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Removal of phenolic compounds by micellar enhanced ultrafiltration using an anionic surfactant

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Abstract

Micellar Enhanced Ultrafiltration (MEUF) of different phenolic derivatives including para-coumaric acid, vanillic acid, and tyrosol, in aqueous solutions has been studied. The MEUF experiments were conducted using an anionic surfactant, namely, Sodium Dodecyl Sulfate (SDS). Two polyethersulfone membranes with different molecular weight cut-off (20 kDa and 50 kDa) were used. Experiments were conducted in a stirred batch cell. The effects of surfactant and solutes concentrations on the permeate flux and on the observed retention have been investigated. The MEUF efficiency was compared to the nanofiltration in terms of flux and rejection. Monovalent and divalent electrolytes were tested for precipitation and recovery of the dodecyl sulfate anion from the retentate stream. The retention of solutes without using any surfactant varies from 1.2 to 6.8% only, whereas, under the same operating conditions, retention increases to about 30 to 67% depending on the nature of solute, the surfactant concentration and the molecular weight cut-off of the membrane. The rise of the surfactant concentration from 5 CMC to 10 CMC increases the phenolic compounds rejection by 15 to 30% only. Our tests demonstrated that the SDS can be removed easily from the retentate by precipitation using $CaCl_2$. The MEUF appears as a promising process for the treatment and valorization of phenolic effluents since it allows higher rejection and reasonable fluxes compared to the nanofiltration which showed a maximum rejection of 40% for vanillic acid and 13% for tyrosol.

Keywords: Micellar Enhanced Ultrafiltration, phenolic compounds, anionic surfactant, rejection.

Nomenclature

Abbreviations
MEUF : micellar-enhanced ultrafiltration.
CMC : critical micellar concentration.
PCA : *p*-coumaric acid.
VA : vanillic acid.
TY : tyrosol.
OD : optical density.
RFR : relative flux reduction (%)
VCF : volume concentration factor.
Symbols
J : permeate flux (l/h.m²).
R : rejection (%).
v : volume.
K_{ow} : octanol-water partition coefficient.
Subscripts
o : initial value.
f : feed.
p : permeate.
r : retentate.
Greek letters
σ : electrical conductivity (mS/cm).

1. Introduction

Industrial wastewater pollution is a serious problem especially in the Mediterranean region where water resources become more and more scarce. Phenolic derivatives are a class of common pollutants of many industries. These organics are phytotoxic and bactericide which consequently resist biodegradation. In the Mediterranean region, more than 30 million tons of phenol loaded agro-food wastewaters (olive mill wastewater, table olive wastewaters, winery and distillery wastewaters, etc.) are produced yearly [1].

Various techniques were tested for the removal of phenols and their derivatives from wastewater, e.g., Photooxidation [2], different advance oxidation processes [3], ozonation [4], nanofiltration [5] and adsorption on: low cost adsorbents [6], different bentonites [7], different types of activated carbon [8], and on other materials [9,10]. Due to the low biodegradability of phenolics, conventional biological wastewater treatment processes are generally found not efficient in treating the phenolic-containing wastewaters [11].

Membrane processes have also been investigated for removing the phenolics from wastewaters. Different kinds of membranes have been used: liquid membranes [12], anion exchange membranes [13], nanofiltration/reverse osmosis membranes [14] and pervaporation membranes [15]. Unfortunately, those processes often show low rejection levels for many small organic molecules such as monocyclic phenolic compounds and are generally energy intensive. As a solution hybrid processes have been developed that combine pressure driven membrane and adsorption processes [16-18] for the separation and/or removal of phenols. Because of their low molecular weights, simple ultrafiltration is ineffective for retaining these compounds. Micellar Enhanced Ultrafiltration (MEUF) could be considered for the retention of these pollutants. In such a process, a surfactant is added into the aqueous stream containing organic matters. When the surfactant concentration rises above the critical micellar concentration (CMC), surfactant monomers assemble and aggregate to form micelles. These macromolecular structures can solubilize organic matters into their hydrophobic core or adsorb on its surface. The aqueous stream is then filtered by an appropriate ultrafiltration membrane with pore sizes smaller than the micelle size. The micelles along with the solubilized organic matters are then rejected into the retentate stream. The MEUF process has been recently reported to separate different organic compounds: *a*-Phenylglycine [19], lactic acid [20], tannic acid [21], aromatic alcohols [22], methylene blue [23], and phenols [24]. As far as the surfactants used can generate some pollution and/or are costly, recycling will be needed prior to final disposal. Different methods were proposed for such a purpose. As an example, foam fractionation was reported as an efficient method for the SDS recovery after a of micellar-enhanced ultrafiltration process [25].

In this study we investigated the possibility to remove some phenolic compounds from model solutions using an anionic surfactant and PES membranes with different molecular weight cut-off. The effects of surfactant and phenolics concentration on rejection and permeate flux were studied. The removal and recovery of the surfactant by precipitation from the retentate stream in the presence of p-coumaric acid (PCA) were also studied.

2. Materials and methods

2.1. Chemicals

Sodium dodecyl sulfate (SDS) was obtained from VWR International (Belgium). 4-Hydroxy-3-methoxy benzoic acid (vanillic acid), trans-4-Hydroxycinnamic acid (p-coumaric acid) and 4-Hydroxyphenylethanol (tyrosol) were supplied by Sigma-Aldrich (Germany). KCl, $CaCl_2$ and isopropyl alcohol were purchased from Merck (Germany). Deionized water produced by a DI4000 (TKA, Germany) system was used in all experiments. The membranes were supplied gratefully by GEA-Wiegand, G.m.b.H (Etlingen, Germany).

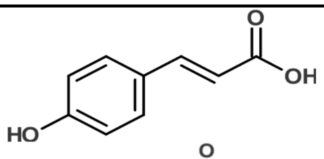
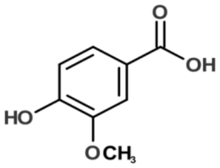
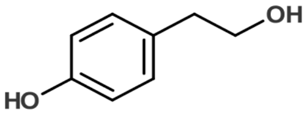
2.2. Model solutions

Phenolics were solubilized in deionized water. Solutions were stirred overnight before adding the SDS at predetermined concentrations. The CMC of SDS was measured at room temperature (25°C ± 2°C) in deionized water using the electrical conductivity method and it was found to be equal to 9.6 mM, which is similar to the value (9.7 mM) reported by Rosen [26]. The pH of the model solutions was adjusted to 2 with a solution of HCl (2N) using a pH-meter (WTW, model pH325). Some physicochemical characteristics of the three studied phenolic compounds are shown in the Table 1.

2.3. Nanofiltration experiments

In nanofiltration experiments, the transmembrane pressure was monitored with pressurized nitrogen gas using a pressure gauge. The experimental set-up is illustrated in Fig. 1. A PES membrane (MicrodynNadir, Germany) with a nominal retention of Na_2SO_4 higher than 30% (NP030) was tested for phenolic compounds removal under a transmembrane pressure of 30 bars. The operating conditions (temperature, feed volume, volume concentration factor and stirring rate) and the experimental set-up were the same as for the MEUF experiments. The rejection coefficient was estimated using the Eq. (2).

Table 1: Physicochemical characteristics of the tree studied phenolic compounds.

Phenolic Compounds	Chemical structure	MW ($g.mol^{-1}$)	Purity (%)	Solubility ($g.l^{-1}$)	Peak of absorbance (nm)
p. coumaric acid		164.16	≥ 98	0.74 [27]	285
Vanillic acid		168.15	≥ 97	1.50 [28]	288
Tyrosol		138.16	≥ 97	124 [29]	275

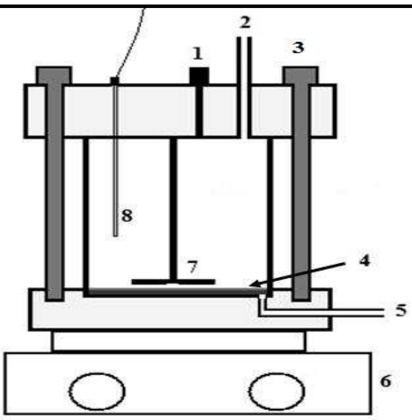


Figure 1: Scheme of the nanofiltration and ultrafiltration experimental set-up: 1. Feed inlet, 2. Connection to the nitrogen cylinder, 3. Screw, 4. Membrane, 5. Permeate outlet, 6. Magnetic stirrer, 7. Magnetic bar, 8. Temperature sensor.

2.4. Ultrafiltration experiments

Experiments were conducted under ambient temperature (25°±2°C) and under a transmembrane pressure of 3 bar. The feed volume was 200 ml and the cell was stirred at 200 rpm using a magnetic stirrer. Two polyethersulfone (PES) membranes (MicrodynNadir, Germany) with different molecular weight cut-off (50 kDa: UH050 and 20 kDa: UP020, data sheet: http://www.microdynnadir.de/cms/pdf/produkte/en/1_katalog_engl_rz_screen.pdf) were used. The effective surface area of the membrane was 50 cm². Permeate fluxes were determined continuously using a graduated cylinder connected to the permeate outlet.

Before and after each run, deionized water was filtered to determine and compare the permeability of the membrane. MEUF experiments were stopped when the volume concentration factor (VCF) reached a value of 2:

$$VCF = \frac{v_o}{v_r} \qquad (1)$$

where v_o is the initial volume and v_r is the volume of the retentate. Phenols rejection was determined by analyzing the permeate and the retentate spectrophotometrically.

The SDS molecules do not absorb at any of the used wavelengths (285 nm, 288nm, 275nm) and the rejection coefficient was calculated as follow:

$$R(\%)=\left(1-\frac{OD_p}{OD_r}\right)\times 100 \tag{2}$$

where OD_p stands for the optical density of the permeate and the OD_r is the optical density of the retentate at the wavelength corresponding to the peak of absorbance of the studied phenolic compound (Table 1). The relative flux reduction (RFR) was calculated from the difference between the permeate fluxes measured at t=0 (J_o) and at the end of the experiment (J_f). The RFR was then calculated as follow:

$$RFR(\%)=\left(1-\frac{J_f}{J_o}\right)\times 100 \tag{3}$$

After removing the feed solution, the membrane surface was externally rinsed twice by filling the cell with pure water (200 ml) and leaving it under high stirring rate (800 rpm) for up to 10 min. The membrane was reused as the water flux reduction after use of membrane did not exceed 5%.

2.5. Surfactant precipitation and recovery

The electrical conductivity (σ) measurements were used to follow the precipitation reactions of the SDS on different systems (SDS/KCl/PCA and SDS/$CaCl_2$/PCA). As precipitation occurs, ions are removed from the solution, the conductivity decreased consequently. An SDS solution (20mM) containing 1.2 mM of PCA was placed in a flask kept at 20°C. The solution was stirred and nitrogen was blown into the flask to reduce the absorption of carbon dioxide into the solution [30]. A salt solution of 8 mM and 16 mM containing 1.2 mM of PCA was added to the SDS solution under stirring rate of 200 rpm. A WTW (Germany) conductivity-meter model 3L5i was used to follow the conductivity of the solution as a function of time during the precipitation reaction. The precipitation efficiency with each cation was compared and the eventual loss of the PCA after the SDS precipitation was evaluated by comparing its absorbance at 285 nm before and after precipitation.

3. Results and discussion

3.1. Nanofiltration

Nanofiltration on the NP030 membrane exhibits rejections up to 14% and 40% for tyrosol and vanillic acid, respectively (Table 2). The permeate fluxes decreased considerably when using phenolic solutions compared to the deionized water flux. The observed retentions of vanillic acid (30% to 40%) are higher than those of tyrosol (9% to 13%). Conversely, the permeate fluxes of the nanofiltration of tyrosol solutions were relatively higher. This may partly originates from the difference of molecular weight between the two phenolics. The vanillic acid (168 Da) is slightly bigger than tyrosol (138 Da). Furthermore, this difference in rejection rates can be due in part to the charge exclusion effect since the vanillic acid has a carboxyl group with a negative charge, so it may be rejected more efficiently by the PES membranes (Table 2).

Table 2: Permeate fluxes and rejection of phenolic compounds by nanofiltration using a PES membrane (NP030).

Phenolic compound (g/l)	Rejection (%)	Flux (L/h/m²)
Tyrosol		
0	--	116
0.25	8.92	25
0.5	10.24	18
0.75	13.56	13
Vanillic acid		
0	--	116
0.25	40.06	18
0.5	29.89	9
0.75	33.43	7

3.2. MEUF processing of phenolic solutions: Permeate fluxes and membrane fouling

The UF permeate flux of the deionized water when using the UH050 membrane was about 2500 l/h.m². It is evident from the Fig. 2 that the permeate fluxes were higher in absence of surfactant. The addition of the SDS above the CMC leads to the formation of large aggregates which may deposit on the membrane surface. The membrane resistance to solvent transfer consequently increased leading to a decrease of the permeate flux. As can be observed from Fig. 2, the fluxes drastically decreased for the solutions containing the SDS. Reductions up to 97% were observed at the highest concentrations of the SDS and the phenolic compounds (Fig. 2). When using the membrane UP020 the decrease of the permeate fluxes (77%-79%) was not as important as that shown by UH050 (Table 3). However, high fluxes were recovered after membrane cleaning inside the UF module by deionized water at high stirring rate (≥800 rpm) for 10 min.

The high water flux recovered after external cleaning of the membrane with water (rinsing) indicates that reversible fouling, caused by concentration polarization, contribute greatly to the overall flux decline. The relative flux reduction (RFR) was estimated to 23% and 63% for UP020 and UH050, respectively. However, longer operating times significantly affect the recovered fluxes revealing that irreversible fouling takes place. The difference of RFR may originates from the nature of the membrane material and also the larger pores of the UH050 membrane may facilitate the penetration of the small micelles into the pores and the adsorption on the walls leading to a narrowing and to greater RFR. Susanto *et al.* [31] reported significant water flux reductions and changes in polyethersulfone membranes surface properties after static adsorption of phenolic compounds.

Table 3: Rejections (R) and initial permeate fluxes (J_o) of the MEUF of phenolic compounds at different concentrations using membrane UP020 and 2 CMC of the SDS.

	p-Coumaric acid		Vanillic acid		Tyrosol	
Concentration	R (%)	Jo (L/h.m²)	R (%)	Jo (L/h.m²)	R (%)	Jo (L/h.m²)
0	0	600	0	600	0	600
0.1	83	129	32	125	26	127
0.2	63	131	43	134	27	126
0.3	61	129	39	130	22	129

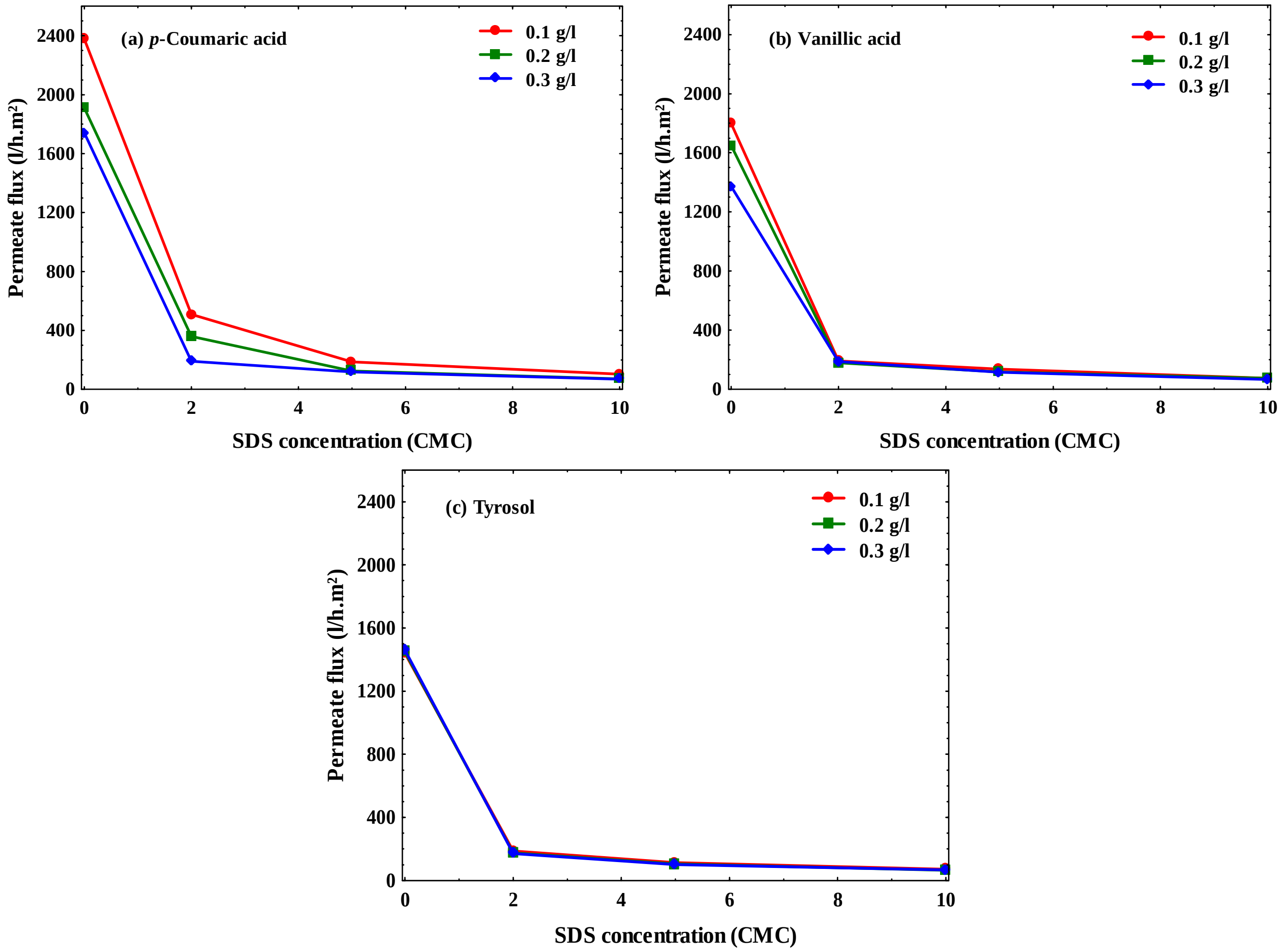


Figure 2: Permeate fluxes against surfactant concentration for the MEUF of different model solutions using UH050 membrane: (a) *p*-coumaric acid, (b) vanillic acid and (c) tyrosol.

3.3. Removal of phenolic compounds

A rejection up to 15% of phenolics was observed in absence of any surfactant (Figs. 3a, 3b and 3c). Most likely some adsorptions of phenolic compounds occur on the membrane material. When the surfactant is used, the retentions of phenolics are increased significantly, depending on the surfactant concentration, to the range of 16%-67%, for the *p*-coumaric acid, 29%-66% for the vanillic acid and 8%-51% for the tyrosol, respectively. This clearly evidences that the solutes are somehow linked to the surfactant micelles, which are subsequently retained by the ultrafiltration membrane. It may also be observed from the Figures 3a, 3b and 3c that the rise of the surfactant concentration from 5 to 10 CMC increases the rejection of different phenolic compounds from 15% to 30%. When 2 CMC of the SDS is used, the retention did not exceed 33%, 30% and 22% for the *p*-coumaric acid, vanillic acid and tyrosol, respectively. However, the decrease of the MWCO of the membrane to 20 kDa improves considerably the rejection at a low concentration of the surfactant (2 CMC) (Table 3). The observed retentions were in the range of 61%-83%, 32%-43% and 22%-26% for *p*-coumaric acid, vanillic acid and tyrosol, respectively. Therefore, the SDS micelles are retained more efficiently when using a membrane with lower MWCO. In aqueous solutions not including electrolytes, the reported micelle aggregation number for SDS is 64 [32,33]. This gives the micelles an equivalent molecular weight of about 18 kDa. When increasing the concentration of the surfactant, the micelles can aggregate and form bigger macrostructures that can solubilize more solutes. Table 3 shows also that the *p*-coumaric acid (PCA) is the most rejected phenolic compound by MEUF using the SDS, it must be the most interacting or/and solubilized in SDS micelles.

PCA exhibits the highest rejection coefficient followed by VA and TY (Table 3, Figs. 3a, 3b and 3c). This finding indicates clearly that the three phenolics are solubilized into the SDS micelles but with different partition coefficients between the water phase and micelles pseudo-phase most likely in respect to the hydrophobicity degree of these phenolics. The hydrophobicity of a chemical can be estimated by the octanol-water partition coefficient (K_{ow}) expressed in log units. The log K_{ow} of PCA, VA and TY are 1.6 [34], 1.42 and 0.69 [35], respectively. From these values we can notice that the rejection coefficients of each phenolic compound are correlated positively to their octanol-water partition coefficients (K_{ow}).

The ionic and structural characteristic of the solutes plays a key rule on their solubilization in the micelles. On the other hand, *p*-coumaric acid and vanillic acid are less hydrophilic compounds than tyrosol since their respective solubility in water did not exceed 0.72 and 1.5 g/l (Table 3). Thus, the *p*-coumaric and vanillic acids are readily solubilized in the hydrophobic core of the micelles and shows higher observed retention.

3.4. Surfactant recovery from the retentate stream

The precipitation of surfactant molecules above CMC was approached by measuring the variation in the electrical conductivity (σ) of the solution against time. When ions form complexes or are incorporated into crystal lattices, the conductivity of the solution must decrease. In the system SDS/KCl/PCA, The potassium cations bind to dodecyl sulfate anion (Appendix A) to form the potassium dodecyl sulfate (KDS) which precipitate and consequently the electrical conductivity decreases (Fig. 4). To test the precipitation of the SDS at low relative concentration of salt, the precipitation was conducted at a mole ratio of K^+ to SDS equal to 0.25 and the decrease of the σ did not exceed 5% of the initial conductivity, σ_o (Fig. 4).

Wu *et al.,* [36] reported that the KDS have similar CMC, water solubility, and ability to solubilize organic solutes in micellar solutions as the SDS. KDS was proposed as a potential surfactant for use in MEUF, with recovery of the surfactant to be accomplished by lowering the system temperature below the Krafft point. KDS has a significantly higher Krafft point than SDS (35°C) [36]. Consequently, it precipitates at ambient temperature. The Krafft point of the SDS is 16°C [37]. The divalent cation (Ca^{2+}) used at a mole ratio to SDS equal to 0.25 leads to a more significant decrease of the conductivity down to 18% of the initial value (Fig. 4). Such a recovery process is rapid, clean and requires only small amounts of the electrolyte. The precipitation was very rapid and occurred during less than 2 minute (Fig. 4 and 5). The induction time which is the time needed before the decrease of conductivity started (precipitation) was about 0.75 min. The precipitation of dodecyl sulfate was more rapid and effective at higher Ca^{2+}/SDS mole ratio (Fig. 5). Furthermore, the increase of the Ca^{2+}/SDS mole ratio decreases the induction time from 0.75min to less than 0.08 min.

When using the precipitation process to remove and recover SDS at large scale, an optimal mole ratio should be determined to assure an efficient precipitation without creating a secondary pollution problem related to the excess of salt. The optimum as shown by the Fig. 5 could be around a mole ratio of 0.5, since the increase of the Ca^{2+}/SDS mole ratio above this value leads to the decrease of the SDS precipitation rate. This finding can be attributed to the fact that the reaction stoichiometry is shown to be 1:2 (Ca^{2+}:DS)(Appendix B). The reaction of sodium dodecyl sulfate with calcium chloride produces the calcium dodecyl sulfate ($Ca(DS)_2$, CDS) which precipitates immediately.

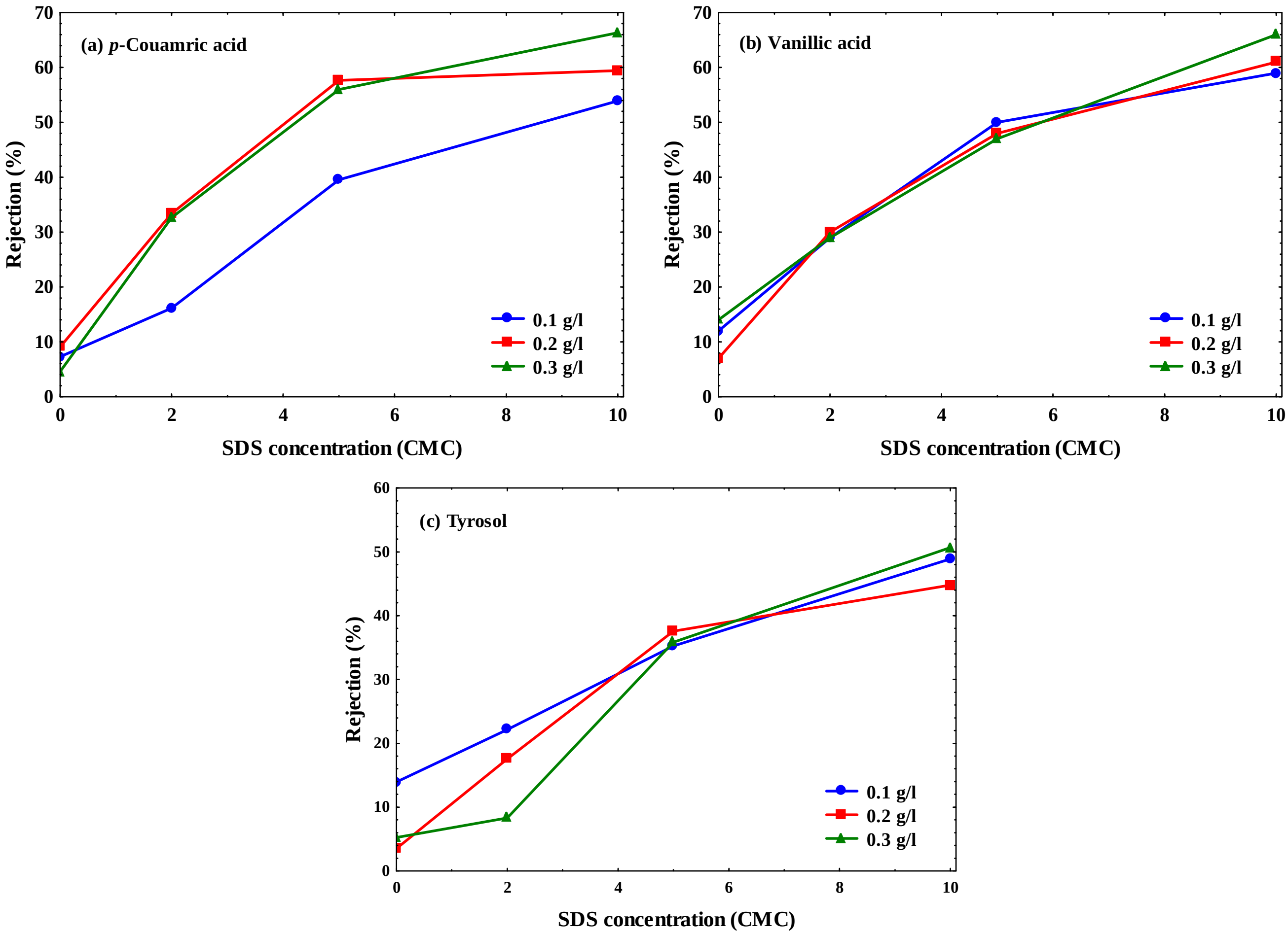


Figure 3: Rejection of phenolic compounds by MEUF against surfactant concentration for different model solutions using UF050 membrane: (a) *p*-coumaric acid, (b) vanillic acid and (c) tyrosol.

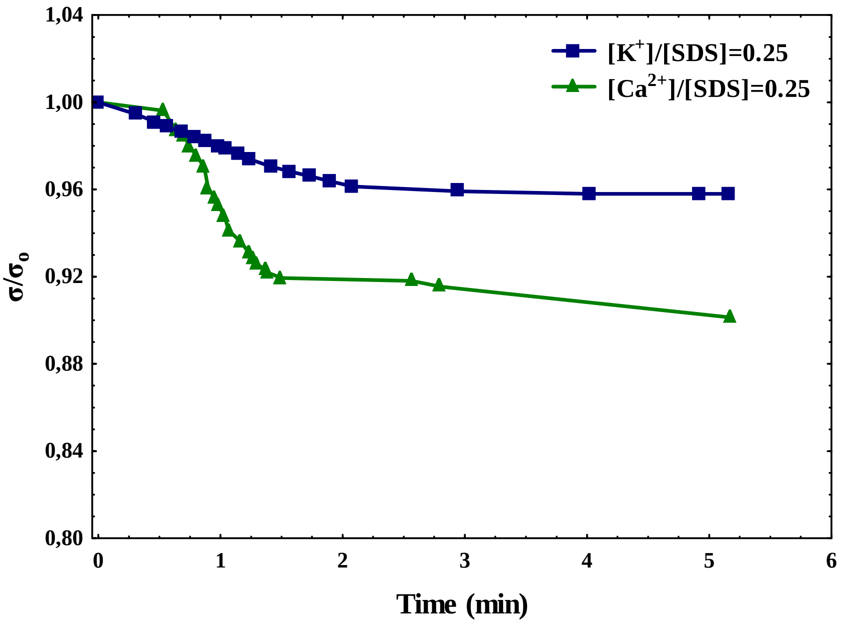


Figure 4: Normalized electrical conductivity time course during the precipitation of the SDS at 30°C using KCl and $CaCl_2$ in presence of 1.2 mM of *p*-coumaric acid.

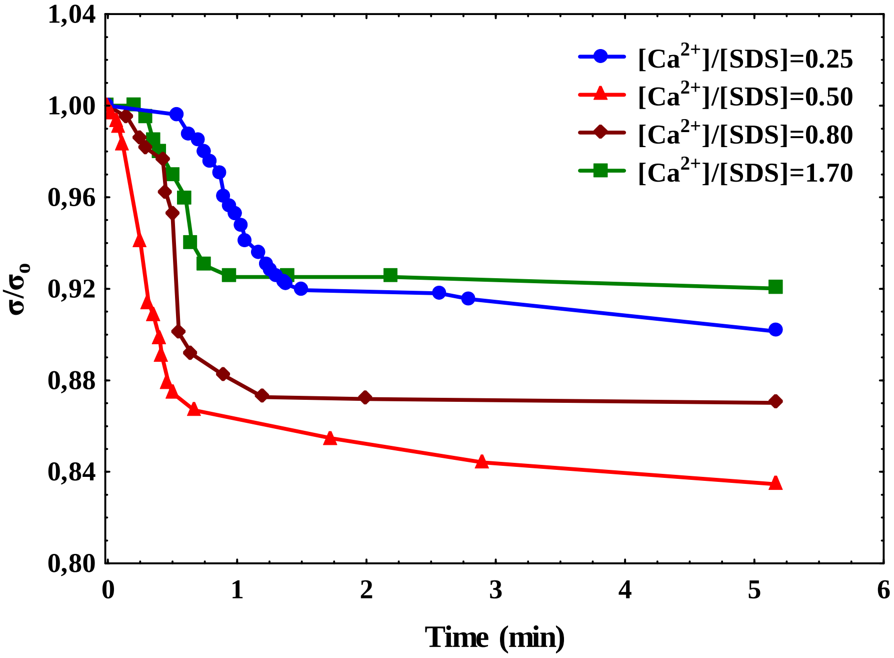


Figure 5: Normalized electrical conductivity time course during the precipitation of the SDS at 30°C using $CaCl_2$ at different mole ratios in presence of 1.2 mM of *p*-coumaric acid.

Micelles act as sequestering agents for the calcium ions. As more Ca^{2+} is added to the system, the additional cations tend to form more $Ca(DS)_2$, reducing the dissolved SDS in the solution. Although, the increase of Ca^{2+}/SDS mole ratio beyond the value of 0.5 leads to a decrease of precipitation rate as followed by electrical conductivity measurements (Fig. 5). The mole ratio between surfactant and cation for the formation of precipitation results in different degrees of charge neutrality of the precipitate. The degree of charge neutrality of the precipitate shows different solubility. This is clearly shown by the decreasing yield of the precipitate with increasing mole ratio of Ca^{2+} to SDS to from 0.5 to 1.7. This same phenomenon was observed when precipitating SDS with a cationic surfactant (CPC) [38].

However, CDS has a low solubility in solutions and cannot be directly recycled to the process. It must be transformed to the monovalent salt (e.g. to SDS by adding Na_2CO_3) before it is re-dissolved in water for recycle [39]. The crystals can be separated by a simple filtration and dried at high temperature or by silica gel at room temperature overnight. The dried CDS can be then used in the MEUF process efficiently but at high temperature since its Krafft temperature is 50°C [40]. The CMC of CDS is approximately at 2.4 mM [29].

Conclusion

Micellar Enhanced Ultrafiltration was more effective to remove phenolic compounds from model solution than nanofiltration. It was found that PES membranes adsorb phenolic compounds. Consequently, the effect of pore size on adsorptive fouling was expected to be very important. The rejection reached satisfactory levels (80%) under some conditions but the use of the SDS involves a considerable decrease in the permeate fluxes.

A precipitation process was proposed to recover the surfactant from the retentate stream. Two systems were tested, SDS/$CaCl_2$/PCA and SDS/KCl/PCA. No substantial loss of *p*-coumaric acid occurs during the precipitation since no significant change in the absorbance at the wave length 285 nm before and after precipitation was observed for both systems. The elimination and recuperation of the surfactant from the retentate will allow the recovery of pure phenolics more efficiently. Further investigations are needed to enhance the rejection with small amount of surfactant and to increase the permeate fluxes. Furthermore, the minimization of the adsorption on the membrane surface can limit the adsorptive fouling.

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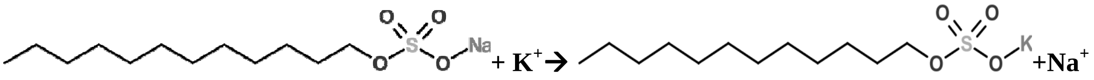
Appendix A

Reactions occurring though the dodecyl sulfate anion precipitation using KCl:

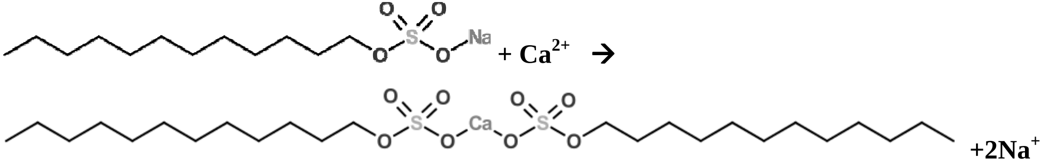
$KCl \rightarrow K^{+} + Cl^{-}$



Appendix B

Reactions occurring though the dodecyl sulfate anion precipitation using $CaCl_2$:

$CaCl_2 \rightarrow Ca^{2+} + 2Cl^{-}$



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