilitates the rejection by size in the membrane [4–8]. Given that the size of the contaminant is a major factor for exclusion in membrane processes, it is evaluated in this study the use of a metal complexing agent prior to membrane treatment. The complexing agent chosen is ethylenediaminetetraacetic acid (EDTA), which has been demonstrating very effectively as a complexing agent for removal of heavy metals in water and soil [9–12]. The complexes formed by these ions with EDTA show great stability, and their formation constants are equal to 5.0×10^{18} for copper and 4.2×10^{18} for nickel [13].

Perhaps the most important issues in membrane technology are membrane fouling and concentration polarization, and this is particularly true to RO and ultrafiltration (UF) systems [14]. One of the ways to address fouling is the configuration of filtration through membrane. There are two: perpendicular to the flow (conventional or dead-end) or tangential flow membrane (in parallel). In dead-end flow occurs a bigger accumulation of components of feed phase on the membrane surface, while in tangential flow mode, the

Table 1
Heavy metal removals by membranes

Author	Process	Membrane material	Metal	Feed conc. (mg/L)	Pressure (MPa)	Rejection (%)
Qin et al. [15]	RO	Polyamide	Ni^{2+}	3–5	0.5–2.0	97.0
Ozaki et al. [16]	RO	Polyamide	Ni^{2+}	50	0.35	99.2
			Cu^{2+}	50	0.35	99.7
			Cr^{3+}	50	0.35	99.9
Taleb-Ahmed et al. [17]	NF	Polyamide	Cr^{3+}	0.01–0.1	0.1–1.5	99.0
			Cr^{6+}	0.001–3.3	0.1–1.5	97.0
Qdais and Moussa [3]	NF	Polyamide	Cu^{2+}	25–200	0.7–1.3	90.0
			Cd^{2+}	25–200	0.7–1.3	97.0
	RO	Polyamide	Cu^{2+}	25–200	0.7–1.3	98.0
			Cd^{2+}	25–200	0.7–1.3	99.0
Bódalo et al. [18]	RO	Polyamide	Cr^{3+}	0.21–1.05	3.92	96.3
	RO	Polyester	Cr^{3+}	0.21–1.05	3.92	80.3
	RO	Cellulose acetate	Cr^{3+}	0.21–1.05	3.92	95.9
	RO	Polyethersulphone	Cr^{3+}	0.21–1.05	3.92	98.2
Zuo et al. [19]	ED	Polystyrene[a]	Zn^{2+}	1.000	–	97.0
			Cu^{2+}	10	–	97.0
			Cr^{3+}	300	–	97.0
Nędzarek et al. [20]	NF	Al_2O_3/TiO_2	Zn^{2+}	1	0.4	97.0
			Cu^{2+}	1	0.4	95.0
			Cd^{2+}	1	0.4	96.0
			Pb^{2+}	1	0.4	90.0
Zhu et al. [21]	NF	Polyethersulfone[b]	Pb^{2+}	1	1	99.4
			Cu^{2+}	1	1	99.6
			Ni^{2+}	1	1	99.2
			Cd^{2+}	1	1	99.5
			Zn^{2+}	1	1	99.2
			Cr^{6+}	1	1	99.1
Gherasim et al. [22]	NF	Polyamide	Co^{2+}	15–1,000	2.0–2.5	97.0
Maher et al. [23]	NF	Polyamide	Pb^{2+}	1	0.6–0.8	86.0
			Ni^{2+}	1	0.6–0.8	93.0
Chaudhari and Murthy [24]	NF	Polyamide	Cd^{2+}	5–250	0.5–2.0	97.3
			Ni^{2+}	5–250	0.5–2.0	98.9
Mehdipour et al. [25]	NF	Polyamide	Pb^{2+}	20–400	1.0–4.0	97.5

[a] Ion-exchange membrane.

[b] Poly(amidoamine) dendrimer grafted on polyethersulfone thin film.

feed flows parallel to the surface of the membrane, higher permeate flux is reached and deposition reduced due to the continuous removal of materials retained on the membrane. In this study, tangential flow was employed.

The four well-established, developed pressure-driven membrane separation processes are microfiltration (MF), UF, nanofiltration (NF), and RO. Although they are conceptually similar processes, the difference in pore diameter (or apparent pore diameter) produces dramatic differences in the way the membranes are used. Given the similarities in membrane constitution (pore size) and applied pressure, there is considerably an overlap in the sizes of particles removed between NF and RO, particularly regarding the removal of heavy metals [2,14].

There are several studies in the literature involving RO and NF to remove heavy metals from industrial effluents indicating highly satisfactory results. Table 1 shows a compilation of some works on this subject. Rejection of heavy metals (as defined in the methodology section, formula 1) was used as a comparative parameter.

Aligned with the work reported in Table 1, in this study, the authors evaluated the use of RO for the removal of nickel (Ni^{2+}) and copper (Cu^{2+}) ions (pure or in mixture) from the aqueous phase. Their choice is justified by their high toxicity, and the fact that they are often generated as waste in different industrial processes. Both ions have six ligands bound to the metal in an octahedral geometry, which is the most common form of arrangement, and in the aqueous phase are found as complex ions such as $Ni(H_2O)_6^{2+}$ and $Cu(H_2O)_6^{2+}$ [13]. The concentration of ions chosen for this work is very low, ranging from 10^{-2} to 10^{-4} mol/L because this is the range where there is advantage for using membranes.

At higher metal concentrations, traditional chemical coagulation–precipitation processes are feasible, achieving great efficiency; however, at low metal concentrations, the process requires the use of proportionally higher concentrations of coagulants, which entails higher operating costs and higher sludge production. Therefore, for low concentrations of metals, the coagulation–precipitation process is not economically advantageous, creating a niche for membranes [26]. Due to the low solubility of the compounds formed by heavy metals, particularly sulfides and hydroxides, concentrations of heavy metals dissolved in the aqueous phase tend to be low.

Heavy metals are dangerous in discharged wastewater even at very low flow concentrations because they are not biodegradable and subjected to bioaccumulation and biomagnification. Even in very low concentrations in aqueous phases, they may appear in toxic concentrations in upper organisms [27,28].

Based on these facts, the present study sought to conduct an evaluation of EDTA as complexing agent associated with RO for removing nickel and copper ions from wastewater. A broad concentration range was employed, particularly focusing on concentrations less than 100 mg/L in feed water. Moreover, the influence of pressure and operating time on the process efficiency was also investigated.

2. Materials and methods

The synthetic effluent tested consisted of aqueous solutions of $Ni(NO_3)_2$ and $Cu(NO_3)_2$ with purity higher than 99% (P.A.) and produced by Vetec. The complexing agent is EDTA, commercialized as a disodium salt by Vetec.

The commercial membrane used is model 4040-X201-TSA, produced by the company Trisep Corporation. Table 2 presents some characteristics of the membrane.

The equipment used include: electronic analytical balance (Mettler Toledo model AL204 brand) and atomic absorption spectrophotometer (Varian, model Spectr AA 220).

The membrane system used was manufactured by PAM Membranas Seletivas Ltda. and has a tangential filtration module for flat membrane permeation and an active area of 77.2 cm. The system is composed of a

Table 2
Membrane operational data (source Trisep Corporation catalog)

Membrane type	Low-fouling RO membrane: X20 fully aromatic polyamide-urea advanced composite membrane
Recommended applied pressure	5–21 bar
Maximum applied pressure	41 bar
Recommended operating temperature	2–45°C
Feedwater pH range	2–11 continuous
Chlorine tolerance	<0.1 mg/L
Maximum turbidity	2 NTU

feed tank with a capacity of 5 L, rotameter, pressure gauge, thermometer, permeate flow indicator, and centrifugal pump coupled to rectifier current, as shown in Fig. 1.

The solution is pumped tangentially to the membrane, forming two streams: concentrate and permeate. The stream that permeates through the membrane is called permeate, while the portion which does not permeate is called the concentrate.

The operation is in batch mode and starts by filling the feed tank with the prepared effluent. Since permeate is collected and no recharge is done, the level on the feed tank drops slightly during operation. Feed tank is open to atmosphere.

The pump is turned on and adjusted to an output pressure of 0.5 MPa by the pressure control valve. Once the experiment begins, the concentrate stream is recirculated into the feed tank and its flow is adjusted to 1 L/min (1.66×10^{-5} m/s), and kept constant during the experiment. The permeate stream is collected and the permeate flow is measured every 30 min. The value reported as “permeate” is the average. Each experiment was done in triplicate.

Each experiment lasted two hours and immediately afterwards, the system was extensively cleaned by circulating distilled water for 1 h. Permeate and concentrate collected after cleaning and analyzed by conductivity showed there was no evidence of contamination of the membrane by heavy metals from the previous experiment. Fig. 1 presents a scheme of the process. Fig. 1 shows the RO system can be operated with recirculation of both concentrate and permeate, but only concentrate was recirculated. So the permeate recirculation valve was always closed.

During the operation, there is a tendency of overall heating because of the pump. The whole system is kept at constant room temperature (25℃) by circulating refrigerant fluid (water at 25℃) at high flow in an external serpentine. There is a thermometer that allows checking the temperature of concentrates at all times.

Effluents were prepared as follows: the quantity (in mg) of salts (nickel, copper, and/or EDTA) calculated to reach the desired concentration of salt in 5 L of solution was weighed on an analytical balance. Then, salts were diluted in distilled water under intensive stirring for 5 min.

The mixtures of copper and nickel are always prepared in a stoichiometric ratio, i.e. 1 mol of nickel to 1 mol of copper. For assays using complexing agent, it was also kept at a ratio of 1 mol of nickel and/or copper to 1 mol of EDTA. Since complexation is a chemical reaction, we decided to prepare the assays in stoichiometric ratio to ensure sufficient EDTA to complex all metals in solution. Each assay was made in triplicate.

Determinations of metal concentrations in permeate and feed streams were made by atomic absorption spectroscopy. The rejection of membrane (R%) was calculated by formula (1):

$$R\,(\%) = \left(1 - \frac{C_p}{C_f}\right).100 \qquad (1)$$

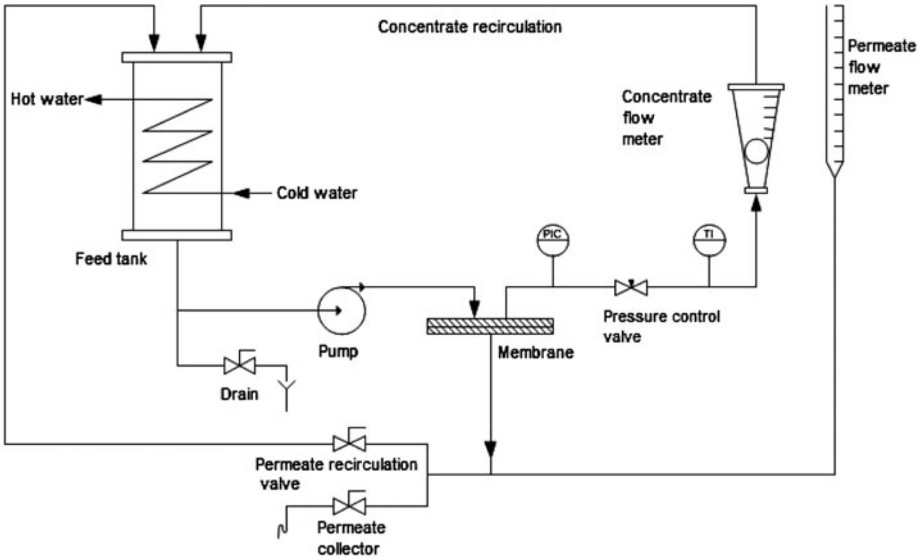

Fig. 1. Flowchart of the RO system.

where C_p is the concentration of the permeate and C_f is the concentration of the feed flow.

3. Results and discussion

Firstly, the use of EDTA as a complexing (or chelating) agent for aqueous solutions containing only one of the metals was evaluated. Fig. 2 shows the results of permeate concentration as a function of feed concentration in experiments with single ion and mixture ion–EDTA in a 1:1 M ratio.

When analyzing the results of Fig. 2(a) and (b), for the same feed concentration, complexation causes lower metal concentrations in the permeate. These results are justified by the larger size of the complex formed. The complex that is formed has a high formation constant, and it creates a steric hindering that enhances process efficiency.

When employing the EDTA disodium salt, complex is formed following the chemical reaction:

$$M^{2+} + Na_2H_2EDTA \rightarrow MH_2EDTA + 2\,Na^+$$

It was found that the fact that chelating increases the size of the complex (favoring steric rejection) offsets the fact that the complex does not present loads, which disfavors rejection by electrostatic repulsion at the membrane surface.

In all experiments, concentrations in the permeate increase proportionally with the increase in the concentrations of the feed. This happens because the concentration increase in feed flow leads to an increase in the solute concentration near the membrane surface, which promotes solute adsorption. Increasing concentration in the membrane surface favors its diffusive transport through the membrane, as observed by the solute transfer equation [29]:

$$J_i = k_i \, \Delta C \tag{2}$$

where J_i = solute flux, k_i = solute mass transfer coefficient, and ΔC = concentration gradient (in the boundaries of membrane).

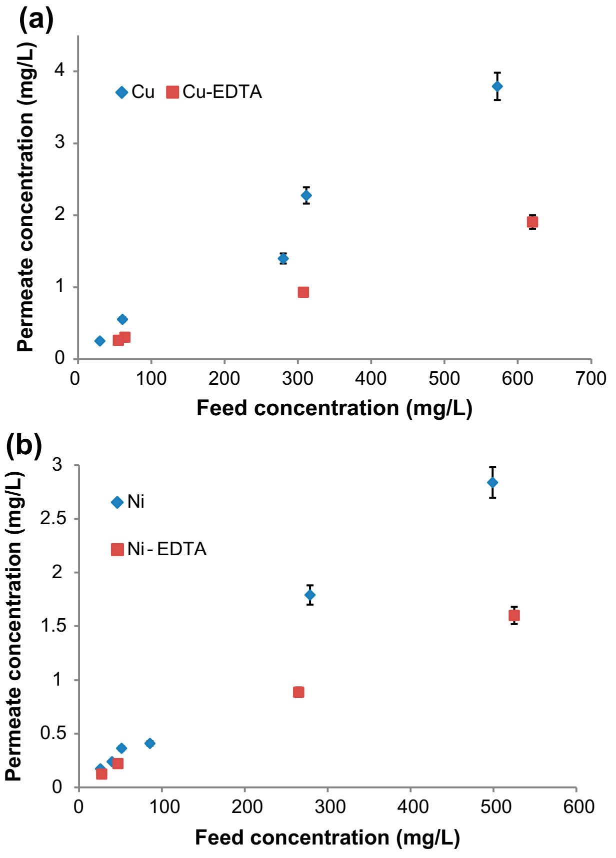

Fig. 2. Metal concentration in the permeate as a function of feed concentration. Metal in aqueous solution in the absence and presence of EDTA: (a) Cu^{2+} and (b) Ni^{2+}.

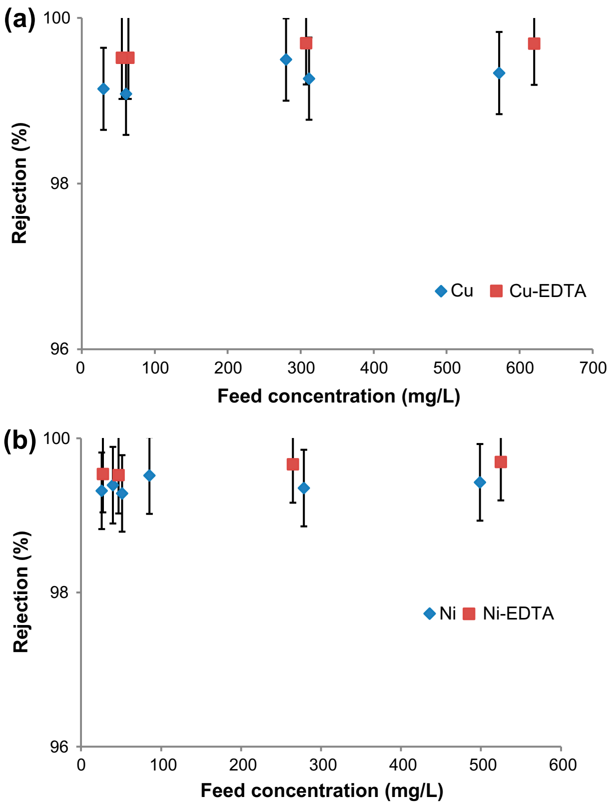

Fig. 3. Metal rejection as a function of feed concentration. Metal in aqueous solution in the absence and presence of EDTA: (a) Cu^{2+} and (b) Ni^{2+}.

From the values of concentrations in the feed and permeate, rejection was calculated for each experiment using Eq. (1). Fig. 3 presents the results.

All experiments obtained superior rejection of metal ions to 99%, which demonstrates the efficiency of the process. As evidenced by Fig. 3, the process is stable because it removes with same efficiency from 50 to at least 500 mg/L contaminant in the feed flow. The use of EDTA as complexing agent has been studied involving complexation and RO for the removal of copper and nickel in the range of 200–600 mg/L. Similar to this work, the authors employed a polyamide membrane and reported high rejections with and without the complexing agent, more than 98% [30].

Secondly, the work focused on solutions containing a mixture of Cu^{2+} and Ni^{2+} in the presence and absence of EDTA. The graphs of Fig. 4 show the results of permeate concentrations as a function of feed concentration, and Fig. 5 presents the results of rejection.

From the analysis of the results presented in Figs. 4 and 5, it can be seen that both Cu^{2+} and Ni^{2+} single or in combination exhibit similar behavior as of concentration in permeate and rejection. Considering that the main mechanisms of separation are steric and electronic effects resulting from each ion, the presence of other ions in solution at dilute concentrations studied did not affect its rejection by the membrane. This result indicates that for these ions, RO is a non-specific treatment as it does not discriminate between metals, so it is feasible, as real industrial wastewater is constituted by a variety of chemicals.

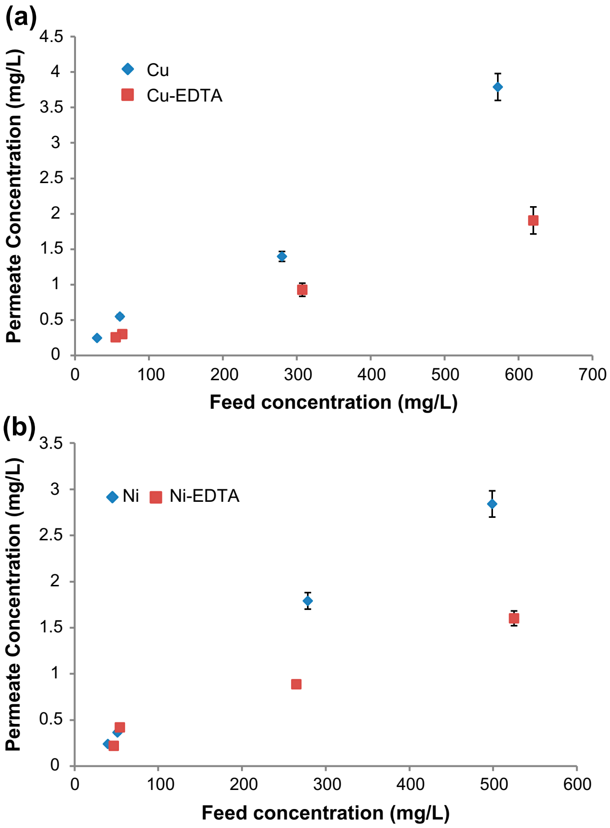

Fig. 4. Metal concentration in the permeate as a function of feed concentration. Mixture of two metals in aqueous solution in the absence and presence of EDTA: (a) Cu^{2+} and (b) Ni^{2+}.

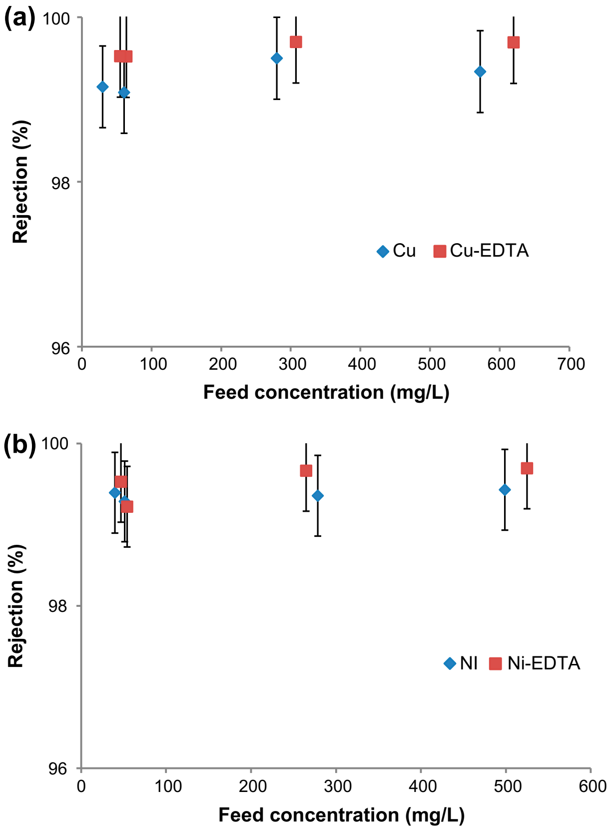

Fig. 5. Metal rejection as a function of feed concentration. Mixture of two metals in aqueous solution in the absence and presence of EDTA: (a) Cu^{2+} and (b) Ni^{2+}.

Permeate flux of all experiments were measured every 30 min for fouling analysis. As expected, no significant fouling was observed. Flow fell very little to justify the inclusion of a discussion of fouling, being 18.7 ± 0.5 L/h m in non-complexed solutions and 20.1 ± 0.5 L/h m in complexed solutions. A number of reasons are reported for no fouling: experiments were run for only 2 h, and the first stage of permeate flux reduction, concentration polarization, takes the first hours of operation to be completed, at least 2 h [31]; the feed solutions are very diluted; feed solution operated under high flow (consequently turbulent flow,

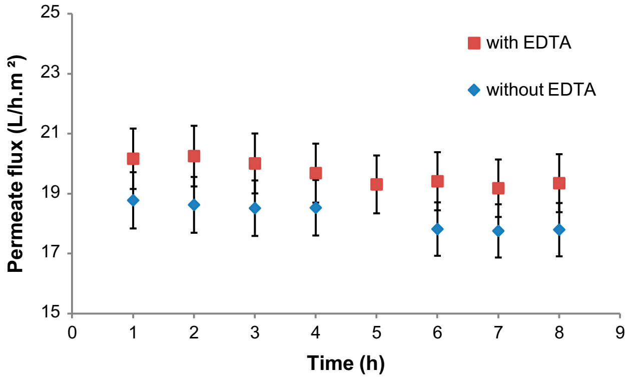

Fig. 6. Results of permeate flux as a function of operating time in the presence and absence of EDTA as a complexing agent.

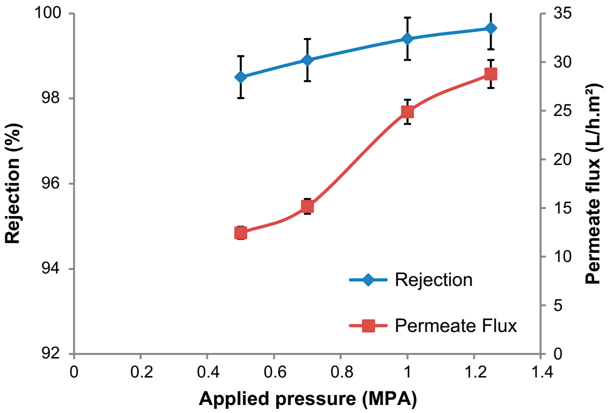

Fig. 7. Rejection of metals and permeate flux as a function of the operating pressure.

avoiding deposition); good flow distribution and cross-flow filtration, with feed solution passing directly across the active surface of the membrane, and producing no stagnant dead spaces that contribute greatly to concentration polarization [14].

To assess the effect of the introduction of the complexing agent on the permeate flux over time, two longer period experiments were run. Permeate flow rate was measured every 30 min for 8 h. The results are shown in the graph of Fig. 6. As it can be seen, there is no significant difference in the permeate fluxes recorded. The values fall within the same error bar.

In a last step, the study was expanded for the mixtures of Ni^{2+} and Cu^{2+} under different operating pressures: 0.5, 0.7, 1, and 1.25 MPA. In Fig. 7, the values of rejection for metals and the permeate fluxes as a function of applied pressure are presented.

The increases in the rejection of ions and permeate flux observed in Fig. 7 can be explained from the analysis of the equation of mass transfer in the boundaries of membrane. The equation is given below [29]:

$$J = k_w(\Delta P - \Delta\pi) \tag{3}$$

where J = water flux through membrane, k_w = solvent mass transfer coefficient, ΔP = applied pressure, and $\Delta\pi$ = osmosis pressure.

By increasing the applied pressure, it is clear from (3) the water flux through membrane will increase, and so will permeate flux and flow. But the solute flux will not be affected because flux is a function of solute mass transfer coefficient and concentration gradient only, as shown in (2). The flow of ions remains virtually unchanged; therefore, rejection of ions in the permeate will appear higher by a dilution effect.

Table 3 presents characterization of raw and treated effluents in terms of pH and conductivity.

By analyzing the results, the treated effluent, compared to the raw one, has a slight increase in pH and a significant reduction in conductivity. The reduction in conductivity is a clear consequence of the removal of ions by RO. The increase in pH is not what would be expected in RO. RO permeate usually has a decrease in pH as a result of removal of dissolved

Table 3
Experimental characterization of raw and treated effluents

Solutions	Feed		Treated	
(50–100 mg/L)	pH	Conductivity (mS cm)	pH	Conductivity (mS cm)
Copper	4.8–5.2	230.0–240.0	5.5–5.8	6.0–7.0
Nickel	5.1–5.5	70.0–80.0	5.5–5.8	2.0–2.5
EDTA	4.6–5.0	70.0–80.0	5.5–5.8	4.6–5.5
Copper + EDTA	3.5–4.0	90.0–100.0	5.5–5.8	5.0–6.0
Nickel + EDTA	3.6–4.0	50.0–60.0	5.5–5.8	4.1–4.5
Copper + Nickel + EDTA	3.3–3.7	190.0–200.0	5.5–5.8	12.1–13.0

ions such as carbonates, but not dissolved gasses like carbon dioxide, which unbalances the equilibrium in media (dissolved carbon is distributed among three species H_2CO_3, HCO_3, and CO_3 as a function of pH) [29]. However, in this case, distilled water was used to prepare the effluents, where we can assume there was no dissolved carbon. The initial pH is lower than distilled water because of the metal complexes formed in the solution; the slight increase in pH is due to the rejection of some hydronium ions by the membrane. Another parameter evaluated was turbidity, but due to the low concentrations studied, both raw and treated effluents showed 0.0 NTU.

4. Conclusions

RO confirmed to be efficient in removing the ions of copper and nickel from aqueous solution. Removal of ions, both single and in mixture, reached over 99.0%. This removal level can be reached for contaminats on feed flow from 50 to at least 500 mg/L; this is complemented by other studies, that show that for 200 mg/L and more, the same removals can be reached, showing the process is very stable.

The use of a preliminary stage of complexation with EDTA resulted in a reduction in the concentration of metals in the permeate due to the larger size of the formed complex relative to the ion in aqueous phase, which created steric hindering that enhances process efficiency. The increase in the size of the complex, favoring steric hindering, more than compensates for the fact that the complex does not present loads, which disfavors the rejection by electrostatic repulsion at the membrane surface.

The effect of the mixture showed no significant interference with the process, which indicates the viability of the process in real industrial effluents. An increase in the operating pressure increases the rejection of metals (by dilution effect).

List of symbols

C_f — concentration of contaminant in the feed flow (mg/L)
C_p — concentration of contaminant in the permeate (mg/L)
UF — ultrafiltration
MF — microfiltration
NF — nanofiltration
R — membrane rejection (%)
RO — reverse osmosis
Ji — solute flux through membrane (M/L t)
k_i — solute mass transfer coefficient (L/t)
ΔC — concentration gradient (M/L)
J — water flux through membrane (L/L t)
k_w — solvent mass transfer coefficient (L t/M)
ΔP — pressure gradient (L)
$\Delta\pi$ — osmotic pressure (L)

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