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Comparison of reverse osmosis and electrodialysis in brassware effluents treatment

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ABSTRACT

The manufacture of the handcrafted items is carried out in a succession of deposit baths and rinses. However, this process gives rise to complex and highly loaded effluents, including a mixture of heavy metals (Cu(II), Ag(I) and Ni(II)). Therefore, to meet the Moroccan discharge limits, it is necessary to treat these effluents before discharging them into the natural environment. The objective of this study is the comparison of electrodialysis (ED) and reverse osmosis (RO) in the reduction of heavy metals in brassware wastewater of the city of Fez (Morocco). The first part is devoted to the study of the influence of the operating parameters of RO (transmembrane pressure (TMP) and recovery rate) on the quality of the permeate. The rejection (R) of metal ions (Cu(II), Ag(I) and Ni(II)) increases with TMP, but decreases with the increase in the recovery rate. The maximum values of 96%, 66.39% and 99.6% have been reached for Cu(II), Ag(I) and Ni(II), respectively. The second part of this work focuses on the feasibility of ED in the removal of heavy metals. The influence of the demineralization rate (DR) on the quality of the dilute is studied. Results show that the removal rate of cations (Cu(II), Ag(I) and Ni(II)) increases with the increase of DR and reaches, respectively 98%, 95% and 97% for a DR of 90%. Finally, a comparison between RO and ED based on the water quality delivered and the energy consumed is performed under optimal operating conditions.

Keywords: Reverse osmosis; Electrodialysis; Brassware wastewater; Heavy metal removal; Copper, silver and nickel ions, Energy consumption

1. Introduction

Fez has a diversified and very old craft tradition. In the heart of this city are hidden treasures of heritage, where workshops of brassware are located which are specialized in the manufacture of handicrafts, containing marvels of craftsmanship. The manufacture of these pieces is carried out in a succession of electroplating baths and rinses. This process gives rise to complex effluents that are highly charged with a mixture of heavy metals (Cu(II), Ag(I) and Ni(II)). The presence of these metals in water presents serious environmental problems, but also problems related to the risk and threat to public health posed by these pollutants. Heavy metals are metallic elements with a density greater than 5 g/cm^3 they are toxic, non-biodegradable and accumulate easily in living organisms, threatening public health and the environment [1]. They cause several health effects, such as respiratory damage, including lung cancer, diarrhoea, low blood pressure, bone defects. With industrialisation, wastewater discharge is one of the main sources of heavy metal releases to the environment [2]. Therefore, the removal of heavy metals from wastewater is attracting a lot of attention from society and industry.

Membrane separation, coagulation–flocculation, precipitation, ion exchange and adsorption are the conventional

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technologies for removing heavy metals from wastewater [3]. Each of these technologies has its own advantages and scope of application. However, some unavoidable limitations encourage researchers to move towards advanced technologies and to improve existing technologies to achieve a more efficient separation of heavy metals from wastewater [3]. Compared to conventional techniques, membrane separation technologies offer several advantages: low energy consumption, no addition of chemicals, environmental friendliness and mature large-scale application [4].

Reverse osmosis (RO) is a physical separation process in which the natural flow of water is forced through a membrane to a more concentrated solution by means of positive hydrostatic pressure to overcome osmotic pressure. RO is used in several fields: seawater desalination, ultrapure water production, food industry, pharmaceutical, medical, cosmetic, chemical, electronic, biotechnological, drinking water production and wastewater treatment [5]. Aljendeel [6] studied the removal of heavy metals by RO from an aqueous solution discharged from some Iraqi factories for mechanical industries. The results revealed that the maximum rejection of copper, nickel and zinc salts was 96.6%, 95.7% and 98.2%, respectively in metal concentration range (50–150 ppm) and duration range (15–90 min). Thaçi and Gashi [7] studied the removal by RO of metal ions such as Pb^{2+}, Zn^{2+}, Cd^{2+}, Co^{2+}, Mn^{2+}, Ni^{2+} at concentrations of 30 mg/L for each ion, from wastewater effluents. The results show that the removal rates of metal ions are close to 99.8%. In other study, Ozaki et al. [8] focused on the removal of heavy metals (Cu^{2+}, Cr^{3+} and Ni^{2+}) by RO from electroplating wastewater containing low concentrations (>10 mg/L). The results showed that heavy metals were rejected at more than 98.75%. Moreover, Algureiri and Abdulmajeed [9] studied the performance of RO in the treatment of industrial wastewater containing nickel, lead and copper ions. The results showed that RO is an effective method for removing nickel, lead and copper, with removal rates of 98.5%, 97.5% and 96%, respectively.

Electrodialysis (ED) is the best-known electro-membrane process that can be applied for the removal of aqueous ions through selective transport of ions through anion-exchange membranes (AEMs) and cation-exchange membranes (CEMs) [10]. Ogütveren et al. [11] studied the removal of Cu^{2+} from electroplating effluent industries in the concentration of 100 mg/L, at 40 V and during 75 min, using IonacMA3475 as an AEM and Nafion423 as a CEM membrane and estimated the energy consumption as a function of time and removal rates. The results showed that a removal rate of 99.9% was achieved with an energy consumption of 65.0 kWh/m^3. Similar results were obtained in a 5-unit cell ED configuration achieving more that 85% removal of Cu^{2+} from synthetic electroplating water [12]. Both works confirmed that similar removal rates can be achieved for both high and low Cu^{2+} concentrations (100–4,000 mg/L) and its dependence on the applied voltage. In addition, Benvenuti et al. [13] studied the removal of Ni^{2+} ions from the wastewater of galvanic processes by ED in order to concentrate and recover them; they were able to achieve a 95% nickel removal rate. In another study [14], they showed that the reuse of the recovered nickel by this technique was able to reduce the cost of chemicals by about 20% during the nickel-plating step.

The case of silver ion removal by ED was a poorly studied. The most relevant work in this case showed a removal of nearly 100% of Ag^+ using two different pairs of IEMs for a duration of only 110 min, providing a relatively short and efficient process for Ag^+ recovery by ED [15]. Benalla et al. [16] investigated the feasibility of ED for the treatment of effluents coming from the copper smelter in the city of Fez, two IEM pairs ACS/CMX and AXE/CMX were studied. The results showed that the best membrane pair was AXE/CMX, which achieved removal rate of 98%, 95% and 97% for copper, silver and nickel, respectively.

The objective of this work is the comparison of ED and RO in the reduction of heavy metals (Cu^{2+}, Ag^+ and Ni^{2+}) in the wastewater of the copper smelter of the city of Fez. The first part is devoted to the study of the influence of the operating parameters of RO (transmembrane pressure (TMP) and recovery rate) on the quality of the permeate. In the second part, the feasibility of ED for the removal of heavy metals is investigated by studying the effect of demineralization rate (DR) on the quality of the permeate. Finally, a comparison of the quality of the treated water and the energy consumed by RO and ED under optimal operating conditions will be carried out.

2. Materials and methods

2.1. Description of RO pilot

RO experiments are performed on an industrial pilot (E3039) supplied by TIA (Applied Industrial Technologies, France) Fig. 1. The applied TMP varies from 5 to 70 bar. The characteristics of the pilot equipment are collected in Table 1. The pilot is equipped with two identical modules in series. The pressure drop is approximately 2 bar. The two spiral modules are equipped with two commercial RO membranes BW30LE-4040 manufactured by the American Group DOW Chemical. The characteristics of this membrane are reported in Table 2.

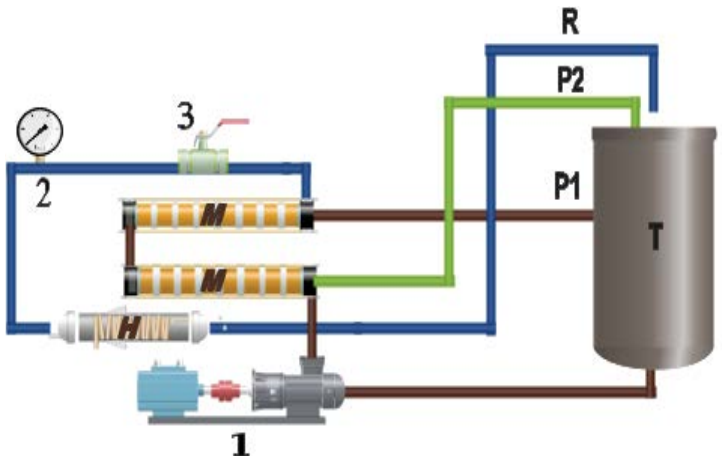
The configuration used is a single pass batch system illustrated by Fig. 2. The permeate is continuously recovered, while the retentate is continuously discharged into the feed tank.

The cleaning of the membranes consisted of washing using several chemical solutions, is carried out in two steps: First, the washing is done with an alkaline solution, extremely effective against organic compounds, and in the second, the washing is performed by an acid to remove metal hydroxides, calcium carbonate and other similar deposits. The cleaning is repeated several times until the initial water flow is obtained, which is measured before and after each test.

The parameters monitored are the recovery rate. The recovery rate is the fraction of liquid that flows through the membrane.

$$Y(\%) = \frac{Q_p}{Q_0} \times 100 \tag{1}$$

where Q_0 and Q_p are the feed and the permeate flow rate, respectively.



T: tank;
M: reverse osmosis module;
P: permeate recirculation;
R: retentate recirculation;
H: heat exchanger;
1: high-pressure pump;
2:pressure sensor;
3: pressure regulation valves.

Fig. 1. Schematic of the reverse osmosis pilot plant.

Table 1
Reverse osmosis pilot equipment characteristics

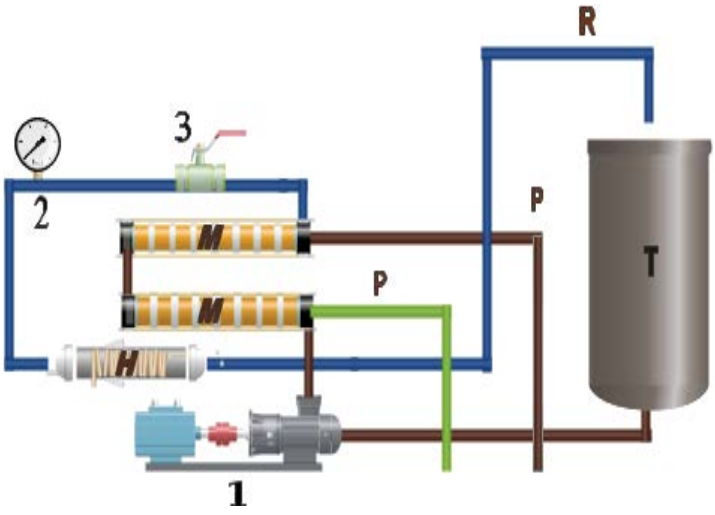
Equipment	Characteristics and models
Membranes	BW30LE-4040
Pumps	Brand: CAT PUMP
	Type: 2531 volumetric piston
	Flow: 3,600 m^3/h
	Engine: WV-DA 132 MB
Needle valves	Type: F O INOX
	Matter: INOX 316 L
Tank	Capacity: 100 L
	Material: 316 L stainless steel
Pressure gauges	Brand: BOURDON-HAENNI
	Series: MEX 5 D31 B31
Thermometer	Brand: JUMO
	Type: V-vertical seed
Chassis	Stainless steel 316 L

Table 2
Characteristics of the BW30LE-4040 membrane

Area (m^2)	7.2
Number of units per tube	1
Diameter (inch)	4
Flow rate produced (m^3/d)	9.5
Pressure (bar)	41
Type	Spiral

2.2. Description of the ED pilot

The ED laboratory driver (Fig. 3) was provided by Eurodia Co., (France) and largely described in previous papers [16,17]. It is equipped with NEOSEPTA ion exchange membranes (IEM), the symbols AXE, ACS and CMX are codes given for cation and anion exchange membranes manufactured by Tokuyama Co., (Japan). The design specifications of this pilot ED are presented in Table 4. Table 5 summarizes the main characteristics of the AXE and CMX membranes.



T: Tank of alimentation ;
M_1 et M_2 : RO module ;
P: Permeate ;
R: Concentrate ;
H: Heat exchanger;
1: High-pressure pump;
2: Pressure sensor;
3: Pressure regulation valves.

Fig. 2. Single pass configuration in semi-batch system.

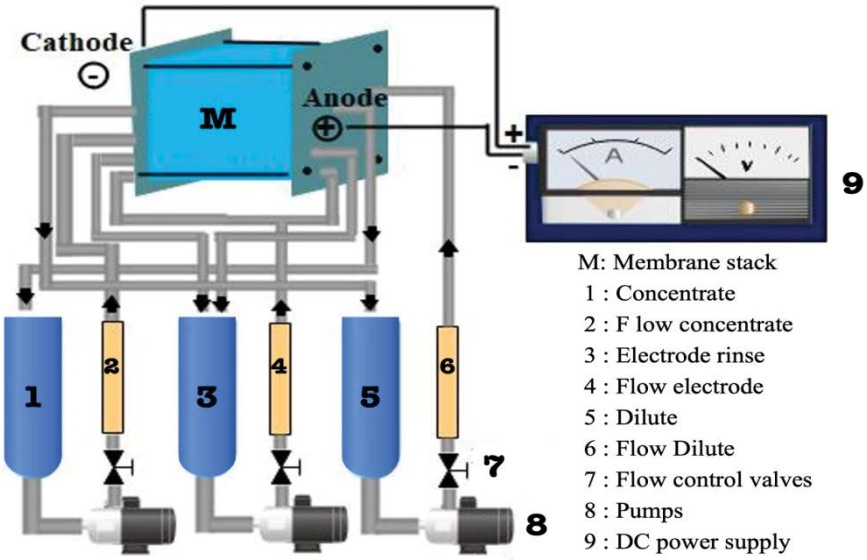


Fig. 3. Schematic of the electrodialysis pilot plant.

Table 3
Electrodialysis laboratory-scale pilot specifications

Parameter		Pilot TS 2-10
Active surface of IEM (cm^2)		200
Number of AEM		10
Number of CEM		12
Number of cells		10
Maximum intensity		9 Å
Maximum voltage		1.5 V/Cell
Diluate	Volume (L)	2
	Flow rate (L/h)	120
Concentrate	Volume (L)	2
	Flow rate (L/h)	120
Electrodes	Volume (L)	2
	Flow rate (L/h)	150

Table 4
Main characteristics of AXE and CMX membranes

Membrane	AXE	CMX
Thickness (mm)	0.17	0.18
Electrical resistance (Ω/cm^2)	1.4	3.0
Exchange capacity (meq/g)	2	1.65
Bursting strength (kg/cm^2)	2.75	5.5
Active area (cm^2)	200	200
Type	Plane	Plane

The demineralization rate reflects the total amount of salts removed. Its value is calculated and from the electrical conductivity (E) according to Eq. (2):

$$\mathrm{DR} = -\left[1-\left(\frac{E_t}{E_0}\right)\right]\times 100 \tag{2}$$

where E_0 and E_t are, respectively, the electric conductivity of the dilute before and after treatment (μS/cm).

Ion rejection (R) is calculated using Eq. (3):

$$R(\%) = \left(\frac{1-C_0}{C_p}\right)\times 100 \tag{3}$$

where C_p and C_o are the permeate concentration and the initial concentration, respectively.

For ED, the energy consumption E which was converted to (kWh/m^3) includes the energy consumed by the three pumps of the pilot (concentrate, diluate and rinse) and the required energy for demineralization. This energy is calculated according to Eq. (4).

$$E = \int I \cdot U \cdot dt \tag{4}$$

where U is the applied voltage (V); I is the stack current (A); t is the duration of ED (h).

For RO the specific energy consumption is calculated according to Eq. (5) [18].

$$E\left(\mathrm{KWh/m^3}\right) = \frac{\mathrm{Pe}\times 100}{\left(\eta \times Y \times 36\right)} \tag{5}$$

where Pe, η and Y are pressure (bar), overall pumping system efficiency and recovery rate (%), respectively.

In all the experiments conducted by RO and ED, a conductivity meter (Inolab WTW, Xylem Analytics Company, Germany) is used to measure the electrical conductivity (E) and temperature of the samples. A pH-meter (Jenway 3510 pH-Meter, Jenway Company, UK) is used to measure the pH of the solutions. Heavy metal concentrations are determined by the ICP-OES technique (Perkin Elmer Optima 8000, PerkinElmer Company, France), bicarbonate ions are analysed by titration (HCl 0.1 M) and finally Ca^{2+} and Mg^{2+} are analysed by atomic absorption spectroscopy.

3. Results and discussion

3.1. Physico-chemical of the brassware effluents

The wastewater for this study is taken from the effluent of the copper smelter in the city of Fez that have been recuperated during the two sampling campaigns (C1 and C2). The characteristics of the treated effluent are presented in Table 5. The effluents C1 and C2 contain a mixture of heavy metals of which the most present are copper, nickel and silver.

In addition, analytical results of Table 5 show that the samples are characterised by a high content of copper, nickel and silver. The content of all metal ions exceeds the Moroccan discharge limits. Moreover, all the samples are characterised by a high electrical conductivity (3,420–6,730 μS/cm), a temperature in the range 20.3°C–21.5°C and an alkaline pH. Finally, the total dissolved solids of C2 is almost double that of C1. This characteristic will guide our experimental choice in the sense that the C1 effluent will be treated by ED, while the C2 effluent will be treated by RO.

3.2. RO treatment

3.2.1. Influence of TMP

Experiments are carried out with industrial wastewater (C2) by adjusting the operating TMP to 10, 15, 20, 25, 30 and 35 bar. Fig. 4 shows the variation of permeate flux and pH as a function of TMP. Fig. 5 shows the variation of the electrical conductivity of the permeate and the ion rate of copper, silver and nickel cations as a function of the applied TMP.

Fig. 4 shows that permeate flux increases linearly with increasing TMP according to Darcy's law. The slight variation of the pH observed is due to the concentration of CO_2 passing through these membranes while bicarbonates are retained by RO membranes [19].

Table 5
Characteristics of untreated effluent

	Brassware effluent (C1)	Brassware effluent (C2)	Discharge limits values [16]
Temperature, °C	20.3	21.5	30
pH	9.62	10.81	6–9
E, μS/cm	3,420	6,730	2,700
Cu^{2+}, ppm	***51.99***	***7.07***	4
Ag^{+}, ppm	***87.26***	***8.033***	0.1
Ni^{2+}, ppm	***50.86***	***20.55***	5
Cd^{2+}, ppm	<DL*	<DL*	1
Cr^{3+}, ppm	0.09	<DL*	5
Pb^{2+}, ppm	<DL*	0.05	1
Zn^{2+}, ppm	<DL*	0.18	10
Ca^{2+}, ppm	42	40.8	–
Mg^{2+}, ppm	16.82	2.65	–
HCO_3^-, ppm	451	3,430	–
SO_4^{2-}, ppm	430	7,518	600
Cl^-, ppm	390	2,197.9	–

*Detention limit

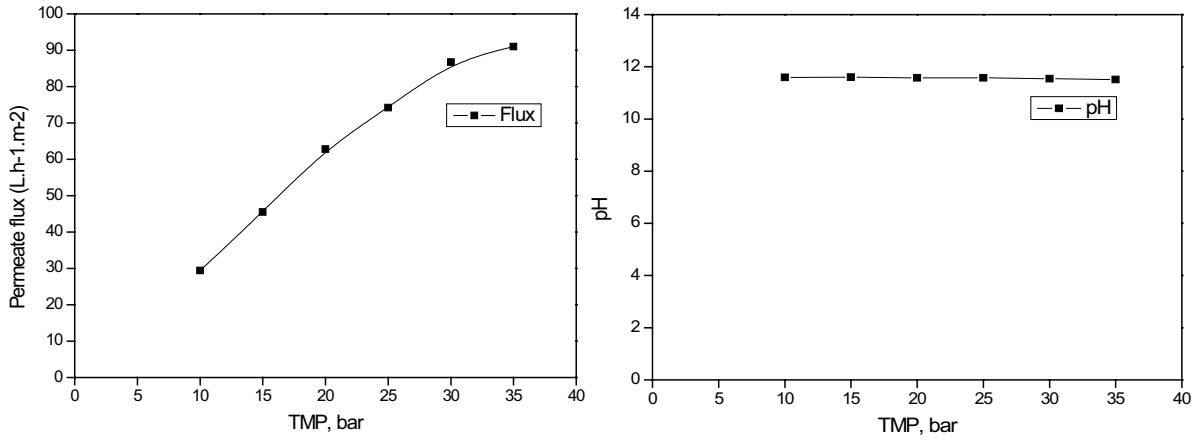


Fig. 4. Variation of permeate flux and pH vs. transmembrane pressure.

According to Fig. 5, the electric conductivity decreases slightly with increasing TMP and the rejection of metal ions remains nearly constant with TMP, reaching 94%, 65%, 99% for Cu^{2+}, Ag^{+}, Ni^{2+}, respectively. The order of membrane selectivity is as follows: $Ni^{2+} > Cu^{2+} > Ag^{+}$. Even if the membrane is dense, other parameters can intervene in the selectivity, namely the charge, the size and the hydration energy of the ion and its initial concentrations. The initial concentration of Ni^{2+} is higher than the concentration of Cu^{2+} and they are both divalent, they exhibit relatively similar rejection behaviour. On the other hand, Ag^{+} ion is a monovalent ion, with a smaller size and low hydration energy.

3.2.2. Influence of the recovery rate

The experiments are conducted under the following conditions: a TMP of 10 bar and a single pass configuration in semi-batch system, as shown in Fig. 2. Sequestrant (AF200) and HCl acid (pH = 7) are added to the crude solution to avoid precipitation. Fig. 6 shows the variation of the flux and pH of the permeate as a function of the recovery rate. Fig. 7 shows the electric conductivity of the permeate, and the rejection of cations (Cu^{2+}, Ag^{+}, Ni^{2+}) as a function of the recovery rate.

Fig. 6 shows that the flux of the permeate, decreases with increasing recovery rate. During the treatment, the total resistance to the transfer increased and the flux drops from 29 to 8 L/m²·h. The drop of flux could be caused by several factors, such as concentration polarisation, gel layer formation and pore clogging [8,20,21]. All these factors induce additional resistance on the feed side to transport across the membrane. The pH remains stable as a function of the recovery rate.

In Fig. 7 a clear decrease of electric conductivity in the permeate up to a recovery rate of 40% is observed, then it increases again. This behaviour is due to the fall of rejection of the various ions by RO membrane. Rejection of copper, silver and nickel ions decreases slightly with the recovery rate and reaches 94%, 23%, 46%, respectively for a recovery rate of 70%. Copper and silver ions contents of the treated water are above the discharge limits but for nickel, the values obtained are below the discharge standards. It is thus obvious that to ensure a good elimination of heavy metals ions by RO, it is necessary to privilege recovery rates below 40%.

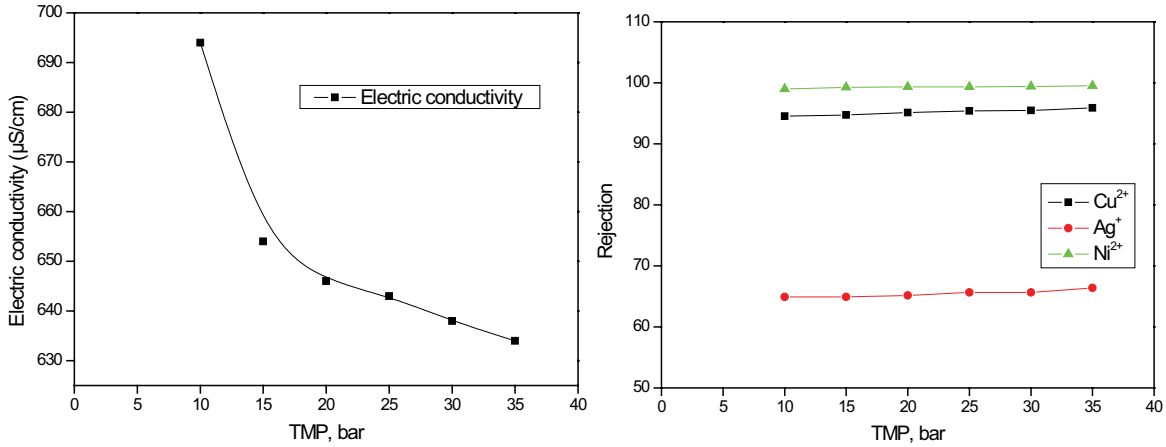


Fig. 5. Electric conductivity and metal ions rejection vs. transmembrane pressure.

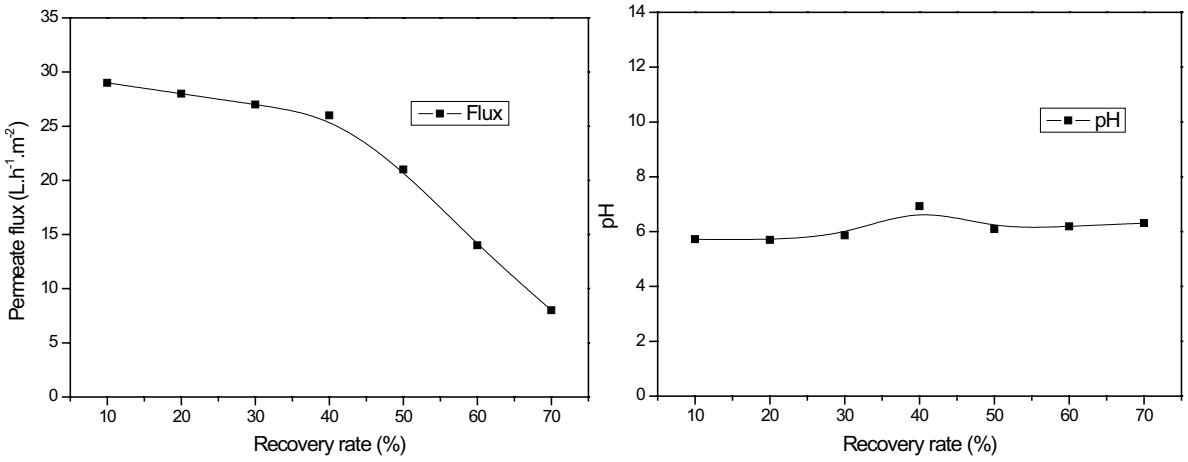


Fig. 6. Variation of permeate flux and pH vs. recovery rate.

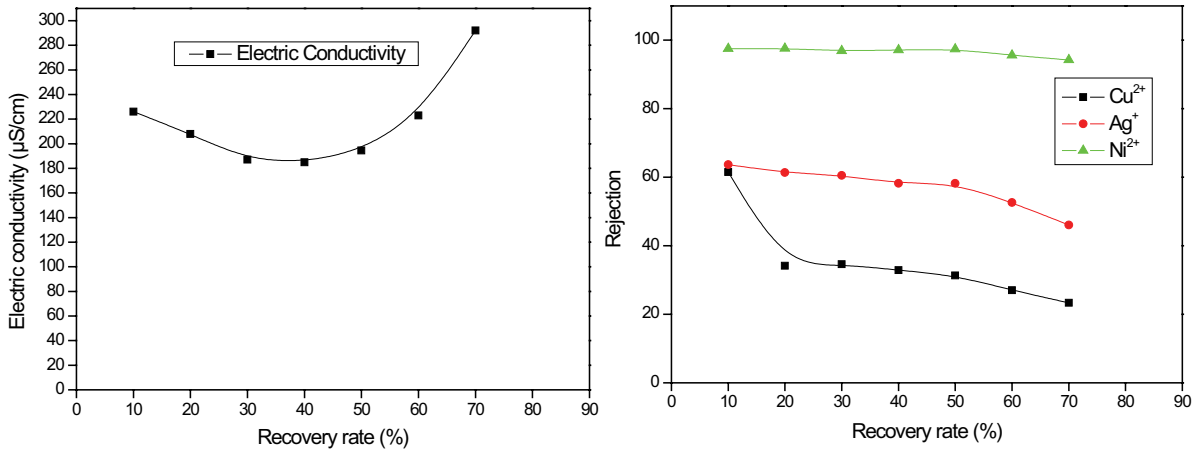


Fig. 7. Electric conductivity and rejection of metallic cations as a function of recovery rate.

Table 6
Characteristics of the treated water by electrodialysis and reverse osmosis at optimal conditions and energy consumption

Parameters	Electrodialysis		Reverse osmosis	Discharge limits values [16]
Temperature, °C	27.2		22.9	30
pH	9.3		6.31	6–9
E, µS/cm	1,026		292	2,700
Cu^{2+}, ppm	***1.545***		***5.420***	4
Ag^{+}, ppm	***6.443***		***4.333***	0.1
Ni^{2+}, ppm	***4.665***		***1.19***	5
Cd^{2+}, ppm	<DL*		0	1
Cr^{3+}, ppm	0.078		0	5
Pb^{2+}, ppm	<DL*		0.031	1
Zn^{2+}, ppm	<DL*		0.01	10
Ca^{2+}, ppm	17		24.48	–
Mg^{2+}, ppm	0.42		0.42	–
HCO_3^-, ppm	148		140	–
SO_4^{2-}, ppm	143.59		47	600
Cl^-, ppm	206		63.81	–
Energy consumer	Cell energy	Pumping energy	49.6	
(kWh/m^3)	8.5	384		

*Detention limit

3.3. ED treatment

The treatment of the effluent from the copper factory (C1) by ED is carried out using the AXE/CMX membrane pair. As shown in Table 6, this effluent is mainly loaded with nickel, silver and copper ions, with contents of 50.86, 87.26 and 51.99 mg/L, respectively. The operating conditions are as follows: the initial current intensity is 0.55 Å, the electrical voltage is 15 V and the volume to be treated is 2 L.

Fig. 8 shows diluate electrical conductivity and pH values as a function of time. The electric conductivity decreases with ED duration, it is lowered by 97% after 41 min of treatment. A slight increase in pH with time is observed which is attributed to the removal of bicarbonate ions [16].

Fig. 9 shows metal cations removal rate as a function of DR. For copper, nickel and silver ions, the removal rate increases with DR and reaches a plateau for DR in the order of 30%. The selectivity of the CMX membrane towards these ions obeys to the following order: $Cu^{2+} > Ag^{+} > Ni^{2+}$. This selectivity order is due to the difference of the charge for Ag^{+} compared to the two ions Cu^{2+} and Ni^{2+} and to their initial concentration. A DR of 30% allows a removal of more than 70% of these three metallic cations. The concentration of Ni^{2+} and Ag^{+} cations are below the discharge limits, but the concentration of Cu^{2+} ions is within the limits.

3.4. Comparison of RO and ED in the removal of metallic cations

Table 6 gives the physico-chemical parameters of the treated water by ED and RO at optimal conditions: DR of 70% for ED which has given the best removal rates for heavy metals and recovery rate of 70 for RO which allowed

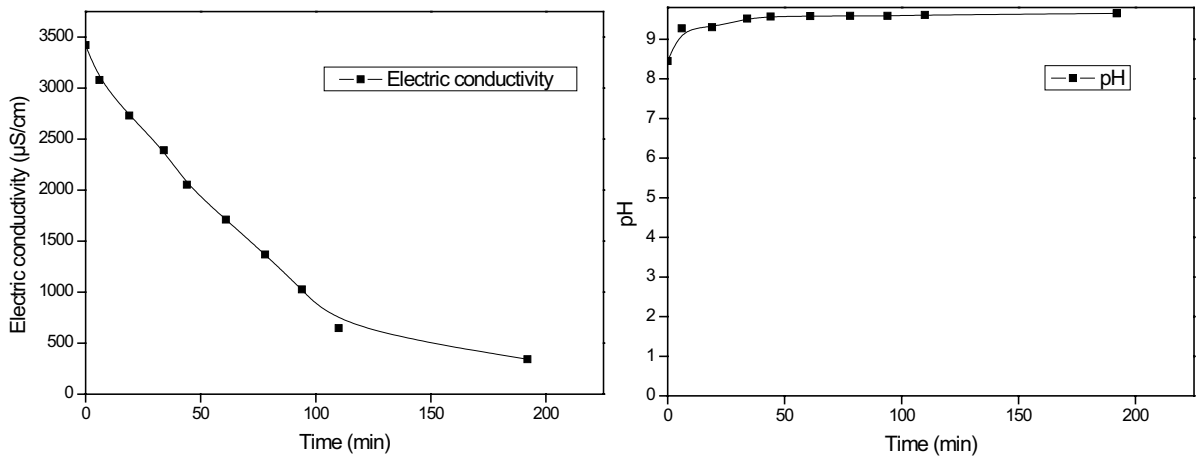


Fig. 8. Electrical conductivity and pH of the diluate as a function of time.

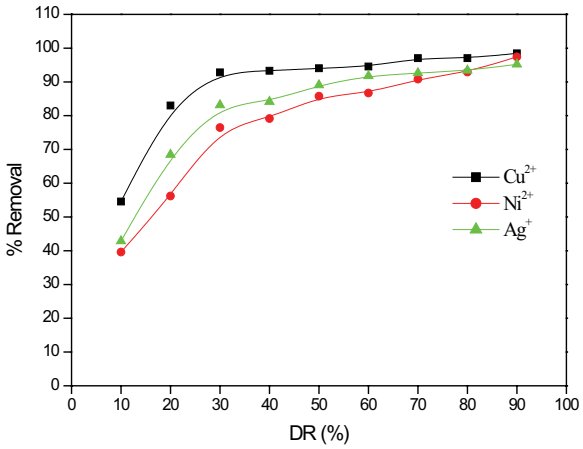


Fig. 9. % Removal of metal cations as a function of demineralization rate.

the largest amount of permeate to be obtained without precipitation in the concentrate. Table 6 also provides the energy consumed by these two technologies under these optimal conditions.

The physico-chemical quality of the water treated by ED for a DR of 70%, a high elimination rate for all ions and contents all within the standards except Ag^+, its concentration is largely above the Moroccan discharge limit values.

For RO, the rejections of metal ions are also important but the obtained content of Cu^{2+} in RO permeate is close to the discharge limits. For Ag^+ it is well above the discharge limits for a recovery rate of 70% and TMP of 10 bar. These conditions allows to obtain a large amount of treated water without precipitation. This permeate can be mixed to other non-contaminated water to bring the concentrations below the limit value.

The energy consumed in kWh/m^3 by ED for a DR of 70% is much higher than that obtained with RO for a recovery rate of 70%. Normally in the range of salinity treated, the energy consumed by ED is lower than that of RO [22], but in our case the high consumption is due to the electrical consumption of the three pumps that the pilot is equipped with, which have a power of 80/100 W each. The water quality obtained by RO is better than that obtained by ED in terms of salinity, hardness and alkalinity. However, the concentrations of heavy metals obtained by ED and RO are below the limit discharge values, except for Ag^+. In this case, the obtained permeate can be blended with non-contaminated water. Other processes could be used to complete the water purification: by ion exchange resins, coupling RO and ED or by implementing a double pass in the case of RO.

4. Conclusion

This study investigates the comparison of two membrane technologies, ED and RO, for the treatment of brassware effluents from Fez city. These effluents are especially loaded with copper, nickel and silver ions known to pose a great risk of contamination. For RO, the results show that the rejection of the three heavy metals increases with the TMP and decreases with recovery rate. For a TMP of 10 bar and a recovery rate of 70%, the rejections of copper, silver and nickel ions are 23%, 46% and 94%, respectively. The concentrations of Ag^+ and Cu^{2+} in the permeate exceed the limits. The RO process can be coupled with other processes such as: coagulation, sand filter, ion exchange resin and pre-chlorination to remove the remaining Ag^+ and Cu^{2+} cations. For ED and for a DR of 90%, the % removal of Cu^{2+}, Ag^+ and Ni^{2+} cations are 98%, 95% and 97%, respectively. The concentrations of these ions in the permeate obtained are below the rejection limits except for Ag^+ that it can be eliminated by combining the two processes. The water quality obtained by RO is better than that obtained by ED and the energy consumed in kWh/m^3 by ED is higher than that obtained with RO. However, additional treatments to improve the permeate quality are possible.

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