Ultrasound membrane hybrid processes for dairy wastewater treatment: pilot-scale analysis

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ABSTRACT

Novel wastewater treatments are necessary to effectively decrease the organic load of dairy wastewater before disposal. In this work the feasibility of a hybrid process of membrane filtration combined with ultrasonication to decrease organic load was investigated. The efficiency of the advanced hybrid process that simultaneously applies ultrasonic irradiation and pressure-driven membrane ultrafiltration was examined. Polyethersulfone ultrafiltration membranes with 20 and 50 kDa molecular weight cut-off were tested. The effects of the continuous and half intermittent ultrasonic irradiation during the ultrafiltration were compared. Ultrafiltration fluxes, chemical oxygen demand retentions and membrane, pore fouling and polarization layer resistances were analyzed in a combined process. Furthermore, mass transfer coefficients, fouling coefficients and concentrations on membrane surface were also calculated and compared. The results showed that the best correlation were achieved by plotting the cake filtration model, but the other models also have given relatively good correlations using gradual pore blocking and intermediate filtration models. For this reason the membrane filtration ultrasonication should be further investigated for future applications.

Keywords: Dairy wastewater; Half intermittent and continuous ultrasonication; Ultrafiltration hybrid processes; Fouling resistances

1. Introduction

Membrane separations have been extensively used in dairy industry for selective separation of different molecules and compounds. In these membrane processes, the pressure difference is the driving force between the two sides of the membrane; therefore, they are called pressure-driven membrane processes or membrane separation technologies. However, these widely used industrial applications have a main obstacle, namely the membrane fouling. Fouling phenomena during the UF process is the retained particle accumulation on the membrane surface causing significant reduction in the separation efficiencies [1]. Ultrasound has been used to improve the permeate flux and recovery of membrane permeability and to reduce

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the rapid flux decline caused by concentration polarization and membrane fouling [2,3].

Over the last decade, various studies have been conducted using ultrasound as a means of mitigating membrane fouling, in ultrafiltration process, and to improve fouled membrane cleaning [4,5]. Ultrasonication (US) treatment has been comprehensively accepted as a powerful method for fouled membrane cleaning in water- and wastewater treatment. US has been regarded as an efficient technique in membrane filtration and fouled membrane cleaning to enhance the permeate flux and membrane permeability recovery [6]. US refers to sound wave propagation into a medium beyond the audible frequency range above 20 kHz or 20,000 cycles per second. For US, surface cleaning generally uses a frequency in the range of 16–70 kHz [7]. US creates a number of physical forces, which can be generated by the alternating compression and rarefaction cycles, like vibration and physical agitation. In addition to the acoustic cavitation microjets, shear, shockwaves, and acoustic streaming are also initiated [8]. Different mechanisms may lead to particle detachment from a particle-fouled membrane surface, including cavitation mechanisms (microstreaming and microstreamers), acoustic streaming (Fig. 1), and micro-jets.

Acoustic vortex microstreaming, henceforward acoustic streaming, on the membrane surface are proven to contribute to the cleaning of the membrane surface [9]. Acoustic streaming is defined as the absorption of acoustic energy resulting in fluid flow, which does not require the collapse of cavitation bubbles [10]. Acoustic streaming in the liquid increases the velocity of the solute and results in eddy currents near the membrane surface [11]. Therefore, this mechanism causes liquid bulk movement toward and away from the membrane surface. It may loosen, break, and disperse the fouling polarization or cake layer, furthermore scour loosely attached particles

from the membrane surface and prevents further deposition. Since the cross-flow velocities in cross-flow filtration is much higher than the feed flow in dead-end filtration, the acoustic streaming effect can be more efficient in dead-end filtration [12].

Dairy industry requires huge volumes of water and generates wastewater and has wide fluctuationsin their effluent quality [13]. In dairy industry technology, water is used throughout a lot of steps generating large volumes of effluents, mainly white waters and it has high biochemical oxygen demand and chemical oxygen demand (COD) contents with high levels of dissolved or suspended solids including fats, oils, and grease, nutrients such as ammonia or minerals and phosphates, milk components like lactose and proteins, and cleaning chemicals, detergents [14,15]. This type of effluents may result in water eutrophication due to the presence of nitrogen and phosphorus when it is discarded without treatment [16,17]. For the mentioned reasons, treating dairy effluents is one of the most crucial tasks not only for the environment, but also for water recycling.

In this work, the feasibility of a hybrid process of membrane filtration combined with ultrasonication for dairy model wastewater treatment was investigated. The efficiency of the advanced hybrid process that simultaneously applies ultrasonic irradiation and pressure-driven membrane ultrafiltration was examined. The effects of the continuous and half intermittent ultrasonic irradiation during the ultrafiltration were compared.

2. Materials and methods

2.1. Ultrasonically assisted ultrafiltration experiments

The experimental equipment and apparatuses are shown schematically in Fig. 2. The UF experiments without ultrasonication (no US) were carried out in
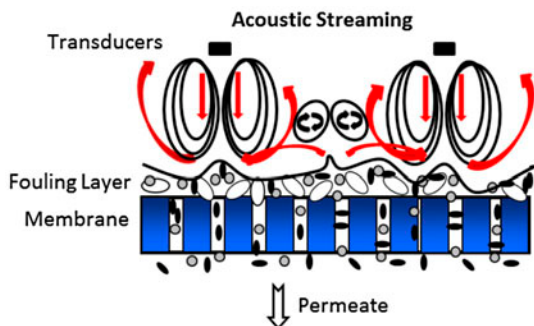

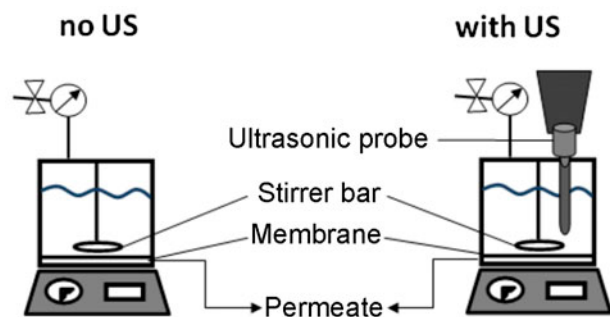Fig. 1. Main mechanism for particle detachment during ultrasonic membrane filtration.

Fig. 2. Experimental setup configurations.

batch solvent resistant stirred cell (Millipore, Model 8400). The UF experiments with ultrasonication (with US) were carried out in a very similar setup, which also consisted of the same batch solvent resistant stirred cell (Millipore, Model 8400) fitted with an ultrasonic probe. The head of this probe was an ultrasonic processor UP100H (Hielscher Ultrasonics GmbH, Germany). The maximal power of the ultrasonic apparatus is 100 W, it was operated at 30 kHz. 100% amplitude and one cycle ultrasonic irradiation was set in continuous US operation mode and 100% amplitude and half cycle ultrasonic irradiation (0.65–0.65 s on-off modes) for half intermittent US irradiation. In our previously reported work, 20 and 30% amplitude were also tested in continuous mode for separation of whey solution using regenerated cellulose ultrafiltration membranes [18].

UF flat sheet membranes of polyethersulfone with 20 and 50 kDa molecular weight cut-off (MWCO) and membrane effective area of 0.004 m² were used (Ultura, Italy). Before conducting the UF experiments, the membrane was immersed into the aqueous solution for one night in order to reach equilibrium. The applied transmembrane pressure (TMP) of 0.1 MPa was constant and achieved by inert nitrogen gas. All measurements were carried out at constant 50°C, as this is an average temperature value for the homogenized dairy wastewaters produced in the industry. The average temperatures at both input (feed) and output of the filtration cell (permeate and retentate) were measured 50 ± 1°C. The initial feed volume was 100 cm³. The UF experiments were carried out until 50 cm³ of the total sample was filtered. The model dairy wastewater in the feed tank was always agitated, in no US mode, using a magnetic stirrer to provide an efficient mixing of 300 rpm, assured a homogeneous solution without excessive vortex formation. In the ultrasonically mixed cell, an equivalent Reynolds number was calculated by measuring the equivalent stirring velocity, resulting the same fluxes as in the stirred batch cell (see Section 2.4). All of the UF experiments were repeated three times to calculate an accurate average. The composition of the feed, retentate and permeate were quantified by different methods. The organic content was measured by COD tests. COD was determined in test tubes (Lovibond, Germany) with an ET 108 digester and a PC CheckIt photometer (Lovibond, Germany). The permeate and concentrate turbidity was determined with a HACH 2100AN turbidimeter (Hach, USA). The conductivity and pH were measured with a multi-parameter analyzer (Consort C535, Belgium). Model dairy wastewater was prepared from skimmed milk powder (5 g dm⁻³) (Szekszárd, Tolnatej Zrt., Hungary) and the anionic surfactant cleaning agent Chemipur CL80 (Nagycserkesz, Hungaro Chemicals, Hungary) at a concentration of 0.5 g dm⁻³. The model dairy wastewater characteristics: $5{,}750 \pm 30$ mg L⁻¹ of COD, 390 ± 45 NTU of turbidity, 7.4 ± 0.1 of pH, and 0.91 ± 0.05 mS cm⁻¹ of conductivity.

2.2. Calculation methods

Fouling is evaluated with a relationship between relative flux (J_{rel}) which is the ratio of the flux at any given time (L m⁻² h⁻¹) during the fouling test to the distilled water flux of the clean membrane (L m⁻² h⁻¹):

$$J_{rel} = \frac{J}{J_W} \quad (-) \tag{1}$$

The total resistance (R_T) is composed of three resistances:

$$R_T = R_M + R_F + R_P \quad (m^{-1}) \tag{2}$$

where R_M is the membrane, R_F is the pore fouling resistance, and R_P is the polarization layer resistance. R_M was calculated as:

$$R_M = \frac{\Delta p}{J_W\,\eta_W} \quad (m^{-1}) \tag{3}$$

where J_W is the water flux of clear membrane (L m⁻² h⁻¹), and η_W is the viscosity of water at 50°C (Pa s). R_F can be measured via the pure water flux (J_{W2}), after washing off the polarization layer from the membrane. R_F and R_P can be calculated as:

$$R_F = \frac{\Delta p}{J_{W2}\,\eta_W} - R_M \quad (m^{-1}) \tag{4}$$

$$R_P = \frac{\Delta p}{J_C\,\eta_{WW}} - R_M - R_F \quad (m^{-1}) \tag{5}$$

where η_{WW} is the viscosity of the wastewater at 50°C (Pa s), J_C is the constant flux at the end of the concentration (L m⁻² h⁻¹). The selectivity of a membrane for a given solute was expressed by the average (apparent) retention:

$$R = \left(1 - \frac{c}{c_0}\right) \times 100 \quad (\%) \tag{6}$$

where c is the average concentration of the solute in the permeate phase, and c_0 is the concentration of the solute in the bulk solution (for COD: (mg L⁻¹)).

2.3. Determination of diffusion coefficients

Diffusion coefficients were determined at 50°C with an Armfield CERb apparatus (Armfield, Great Britain) [19]. A specially designed diffusion cell is mounted on top of the stirred vessel and clamped into the desired position using the locking screw. The equipment consists of a variable speed magnetic stirrer and stirring bar for agitation of the test solution. The diffusion apparatus uses vertical capillaries 5 mm long with a diameter of 1 mm, which restricts the diffusion to one dimension. The concentration at the lower ends is assumed constant and the concentration at the top ends is effectively zero during the experiment. The diffusion coefficient was determined on the basis if Fick's law by Eqs. (7) and (8), as it was discussed in our earlier study [18]:

$$\frac{V}{C_M}\frac{dk}{dt} = -D\frac{d^2\pi}{4}N\frac{c_0}{x} \tag{7}$$

$$D = -\frac{4Vx}{d^2\pi N c_0 C_M}\frac{dk}{dt} \tag{8}$$

where V is volume of water in outer vessel in liters (L), x is the length of capillaries (cm), d is the diameter of capillaries (cm), N is the number of capillaries ($N = 121$), c_0 is the concentration of the dairy wastewater (g dm^{-3}), C_M is the turbidity change for unit concentration change (NTU), dk/dt is the rate of change of turbidity with time (NTU s^{-1}). Measurements were carried out with 5.5 g dm^{-3} model dairy wastewater, by measuring the change of turbidity and conductivity in distilled water. The measurements were triplicated, the diffusion coefficient at 50°C was found to be $1.902 \times 10^{-10} \pm 5.94 \times 10^{-12}$ m^2 s^{-1}.

2.4. Theory

2.4.1. Determination of CMC

Critical micelle concentration experiments were carried out by adding different amounts of 55 g dm^{-3} model dairy wastewater in distilled water and measuring the conductivity. The temperature was kept at 50 ± 1°C using a heatable magnetic stirrer. The conductivity was measured with a multi-parameter analyser (Consort C535, Belgium).

2.4.2. Determination of Reynolds numbers

Since the Reynolds number cannot be used in the ultrasonically mixed cell, equivalent Reynolds numbers were calculated [20] by measuring the equivalent stirring velocity, resulting in the same fluxes as in the mixed ultrafiltration batch cell. In these ultrasonication cases, the characteristic radius was the radius of the membrane. It was found that the effect of half intermittent ultrasonication and continuous ultrasonication mixing are the same as stirring with $n = 716$ and $1,458$ and $1,223$ and $1,296$ min^{-1} rev in the case of 20 and 50 kDa membranes, respectively.

2.4.3. Determination of mass transfer coefficients and concentrations on membrane surface

Cake filtration, intermediate filtration, standard pore blocking, and complete pore blocking are commonly used models to describe the fouling mechanisms in membrane filtrations [21–23]. These filtration laws are summarized in Table 1.

In Eqs. (12)–(19), J is the flux, J_0 is the initial flux, the various k are the fouling coefficients. The various K terms are the mass transfer coefficients for the associated filtration laws. In a stirred batch cell, the solute mass transfer coefficient can be calculated from the following correlations [23]:

$$K_c = 0.0443\frac{D}{b}\ Re^{0.75}\ Sc^{0.33}\quad \text{if } Re > 3200 \tag{9}$$

where $Re = \frac{\omega\,b^2\,\rho}{\eta}$, $Sc = \frac{\eta}{\rho\,D}$, b is the stirring radius, ω is the stirring velocity (rad s^{-1}) and D is the diffusion coefficient (m^2 s^{-1}).

With the assumption of a convection-diffusion mechanism during filtration, the flux is generally expressed by a simplified equation [24]:

$$J = K_c\ \ln\left(\frac{c_M - c_P}{c_F - c_P}\right) \tag{10}$$

where c_M is the concentration on the membrane surface, c_P is the permeate concentration, and c_F is the feed concentration. The polarization layer concentration was calculated according to the following equation:

$$c_M = (c_F - c_P)e^{\frac{J_c}{K_c}} + c_P \tag{11}$$

3. Results and discussion

3.1. The determination of CMC of model wastewater

The critical micellar concentration (CMC) of model wastewater was estimated by measuring the

Table 1
Filtration laws [21–23]

Fouling mechanism	Filtration law		Constant pressure filtration $J_0 A$ = const.	
Complete pore blocking	$J = J_0 e^{-k_b t}$	(12)	$\ln J = \ln J_0 - k_b t$	(16)
Gradual pore blocking (standard pore blocking)	$J = J_0 \left(1 + \frac{1}{2} K_S (A J_0)^{0.5} t\right)^{-2}$	(13)	$\frac{1}{\sqrt{J}} = \frac{1}{\sqrt{J_0}} + k_s t \quad k_s = 0.5 K_s A^{0.5}$	(17)
Intermediate filtration	$J = J_0 (1 + K_i A J_0 t)^{-1}$	(14)	$\frac{1}{J} = \frac{1}{J_0} + k_i t \quad k_i = K_i A$	(18)
Cake filtration	$J = J_0 (1 + 2 K_c (A J_0)^2 t)^{-0.5}$	(15)	$\frac{1}{J^2} = \frac{1}{J_0^2} + k_c \quad k_c = 2 K_c A^2$	(19)

conductivity of the solution at 50°C (Fig. 3). Since the presence of milk powder affects the experimental value of CMC of the detergent, the conductivity was measured dropping a mixed wastewater solution (using an extreme high concentration of 55 g dm^{-1} model wastewater). The CMC value of the model wastewater was obtained at 22.38 g dm^{-3} at 50°C as shown in Fig. 3.

3.2. The ultrafiltration fluxes

During our UF experiments, the ultrasonication from the feed side was used as described in the materials and methods, since Kyllönen et al. found that this kind of configuration is more effective in ultrafiltration, than the irradiation from the permeate side [25].

In the ultrafiltration experiments model dairy wastewater was tested. The ultrafiltration membrane fluxes without ultrasonication, (no US, Fig. 4), ultrafiltration with continuous ultrasonication (with continuous US), and with half intermittent ultrasonication (with half intermittent US) were primarily studied. Studies were carried out with 20 and 50 kDa MWCO PES membranes and the relative fluxes (J_{rel}: calculated with Eq. (1)) are shown in Fig. 4. The highest average flux was observed with continuous ultrasonication. It is generally accepted that the US increases the flux primarily by changing the polarization layer and decreasing the solute concentration at the membrane surface [26]. It is interesting to note that the flux increasing effect of ultrasonication was more pronounced using 50 kDa membrane.

The linearized filtration laws (Eqs. (16)–(19)) were plotted against measured data (Fig. 4). It was found that the best correlations were achieved by plotting the cake filtration model, but the other models the gradual pore blocking and intermediate filtration models also have given relatively good correlations
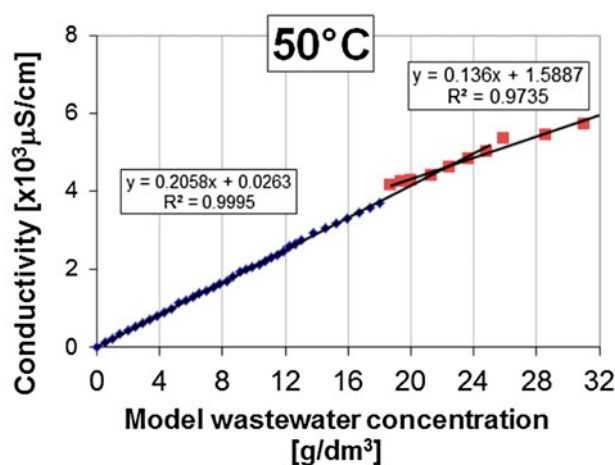
Fig. 3. Determination of CMC by conductivity measurements at 50°C.

using Eqs. (13) and (14); R^2 varies between 0.90 and 0.98. It suggests that the ultrafiltration of model dairy wastewater is not squarely gel layer controlled, the real mechanisms is probably more complex. Based on Eqs. (9)–(19), the mass transfer coefficients and the fluxes of the cake filtration model were calculated. As Fig. 4(a) and (b) show, the simulated flux data are in a good correlation with the measured data. The mass transfer coefficients in the mixed cell increased with ultrasonication and a higher K_c value was observed with continuous ultrasonication (Table 2). The fouling coefficient, k_c decreased with ultrasonication and continuous ultrasonication had the lowest value in case of both membranes. The calculated concentration on the membrane surface (c_M) increased with ultrasonication. The conductivity retention increased with ultrasonication and half intermittent ultrasonication had the highest value; except of continuous ultrasonication using 50 kDa membrane the concentration at the surface of the membrane reached the CMC value.
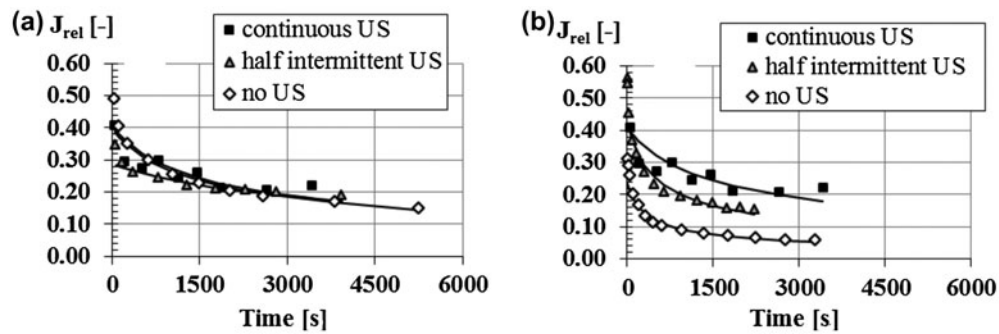
Fig. 4. Ultrafiltration membranes relative fluxes: (a) 20 kDa membrane and (b) 50 kDa membrane; $T = 50°C$; TMP = 0.1 MPa). Continuous lines represent the fitting results of fouling models.

Table 2
Calculated mass transfer coefficients and fouling coefficients in a stirred or ultrasonically mixed batch cell

Membrane (kDa)	Ultrafiltration	K_c (m s^{-1})	k_c (m^4 L^{-6} h^{-1})	R_{cond} (%)	c_M (wt%)
PES20	Without ultrasonication	1.48×10^{-5}	1.67×10^{-2}	10.35	20.33
	With half intermittent ultrasonication	2.83×10^{-5}	1.20×10^{-2}	16.53	23.47
	With continuous ultrasonication	4.83×10^{-5}	9.90×10^{-3}	14.48	23.78
PES50	Without ultrasonication	1.48×10^{-5}	4.95×10^{-3}	3.59	20.28
	With half intermittent ultrasonication	4.23×10^{-5}	1.15×10^{-3}	7.86	23.17
	With continuous ultrasonication	4.42×10^{-5}	5.74×10^{-4}	7.01	21.97

3.3. The ultrafiltration resistances

The membrane resistances (R_M), fouling resistances (R_F), polarization layer resistances (R_P), and total resistances (R_T) were calculated using Eqs. (2)–(5). It is obvious from the data in Fig. 5, that with both membrane types the total resistances decreased with ultrasonication and the continuous ultrasonication had more pronounced effects. A comparison of the calculated R_T and the k_c indicated that these parameters decreased with increasing ultrasound intensity. The retention results shown in Fig. 6 support this statement.

From our measurements it was obvious that in the ultrafiltration laboratory stirred cell device the resistances might have a noticable impact on filtration, but the ultrasonication effect can vary the values of R_F and R_P from lot-to-lot. This results also show that (similarly than it was found by plotting filtration models) in case of ultrasonically enhanced ultrafiltration the mechanism of filtration is complex, to find an exact model to describe the flux decline in the function of the time further examinations are necessary.

3.4. The ultrafiltration retentions

Fig. 6 shows the COD retentions (R_{COD}: calculated with Eq. (6)) of the ultrafiltration experiments without US, with half intermittent US and with continuous US modes. The retentions increased from 53.1 to 65.4% and to 74.4% using half intermittent and continuous ultrasonication, respectively. The observed COD rejections were found to be in accordance with the results obtained from c_M results (concentration on the membrane surface). The c_M values are important to characterize the back diffusion of the accumulated particles on the surface of the membrane which are important in determining the membrane rejections. In Table 2 it was found that the c_M values were higher than the CMC value of the model wastewater (22.38 g dm^{-3}) in the case of both ultrasonication with 20 kDa membrane and half intermittent US with 50 kDa. In these cases, the rejections of the COD were also higher than without ultrasonication. These results could be explained in terms of concentration polarization effects, since the formed more dense accumulated layer rejected the particles more efficiently.
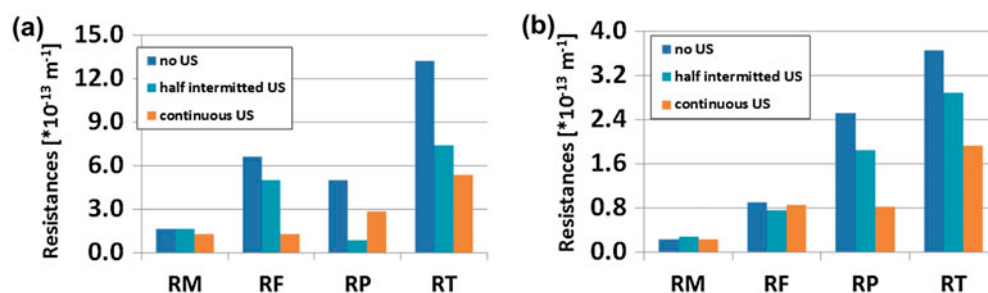
Fig. 5. Filtration resistances during ultrafiltrations: (a) 20 kDa membrane and (b) 50 kDa membrane; $T = 50\,°C$; TMP = 0.1 MPa; R_M: membrane resistances; R_F: fouling resistances; R_P: polarization layer resistances.
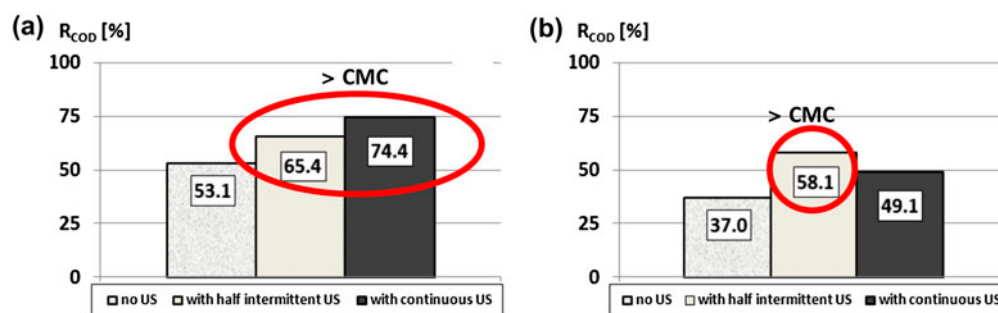

Fig. 6. COD retentions: (a) 20 kDa membrane and (b) 50 kDa membrane; $T = 50\,°C$; TMP = 0.1 MPa.

4. Conclusions

In this study, the feasibility of a hybrid process of membrane filtration combined with ultrasonication for dairy model wastewater treatment was investigated. From filtration law models it was found that the best correlation were achieved by plotting the cake filtration model, but the other models also have given relatively good correlations. It suggests that the ultrafiltration of model dairy wastewater was not squarely gel layer controlled, the real mechanisms is probably more complex.

However, dead-end stirred cell device is commonly used in laboratories to characterize microfiltration and ultrafiltration membranes, ultrasonication efficiency and filtration model classification should be further investigated for future applications in filtration operations. Furthermore, the use of the intermittent mode compared to the continuous mode to apply ultrasound irradiation should be explored in order to minimize energy consumption, with the aim to develop this technology in industrial scale.

Acknowledgments

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