



## Simultaneous removal of aniline and nickel from water by micellar-enhanced ultrafiltration with different molecular weight cut-off membranes



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### ABSTRACT

The aim of this study was to evaluate the performance of the micellar-enhanced ultrafiltration (MEUF) process for the simultaneous removal of aniline and nickel from the aqueous phase. A prepared polysulfone (PSf) membrane and two commercially available membranes (NP010 and UFX5) were used to investigate the effects of the molecular weight cut-off of the membranes on the performance of MEUF which highlighted the significance of the mean micelle size in this process. Increasing of sodium dodecyl sulfate (SDS) concentration and the addition of aniline and nickel into the solution would change the SDS micelle size. The best rejection of nickel and aniline, 97% and 70%, respectively, was achieved with NP010 membrane which has the smallest pore size (1 kDa). The results demonstrated that the rejection of nickel and aniline are influenced by the co-presence of them in solution. The presence of nickel ions in the solution increased the aniline rejection. Moreover, the presence of aniline molecules enhanced the nickel rejection. In the mixed surfactant solution (SDS-Brij 35) for the prepared PSf membrane, nickel rejection and permeate flux achieved over 90% and 100 kg/m<sup>2</sup> h, respectively at a pressure of 2.5 bar.

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### 1. Introduction

A common problem encountered in most industries is the disposal of large volumes of wastewater containing both inorganic (such as heavy metals) and organic (such as phenols and aniline) contaminants which are hazardous to humans and also to the environment [1]. Therefore, wastewaters containing these compounds need to be treated before discharge into the environment.

Conventional separation processes such as distillation, extraction, adsorption and precipitation are not very attractive for this purpose because of high energy consumption and sometimes difficult regeneration or because some of the processes produce waste that must be treated appropriately [2]. Membrane separation processes, especially microfiltration, reverse osmosis, ultrafiltration and recently nanofiltration have been used for the treatment of these compounds. Recently, integrated more cost-efficient processes, such as micellar-enhanced ultrafiltration (MEUF), have received increasing attention [3–6].

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The MEUF has been applied to the separation of dissolved low molecular weight substances using surfactants at their critical micelle concentration (CMC). Above the CMC, surfactant monomers begin to assemble and form micelles whose size or diameter is larger than the membrane pore size. One of the most important results of micellization is that micelles can solubilize a certain amount of organic compounds as a solubilize and adsorb metal ions on their own surface. Organic materials, depending on their chemical structure, may be dissolved at different locations within micelles, such as the hydrophobic core, palisade layer, and micelle surface [7]. They tend to situate in regions within the micelle that are similar chemically and in polarity to these molecules [7]. The micelles, according to their surface charge, can bind metal ions with an opposite charge on their surface.

Most studies on MEUF have concentrated on the removal of heavy metals or organic matters in the single-pollutant systems [4,8–12] and some researchers have reported the elimination of these contaminants in multi-pollutant systems [6,13,14]. In multi-pollutant systems, competition may take place due to differences in the binding power of contaminants, and it prevents the binding of pollutants with weaker binding power. The effective

removal of two contaminants by MEUF has also been reported feasible when the contaminants co-exist in wastewater.

Wastewaters from industrial operations such as coal refining, textiles, dyes and synfuel processing contain both dissolved organics and heavy metals [15]. In this study, aniline as a non-ionic organic contamination and nickel as a divalent metal ion were selected. Nickel and aniline are toxic carcinogen chemicals, and both are present in dyeing industry wastewaters. If these wastewaters were discharged to the environment, they would generate a serious risk for humans and animals. As the global dyeing industry is extensive, the removal of these contaminants from water is important and essential [16]. No reports about the simultaneous removal of aniline and nickel by MEUF have been observed.

The simultaneous removal of organic and metal ions such as  $\text{Cr}^{3+}$  and phenols [17], chromate and chlorinated aromatic hydrocarbons, nitrate [18],  $\text{Cu}^{2+}$  and phenol [19], uranyl ions, dissolved DBP and TBP [3],  $\text{Cd}^{2+}$  and methylene blue [5], and  $\text{Cd}^{2+}$  and phenol [15] with MEUF were reported by several authors. Their studies focused on high removal of dissolved pollutants by MEUF and the measurements of residual surfactants in permeate. These studies, however, did not investigate efficiency of pollutants removal by membranes with different molecular weight cut-off and changing of sodium dodecyl sulfate (SDS) micelle sizes as an important parameter with increasing the concentration of SDS and pollutants in the solution.

The objectives of this study were: (1) to investigate the effects of membrane pore sizes on the separation efficiency of aniline and nickel and the permeate flux in the MEUF process, (2) to investigate the change of SDS micelle sizes with increasing of SDS and pollutants concentration in the solution and its effect on separation performance, and (3) to study the effect of mixed non-ionic and anionic surfactants on the removal of nickel and aniline.

## 2. Experimental

### 2.1. Surfactants and membranes

SDS ( $\geq 99.0\%$ ) and polyethylene glycol lauryl ether (Brij 35) as anionic and non-ionic surfactants were purchased from Sigma Aldrich.  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$  and aniline were supplied by J.T. Baker and the Merck Group, respectively. For the solution preparation, deionized (DI) water with a conductivity of approximately  $0.8 \mu\text{S}/\text{cm}$  was used. The commercially available membranes used in this work were polysulfone (trade name: UFX5-pHt) and polyethersulfone (trade name: NP010). Their characterizations are shown in Table 1. Udel P-1700 polysulfone (polymer) and NMP (n-methyl-2-pyrrolidone as solvent) were used to prepare the casting solution in the case of PSf membrane preparation by the phase inversion method. The NMP solvent was purchased from Sigma–Aldrich.

### 2.2. Chemical analyses

The concentration of nickel was measured by a thermo scientific iCE 3000 series Atomic Adsorption Spectrometer (AAS) from China. The concentration of aniline was measured by UV absorp-

tion at a wavelength of 280 nm with an UV–Vis spectrophotometer JASCO V-670 manufactured in Japan. The SDS concentration was determined by titrating the SDS solution with a cationic solution (poly DADMAC, 0.001 N) in a Mütek Particle Charge Detector (PCD 02, Germany).

### 2.3. Structural analyses of membranes

The structure of the prepared membrane was characterized by a scanning electron microscope (SEM). Cross-sections of prepared membrane samples were scanned using JEOL (JSM-5800) at 10 kV. The prepared samples were coated with a very thin layer of gold in a vacuum. After sputtering these samples with gold, they were moved into the microscope chamber.

### 2.4. Micelle size measurement

The mean micelle sizes were measured using a Zeta Sizer Nano ZS apparatus (Malvern Instruments Ltd.) as a laser particle sizer. The instrument allowed measuring the particle size, taking advantage of optoelectronic systems.

### 2.5. Permeation and rejection properties of membranes

The permeate flux of the ultrafiltration membrane was defined as:

$$J_i = \frac{Q_i}{A} \quad (1)$$

where  $J_i$  is the permeate flux ( $\text{kg}/(\text{m}^2 \text{h})$ ),  $Q_i$  is the mass rate ( $\text{kg}/\text{h}$ ) and  $A$  is the effective area of the membrane ( $\text{m}^2$ ). Furthermore, the volume concentration factor (VCF) was defined as:

$$\text{VCF} = \frac{V_{ini}}{V_{fin}} \quad (2)$$

where  $V_{ini}$  and  $V_{fin}$  are the initial and final volumes of the solution in the batch stirred cell, respectively. The rejection of nickel and aniline ( $R\%$ ) was defined as:

$$R\% = \left(1 - \frac{2C_p}{C_{F1} + C_{F2}}\right) \times 100 \quad (3)$$

where  $C_p$  is the solute concentration in the permeate,  $C_{F1}$  is the solute concentration in the feed solution just before the experiment begins (i.e.  $t = 0$ ), and  $C_{F2}$  is the solute concentration in the feed solution at the end of the experiment.

### 2.6. Ultrafiltration procedure

Experiments were performed at  $25^\circ\text{C}$  using an Amicon stirred cell type filter (model 8400) with a feed volume of 300 mL. The membrane samples used in this work had an effective surface area of  $40 \text{ cm}^2$ . Before each experiment, the membrane samples were kept in deionized water for 2 h. The pre-treated membrane was placed into the cell and compacted at 540 kPa for approximately 1 h using deionized water.

**Table 1**  
Characterizations of the used commercially available membranes.

Membrane code	Manufacturer	Membrane property	MWCO <sup>a</sup> (kDa)	M.O.P. <sup>b</sup> (bar)	M.O.T <sup>c</sup> ( $^\circ\text{C}$ )
NP010	Microdyn <sup>®</sup> Nadir	Hydrophilic	1	40	95
UFX5-pHt	Alfa Laval	Hydrophilic	5	15	75

<sup>a</sup> Molecular weight cut-off.

<sup>b</sup> Maximum operation pressure.

<sup>c</sup> Maximum operation temperature.

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