



## Removal of metal ions at low concentration by micellar-enhanced ultrafiltration (MEUF) using sodium dodecyl sulfate (SDS) and linear alkylbenzene sulfonate (LAS)

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### ABSTRACT

Micellar-enhanced ultrafiltration (MEUF) has been used to remove dissolved metals including Cd<sup>2+</sup>, Cu<sup>2+</sup>, Ni<sup>2+</sup>, Pb<sup>2+</sup>, and Zn<sup>2+</sup> from synthetic water using two anionic surfactants: sodium dodecyl sulfate (SDS), and linear alkylbenzene sulfonate (LAS) in a lab-scale membrane system. The effect of surfactant concentration, pH and conductivity on metal retention was studied. The molar concentration ratio of the surfactant to metal (*S/M* ratio) is higher than 5 in all experiments. When the initial SDS concentration was below the critical micelle concentration (CMC), metal retention higher than 90% was unexpectedly obtained (at conditions used in these experiments), except for Ni<sup>2+</sup>. Moreover, it was shown that complete removal of metal ions, except for Ni<sup>2+</sup>, could be achieved at an LAS concentration below CMC. The addition of NaCl provokes the complexation of metal cations with chloride ions and the adsorption competition of sodium cations with metal ions, therefore increasing conductivity decreases metal retention. The presence of the NaCl in the feed surfactant solution had a drastic adverse effect on metal retention by SDS and LAS.

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### 1. Introduction

The direct discharge of toxic organic compounds and metallic ions (non-biodegradable, highly toxic, and potentially carcinogenic) into sewage systems may seriously damage the operation of biological treatment plants [1,2]. Consequently, the removal of metal ions and/or organic contaminants from aqueous solutions is a problem frequently encountered in the treatment of industrial wastewaters.

The stringent environmental and ecological requirements have spurred the search for industrial waste treatment options with low energy, labour, and capital costs [3], but the traditional techniques for the removal of metal ions from aqueous effluents are incapable of reducing concentration to the levels required by law (process of reduction or lime precipitation) or prohibitively expensive (process of ion exchange, activated carbon adsorption, electrolytic removal).

The use of a membrane separation process to treat wastewater containing toxic metal ions is today an attractive and suitable technique, but microfiltration (MF) or traditional ultrafiltration (UF) are usually limited to the separation of molecules with high molecular weights and are not sufficient to retain all the contaminants [4]. In order to remove metallic ions, reverse osmosis (RO) (or at least

nanofiltration (NF)) can be used due to the size of the ions in aqueous solutions. Although, one of the disadvantages of RO membrane separation is that permeate flux is limited due to the dense polymeric membranes used [5], as a result the capital and operating costs will be increased significantly [6].

Since the pioneer work of Leung [7], micellar-enhanced ultrafiltration (MEUF) has been shown to be an effective separation technique to remove metal ions and/or soluble organic solutes from aqueous environments using several attractive forces such as electrostatic or hydrophobic interactions [2,3,8–11]. It combines the high selectivity of RO and the high flux of UF [12]. The advantages of this method are the low-energy requirements involved in UF processes [13] and its high removal efficiency owing to the effective trapping of solutes by the micelles [10]. This separation technique is based on the addition of surfactants to the solution to be filtered. When surfactants are present in aqueous solutions above a certain concentration, i.e., the critical micelle concentration (CMC), aggregates are formed that may entrap a solute existing in the solution. The increased hydrodynamic size of the solutes enables their rejection by polymeric UF membranes. The micellar solution is then passed through a UF membrane having pore sizes small enough to reject the micelles containing the attracted metal ions or solubilized organic solutes. The permeate side will consequently contain very low concentrations of unbound metal ions, organic molecules will not be trapped in micelles and surfactant monomers (Fig. 1), resulting in a clean permeate which can be recycled or discarded.

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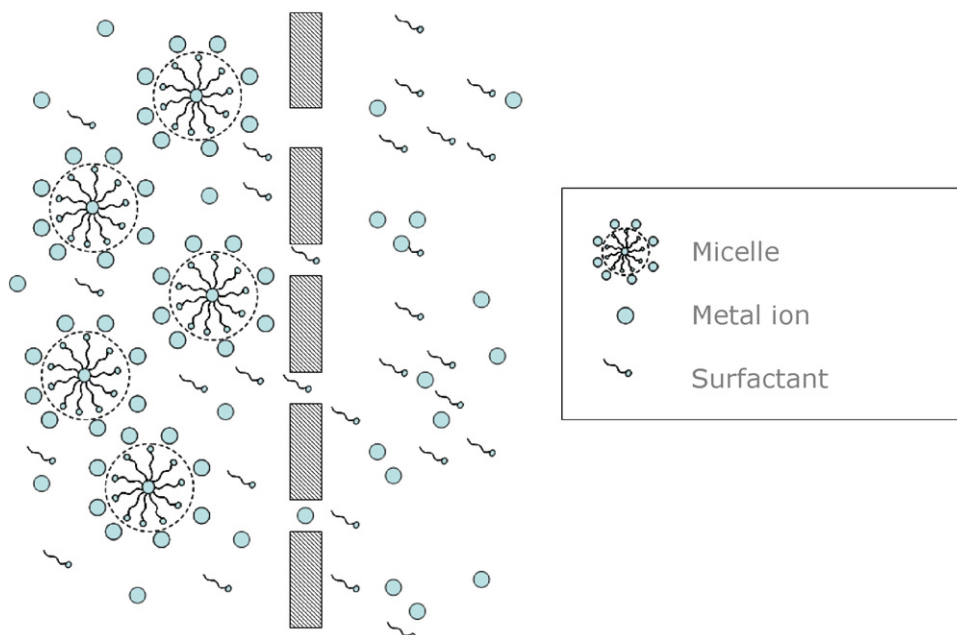


Fig. 1. A schematic diagram illustrating micellar-enhanced ultrafiltration for the removal of metal ions.

Metal removal efficiency by MEUF depends on the characteristics and concentrations of the metals and surfactants, solution pH, ionic strength, and parameters related to membrane operation (such as transmembrane pressure, filtration flow rate, membrane pore size, etc.). Operating pressure was found to play a dual role in the separation of the solute. An increase in pressure resulted in the enhancement of solubilization capacity of the micelles. On the other hand, the permeate concentration of the surfactant also increased with increased pressure [14]. Huang et al. [15] reported that two important parameters (surfactant concentration and surfactant to metal molar ratio ( $S/M$  ratio)) controlled the removal efficiency of metals. To reach the maximum metal removal efficiency (>99% for most of metals investigated), the surfactant concentration has to be higher than the CMC of the surfactant [3]. A high ion concentration or the addition of a buffer may affect the removal of metal ions. In addition, dissolved electrolyte in an aqueous surfactant solution causes a decrease in the CMC of the solution; this depression is mainly due to the decrease of the electrostatic repulsion between the ionic head groups on the micelles [9].

Metal removal combining SDS and UF membranes has been extensively studied [2,6,8–10,15–17] but LAS surfactant has been rarely used [18] for metal removal in MEUF processes. This surfactant has a lower CMC than SDS, therefore it results in a smaller monomeric surfactant concentration in the permeate. Furthermore, a surfactant with a lower CMC would cause a higher micellar concentration.

The aim of this study is to discuss and compare the retention characteristics of single metal ions including  $Cd^{2+}$ ,  $Cu^{2+}$ ,  $Ni^{2+}$ ,  $Pb^{2+}$ , and  $Zn^{2+}$  in the presence of anionic surfactants: sodium dodecyl sulfate (SDS) and linear alkylbenzene sulfonate (LAS) by UF. A metal concentration of  $10\text{ mg l}^{-1}$  was selected. The influences of solution pH, and conductivity on metal retention were studied.

## 2. Experimental

The two surfactants used were sodium dodecyl sulfate (SDS) (>99% pure, purchased from Sigma–Aldrich), and linear alkylbenzene sulfonate (LAS) (42.5% pure, obtained from PETRESA (Petroquímica Española S.A.)). Metal nitrates and other inorganic

chemicals: (HCl, NaOH and NaCl) were all supplied by PANREAC, at analytical reagent grade. The aqueous solutions were prepared by dissolving  $10\text{ mg l}^{-1}$  of a single metal and a given amount of SDS or LAS in deionised water. If necessary, the solution pH was adjusted by adding a small amount of 0.1N HCl or NaOH. The ionic strength was adjusted by adding 6M NaCl.

The UF experiments were carried out in a batch stirred cell (Amicon 8200, Millipore), and the aqueous mixture was stirred at a constant speed of 200 rpm. The transmembrane pressure was maintained at 100 kPa using high pressure nitrogen gas, and the temperature at  $22 \pm 2\text{ }^\circ\text{C}$  to avoid any precipitation because the Kraft points of the LAS and SDS are  $<1\text{ }^\circ\text{C}$  (data of supplier) and  $14\text{ }^\circ\text{C}$  [19], respectively. A schematic diagram of the experimental apparatus is shown in Fig. 2. The UF membranes used are manufactured by Millipore (Biomax polyethersulfone PBGC), which have a nominal molecular weight cut-off (MWCO) of 10 kDa and an effective filtration area of  $30.19\text{ cm}^2$ . The feed volume was  $200\text{ cm}^3$ . UF experiments were stopped when the permeate volume reached  $100\text{ cm}^3$ .

Firstly the membrane is compacted using deionised water at a pressure of 400 kPa for 8 h, after that the deionised water flow rate of the membrane was measured at 100 kPa. After UF experiments, the membranes were immediately flushed with deionised water and were used again only if the deviation of clean water flux at 100 kPa was less than 5% [16].

CMC of LAS and SDS were determined using the conductivity-meter method. Experimentally, the CMC is usually found by plotting a graph of electric conductivity versus of concentration. An abrupt change of slope marks the CMC. Its plot against the concentration of surfactant yields two straight lines from whose intersection the CMC is obtained. This procedure is perfectly applicable to single-component micelle systems [20].

Conductivity, pH and dissolved organic carbon (DOC) were measured with a Crison microCM 2200 conductivity meter, a Crison micropH 2000 pHmeter and a Shimadzu TOC 5000A analyser, respectively.

The permeate flux was measured continuously and gravimetrically using a Cobos CB Complet laboratory scale that was connected to and monitored via a computer. The cumulative weights are converted to cumulative volumes and from the slope of the cumulative

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