

Treatment of concentrated arsenic(V) solutions by micellar enhanced ultrafiltration with high molecular weight cut-off membrane

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Abstract

In this work arsenic removal by micellar enhanced ultrafiltration (MEUF) was investigated using cetylpyridinium chloride (CPC) and a cross-flow polyethersulphone (PES) membrane apparatus. The effects of some operating factors on permeate flux, arsenic and CPC rejections were investigated and, in particular, transmembrane pressure, pH, CPC concentration, As concentration and ionic strength. The novel aim of this work is evaluating the possible advantages of using large molecular weight cut-off membrane (100 kDa) and reduced surfactant concentrations (1–3 mM) for treating high fluxes of concentrated arsenic-bearing solutions (6–10 ppm).

The experimental results reported in this paper show that PES membrane apparatus with high molecular weight cut-off allowed to treat large fluxes of concentrated arsenic-bearing solutions (6–10 ppm) even by using low surfactant concentration (1–3 mM). In particular arsenic removal ranged from 93–98% to 70–74% depending on initial As concentration (6 and 10 ppm, respectively). In addition surfactant leakage in the permeate was always below CMC due to presieving of concentration polarisation layer. The favourable combination of high MWCO membranes and low surfactant concentration can benefit to overall process economics for the lower membrane area requirement (due to greater flux) and the reduced surfactant consumption.

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1. Introduction

The stringency of modern environmental standards encourages the search for industrial waste treatments requiring low energy, labour, and capital costs. Among membrane processes, nanofiltration and reverse osmosis can be used to remove inorganic ions (such as heavy metals) by capillary flow or solution diffusion mechanisms. Nevertheless in both these processes dense membranes are used, which are characterised by high operating pressures, very low fluxes and pore fouling. Unlike nanofiltration and reverse osmosis, the separation in ultrafiltration (UF) and microfiltration processes is accomplished by mechanical sieving allowing high fluxes at low pressures and without requiring pretreatment of source water. UF processes can remove only partially heavy metals in solution because

of the little dimensions of these ionic species in comparison with membrane pores [1]. Nevertheless the attractive low-energy characteristics of UF can be exploited also for heavy metal removal by using surfactant-based separation processes such as micellar-enhanced ultrafiltration (MEUF) [1–9]. An anionic or cationic surfactant at a concentration greater than its critical micellar concentration (CMC), is added to the aqueous solution containing the dissolved solutes (cations or anions, respectively). The surfactant, present in the micellar form, can bind ions on the surface of the oppositely charged micelles. An UF membrane with pore sizes small enough to block the passage of micelles, is then used to filtrate this metal-bearing emulsion. By this way ions sorbed onto the micelles are rejected and only unbound ions and surfactant monomers can pass in the permeate stream.

The effectiveness of MEUF processes is strictly related to the achievement of high permeate fluxes and high rejections of both heavy metals and surfactant. Metal retention in MEUF is mainly governed by electrostatic interactions for the exchange of metal

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ions at the micellar surface [7]. Nevertheless the complexity of wastewaters often requires that also the metal speciation in solution be specifically taken into account. In fact every change in the electric charge of the metal ion species (caused by pH changes or complexation by ligands in solution) can drastically modify the interaction with the micellar surface and then the distribution of the cation between charged micellar phase and bulk phase.

Operational targets related to permeate fluxes and surfactant rejection can be optimised by taking into account two main phenomena: pore fouling and concentration polarisation on the membrane. Concentration polarisation generates a concentrated surfactant layer close to the membrane causing additional resistance and increase of osmotic pressure across the membrane. Low permeate fluxes are then observed especially working at high pressure, low retentate flow velocity, and high viscosity of feed solution [10].

Surfactant rejection is increased by high retentate flow velocity due to the attenuation of concentration polarisation produced by fast retentate flux (reduced surfactant gradient across the membrane) [10]. On the other side opposite effects on surfactant rejections were observed by increasing transmembrane pressure: the augment of surfactant in the permeate as pressure increases was explained by taking into account micelle deformation, micelle decomposition and high surfactant concentration near the membrane (increased surfactant gradient across the membrane), while the opposite effect (diminution of surfactant in permeate as pressure increases) was explained by presieving effect sometime observed for concentration polarisation [10,11].

The improvement of surfactant rejection can be then approached by the modification of membrane module or material in order to reduce the concentration polarisation [12]. Nevertheless concentration polarisation itself can be also exploited to design MEUF working below CMC and reducing surfactant release and consumption. In fact the concentration polarisation can result in the formation of micelles near the membrane surface even below the surfactant CMC [13]. In addition the presieving of gel layer associated to concentration polarisation can have a crucial effect on metal rejection when surfactant concentration was lower than its CMC [2,5,14].

Membrane pore size also plays an important role in this context: increasing the molecular weight cut-off (MWCO) of the membrane can cause an earlier development of concentration polarisation regime and reduce the surfactant release in the permeate for presieving effect. As a consequence even for very large pore size (50 kDa MWCO) the vast majority of micelles can be rejected [11]. According to this finding, high MWCO membrane present extremely good rejection characteristics with minimum membrane area requirement and capital cost.

In this work arsenic removal by MEUF was investigated using cetylpyridinium chloride (CPC) and a cross-flow polyethersulphone (PES) membrane apparatus. The effects of some operating factors on permeate flux, arsenic and CPC rejections were investigated and, in particular transmembrane pressure, pH, CPC concentration, As concentration and ionic strength. The novelty is evaluating the possible advantages of using large molecular weight cut-off membrane (100 kDa) and reduced surfactant con-

centrations (1–3 mM) for treating high fluxes of concentrated arsenic-bearing solutions (6–10 ppm). This operative combination aimed at improving the cost effectiveness of the process taking advantage of the concentration-polarisation and presieving effects associated to high fluxes, but also minimising membrane fouling by high MWCO and low surfactant concentration. This would allow the reduction of surfactant leakage on one hand, and the economical treatment of large streams of highly polluted waters on the other.

2. Materials and methods

Micellar enhanced ultrafiltration tests for As removal were carried out using a polyethersulphone (PES) membrane with 100 kDa molecular weight cut-off (MWCO) and a total area of 36 cm².

An emulsion of surfactant (cetylpyridinium chloride, CPC) and arsenic (As₂O₅) was introduced in a temperature controlled glass reactor (liquid volume 100 mL, temperature 30 °C) and fed through the membrane module by a peristaltic pump. The mixing in the reactor was ensured by magnetic stirring.

In permeability tests transmembrane pressure was varied (0.4–2 kPa) and correspondently permeate flux was measured by collecting permeate volume at fixed time. Both permeate and retentate were sampled for pH, Eh, As and CPC measurements. Both permeate and retentate streams were recirculated in the reactor. Experimental conditions investigated for permeability tests were reported in Table 1.

In diafiltration test only the retentate was recirculated inside the reactor by a peristaltic pump. The liquid volume in the reactor was kept constant (0.5 L) by manual tuning of the feed stream pump. Diafiltration was performed according to the following operating conditions: TMP = 1.85 kPa; pH 7; 2 mM surfactant feed concentration; 10 ppm As feed concentration.

pH and Eh were measured by specific electrodes. Cetylpyridinium was determined by means of kit LANGE LCK 331: the cationic surfactant reacts with bromophenol blue and forms complexes that are extracted into chloroform and read at 410 nm by a CADAS 50 Dr. LANGE spectrophotometer. Repeated determinations revealed an experimental error of about 5%. Arsenic was determined by a flame atomic absorption

Table 1
Operating conditions adopted in permeability tests

Test set	Test number	[CPC] (mM)	[As] (mg/L)	[NaNO ₃] _F (mM)
SET 1	1	0.97	6.5	–
	2	1.41	6.6	–
	3	2.86	6.5	–
	4	1.03	6.1	–
	5	1.78	6.3	–
	6	2.14	6.3	–
SET 2	7	1.93	11.1	–
	8	3.03	11.5	–
	9	1.99	4.4	–
SET 3	10	0.99	9.0	0.1
	11	2.85	9.2	0.1

spectrophotometer (Varian SpectrAA200), with an arsenic quantisation limit of 1 ppm. The same procedure was used both in the presence and in the absence of surfactant, as verified by means of determinations on control samples. Measures were performed in triplicate.

3. Results and discussion

3.1. Permeability tests

Permeability tests were performed to evaluate the effect of transmembrane pressure on permeate fluxes, As and CPC retention coefficients according to the operating conditions reported in Table 1.

3.1.1. Arsenic speciation

Both pH and Eh in retentate and permeate streams were monitored during permeability tests. These variables remained quite constants as transmembrane pressure was increased (experimental data not shown here). Mean values of pH and Eh in the retentate (pH_R and Eh_R , respectively) were reported in Table 2. In the investigated range of conditions the lower boundary of potential (LB Eh) for As(V) species was calculated for the different pH conditions by empirical correlations [1] (LB Eh in Table 2). Comparing measured and calculated Eh, arsenic is mainly present as arsenate ions in the form of $H_2AsO_4^-$ (pH between 2.24 and 6.88) and $HAsO_4^{2-}$ (pH between 6.88 and 11.44). These As negative ions can be also retained in UF tests without micelles due to the negative charge of membrane matrix (Donnan-exclusion effect) [14]. Nevertheless blank tests without CPC (experimental data not reported here) denoted that PES membranes can provide only low As rejections due to the slight negative surface charge of this polymeric material as determined by streaming potential measurements [1,15].

3.1.2. Membrane resistance

Permeate flux (J_p) dependence on transmembrane pressure (TMP) can be represented by linear trend under the hypothesis of pressure control [16]. The apparent total membrane resistance

(R_{TOT}) can be then evaluated as

$$R_{TOT} = \frac{TMP}{J_p} \quad (1)$$

Linear regressions of J_p versus TMP data for the different permeability tests were performed and regressed values of R_{TOT} are reported in Table 2. The coefficients of determinations (R^2) show that linear model is an adequate approximation of the system for SET 1 and SET 2 meaning that there is not any additional resistance to flow within the hydrodynamic boundary layer next to the membrane [11]. As a consequence, in these conditions hyperbolic models considering the additional resistance are not necessary. Nevertheless a partial reduction of the permeate fluxes with respect to pure solvent tests is also observed. Mean value of R_{TOT} for pure water tests is 1.2 MPa h m^{-1} denoting that in all the investigated conditions there was a partial reduction of the permeate flux due to membrane fouling and concentration-polarisation effect.

Experimental data of SET 3 denote the effect of ionic strength in permeability tests. Feed solution containing 0.1 M $NaNO_3$ drastically changes the operative conditions in the systems especially for membrane fouling due to the high nitrate amount reacting with the micelles. This is evident from the larger values of membrane resistance reported in Table 2. The lower R^2 for SET 3 data also denote the inadequacy of linear model for regressing fluxes versus transmembrane pressure data.

R_{TOT} values for the different permeability tests are reported in Fig. 1 as a function of CPC concentration and pH. R_{TOT} is not significantly affected by CPC concentration in the range of investigated conditions here considered. This is in accordance with the observed linearity of permeate flux vs. transmembrane pressure denoting that minor interactions among surfactant, micelles and membrane pore and surface take place. On the other hand, the increase of As concentration in the system (SET 2) determines higher membrane resistance: this can be due to the larger

Table 2
Arsenic speciation indicators (pH–Eh) and total membrane resistance observed in permeability tests (see text for details)

Test set	Test number	pH_R	Eh_R (mV)	LB Eh (mV)	R_{TOT} (MPa h m^{-1})	R^2
SET 1	1	7.17	168.5	-36.9	1.399	0.955
	2	7.09	193.3	-26.9	1.384	0.979
	3	6.66	200.6	20.3	1.394	0.943
	4	8.57	163.4	-219.6	1.459	0.957
	5	7.87	180.0	-127.8	1.302	0.921
	6	8.63	143.4	-226.7	1.478	0.957
SET 2	7	9.10	126.3	-279.4	1.519	0.982
	8	10.17	87.0	-351.8	1.687	0.978
	9	7.02	223.6	-18.1	1.613	0.965
SET 3	10	9.93	89.4	-335.6	2.196	0.583
	11	10.08	54.3	-345.7	2.226	0.760

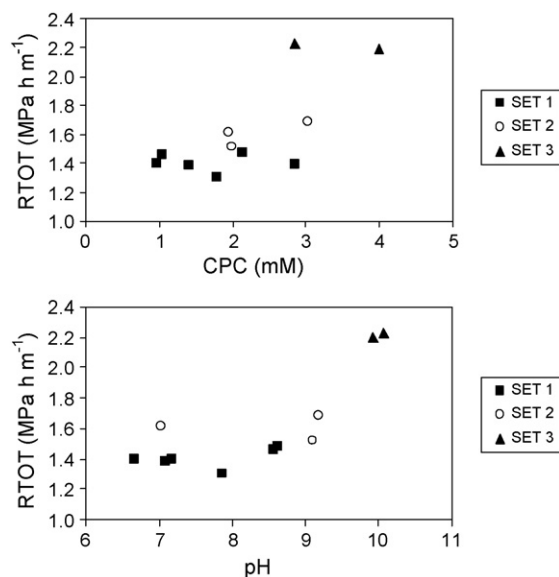


Fig. 1. Membrane resistance (R_{TOT}) vs. [CPC] and pH in permeability tests (see Table 1 for details on operating conditions).

dimensions of As-micelles with respect to Cl-micelles causing membrane pore plugging and flux decrease. A slight effect of pH was also observed probably due to the closer packing of As-micelles with HAsO_4^{2-} which can bind two CPC head groups. As a consequence the shape and aggregation number of the micelles can change giving closely packed large surfactant aggregates, which causes a permeate flux decline [1].

3.1.3. Arsenic and CPC retention coefficients

As and CPC retention coefficients (σ_{As} and σ_{CPC}) were evaluated by comparing retentate and permeate concentrations for both components:

$$\sigma = 1 - \frac{C_P}{C_R} \tag{2}$$

Retention coefficients measured during permeability tests were not significantly affected by the change of transmembrane pressure (experimental data not reported here). Mean values were then considered in the following discussion and reported in Table 3.

In SET 1 and SET 2 tests, arsenic retention coefficients ranged from 0.74 to 0.98 showing the effectiveness of CPC addition in comparison with previously cited blank tests without CPC ($\sigma_{\text{As}} = 0.5$ at pH 8).

σ_{As} values are positively affected by CPC concentration due to the consequent increased number of micelles in the system able to bind As(V) species (Fig. 2). For larger As concentration (SET 2) lower retention were observed requiring larger CPC concentration in the systems.

Nevertheless it should be noted that the system was operated with As concentrations (6–10 ppm) that are larger than those generally used in MEUF studies (<1 ppm) [1,14,17,18] and with CPC amounts (1–3 mM) that are lower than the usual ones (10–100 mM) [1,11]. These specific choices were made to evaluate possible favourable effects of these operating conditions combined with a large MWCO membrane allowing high flow rate (100 kDa against 5–10 kDa) [1]. By this way even large stream of extremely concentrated solutions could be treated minimising both operative costs (lower membrane surface) and surfactant use. This is the case of acid mine drainage generated from dismissed sulphite mine area where As is generally

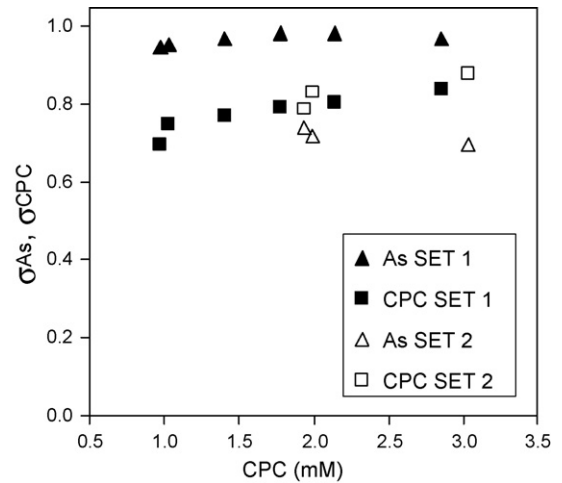


Fig. 2. Arsenic and CPC retention coefficients (σ_{As} and σ_{CPC}) as a function of surfactant concentration in permeability tests (SET 1 and SET 2).

present at high concentration as the product of dissolution of arsenopyrite [19].

CPC retention coefficients present a slight positive dependence on CPC concentration regardless arsenic concentration in the system (Fig. 2).

As for the effect of pH, not significant effect can be noted: only a slight increase of both As and CPC due to formation of dianionic HAsO_4^{2-} species which interact with CPC more strongly also causing micelles aggregation.

The effects of pH and CPC concentration on σ_{As} and σ_{CPC} were isolated by regressing SET 1 and SET 2 data by a linear model neglecting pH-CPC interactions (Table 4):

$$\sigma_i = \sigma_i^0 + \sigma_i^{\text{pH}} \text{pH} + \sigma_i^{\text{CPC}} [\text{CPC}] \tag{3}$$

where i is As or CPC.

The values of the independent variables (pH and [CPC]) fall in the same range (0–10) and, as a consequence, the regressed parameters (σ_i^{pH} and σ_i^{CPC}) can be considered a direct measure of the sensitivity of the dependent variables (σ_{As} and σ_{CPC}) with respect to pH and [CPC] changes. It is possible to see that both σ_{As} and σ_{CPC} are mainly influenced by [CPC] change and this effect is more pronounced for σ_{CPC} , while pH variation has minor effects on both retention coefficients (Fig. 3).

The presence of NaNO_3 (0.1 M) drastically reduced arsenic retention. In fact arsenic retention coefficients were found to be not higher than 0.1, under tested conditions in SET 3 (Table 3). This is due to competition between arsenate and nitrate ions, the latter being present in a concentration significantly higher than the former. A similar trend was also observed by Aoudia et al.

Table 3
Arsenic and CPC retention coefficients (σ_{As} and σ_{CPC})

Test set	Test number	σ_{As}	σ_{CPC}
SET 1	1	0.90	0.70
	2	0.93	0.77
	3	0.90	0.84
	4	0.90	0.75
	5	0.98	0.79
SET 2	6	0.98	0.81
	7	0.92	0.83
	8	0.92	0.88
	9	0.74	0.79
SET 3	10	0.00	1.00
	11	0.13	1.00

Table 4
Linear modelling (Eq. (3)) of arsenic and CPC retention coefficients vs. pH and CPC concentration

Adjustable parameters	σ_{As}	R^2	σ_{CPC}	R^2
σ_i^0	0.889		0.568	
σ_i^{pH}	0.007	0.999	0.012	0.999
σ_i^{CPC} (mM^{-1})	0.016		0.069	

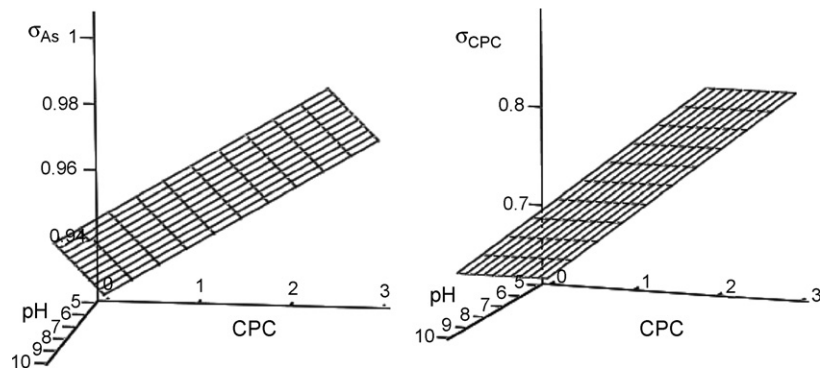


Fig. 3. 3D representation of the linear models for σ_{As} and σ_{CPC} vs. pH and [CPC].

[20] in the presence of sodium chloride. On the other side CPC retention is increased due to the presieving effect associated to membrane fouling and pore plugging in accordance with the larger R_{TOT} observed for SET 3 tests.

3.1.4. CPC release

Surfactant leakage in the permeate is one of the most important parameter to be optimised in MEUF process in order to minimise both costs and environmental problems associated to this release. Operating conditions considered in these tests denoted that CPC concentrations in the permeate are always lower the critical micellar concentration (CMC) of this surfactant (0.9 mM) [1] (Fig. 4). This favourable result can be explained by the combined effect of working with high fluxes and low surfactant concentrations. In these conditions concentration-polarisation is maximised allowing presieving effect also towards CPC monomers. In addition due to the high MWCO monomers can also interact with the membrane by filling the pores and adding a further filtering effect towards surfactant. Nevertheless these interactions do not affect negatively permeate flux according to the apparent membrane resistance observed during the different permeability tests. As a prove of this hypothesis, in SET 3 with 0.1 M nitrate added in the feed solution, the observed membrane fouling caused the complete retention of CPC in the system (Fig. 4).

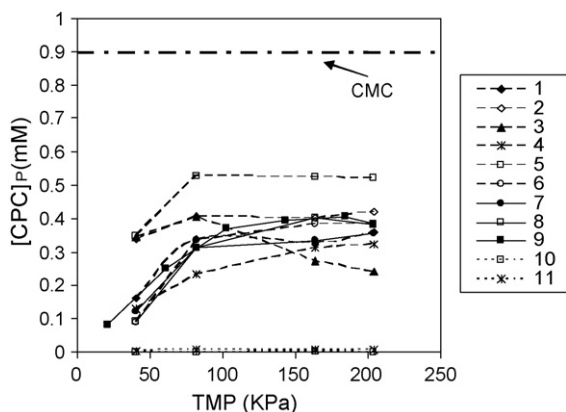


Fig. 4. CPC release in the permeate stream for permeability tests (see Table 1 for treatments conditions).

3.2. Diafiltration test

A diafiltration test was performed in a laboratory scale continuous operation in order to simulate possible larger scale wastewater treatment (Fig. 5).

Experimental data of As concentration in retentate and permeate, denoting the retention properties of the systems, can be adequately simulated by a simple dynamic model based on the metal balance in the system [21,22]:

$$\frac{d(C_R V)}{dt} = F(C_{IN} - C_{OUT}) \quad (4)$$

where V is the volume reactor (L), F the volumetric flow rate (L/h), C_{IN} , C_R and C_{OUT} are the arsenic concentrations (ppm) in the feed, the retentate and the permeate, respectively.

The amount of metal bound to the micelles can be expressed by using the metal retention coefficient as

$$C_{OUT} = C_R(1 - \sigma) \quad (5)$$

Eq. (5) was inserted in the metal balance, which can be solved analytically for a constant σ value (0.9) evaluated by comparing

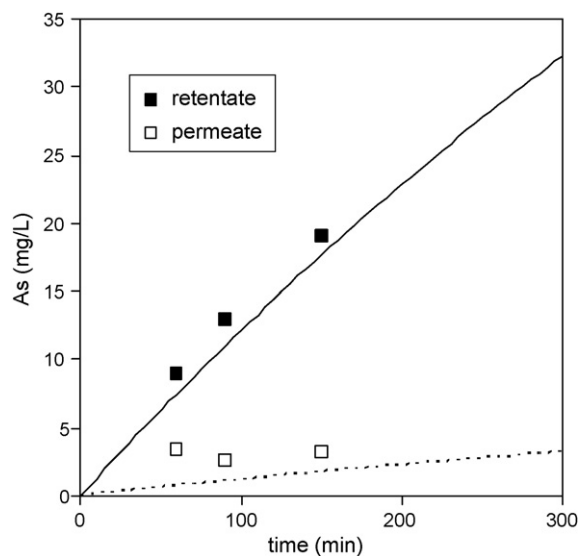


Fig. 5. Arsenic concentration in the retentate and in the permeate during diafiltration test. Lines for retentate (—) and permeate (---) concentrations were calculated by Eqs. (6) and (7), respectively.

retentate and permeate concentration during the test:

$$C_R = \frac{1}{1-\sigma} C_{IN} \left\{ 1 - \exp \left[-\frac{t}{\tau} (1-\sigma) \right] \right\} \quad (6)$$

$$C_{OUT} = C_{IN} \left\{ 1 - \exp \left[-\frac{t}{\tau} (1-\sigma) \right] \right\} \quad (7)$$

Simulated profiles calculated by Eqs. (6) and (7) were compared with the experimental data reported in Fig. 5 showing the adequate representation of the dynamic behaviour of the system.

4. Conclusions

The experimental results reported in this paper show that PES membrane apparatus with high molecular weight cut-off allowed to treat large fluxes of concentrated arsenic-bearing solutions (6–10 ppm) even by using low surfactant concentration (1–3 mM). In particular arsenic removal ranged from 93 to 98% for SET 1 conditions and from 70 to 74% for SET 2 and surfactant leakage in the permeate is always below CMC. This favourable combination can benefit to overall process economics for the lower membrane area requirement due to greater flux and the reduced surfactant consumption. Nevertheless, some crucial aspects still remained open for future research. First of all the problem of water recovery: the aforementioned advantages of using low surfactant concentrations are accompanied by the necessity of a concentration step for the final disposal of low-concentrated retentate. Finally, surfactant leakage remained to be addressed even using low-concentrated emulsion. This is the reason why many authors in the literature are studying processes where the ultrafiltration technology is integrated with water soluble polymers rather than with surfactants [23]. The challenge for further work will be the practical application of MEUF, looking for experimental conditions which allow the achievement of arsenic retention, no surfactant leakage and high permeate fluxes.

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