



Transport mechanisms of detergent ingredients through ultrafiltration membranes



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ABSTRACT

The extensive use of anionic surfactant sodium dodecyl sulfate (SDS) and the chelating agent ethylenediaminetetraacetic sodium salt (Na₄EDTA) in industrial detergent formulations suggests the need to study the possibility of their recovery and reuse from waste effluents in order to decrease the cost of the cleaning process and to avoid environmental drawbacks, as well as to save water. A lab-scale plant was provided with a 5 kDa ceramic membrane and used to study the permeation and retention of these compounds in single solutions as well as several mixtures. The presence of micelles in the concentration polarization layer plays an important role in the permeate flow rates and the retention of both compounds. Finally, the decrease in SDS and Na₄EDTA retention when NaOH is added is attributed to micelles and pre-micelles destabilization and membrane charge shielding, respectively.

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1. Introduction

Industrial wastewater treatment is one of the main current environmental issues. As water resources are limited, its treatment and recovery is of special importance. Furthermore, the discharge of contaminated water must be conscious and the legislation of water disposal must be abided by. In terms of the effluents, detergents coming from detergent plants or industrial cleaning processes can cause important environmental problems to aquatic media [1–6].

The recovery and reuse of cleaning agents in industrial cleaning processes permits lower discharge into the environment and reduces costs. In wastewater treatments, membrane technology has shown many advantages with respect to other methods as it is less time-consuming, easy to operate, lower energy costs, the requirements of additional chemicals is minimized and the scale-up is easy [4]. A review of the recovery of detergent compounds by means of membrane technology has previously been published [7].

Cleaning processes are hugely water-consuming in industries where large volumes of wastewater are generated for disposal [8]. These wastewaters contain mixtures of cleaning agents from detergent formulations (in some cases were described more than 25 ingredients [9]) and soils. Although the variability of compounds in these streams is high (detergent agents, fats, salts,

carbohydrates, proteins, etc.), there are some molecules that are commonly present in these streams which come from commercial detergent formulations, that is surfactants and chelating agents.

Sodium dodecyl sulfate (SDS) is one of the most studied anionic surfactants. The monomer has a hydrophobic tail (hydrocarbon chain) and a hydrophilic head (sulfate group), and this amphiphilic character allows it to easily attach to interfaces. Foaming and emulsifying are the main properties of this type of compound. The formation of aggregates of monomers, called micelles, is one of its particular characteristics. Micelles are spherical structures, in which the hydrophobic tails are oriented to the interior and the hydrophilic groups point towards the aqueous medium. This configuration occurs with a minimum concentration of the surfactant, called critical micellar concentration (CMC). Rod-shaped structures are also described when the concentration is higher than the CMC value [10]. A scheme of the micellar structures is shown in Fig. 1.

Ethylenediaminetetraacetic sodium salt (Na₄EDTA) is a chelating agent that is widely used in household and industrial detergent formulations. Its main function is the chelation of Ca⁺², Mg⁺² and heavy metals to prevent precipitation and incrustation [11–14].

The discharge of these two agents into the environment without suitable pre-treatment contributes to the pollution of water, as the chemical oxygen demand (COD) in aqueous solutions varies from 910 to 9080 mg O₂/L for the anionic surfactant and between 235 and 3155 mg O₂/L for the chelating agent in the range of 0.05–0.6% w/w.

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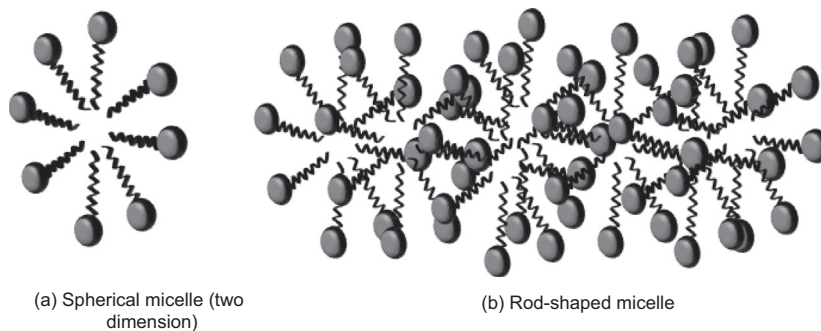


Fig. 1. Structures of SDS micelles.

In membrane technology (ultrafiltration and nanofiltration), one of the mechanisms involved in the transport of ionic compounds is the electrostatic interaction (repulsion/attraction) between them and a charged membrane. Ions with the same charge as the membrane (co-ions) are repelled and ions with the opposite charge (counter-ions) are attracted to the membrane surface. This difference of ions concentrations between the membrane surface and the bulk solution generates a potential difference at the interface. Then, ions are repelled and attracted in order to maintain electrochemical equilibrium (Donnan effect) [15]. However, if some components are able to form micelles (as it is the case for SDS) and/or there are other ions present in the feed (salts, H^+ , OH^- , etc.), the transport mechanisms can change and transmission through the membrane can be modified. In such cases, the effects of transmembrane pressure and solutes concentration must be studied.

The objective of the present work is to study the transport through a 5 kDa ceramic membrane of SDS and mixtures of SDS/Na₄EDTA/NaOH. The presence of NaOH was included because of the high number of commercial alkaline formulations used in industry [8] and owing to the fact that caustic solutions have been used traditionally as chemical cleaning formulations [6,16,17]. These mixtures simulate the detergent formulations used in many industrial applications. A preliminary study of the permeation and retention of single SDS solutions has been included. In a previous work, the transmission of Na₄EDTA through UF membranes has been extensively described [14].

2. Experimental

2.1. Detergent agents

Na₄EDTA (purity 98%) and SDS (92.5–100.5%, based on total alkyl sulfate content basis) were purchased from Sigma Aldrich (Spain). Sodium hydroxide – pellets – were supplied by Panreac Química Sau (Spain).

2.2. Analytical methods

SDS (288.38 Da) was quantified in permeate and retentate streams by means of spectrophotometric measurements at 650 nm with a Thermo Electron Corporation Helios α spectrophotometer. The standard methylene blue active-substances method consists of several extractions of the colored surfactant-cationic dye complex by using chloroform. However, in this study, the procedure was simplified with only one extraction, following the method developed by Jurado et al. [18]. The estimated error for SDS determination using this method was evaluated as $\pm 6\%$. All samples were analyzed at least by duplicate. Methylene blue, disodium tetraboratedecahydrate and HCl were provided by VWR

(Spain). Chloroform, for analysis, was purchased from Merck (Germany).

High-performance liquid chromatography with an ultraviolet detector (HPLC-UV) was used to quantify the chelating agent Na₄EDTA (380.17 Da) in the permeate and retentate samples, using the signal obtained at 254 nm for the NaFeEDTA complex that was previously formed. The analytical procedure was developed in a previous work [19] and the error was established to be lower than 1.5%. Iron (III) chloride hexahydrate (purity >99%), sodium formate (purity >99%), formic acid (50% in water), tetrabutylammonium bromide (purity >99%), water (HPLC-grade) and methanol (HPLC-grade) were provided by Sigma Aldrich (Spain).

The membrane retention (R) of SDS and Na₄EDTA was calculated as follows

$$R(\%) = 1 - \frac{C_p}{C_R} \cdot 100$$

in which C_p and C_R are the concentrations of the solute in the permeate and retentate streams, respectively.

The conductivity and pH values of the solutions were measured with a portable Crison MM40 conductivity meter (the error of the conductivity measurement was lower than 0.5%). The CMC value of the anionic surfactant at different temperatures was determined by the change in the slope of the plot of conductivity vs. SDS concentration [20–23].

The viscosity measurements were carried out with a Cannon Fenske viscosity meter (series 25) for clear liquids at 40 °C.

2.3. Equipment and procedure

An ultrafiltration rig with a 5 kDa ceramic membrane, similar to one previously used by the authors [14], was employed to carry out the experiments (the membrane characteristics are shown in Table 1). The solutions at different concentrations were prepared in a feed tank (15 L) and the temperature was fixed at 80 ± 2 °C. The temperature was selected to be similar to many cleaning processes in industry. The permeate and retentate streams were recirculated to the feed tank in all experiments.

Table 1
Main characteristics of the tubular ceramic membrane KERASEP™.

Parameter	Value
MWCO (Da)	5000
Channels	19
Filtration area (m ²)	0.245
Membrane support material	TiO ₂ -Al ₂ O ₃
Membrane material	ZrO ₂
Isoelectric point	Slightly below pH 6
Water permeability (80 °C, 3 bar) (L/h m ²)	540

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