

Zinc and chromium removal from liquid wastes by using micellar enhanced ultra filtration

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ABSTRACT

Water or groundwater contamination by heavy metals is a major problem since once metal ions are introduced into the environment, they are not biodegraded and are transported to the ecosystem via various pathways. Moreover, if directly discharged in sewers, metal ions may seriously damage the subsequent biological treatments in depuration plants and render treatment sludge not reusable such as in agriculture. Liquid wastes effluents from several industrial activities often contain metals ions; for example the residual solutions from hydrometallurgical processes of waste electrical and electronic equipment can contain metals as iron, cadmium, chromium, zinc and other dangerous elements. Ultrafiltration process with the addition of a surfactant (micellar-enhanced ultra filtration, MEUF) is a reliable technique for almost complete heavy metal removal. This research group has already applied with success this method for arsenic and lead removal. In this paper, the MEUF technique is applied to the removal of Zn and Cr ions from synthetic liquid wastes aimed at water reuse, by using; a UF membrane (monotubular ceramic membrane of molecular weight cut-off: 210 kDa) with adding an anionic surfactant (SDS) below and above the CMC. The synthetic liquid waste contains metal ions in a concentration of 10 mg L⁻¹. The preliminary results show that very good removal percentages of these metal ions are achieved. An ANOVA test on zinc and chromium ultrafiltration results allowed to determine the best operating conditions for metal ions' removal.

Keywords: Ultrafiltration; Surfactant; Wastewaters; Heavy metals; Chromium; Zinc

1. Introduction

Wastewaters from urban and more relevant from industrial activities contain a lot of dangerous metals. Heavy metal pollution of civil and industrial wastewaters represents a major problem for the environment since metal ions are non-biodegradable. These liquid wastes cannot be directly discharged in sewers or reused for further activities but a treatment is mandatory to reduce the metal concentrations up to fulfill emission limits [1,2]. The choice of the right treatment

train depends on the metal type and concentration and on fate of the treated water (e.g. if it is intended for water reuse [3]). In the scientific literature some reviews regarding the removal of heavy metals from wastewater using several types of techniques can be found [4,5]. Adsorption and chemical precipitation are the most widely used techniques to remove heavy metals from wastewaters [6]. The main disadvantages of these methods are that the removal of metals in some cases is not complete, the processes are expensive and have low selectivity [7]. Membrane processes represent a viable and almost cheap solution in the treatment of metal contain-

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ing wastewaters since membranes can be added as a retrofit of existing plants, moreover efficiency removal of 99% can be almost easily gained [8,9]. Membrane processes include nanofiltration, reverse osmosis, ultra filtration and micellar enhanced ultra filtration (MEUF). In the last one, a surface active agent is added to the liquid stream in a concentration higher than critical micellar concentration (CMC), thus forming micelles; when metal ions are in solution, micelles trap metal ions on their surfaces forming micelle-metal complexes [10,11]. The stream is then fed to an ultrafiltration membrane, which retains complexes on its surface (differently from metal ions that it alone would pass through the membrane). To obtain the highest retentions, surface active agents of electric charge opposite to that of the ions to be removed have to be used. At the moment, MEUF is mostly used to remove single pollutants through the addition of anionic surfactants, cationic surfactants or polyelectrolytes. More interesting is the simultaneous removal of two or more chemical species in the same stream, which is made complex by the different ions affinity towards micelles, with the subsequent inhibition of the component with the lowest binding power [12,13]. The present manuscript reports the experimental data for zinc and chromium removal from synthetic solutions that simulate industrial wastewaters coming from waste electronic and electric equipment (WEEE) hydrometallurgical processes. In according to Dlg.s 152/06 the concentrations for sewer discharge of these metals should be $< 1 \text{ mg L}^{-1}$ and $< 4 \text{ mg L}^{-1}$ for zinc and total chromium, respectively. In literature it is possible to find several studies that describe the removal of zinc from wastewater by using MEUF. Huang et al., [14] studied the removal of zinc using ultrafiltration membrane with sodium dodecyl sulfate (SDS) as surfactant. The formation of micellar and the adsorption mechanism were investigated varying in particular the influence of the ratio of SDS to zinc ions on the micelle quantity, the micelle ratio, the gross adsorptive capacity, the rejection of zinc ions was reported. The results showed that the rejection rate of zinc ions reached 97% and the adsorption of zinc ions on SDS was described by the Langmuir adsorption isotherm. Aguirre et al. [15] studied the zinc removal using MEUF and SDS as surfactant. A full factorial plan with two levels was carried out to investigate the influence of pressure, membrane nominal molecular weight limit (NMWL), zinc initial concentration and SDS concentration on zinc removal yields and on permeate absolute flux. It was found that NMWL, pressure, and their respective interaction presented the largest influence on the permeate flux, and a negligible effect on the rejection coefficient. Moreover zinc and SDS initial concentrations and their interaction had a major influence on the rejection coefficient and negligible effect on the permeate flux. The maximum of removal (99%) was achieved when the surfactant/metal molar ratio was above 5. Rahmanian et al. [16] removed zinc from synthetic solutions by spiral-wound ultrafiltration membrane. The effects of different operating conditions on the separation performance of membrane were investigated. The transmembrane pressure had the largest influence on the permeate flux, but it had negligible effect on the rejection coefficient. The results showed that the permeate flux and removal percentage of zinc by anionic surfactant (SDS)–MEUF depended on the ligand-to- Zn^{2+} ratio. The maximum removal yield was of 98%. Regarding the other metal, the removal of chromium has been a topic of several authors [17–19]. Sadaoui et al. [17]

described the removal of hexavalent chromium from wastewaters by ultrafiltration membrane. Cetyltrimethylammonium bromide (CTABr) was used as an adsorbent. The effects of various experimental parameters on equilibrium adsorption were investigated using batch adsorption experiments. It was found that the capacity of chromium adsorption on CTABr increased with initial metal concentration and in a lesser extent with pH solution. Total chromium adsorption decreased slightly with a rise in temperature suggesting an exothermic adsorption of chromium. Moreover the capacity of chromium adsorption decreased with the mass of adsorbent and concentration of other ions present in the solution. Freundlich isotherm represented well the metal ion adsorption. Ghosh et al. [18] reported their experiments performed to analyze the adsorption of chromate ions on the surfaces of the surfactant micelles. It was observed that at high feed concentration, the percent retention of chromate ions increased. However, the flux declined over a varying range of feed concentration. Further, at a much higher surfactant concentration (60 mM), the retention of chromate ions slightly decreased. Aoudia et al. [19] studied chromium removal by MEUF. The permeate flux and rejection characteristics of SDS and nonyl-phenolethoxylated $\text{C}_9\text{H}_{19}\text{C}_6\text{H}_4(\text{CH}_2\text{CH}_2\text{O})_5\text{CH}_2\text{CH}_2\text{OH}$ (NPE) micellar solutions for a ceramic tubular membrane (zirconium oxide–titanium oxide) were investigated. For both surfactants, micelle rejection increased with pressure and reached an asymptotic value at $\Delta P = 250 \text{ kPa}$ beyond which micellar rejection remained unchanged ($\sim 99\%$). Within the optimum composition and $\Delta P = 250 \text{ kPa}$, unexpectedly high rejection (33.50%) was measured below the critical micellar concentration (CMC). Above the CMC, high Cr^{3+} rejections ($+99\%$) were obtained. This rejection was found to be independent of the feed surfactant concentration within the range investigated in this study ($3 \times \text{CMC} - 30 \times \text{CMC}$), suggesting that Cr^{3+} rejection was not a function of the surfactant concentration in the bulk retentate solution but a function of the surfactant concentration nearby the membrane surface.

From this literature review appeared that no study on the simultaneous removal of both metals has been so far carried out. MEUF technique was already studied by this research group for the removal of heavy metals as lead, arsenic, nickel and cobalt from wastewaters [7,20–22] and in the present work, the simultaneous removal of zinc and chromium ions from synthetic industrial wastewaters by surfactant-enhanced ultrafiltration is investigated. A monotubular ceramic membrane of molecular weight cut-off: 210 kDa is used with adding an anionic surfactant (SDS) with concentration below and above the CMC.

2. Materials and methods

2.1. Apparatus description

Experimental studies have been carried out in a tangential flow Membralox® XLAB 3 (Exekia, Bazet, France) laboratory pilot plant with a single tube Membralox® TI-70 ceramic ultra filtration membrane (Fig. 1).

The recirculation pump gives a fixed tangential velocity of about 7 m s^{-1} . All experiments were performed at room temperature; for the cleaning procedure, with water temperature at 40°C , a Crioterm 10–80 thermostat was used.

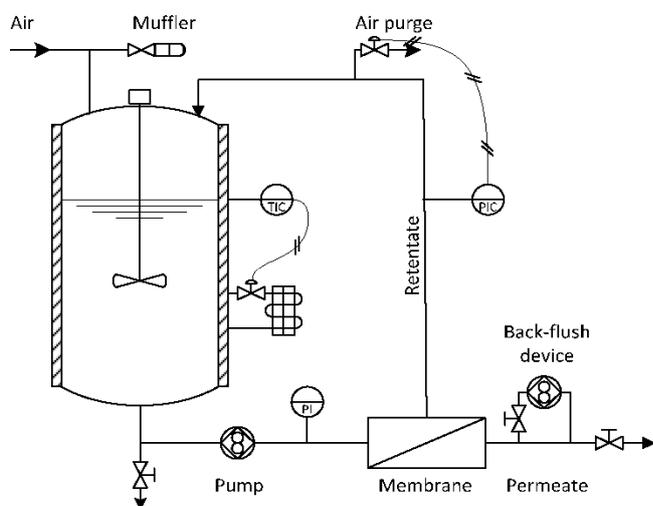


Fig. 1. Sketch of the experimental apparatus.

The plant is equipped with a back flush system BF3, controlled by an electro valve (pressure 7 bar, re-injected volume 3 ml). Back flush was used only during membrane cleaning (frequency 2 min, length 1 s, approximately). The pore size of membrane used in experimental work was 20 nm (MWCO: 210 kDa).

2.2. Feed water characteristics

Synthetic solutions were used to perform the laboratory tests. Chromium chloride hexahydrate (Carlo Erba), zinc sulfate-heptahydrate (Sigma) was used to prepare the solutions, dissolving the salts in distilled water. The volume and the initial Zn and Cr concentrations were 3.5 L and 10 mg L⁻¹, respectively. Distilled water was produced by a D10-T distiller (Enrico Bruno Company). Sodium dodecyl sulfate (SDS, Merck Millipore) was used as a surfactant at two concentration levels $X_1 = 4$ mM and $X_2 = 10$ mM, below and above the CMC, respectively.

2.3. Ultrafiltration procedure

Seven ultrafiltration experiments have been carried out; a summary of this experimental plan is shown in Table 1. For each ultrafiltration test was used a different solution:

- only surfactant solution, over CMC (X_2) for V1 test;
- solution with 10 mg l⁻¹ of chromium in distilled water for V2 test;
- solution with 10 mg l⁻¹ of zinc in distilled water for V3 test;
- solution with 10 mg l⁻¹ of chromium and SDS under CMC (X_1) in distilled water for V4 test;
- solution with 10 mg l⁻¹ of zinc and SDS under CMC (X_1) in distilled water for V5 test;
- solution with 10 mg l⁻¹ of chromium and SDS over CMC (X_2) in distilled water for V6 test;
- solution with 10 mg l⁻¹ of zinc and SDS over CMC (X_2) in distilled water for V7 test.

Table 1
Experimental plan

Test	Surfactant	Cr (mg l ⁻¹)	Zn (mg l ⁻¹)
	SDS (mM)		
V1	X_2	–	–
V2	–	10	–
V3	–	–	10
V4	X_1	10	–
V5	X_1	–	10
V6	X_2	10	–
V7	X_2	–	10

Table 2
Cleaning procedure

Cleaning solution	Concentration	Back flush	TMP [bar]	Time [min]	T [°C]
P3-Ultrasil 25	2% v/v	yes	1.3	30+30	room
Distilled Water	–	yes	1.8	30	50
Distilled Water	–	yes	1.8	30	50

After this measurement, feed tank was filled with 3 L synthetic solution and TMP was adjusted at 0.8 bar with permeate and retentate valves closed; the apparatus was pressurized by nitrogen, so it was not possible to work in a continuous way. Liquid samples were analyzed by atomic absorption spectrophotometry (SpectrAA 200, Varian) to measure the concentrations of zinc and chromium. After each experiment equipment and membrane were washed with alkaline detergents (P3-Ultrasil 25) and rinsed with distilled water until pH returned to the value of about 7, according to the cleaning procedure described in Table 2.

3. Results and discussion

3.1. Flux decay experiments

The following figures show the flux pattern at various transmembrane pressures for each test, as reported in Table 1. Three kinds of data sets are available:

- Metal solution: indicating flux patterns in the presence of the solution to be filtered;
- Dirty membrane, distilled water: referring to the values of the water flux with dirty membrane, *i.e.* with passing distilled water through the membrane once the filtration of the metal solution was finished;
- Clean membrane, distilled water: indicating the values of flux patterns with clean membrane, by using distilled water.

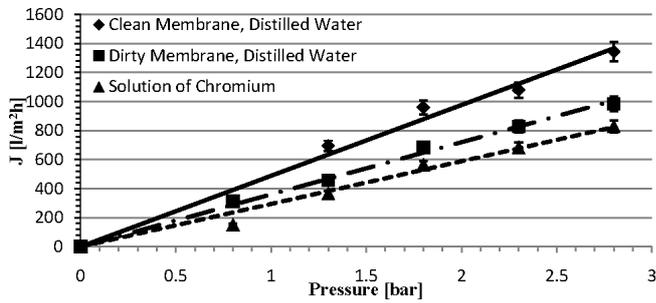


Fig. 2. Permeability curve of V2 test.

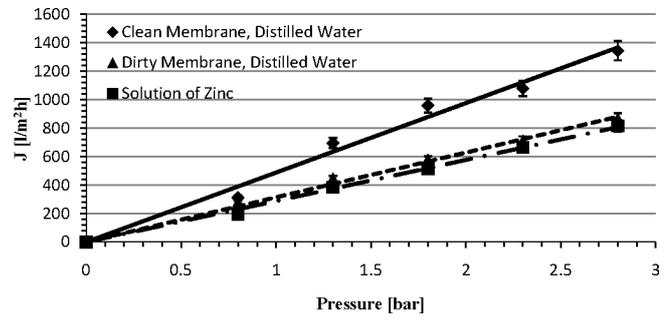


Fig. 5. Permeability curve of V3 test.

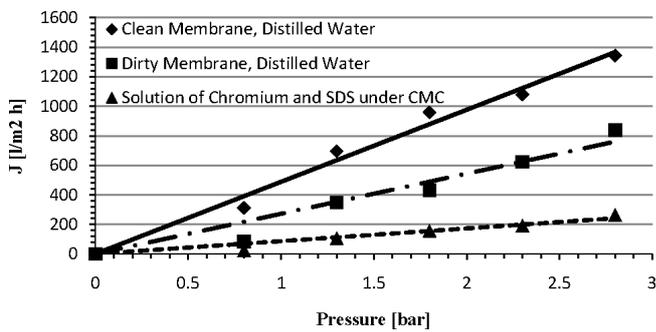


Fig. 3. Permeability curve of V4 test.

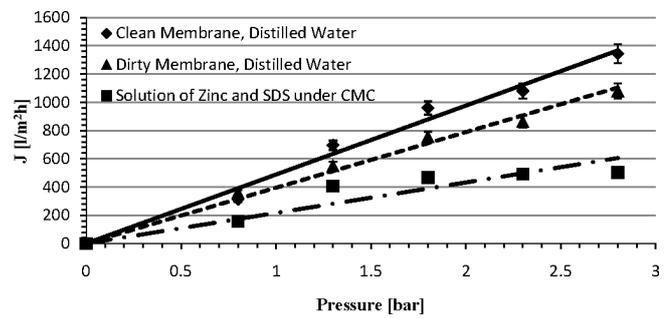


Fig. 6. Permeability curve of V5 test.

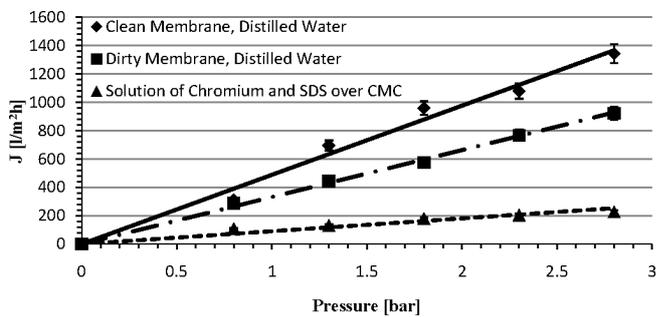


Fig. 4. Permeability curve of V6 test.

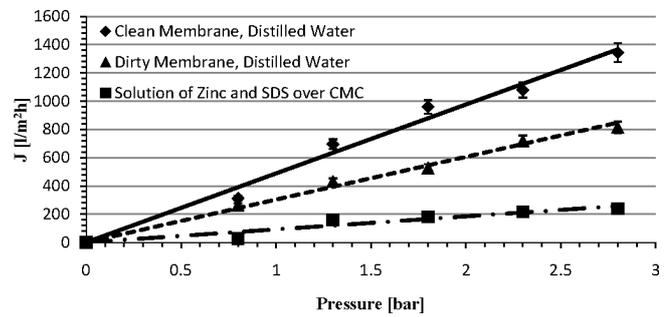


Fig. 7. Permeability curve of V7 test.

Fig. 2 reports flux patterns at various transmembrane pressures for the chromium test without surfactant.

It was possible to observe that the flux was higher for the clean membrane followed by dirty membrane and finally for the chrome solution; this is a logical consequence of different fouling of the membrane. Fig. 3 reports the flux pattern at various transmembrane pressures, for the test with chrome and SDS under CMC.

Fig. 4 reports flux patterns at various transmembrane pressures, for the chrome test with SDS over CMC.

There is a net difference between the test with dirty membrane-distilled water and the solution of chrome and SDS. The difference is probably due to the different fouling of the membrane that was previously fouled in the chrome-SDS test.

Fig. 5 reports flux patterns at various transmembrane pressures for the zinc test without surfactant.

As already noted in Fig. 2, for each pressure flux sequence is: clean membrane/dirty membrane/metal solution; this is a logical consequence of different fouling of the membrane.

Fig. 6 reports flux patterns at various transmembrane pressures, for the zinc test with SDS under CMC.

Fig. 7 reports flux patterns at various transmembrane pressures, for the zinc test with SDS over CMC.

There is a net difference between the test with dirty membrane-distilled water and the solution of zinc and SDS. The difference is probably due to the different fouling of the membrane that was previously dirtied in the zinc-SDS test.

Resuming, in the presence of SDS permeate fluxes are considerably lower than in its absence as expected, but these results are of poor meaning if they are not compared to metal rejections, reported in the following Section 3.3.

3.2. Membrane resistances

Starting from the curves of permeability it is possible to obtain the values of the resistances opposed from the membrane to the mass transfer, calculated as reciprocal per-

meability. The permeability is the slope “ m ” of the curve of permeability, therefore R is:

$$R = \frac{1}{m} \quad (1)$$

where R is expressed in $[\text{m}^2 \text{ h bar l}^{-1}]$

For example, considering permeability curves of V7 test, according to the resistances in series model, the total resistance of the membrane to mass transfer can be considered as composed of three terms:

$$R_{tot} = R_m + R_f + R_p \quad (2)$$

where: R_{tot} is the total resistance; R_m is the membrane resistance; R_f is the fouling resistance; R_p is the concentration polarization resistance.

In the test with distilled water, R_m is the only resistance and it can be calculated using Eq. (3):

$$R = \frac{1}{m_w} \quad (3)$$

where m_w represents the slope of the straight line obtained in the test with distilled water and clean membrane. Referring to Fig. 7, m_w is equal to $488.21 [\text{l m}^{-2} \text{ l}^{-1} \text{ bar}^{-1}]$ and is thus $R_m = 0.002 [\text{m}^2 \text{ h bar l}^{-1}]$.

By the straight line relating dirty membrane and distilled water, however, it is possible to calculate the resistance R_f , equal to the sum of R_m and R_f :

$$R_f = R_m + R_f \quad (4)$$

As a matter of fact, during the test performed with distilled water and with using dirty membrane, the concentration polarization resistance, due to fouling, is lacking. If m_w^d is the slope of the relative membrane dirty and distilled water, R_f is calculated using the following equation:

$$R_f = \frac{1}{m_w^d} \quad (5)$$

In according to the data of the Figure 7, m_w^d and R_f are equal to $302.89 [\text{l m}^{-2} \text{ l}^{-1} \text{ bar}^{-1}]$ and $0.0033 [\text{m}^2 \text{ h bar l}^{-1}]$, respectively.

Finally, the slope of the line relative to the solution, referred to as m_{SOL} is the inverse of the resistance R_{tot} :

$$R_m = \frac{1}{m_{SOL}} \quad (6)$$

Referring to Fig. 7, m_{SOL} is $91.97 [\text{l m}^{-2} \text{ l}^{-1} \text{ bar}^{-1}]$ and R_t is $0.0108 [\text{m}^2 \text{ h bar l}^{-1}]$.

The values of the resistances due to the fouling and to the concentration of polarization, respectively, can be calculated as follows:

$$R_f = R_t - R_m \quad (7)$$

$$R_p = R_{tot} - R_t \quad (8)$$

Table 3
Calculated resistances $[\text{m}^2 \text{ h bar l}^{-1}]$ during MEUF process

	TEST					
	V2	V3	V4	V5	V6	V7
R_m	0.002	0.002	0.002	0.002	0.002	0.002
R_{tot}	0.003	0.0034	0.011	0.0046	0.011	0.0108
R_f	0.0027	0.0031	0.0036	0.0025	0.003	0.0033
R_p	0.0007	0.0011	0.0016	0.0005	0.001	0.0013
R_p	0.0003	0.0003	0.0074	0.0021	0.008	0.0075

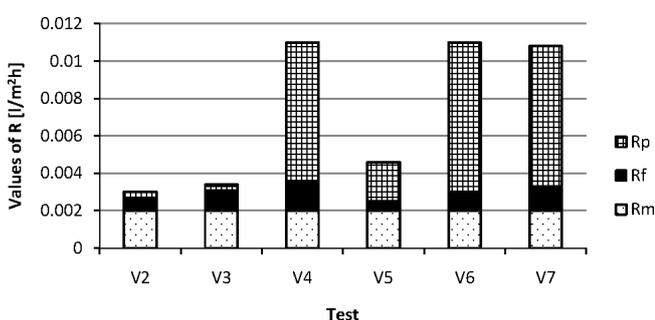


Fig. 8. Membrane resistances in zinc and chromium ultra filtration test.

Table 4
Removal percentages for zinc and chromium

Metal	Percentage rejections [%]		
	Without SDS	SDS below CMC (4 mM)	SDS above CMC (10 mM)
Chromium	4.97	83.05	96.65
Zinc	5	81.28	98

Table 3 and Fig. 8 summarize the values of the obtained resistances for each test.

It is possible to note that the values of R_m are the same for each test being the membrane equal for all. The values of R_{tot} in V2 and V3 test are similar but this value is slightly higher for the V3 test.

In the presence of SDS with a concentration less than CMC (V4 and V5 tests) the total resistances are higher with respect to other experiments; this is a consequence of the ability of micelles to retain the zinc and chromium ions, as already demonstrates by the rejections yields (Table 4).

3.3. Metal removal

Table 4 reports zinc and chromium removal in the presence and in the absence of surfactant.

The presence of SDS improves the removal percentage of the metals from solutions. For chromium the percentage of removal was around 5% without SDS and reaches 96% with SDS with concentration greater than CMC. For zinc

alone the removal is very low (5%), with using SDS the percentage increases until around 98%.

Therefore, it can be concluded that even if the presence of surfactant significantly reduces the fluxes through the membrane (see Section 3.1), however it allows the retention of metal ions, that otherwise would pass through the pores.

3.4. ANOVA zinc and chromium

Finally, a statistical analysis has been carried out on the obtained results. Yates' algorithm was used to evaluate whether effects and interactions among factors are significant with respect to the experimental error as for the zinc and chromium removal tests. The significance of main factors and their interactions was assessed by F-test method with a confidence level of 95%.

The experimental error was calculated using the data of the central point. The variance of the experimental error calculated was 0.1152 with 1 degree of freedom. The ANOVA data as for zinc is reported in Table 5. The maximum zinc removal (98% of rejection) is obtained using a membrane with 210 kDa, in the presence of surfactant over CMC and 0.8 bar of pressure. Good removal efficiencies, around 80%, are also recovered during the central tests (I, II).

Elaboration of ANOVA analysis are reported in Table 5 and Fig. 9.

It is possible to observe that factor A (pressure), factor B (SDS concentration) were significant at 95% with negative and positive effect, respectively. The rejection yields decreased with the increasing of the pressure, instead the presence of surfactant resulted to be very important to obtain a good zinc removal efficiency. The interaction

Table 5
ANOVA analysis for zinc rejection [%]

Test	Pressure	SDS	Rejection [%]	Effects	Significance [%]
1	0.8	0	5	Effects	
2	2.8	0	0.25	A -9.875	98
3	0.8	X2	98	B 87.875	100
4	2.8	X2	83	AB -5.125	96
I	1.3	X1	80.8	-	-
II	1.3	X1	81.28	-	-

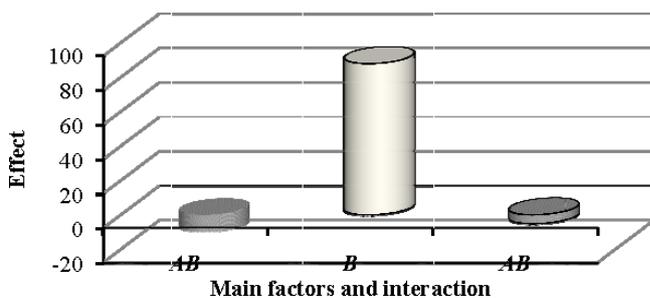


Fig. 9. Main factors and interaction obtained using ANOVA analysis for zinc (A= pressure, B= surfactant concentration).

AB (pressure–SDS concentration) had a negative effect as shown in Fig. 10:

The figure shows, again, the strong positive effect of SDS concentration on zinc removal yields. When the factor B was at low level (surfactant concentration equal to zero) the efficiencies were very low and the yields decreased moving from low to high pressure. The negative effect of this last factor was also noticed when the factor A was at high level, concentration of SDS was equal to X₂. The removal of zinc decreased from 98% to 83% moving from 0.8 bar to 2.8 bar.

It was clear that to maximize the zinc extraction the operating conditions required low pressure and the presence of surfactant.

The ANOVA elaboration for chromium, reported in Table 6, shows that the maximum chromium removal (96.65% of rejection) is obtained using a membrane with 210 kDa, in the presence of surfactant over CMC and 0.8 bar of pressure. Also good removal efficiencies, around 80%, were recovered during the central tests (I, II) with SDS under CMC at 1.3 value of pressure. Elaboration of ANOVA analysis were reported in Table 6 and Fig. 11.

It is possible to observe that factor B (SDS concentration) is significant at 95% with positive effect while factor A (pressure) as well as interaction between A and B factors are not significant with negative effects.

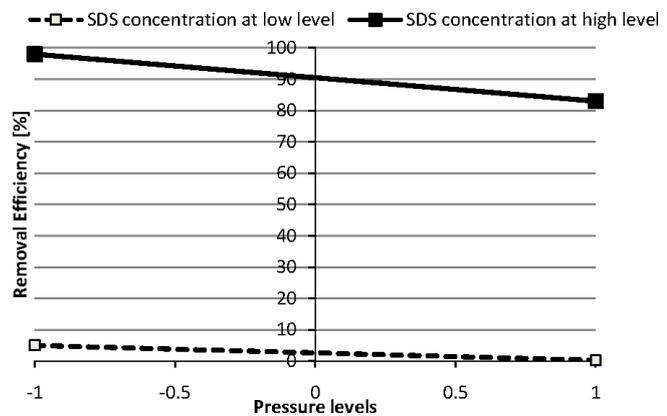


Fig. 10. Interaction AB – pressure and SDS concentration.

Table 6
ANOVA analysis for chromium rejection [%]

Test	Pressure	SDS	Rejection [%]	Effects	Significance [%]
1	0.8	0	4.97	Effects	
2	2.8	0	0.31	A -10.805	93
3	0.8	X2	96.65	B 85.535	99
4	2.8	X2	79.7	AB -6.145	89
I	1.3	X1	83.05	-	-
II	1.3	X1	81.48	-	-

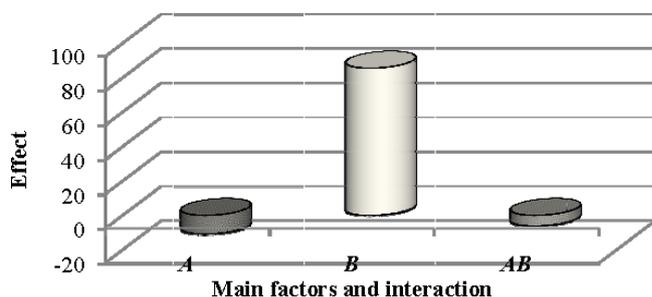


Fig. 11. Main factors and interaction obtained using ANOVA analysis for chromium (A= pressure, B= surfactant concentration).

4. Conclusions

In this paper, the removal of chromium and zinc ions from industrial wastewaters by surfactant enhanced ultrafiltration has been investigated. The surface active agent used is anionic (dodecylbenzenesulfonic acid) which has been added to a synthetic industrial liquid waste obtained by the addition of chromium and zinc ions to distilled water. The solutions simulated a possible wastewater produced in a hydrometallurgical process for the treatment of WEEE. The mixture was forced through an ultrafiltration membrane with molecular cut-off of 210 kDa. Surfactant has been tested at two concentration levels *i.e.* 4 mM and 10 mM, below and above the CMC, respectively. The best removal percentages of the metals were around 90%, obtained in the presence of the surfactant. In the experiments without surfactant the percentages of rejection were 31% and 5% for chromium and zinc, respectively. An Anova analysis of the results obtained both with zinc and chromium solution, allowed to see in a clear way that to maximize metal removal, the operating conditions required are low pressure and the presence of surfactant. However, the obtained percentage removals, depending on metal initial concentration, could be satisfactory for discharge according to the Law limit. It is to point out that with a slightly lower membrane cut-off or a pre-treatment step the performances could surely be improved.

The future work will be thus aimed at studying the removal of the metals varying experimental conditions, as the SDS concentrations and membrane cut off, as well as the recovery of the surfactant to render for making more affordable the costs of the whole process. As a matter of fact, since surfactants are expensive and non-biodegradable, the environmental hazard of them remained in effluent is a serious disadvantage of complexation-membrane separation methods and has to be faced.

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