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Membrane Ultrafiltration for Oil-from-Water Separation: Multidisciplinary Lab Experiment*

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An ultrafiltration membrane module was assembled and used for treating an oil-in-water (O/W) emulsion. This lab set-up was implemented to teach membrane separation processes within Chemical or Environmental Engineering programmes. The experimental set-up and procedure are described, as well as typical results obtained by students, underlining the main objectives and reasoning expected to be accomplished in each stage of the work. Particular emphasis is given to the discussion of the impact of some variables, such as pressure driving force, surface velocity and emulsion concentration, on concentration polarization, recovery and rejection. The use of this technology at an industrial scale is also discussed. The implemented lab experiment has a relevant pedagogic impact and facilitates students to grasp the inherent theoretical concepts, as perceived from their reports and oral discussions. Moreover, the work has been well accepted and appreciated by students, as can be inferred from the questionnaire; their assessment also showed the fulfilment of the established technical and pedagogic objectives. Particularly relevant is the importance that students attribute to the execution of the experimental work to comprehend the concepts (i.e. importance of a hands-on approach).

Keywords: ultrafiltration; chemical and environmental engineering; membrane module; oil and water

Nomenclature

A_w	permeance	L m^{-2} h^{-1} bar^{-1}
c_f	solute concentration in the feed side	% (v/v)
c_p	solute concentration in the permeate side	% (v/v)
N_w	solvent (water) flux	L m^{-2} h^{-1}
ΔP	pressure difference between the feed and permeate sides	bar
Q_f	feed flow rate	L h^{-1}
Q_p	permeate flow rate	L h^{-1}
r	membrane pore radius	m
Rec	recovery	%
Rej	rejection (or yield)	%
T	temperature in the recirculation tank	K
Greek letters		
δ	membrane thickness	m
ε	membrane porosity	–
η	water viscosity	Pa s

1. Introduction

1.1 Theoretical concepts

Membrane separation technologies have been assuming a growing importance in the industrial treatment of liquid and gas effluents, among several other applications [1, 2]. A membrane is defined as a structure having lateral dimensions much greater than its thickness, through which transfer may occur under a variety of driving forces [3]. Membranes can be flat or tubular; in a membrane module they are arranged in 'plate and frame', spiral wound or tubular membrane configurations. Tubular membranes are named hollow fibers if they have a diameter smaller than 0.5 mm; those between diameters of 0.5 mm and 5 mm membranes are named capillary, and above 5 mm they are named tubular [4]. Membranes can be polymeric but can also be made of ceramic, metal, glass, carbon or liquid materials [4]; they can be porous, microporous or dense (non-porous).

At least three streams should be considered in a membrane process: 1) feed; 2) retentate and; 3) permeate. A fourth stream might as well be present: the sweep stream. The feed and retentate streams are the input and output of the retentate chamber, while the sweep and permeate streams are the input and output of the permeate chamber, respectively.

An important group of membrane processes involves application of pressure to force the passage of a liquid solvent (aqueous or not) through the membrane. This class of membrane separation process is named filtration. Depending on the size of the pores, the separation process is named microfiltration, ultrafiltration, nanofiltration or reverse osmosis, the latter being also known as hyperfiltra-

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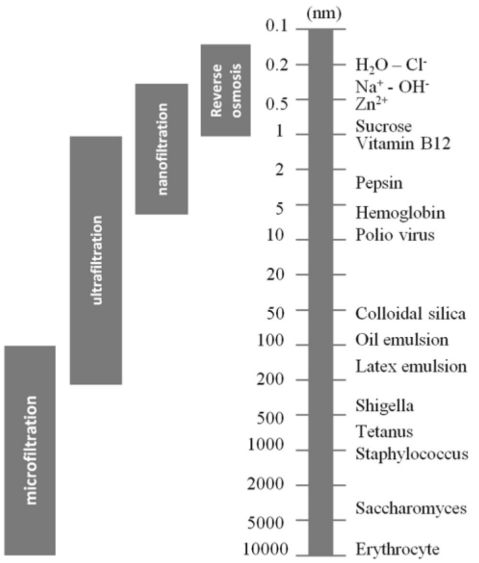

Fig. 1. Application of the microfiltration, ultrafiltration, nanofiltration and reverse osmosis processes as a function of the membrane pore size; adapted from [4].

tion, Fig. 1 [4]. Microfiltration membranes have the largest pores and are used mostly for removing particles from liquid streams. Ultrafiltration and nanofiltration have smaller pores, therefore demanding greater driving pressures; nanofiltration membranes can be microporous if pores are smaller than 2 nm. Membranes for reverse osmosis are non-porous. The most common materials of filtration membranes are polymeric and ceramic.

The ability of a microfiltration or ultrafiltration membrane to permeate a given solute depends on its size, shape and shape adaptation during the filtration process, but also on the chemical nature of both membrane and solute. However, pore size is the most important parameter to characterise microfiltration and ultrafiltration membranes in terms of their ability to perform a given separation. Since pore size is difficult to determine, manufacturers normally use the mass cut-off to characterise the membrane's ability to perform a given separation. This is defined as the molecular mass that is 90% rejected by the membrane [4]. Though IUPAC recommends the use of atomic units (au) to characterise the molar mass, in fact most suppliers still present data using the Dalton unit.

The work herein described deals with the treatment of an effluent containing an oil-in-water emulsion (O/W). This is used as a metalworking fluid in machining processes for the lubrication and cooling of cutting tools [5, 6]. The dimension of the oil droplets is typically about 0.1 μm, making the removal of the oil from the aqueous medium an ultrafiltration application (see Fig. 1).

Industrially, ultrafiltration membrane processes are used for important applications like: (1) the food industry (pre-concentration of milk before making cheese or clarification of fruit juices); (2) water treatment (car washing, oil removal from metalworking operations or colour removal from Kraft black liquor in papermaking) and (3) the pharmaceutical industry (recovery of vaccines and antibiotics from fermentation broth) [7, 8].

The solvent (water in this case) flux, N_w, that permeates through the membrane is proportional to the pressure difference (ΔP) between the two sides of the membrane (feed and permeate) [9]:

$$N_w = A_w \Delta P \tag{1}$$

where A_w is the membrane permeance towards the solvent. The permeance is normally expressed in units of L m^{-2} h^{-1} bar^{-1} [4]. It should be emphasised here that permeance and permeability are properties of the membrane and not of the permeate, and then one should not write 'solvent permeance' nor 'solvent permeability'.

During the treatment of the O/W emulsion, water permeates the membrane while the oil droplets are retained. As the total pressure difference increases, the oil concentration increases (and the water concentration decreases) at the membrane surface on the retentate side. As a consequence, the driving force for water permeation decreases. This reversible phenomenon is known as concentration polarization. It consequently imposes a practical limit to the maximum value of the permeate flux (N_w^{∞}). Two strategies are normally followed to minimize concentration polarization: (1) the use of turbulence promoters, known as spacers, and (2) the increase of feed velocity, which decreases the thickness of the stagnant film. In practice a filtration process should be operated within the linear regime defined by Equation (1), since after the onset of concentration polarization, the additional energy used in increasing the driving force has minimal or no effect on permeate flux, Fig. 2.

When the concentration at the surface reaches a limit value, a gel may be formed, covering the membrane surface. Concentration polarization is a reversible phenomenon that increases as the solution concentration and permeance increase. Contrarily, when a permanent loss on membrane permeance is observed, this is normally related to fouling. This results from permanent solute deposition at the membrane surface and pore network, progressively impairing the membrane transport properties [8].

In ultrafiltration, permeance is related to factors inherent to membrane morphology (e.g., porosity and pore size distribution) and the chemical nature of membrane and permeants. As mentioned above,

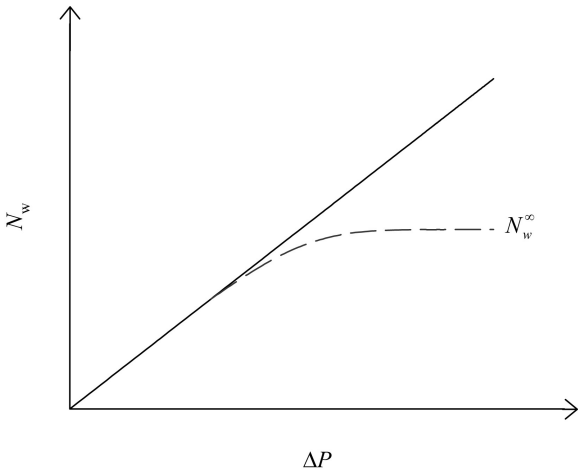

Fig. 2. Permeate flux versus pressure difference: continuous line—linear relationship (Equation (1)); dashed line—deviation from Equation (1) caused by the occurrence of concentration polarization phenomenon, where a limiting flux is obtained (N_w^∞).

ultrafiltration membranes can be polymeric or ceramic [4, 8]. The latter are more expensive and fragile, and modules are less compact. However, they have longer lifetime and are suitable for operation under harsh environments; additionally, ceramic membranes are easier to regenerate [4]. In ultrafiltration, the applied pressure difference ranges between 1 bar and 5 bar and the permeance ranges between 10 L m^{-2} h^{-1} bar^{-1} and 200 L m^{-2} h^{-1} bar^{-1}.

Ultrafiltration is normally used to concentrate solutions or mixtures; membrane permeance is therefore of key importance. Assuming laminar flow of the solvent through the membrane cylindrical pore network, mass transport is given by the Hagen–Poiseuille equation [4, 8]:

$$N_w = \frac{\varepsilon r^2}{8\eta\delta}\Delta P, \tag{2}$$

where ε is the membrane porosity, r the pore radius, δ the membrane thickness and η the liquid viscosity.

Usually, the membrane is selected so that the solute is completely (or almost completely) rejected. The performance of an ultrafiltration membrane is characterised in terms of recovery (or yield) and/or rejection, apart from permeance. The recovery is the ratio between the permeate, Q_p, and the feed, Q_f, flow rates:

$$Rec(\%) = \frac{Q_p}{Q_f} \times 100 \tag{3}$$

Industrial systems are usually designed so that the recovery is the largest possible. On the other hand, rejection evaluates the efficiency of the membrane in hindering the solute passage to the permeate stream:

$$Rej(\%) = \left(1 - \frac{c_p}{c_f}\right) \times 100, \tag{4}$$

where c_p is the solute concentration in the permeate and c_f the concentration in the feed. Therefore, a rejection of 100% is equivalent to obtaining a permeate stream free of solute.

Few experimental works are reported in the pedagogical literature concerning ultrafiltration. Silva et al. [10] described a similar ultrafiltration experiment, though their analysis focus essentially on the concentration polarization using an aqueous suspension of a yeast, which easily forms a gel layer on the retentate side. Conlee et al. [11] discussed the use of ultrafiltration to treat dairy feeds and its applicability to chemical engineering, as well as process performance and controlling parameters. These authors also concentrated their analysis on the concentration polarization phenomenon.

This lab experiment was designed to be an introductory experimental work to the topic of ultrafiltration, although concepts such as the role of the chemical nature of the membrane material on separation performance, and the thumb rules for process optimization and phenomenological modelling are not tackled. Even so, the paper (as well as the tutorial provided to students) provides the relevant scientific background, the description of the experimental set-up and proposed experimental procedure; the learning impact on the students is also herein addressed. The lab experiment was designed to be cheap to acquire and to operate, be environmentally friendly, and to be easy to operate and to provide easily understandable results, in line with other experiments developed by the same educators [12–13]. Since membrane filtration processes are quite relevant among the separation processes within chemical, environmental and bio-engineering, this experimental work allows students to embed the working principles of ultrafiltration, develop experimental skills, and to acquire relevant knowledge concerning this separation process.

1.2 Pedagogical objectives

Membrane separation processes find numerous industrial applications. The fundamental concepts involved are common to different engineering degrees. In our particular case, at the Faculty of Engineering of the University of Porto, this experimental work is carried out by students attending the 4th year of both Chemical and Environmental Engineering Integrated Masters.

After the completion of this work, students are expected to:

- operate an ultrafiltration laboratorial unit and describe its components, establishing a relationship with the equipments used in industry;
- describe the physical phenomena involved in the purification/concentration of liquid streams using membrane technology;
- interpret the plots of permeate flux as a function of pressure for different oil-in-water emulsion concentrations, identifying the most appropriate operating conditions.

The work is performed in a 3 hour lab session by groups of 2–3 students, depending on the total number of students in each session. This means that each group has not enough time to test different emulsions. Therefore, the group should compile the results obtained by other colleagues, allowing them to better understand what happens for different O/W concentrations, and discuss them on a written report or in an oral discussion.

2. Material and methods

2.1 Experimental set-up

A monotubular Carbosep® M2 membrane was used in this study. The technical data provided by the manufacturer are presented in Table 1.

Table 1. Membrane characteristics and recommended operating limits

Characteristic	
Support	Carbon
Membrane active coat	ZrO_2–TiO_2
Cut-off (au)	15 000
External diameter (mm)	10
Hydraulic diameter (mm)	6
Length (mm)	400
Operating limit	
Pressure (bar)	≤ 5
pH	0–14
Temperature (°C)	≤ 100

A sketch of the experimental set-up is shown in Fig. 3. The tubular membrane is enclosed in a metal case and fed to the bore side (Fig. 4). In this set-up both permeate and retentate streams are directed to the feed tank. Students should, however, be aware that in practice the permeate stream constitutes the treated effluent, and the retentate can be partially recirculated back to the membrane module.

By regulating valve V2 (cf. Fig. 3), the pressure inside the tubular membrane may be varied. The feed stream is sent to the membrane module using a displacement pump (E.M.G. Elettromeccanica, model 71/4). The feed flow rate is kept approximately constant as the pressure increases by regulating valve V1 that gives access to a bypass circuit, Fig. 3. The retentate flow rate is measured with a turbine flowmeter (RS Amidata, model 257-026) and may be assumed equal to the feed flow rate (students should realize this when measuring the permeate flow rate, because this is almost negligible as compared to the feed one). Two pressure sensors (Keller, type PR-21SR) allow calculating the average pressure inside the membrane (feed side). Since the internal diameter of the membrane is relatively high, the pressure drop is small and the two pressures should be very similar. A type K thermocouple reads the temperature history of the fluid inside the recirculation tank. Generally, the use of a heat-exchanger to keep constant the feed stream temperature is preferred (see [6]).

The oil-in-water emulsion used is a hydrocarbon mixture with an anionic emulsifying agent (Sunoco DRY), used in the metal-mechanic industry for lubrication of metal cutting tools. The oil content in the permeate stream is analysed continuously measuring the absorbance with an UV/Vis spectrophotometer (Jenway, model 6305), making use of a flow-through cell, thus allowing the calculation of the respective concentration from a previously obtained calibration curve. Measurements were

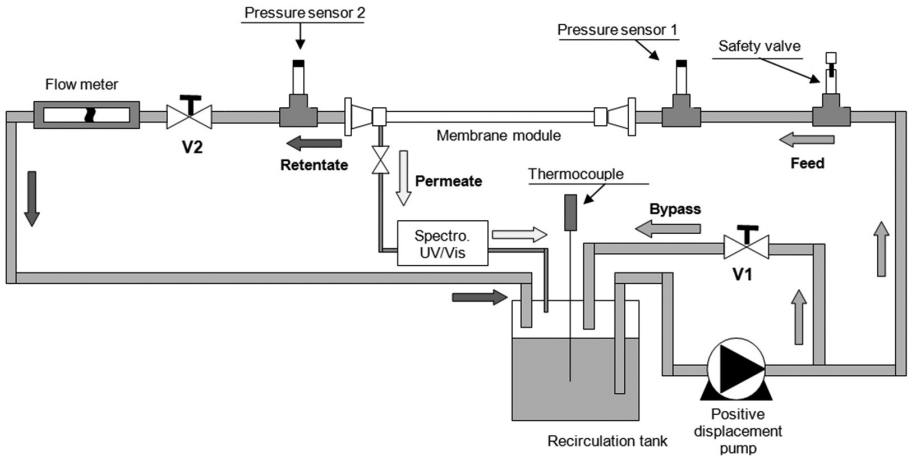

Fig. 3. Schematic representation of the experimental set-up.

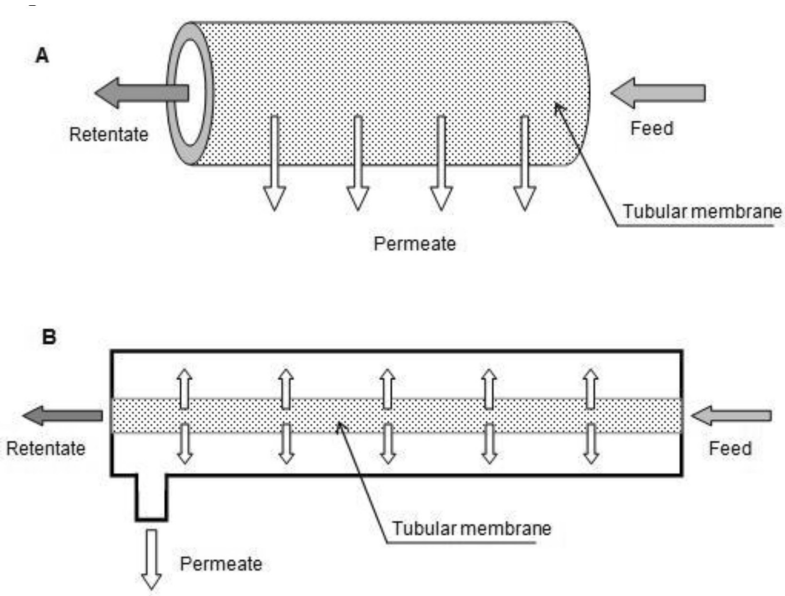

Fig. 4. Streams involved in permeation through a tubular membrane (A) and in a membrane module without sweep stream (B).

obtained at 734 nm (maximum absorbance that is characteristic of the emulsion in use).

2.2 Experimental procedure

At the beginning of the class, and before starting operating the unit, students are asked to familiarize themselves with the set-up and clarify any doubts with the professor or lab technician. Students are expected to have read the lab manual in advance.

First of all, the base line of the spectrophotometer used to quantify the concentration of the oil emulsion in the permeate stream (see Fig. 3) is established using distilled water. Afterwards, the wavelength of 734 nm at the spectrometer is checked / established.

After pouring the feed solution into the tank and after assuring that valves V1 and V2 are completely open, the pump is turned on. The desired pressure difference and feed flow rate for each run is obtained regulating valves V2 and V1, starting with values of ca. 1 bar (pressure difference between feed and permeate sides). Data acquisition (pressure in sensors 1 and 2, flow rate of retentate and absorbance) is performed with a computer running a Labview application.

Students are asked to measure the permeate flow rate with the help of a beaker and a stopwatch, after checking that the absorbance exhibits a stable behaviour, with intervals of ca. 5–10 min., until steady state is attained. As discussed below, this is not observed unless the permeate flow rate is corrected for the water viscosity dependence with temperature, since it was chosen not to use a heat-exchanger to control the feed stream temperature. For that, the temperature in the feed tank must be also recorded when the permeate flow rate is measured.

After obtaining the stable corrected permeate flow rates at ca. 1 bar pressure difference, it is suggested that one obtains data at ca. 2 and 3 bar. For that, valves V2 and V1 should again be manipulated according to the above-described procedure. While regulating valves V2 and V1, one must be careful so that the pressure difference never exceeds 4 bar, in order to avoid damaging the membrane; this is clearly indicated in the protocol given to the students, in the Special Operation and Safety Indications section. At the end of the experimental session, students should check that the pump, spectrophotometer and computer are turned off, and are required to leave the set-up properly cleaned and valves V2 and V1 completely opened.

The system is periodically back-flushed with water for cleaning the membrane, particularly between experiments performed with different oil concentrations.

3. Technical results

3.1 Determination of the oil droplet diameter

The oil droplet size distribution was determined using a Coulter LS230 light scattering particle size analyser. Most of the oil droplets formed in the O/W emulsions used in this experimental work (with oil volume concentrations of 5%, 10% and 15%) have a diameter between 0.04 μm and 0.20 μm (c.f. Fig. 5). This means that the water can be separated from the oil by ultrafiltration (see Fig. 1) using the proposed membrane (cut-off is 15 000 au $\approx$ 0.01 μm).

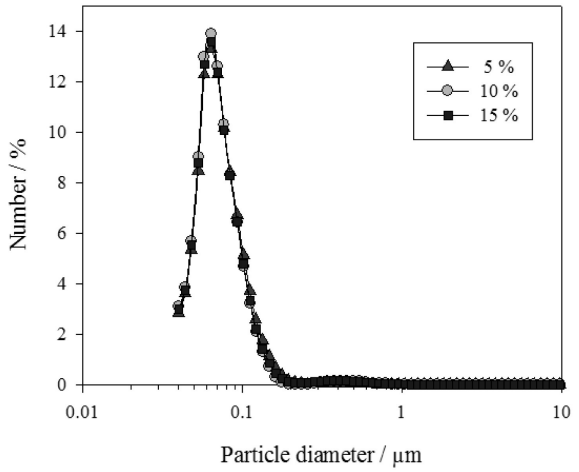

Fig. 5. Oil droplet size distribution for the different O/W emulsion concentrations used.

3.2 Permeation experiments

Figure 6 shows a typical calibration curve, which is not obtained by students during the class, but is made available for them to perform the necessary calculations. In particular, it is important to transform the measured absorbance data into oil concentration in the permeate stream, allowing calculation of the rejections reached by the membrane under different conditions. Of course, the calibration curve refers to very low oil concentrations, which are typically found in the permeate stream due to the very good separation reached.

As mentioned above, during the lab class each group of students uses an oil emulsion with a given concentration. Later on, they complete the report with data from other groups.

For three feed pressures (2, 3 and 4 bar), students plot the permeate flow rates as a function of time. Figure 7 shows a typical run, for a 5 vol.% O/W emulsion, and a total pressure difference across the membrane of approximately 1 bar (the value considered in calculations is actually the difference between the average retentate pressure, measured along the run, and the atmospheric pressure).

Students should note that the measured permeated flow rate increases steadily along the run, as illustrated in Fig. 7. Due to closed loop operation, heat released by the pump accumulates in the liquid, causing its temperature to rise continuously (cf. Fig. 7). Consequently, the viscosity decreases, leading to an increase in permeation flux. To account for this effect, students should recall Equation (2), which shows that the permeate flux (and inherently flow rate) is inversely proportional to viscosity; therefore, they should correct the permeate flow rate to a reference temperature. This is achieved using the following relation for the water viscosity dependence on the temperature [14]:

$$\eta = \frac{e^{\left(A+\frac{B}{T}+CT+DT^2\right)}}{1000}, \qquad (5)$$

where $A = -2.471 \times 10^1$, $B = 4.209 \times 10^3$, $C = 4.527 \times 10^{-2}$ and $D = -3.376 \times 10^{-5}$. The temperature, T, is given in kelvin, whereas the viscosity, η, is in Pa·s. Thus, from Equation (2), students can deduce the following relationship to correct the flow rate:

$$Q_{p,corrected} = Q_{p,uncorrected} \frac{\eta_{uncorrected}}{\eta_{corrected}}, \qquad (6)$$

where $Q_{p,uncorrected}$ stands for the permeate flow rate at a given instant and temperature for which the water viscosity is assessed through Equation (5); the corrected permeate water flow rate is obtained assuming a reference temperature of 25 °C.

Figure 7 also shows the corrected permeation flow rates. A more constant plot is observed, showing that the measured flow rate increases only due to the variation of the temperature. Temperature increase is mostly related to the power of the pump, increasing in the present case ca. 11 °C in 90 min.

Figure 8 shows the average corrected permeate fluxes at steady state as a function of the pressure difference across the membrane, for the three oil emulsions tested.

The water flux that permeates through the membrane increases, as expected, with the driving force; such dependence is nearly linear, for all O/W emulsions and range of pressures studied, except for the more concentrated emulsion and for a feed flow rate of only 5.9 L min^{-1} (Fig. 8). In this case, the permeate flow rate decreases noticeably when increasing ΔP to 2 and particularly to 3 bar. Students are prompted to discuss this fact, and analyse the possibility of occurring concentration

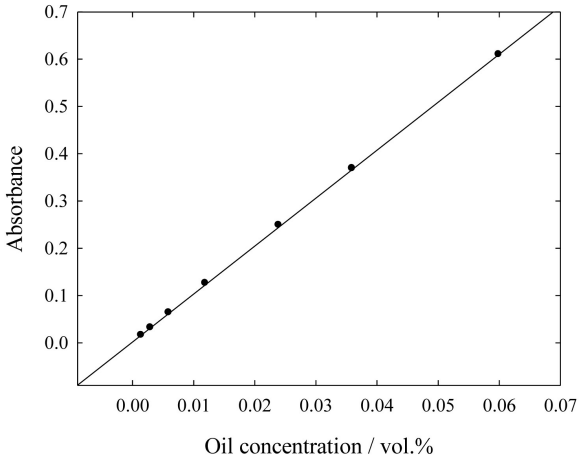

Fig. 6. Calibration curve: absorbance versus oil concentration.

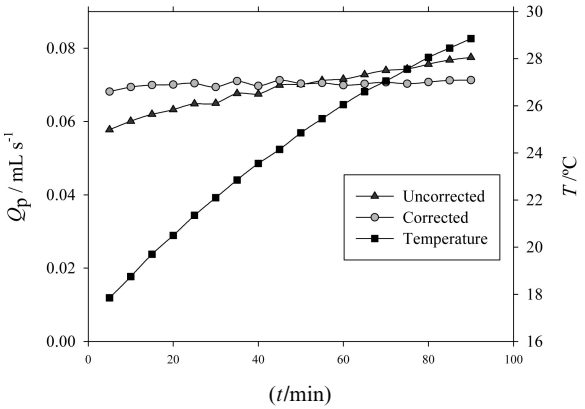

Fig. 7. Corrected (due to viscosity variation) and non-corrected permeate flow rate along the time for a 5 vol.% O/W emulsion and a total pressure difference of 1 bar. O/W temperature along the time is also presented. Lines are for eye guidance.

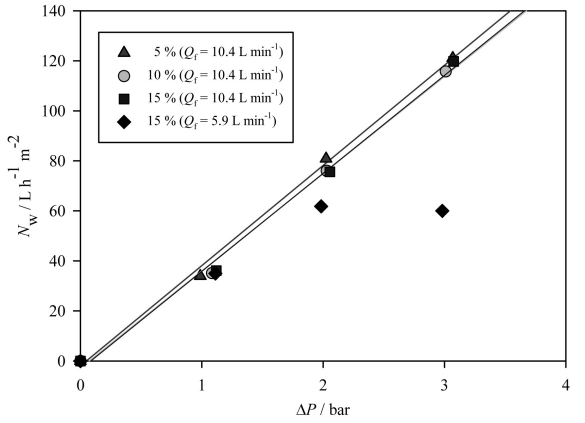

Fig. 8. Permeate flux as a function of pressure difference between the feed and permeate sides, for different emulsion concentrations and feed flow rates. Lines show the fitting by linear regression to experimental data (c.f. Equation (2)).

polarization, as described in the Introduction section.

For each O/W emulsion, permeance can then be easily obtained by students, as described by Equation (1). Basically and for the reference temperature, this is taken from the slopes of the corrected permeate fluxes as a function of the pressure difference plots, in the linear region (i.e., when no polarization concentration phenomenon occur). Table 2 shows permeance values computed from the slopes of the linear portions of the permeate flux versus pressure difference plots reported in Fig. 8. Students should realize that these results are within the typical values for ultrafiltration (10 to 200 L m^{-2} h^{-1} bar^{-1}). The results in Table 2 show that the membrane permeance towards water is nearly independent of the O/W concentration, as long as the feed flow rate is high enough to prevent the occurrence of concentration polarization.

Table 3 shows recoveries for the experiments

Table 2. Membrane permeance obtained for different oil concentrations and for a feed flow rate of 10.4 L min^{-1}

Oil feed concentration (vol. %)	A_w(L m^{-2} h^{-1} bar^{-1})
5	40.1
10	38.8
15	39.9

Table 3. Recovery as a function of feed oil concentration and pressure difference across the membrane and for a feed flow rate of 10.4 L min^{-1}

ΔP (bar)	Oil feed concentration (vol.%)	Recovery (%)
1		0.040
2	5	0.098
3		0.144
1		0.041
2	10	0.096
3		0.137
1		0.043
2	15	0.098
3		0.146

reported, assessed using Equation (3). Students should realize that these values are quite low, as a consequence of the low permeation flow rates. Recoveries increase for higher-pressure differences across the membrane, which is the driving force for water permeation. However, extrapolation of data shown in Fig. 8 allows easily anticipating that such increase would not be considerable, or could even not occur, namely for lower feed flow rates and concentrated emulsions (see also Fig. 2).

Equation (4), together with the spectrophotometer calibration curve (Fig. 6) allows obtaining the rejection for each assay. The results obtained are always quite high (above 99%, data not shown), indicating a very good performance of this membrane towards separation of the O/W emulsion.

Students are also asked to compare the oil concentration in the permeate side with the Portuguese wastewater legislation standards—15 mg L^{-1}, as defined in decree law no. 236/98. An additional information is herein required, i.e., that a 5 vol.% suspension corresponds to a concentration of 38 g L^{-1} [6]. Figure 9 shows the oil concentrations obtained in the permeate side, for the different oil feed concentrations and pressure differences across the membrane.

Based on the data shown in Fig. 9, it is clear that for the operation conditions used, ultrafiltration allows obtaining low oil content downstream effluents, quite below the Portuguese limit value (15 mg L^{-1}), as long as pressure differences across the membrane are below 3 bar and the feed flow rate at least 10.4 L min^{-1}. Therefore, students should be

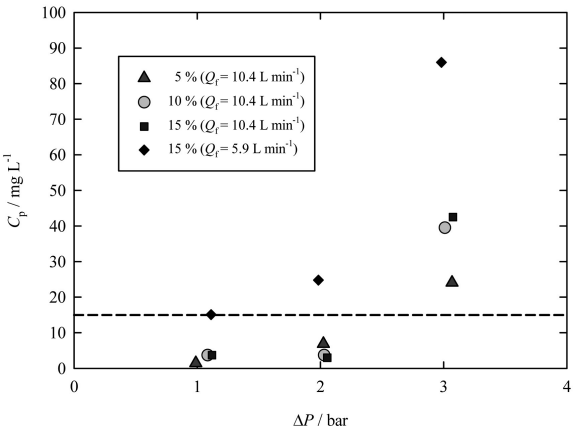

Fig. 9. Oil permeate concentration as a function of the pressure difference across the membrane for different feed oil concentration and feed flow rates. The dashed line stands for the Portuguese limit value of oil concentration in wastewaters.

aware of the trade-off between high recovery and low rejection when operating at high-pressure differences.

In their reports, students are also required to describe the practical relevance and the industrial applicability of this type of technology (ultrafiltration), indicating the advantages, disadvantages and alternatives. In particular, they realize that on an industrial scale, and to increase the permeate flow rate up to reasonable values, parallel arrangements of membrane modules are used, hosting multitubular membranes. They have been reporting numerous and interesting examples that they have found in quite different industrial sectors (from food and beverage industries to pharmaceutics, besides the most obvious water/wastewater treatment companies).

4. Students' assessment

Around 35% of the total number of students (147) that have performed this work in the last two years (Integrated Master in Chemical Engineering at FEUP) answered the questionnaire provided in the Supporting Information (available at http://paginas.fe.up.pt/~fdmagalh/IJEE-ultrafiltration). The questions concerned how students evaluate the experimental work organization and lab protocol, the importance they attribute to this work and related technology, the contribution of the people with whom they interacted during and after the experiments, and also the relevance of this lab experiment to better understanding theoretical concepts of ultrafiltration. In particular, this questionnaire enabled one to assess if the technical and educational objectives mentioned above were achieved. An average punctuation of 3.9 (in a scale of 1 (not relevant/totally disagree) to 5 (very relevant/totally agree)), with an average standard deviation of 0.8, was obtained to the questions; see Supporting Information. The majority of the answers were essentially centred on the average; however students gave a particularly positive evaluation to the role of performing the lab session on comprehending the involved concepts of ultrafiltration, which is a major goal of this work.

5. Conclusions

The membrane separation lab set-up described is easy to operate, low cost and poses no problems in terms of residues disposal; it is also safe to operate and with a relevant pedagogic impact. In addition, it is multidisciplinary, being useful for students in different engineering areas.

Its impact on student's perception of the inherent concepts is easily understood from their written reports and oral discussions, as well as from the questionnaire made (see Supporting Information). Operation of the membrane module in different conditions facilitates grasping the concepts usually taught in separation processes subjects.

Students became aware of the importance of this technology when asked to discuss industrial examples where membrane processes (ultrafiltration or otherwise) are used. Gathered information during field trips allows students to realize the relevance of parameters such as recovery and rejection, and understand better how membrane modules are inserted in the whole industrial process. Student assessments showed that both the technical and educational objectives were achieved. Students considered that this work allowed them to better comprehend the concepts of ultrafiltration.

Supporting information

Supporting information containing the results of the surveys made to students is available at: http://paginas.fe.up.pt/~fdmagalh/IJEE-ultrafiltration

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