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Nanofiltration of sweet whey by spiral wound organic membranes: Impact of hydrodynamics

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Abstract — The influence of permeation flux J and wall shear stress τ_w , on performances of the nanofiltration of concentrated sweet whey at volume reduction ratio 2, 18 °C and using organic membranes was studied. It was concluded that sweet whey nanofiltration could be optimised through the parameter J/τ_w , since J and τ_w act together in a competing mechanism of convection/erosion at the membrane surface, that governs membrane fouling and selectivity. Hence, the overall fouling was strongly dependent on the ratio J/τ_w , with a critical J/τ_w value of approximately 6.0. When $J/\tau_w < 6.0$, the total membrane fouling was relatively low and almost independent on operating conditions. For higher ratios of J/τ_w , there was a spectacular increase in fouling and a very strong dependence between the fouling index and J/τ_w . Simultaneously, selectivity was altered under these conditions, with higher retention of monovalent ions and lower retention of lactose. Finally, optimal hydrodynamic conditions were defined in the range 3–6. Under these conditions, longer operating times, lower chemical oxygen demand in permeate and higher demineralization could be expected at lower processing costs.

nanofiltration / hydrodynamic / sweet whey / fouling

1. INTRODUCTION

Nowadays, NF (membrane nominal molecular weight cut-off ranging between 0.2 and 1) is widely used in the dairy industry for the concentration and selective dem-

ineralization of acid and sweet whey. There would be about 150 000–200 000 m² of NF membranes installed for the dairy industry all over the world, either by substituting NF for RO membranes or as totally new equipment. NF membranes which are suitable for

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dairy applications generally exhibit a high permeability for monovalent ions (between 40–90%), and a low or very low permeability for multivalent ions (between 5–20%) and organic compounds (proteins, lactose, urea) [12, 17]. However, NF membrane operation is not always efficient, and there are frequently problems with selectivity (losses of lactose in the permeate), and with productivity (fouling, reduction of the duration of functioning) [4].

In most practical applications with pressure-driven processes, fouling of the membrane occurs during the process, and this represents one of the major operating problems resulting in the reduction of selectivity and productivity of the process. The general consensus is that a surface layer (also called dynamic membrane) is formed on the membrane surface during the operation, and once this surface layer is formed, it regulates the permeability and the selectivity of the membrane, regardless of its intrinsic properties [13]. In crossflow filtration of whey using organic membranes, the most important fouling components are the whey proteins, which adsorb to the membrane and inside the pores [16]. Whey minerals such as calcium and phosphate cause severe fouling, especially in the case of acid whey, due to the presence of higher contents of insoluble salts (i.e. colloidal calcium phosphate) [5, 16]. Lactose, small nitrogenous components, like peptides, urea, amino acids, and whey lipids, have only a minor contribution to fouling.

Some studies have demonstrated that the hydrodynamic operating conditions of membrane crossflow processes are the most important parameters responsible for its adequate performance. In this way, at a constant VRR, the permeation flux (J) governs mass transport by convection towards the membrane, whereas the wall shear stress (τ_w) governs the transport by erosion, hydrodynamic diffusion of solutes from the membrane towards the feed fluid. Therefore, performance of the process (selectivity and

productivity) greatly depends on these parameters: J , τ_w and VRR and hence, at a constant VRR, on the ratio J/τ_w , which is characteristic of the membrane, the fluid, and independent of the characteristics of the installation.

Considering that only few studies have been related to the role of J/τ_w on NF membrane performance, our purpose was to determine an optimum J/τ_w for the best NF separation features of sweet whey using spiral wound organic membranes. In order to simulate the behaviour of one stage of an industrial multistage NF plant, each experiment was carried out at constant volume reduction ratio (VRR), temperature, J and τ_w .

2. MATERIAL AND METHODS

2.1. Nanofiltration

2.1.1. Experimental equipment

The NF unit was an U01-Lab installation (Filtration Engineering, New Hope, Minnesota, USA) equipped with a Desal 5 (DK 3840 C, Osmonics, Minnetonka, Minnesota, USA) spiral wound NF membrane, with a total area of 7.9 m² and a molecular weight cut-off of 200 g·mol⁻¹ [10].

2.1.2. Cleaning procedure

Cleaning of the installation and the membrane was performed before and after NF experiments as described by Jeantet [9]. The cleaning procedure was repeated until the resistance of the clean membrane (R_m) was recovered ($\pm 5\%$).

2.1.3. Whey concentration

Emmental cheese whey obtained from a local dairy plant (Préval, Montauban, France) was concentrated to a VRR 2 at 45 ± 1 °C, flow velocity 1.1 m·s⁻¹, and TP between $25\text{--}30 \times 10^5$ Pa. The concentrated

whey was then cooled to 2–4 °C and sodium azide (0.2 g·kg⁻¹) was added in order to prevent microbial growth.

The composition (g·kg⁻¹) of the concentrated whey (VRR 2) used in the experimental study is shown in Table I. The pH of all whey samples immediately after concentration was 6.2 ± 0.2, and did not change to any appreciable extent during the different NF experiments.

2.1.4. Nanofiltration experiments

Experiments were performed at 18 ± 1 °C and VRR constant (VRR 2) by recycling both the permeate and the retentate into the feed tank until a filtered volume (FV) of 20 L·m⁻². Twenty-five experiments were performed for J/τ_w ratios within the range 1.0 and 9.3, corresponding to J and τ_w lying between 10–30 L·h⁻¹·m⁻² and 3.1–11.6 Pa, respectively.

The wall shear stress (τ_w , Pa) was calculated as follows:

$$\tau_w = \frac{\Delta P \cdot d}{4 \cdot l}$$

where, ΔP = pressure drop across the membrane (Pa) ranging between 0.15×10^5 and 0.65×10^5 Pa during the experiments; l = length of the module (m), and d = hydraulic diameter of the membrane (m), calculated according to van Gauwbergen and Baeyens [19].

2.1.5. Assessment of fouling

The hydraulic resistances of the clean membrane (R_m), the overall fouling (R_f), the irreversible fouling (R_{if}) and the reversible fouling (R_{rf}) were calculated according to Daufin et al. [3].

2.2. Physicochemical analyses

The pH of samples was determined at 20 °C on a Radiometer pH-meter. Total solids content was determined according to

Table I. Composition (g·kg⁻¹) and mineral content (g·kg⁻¹) of the concentrated sweet whey (VRR 2) used for nanofiltration experiments.

pH	6.2 ± 0.2
Total solids	127.2 ± 2.7
Protein	17.1 ± 0.4
NPN	3.4 ± 0.1
Ash	8.1 ± 0.1
Lactose	102.2 ± 1.9
Ca ²⁺	0.67 ± 0.04
Mg ²⁺	0.15 ± 0.01
Na ⁺	0.62 ± 0.01
K ⁺	2.41 ± 0.05
Cl ⁻	1.25 ± 0.04

the standard method by desiccation at 102–105 °C for 7 h [7]. Total protein and non-protein nitrogen (NPN) were determined by the Kjeldahl procedure [8].

Ash was determined by incineration of 10 mL of sample at 550 °C for 4 h [1]. The mineral content (Ca²⁺, Mg²⁺, K⁺, Na⁺) of the samples were quantified by atomic absorption spectrometry on a Varian AA 300 equipment (Sunnyvale, USA), according to the method described by Brulé et al. [2]. Chloride content was determined directly on a Corning Model 926 equipment (Humeau, La Chapelle, France).

Lactose content of the samples was determined from mass balance (total solids – ash – protein). The chemical oxygen demand (COD) of the permeate was determined by using a LCK 014 DCO test (Dr. Lange, Düsseldorf, Germany).

Solute retention (R) by the membrane was calculated as follows:

$$R (\%) = \frac{C_r - C_n}{C_n} \times 100$$

where C_r = concentration of solute in the retentate and C_n = concentration of solute in the nanofiltrate.

2.3. Economic analysis

NF processing cost, including operating (without cleaning) and capital costs, was estimated for each couple (J, τ_w) on the basis of a NF plant functioning in recirculation mode at constant VRR 2. This plant is supposed to produce a permeate flow rate \dot{V}_P ($\text{m}^3 \cdot \text{s}^{-1}$) with a total membrane area A (m^2), depending on the constant permeate flux value J :

$$A = \frac{\dot{V}_P}{J}.$$

The hydraulic setup includes:

- a positive feed pump delivering a flow rate $\dot{V}_F = 2 \dot{V}_P$ ($\text{m}^3 \cdot \text{s}^{-1}$) under the NF operating pressure TP (Pa) required to keep J constant;

- a recirculating pump on the retentate loop, delivering a flow rate \dot{V}_R ($\text{m}^3 \cdot \text{s}^{-1}$) under the pressure drop ΔP (Pa) required to keep τ_w constant.

The power P_p (W) required for this setup is given by:

$$P_p = \frac{\dot{V}_F \cdot TP}{\xi_F} + \frac{\dot{V}_F \cdot \Delta P}{\xi_R}$$

with $\xi_F = 0.5$ and $\xi_R = 0.5$, the global efficiencies of the feed pump and the recirculating pump, respectively. The corresponding energy consumption per permeate volume E_p ($\text{J} \cdot \text{m}^{-3}$) is:

$$E_p = \frac{P_p}{\dot{V}_P}.$$

This energy input will induce a rise in temperature of the recirculating retentate, that should be cooled by a heat exchanger to keep temperature constant. The cooling plant energy consumption per permeate volume E_C ($\text{J} \cdot \text{m}^{-3}$) can be estimated from:

$$E_C = E_p \times \frac{\xi_E}{\xi_C}$$

with $\xi_E = 0.9$ and $\xi_C = 2$, the pump engine efficiency and the cooling plant global effi-

ciency, respectively. The total operating costs OC ($\text{\$} \cdot \text{m}^{-3}$) is then:

$$OC = \left(\frac{E_p + E_C}{3600} \right) \times e$$

where e is the electricity cost ($0.05 \text{ \$/kWh}^{-1}$). On the other hand, capital costs CC ($\text{\$} \cdot \text{m}^{-3}$) are estimated from the following:

$$CC = \frac{PC \times CF}{t}$$

where PC is the plant cost ($\text{\$} \cdot \text{m}^{-3} \cdot \text{h}$), CF the capital factor (yr^{-1}) and t the total operating time ($\text{h} \cdot \text{yr}^{-1}$). PC is a function of the fixed costs (minimal configuration and automation), the total membrane area A , and the membrane and equipment cost: this lies between 1000 and 560 $\text{\$} \cdot \text{m}^{-2}$ for A in the range 30 to 100 m^2 , respectively. CF includes depreciation, interest and maintenance, and is equal to 0.4 yr^{-1} .

The greater the membrane fouling, the lower the total operating time because of longer periods of membrane cleaning. Arbitrarily considering a 10% decrease of operating time per increase of 1 of R_f/R_m , t was calculated on the basis of a theoretical operating time of 8 000 $\text{h} \cdot \text{yr}^{-1}$.

3. RESULTS

3.1. Assessment of fouling

Figure 1 shows the overall fouling (R_f/R_m) at a FV of $20 \text{ L} \cdot \text{m}^{-2}$ as a function of J/τ_w , each point corresponding to one of the 25 experiments. The hydraulic resistances of the clean membrane (R_m) were between 9.1×10^{13} and $9.8 \times 10^{13} \text{ m}^{-1}$, and did not change to any appreciable extent throughout the experimental study. The reversible fouling (R_f/R_m) accounted for 92–98% of the overall fouling.

The evolution of R_f/R_m as a function of J/τ_w could be divided into two parts around a critical J/τ_w value of 6.0. The first part

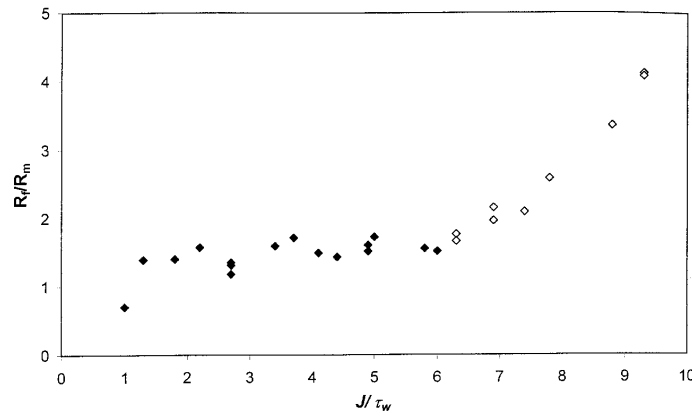


Figure 1. Normalized overall fouling hydraulic resistance (R_f/R_m) as a function of the J/τ_w ratio, for the nanofiltration processes of concentrated sweet whey. VRR 2; 18 °C; FV 20 L·m⁻².

($J/\tau_w < 6.0$), corresponded to an overall fouling of relatively little importance (R_f/R_m between 0.7 and 1.5), which slightly increased with J/τ_w (Fig. 1).

Conversely, for higher J/τ_w ratios (> 6.0), there was a very strong dependence between total fouling at the membrane and the ratio J/τ_w : the higher the J/τ_w , the higher the R_f/R_m . Indeed, there was a 2.7 fold increase of R_f/R_m from J/τ_w 6.0 to J/τ_w 9.3 (Fig. 1).

3.2. Selectivity of the process

The J/τ_w conditions greatly altered the selectivity of the membrane as shown by large variations in permeate sample composition (Tab. II). Higher losses (20%) of NPN in the permeate were obtained for lower J/τ_w ratios (J/τ_w 1.0). Likewise, the higher the J/τ_w , the higher the lactose loss in the permeate. Losses of lactose were 0.4 and 1.2 g·kg⁻¹ for J/τ_w 1.0 and 9.3, respectively, equivalent to 0.4 and 1.1% of the raw concentrated whey. Consequently, the highest permeate COD values were obtained in the permeate at the highest J/τ_w (Tab. II).

Divalent cations (Ca²⁺ and Mg²⁺) were almost completely retained whatever the operating conditions used ($R = 97$ – 99% and $R = 100\%$ for Ca²⁺ and Mg²⁺, respectively) (Fig. 2). However, a higher rejection of

monovalent ions was obtained for higher J/τ_w ratios. Rejection of Na⁺ and K⁺ increased from about 15%, and the rejection of Cl⁻ from about 23%, between J/τ_w 1.0 and 9.3. As a result of the differences found between the monovalent mineral rejection in the experimental study, the ash content of the permeate samples obtained at a FV 20 L·m⁻² was higher when a lower J/τ_w ratio was used (J/τ_w 1.0 and 3.4, Tab. I).

3.3. Economic analysis

Costs were minimized for J/τ_w ratio in the range 3.0–6.0, at approximately 2 \$·m⁻³ (Fig. 3). Under J/τ_w 3, capital costs were increased because of higher total membrane area A (low J value). Above J/τ_w 6, operating and capital costs were increased because of higher operating pressure (high J value) and lower operating time t (higher membrane fouling), respectively.

4. DISCUSSION

Fouling occurring during crossflow membrane separation processes of dairy products is a very complex phenomenon, especially in the case of whey, due to the large diversity of dairy components participating in fouling. Generally speaking, the reversible

Table II. Composition ($\text{g}\cdot\text{kg}^{-1}$), chemical oxygen demand (COD, $\text{mg}\cdot\text{kg}^{-1}$) and mineral content ($\text{mg}\cdot\text{kg}^{-1}$) of the permeate samples obtained from the nanofiltration processes of sweet concentrated whey (VRRR 2), as a function of the J/τ_w ratio (18 °C; FV 20 $\text{L}\cdot\text{m}^{-2}$).

J/τ_w	Ash	NPN ^a	Lactose	TS ^b	COD	Ca ²⁺	Mg ²⁺	Na ⁺	K ⁺	Cl ⁻
1.0	2.3	0.7	0.4	3.6	500	17.8	3.3	252.1	847.4	1126.2
3.4	2.4	0.5	0.7	3.9	625	16.8	3.2	270.8	869.4	953.6
6.3 ^c	1.9 ± 0.2	0.5 ± 0.0	0.8 ± 0.1	3.3 ± 0.4	600 ± 20	14.8 ± 1.7	2.9 ± 0.6	218.5 ± 46.3	714.5 ± 154.9	786.7 ± 130.9
9.3	1.7	0.6	1.2	3.6	1100	14.4	3.3	168.0	505.1	882.1

^a NPN: non-protein nitrogen. ^b TS: Total solids. ^c Means and standard deviations of 3 trials.

part of fouling based on the concentration polarization (CP) phenomenon is dominant during whey nanofiltration. In the present study, reversible fouling (which involves CP phenomenon) accounted for 92–95% of the overall fouling. Thus, the control of fouling of sweet whey nanofiltration implied the control of the CP phenomenon. This can be understood as the result of the accumulation of the retained species and of the depletion of the permeating components in the boundary layer adjacent to the membrane, which causes a concentration gradient build up on the latter. Thus, in the present study, from the analysis of nanofiltrate composition and comparing the experimental retentions of whey components, the CP layer was expected to consist mainly of divalents salts, lactose and proteins. Consequences of the CP build up could be the following. Firstly, the boundary layer characteristics would result in an intrinsic mass transfer coefficient, related to the solute diffusivity within the boundary layer, the boundary layer thickness and compaction. This coefficient would control the alteration of flux and selectivity of the fouled NF membrane. Secondly, the over-concentration of negatively charged whey proteins in the CP layer would increasingly trap small inorganic counter ions around the proteins and simultaneously small inorganic co-ions would be electrically repelled. Thirdly, the over-concentration of lactose would enhance the leakage of lactose into the permeate by diffusion across the NF membrane. And finally, as soon as the calcium phosphate concentration at the membrane surface exceeds saturation, precipitation is liable to occur. This would then cause a far more severe fouling as shown by Kelly et al. [11]. Some authors have even linked the increase of the fouling index, due to the formation of a CP layer, to the increase of precipitation of calcium phosphate at the membrane surface [14].

Methods to reduce the CP phenomenon by a proper choice of membranes and installation [20], by product pretreatment [5–6]

Figure 2. Percentage of rejection R (%) of mineral species during the nanofiltration process of concentrated whey (VRR 2), at 18 °C and for different ratios J/τ_w (Ca^{2+} : ●; Mg^{2+} : ○; Na^+ : □; K^+ : ■; Cl^- : ◆).

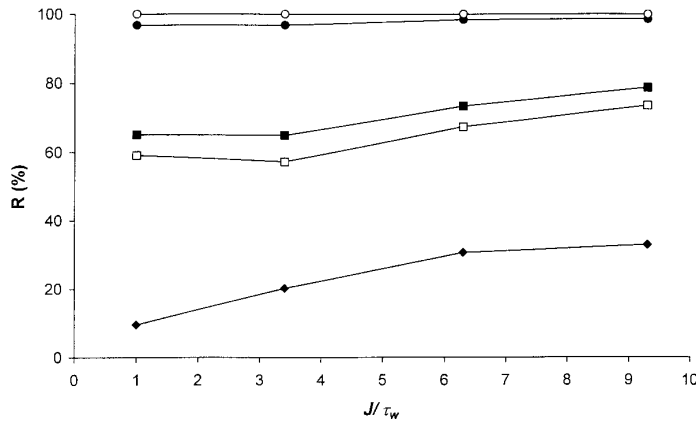
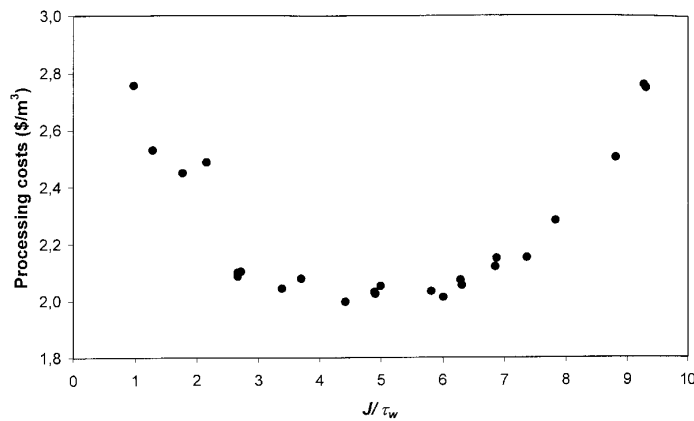


Figure 3. Nanofiltration estimated processing costs ($\$/\text{m}^3$) as a function of the J/τ_w ratio. VRR 2; 18 °C; FV $20 \text{ L}\cdot\text{m}^{-2}$.



or by the control of the operating variables [4, 13, 15] are largely described in the literature. Results of the present study highlighted the crucial influence of the ratio between the permeation flux J and the wall shear stress τ_w on the fouling index and on the alteration of process selectivity during the NF of sweet whey. From the knowledge being gathered today in every area of membrane liquid separation, it could be assumed that the two parameters J and τ_w act together in a competing mechanism of convection/erosion. The higher the shear rate, the larger the amount of matter scrapped off the fouling layer. The higher the permeate flux, the larger the amount of matter brought

towards the membrane surface and hence the higher the concentration profile in the CP layer. The competition of both parameters, through the J/τ_w ratio, would thus control the following characteristics of the CP layer: concentration profile, thickness, compaction and solute diffusivity, and hence, the mass transfer coefficient within the boundary layer.

So, the present results can be understood with regard to the competing mechanism of convection/erosion. As expected from literature, the fouling index was essentially linked to the formation of a CP layer and this constantly increased with J/τ_w . As long as J/τ_w was under a critical value of around 6,

the fouling index increased slowly which indicated that mass transport by erosion/diffusion was greater than mass transport by convection. A more compact, thinner CP layer was then expected to be formed, which would be probably less able to capture small solutes. Then, as soon as a critical J/τ_w was reached (around 6.0), R_f/R_m increased drastically. Some nanofiltration runs even have to be stopped for the highest values of J/τ_w . Under these hydrodynamic conditions, the mass transport by convection towards the membrane was far more important than the mass transport by erosion/diffusion, and hence a thicker and looser CP layer settled fast. Because of its specific presumed structure, it was expected to be more liable of capturing the small solutes.

Hence, the selectivity of the NF membrane was greatly altered by J/τ_w . With respect to monovalent ions, rejection increased for higher J/τ_w ratios. The interpretation of these results rests on the assumption that the small counter ions of negatively charged whey proteins (Na^+ and K^+) were trapped electrically around the proteins, and over concentrated in the CP layer. On the other hand, the small co-ions of the proteins (Cl^-) were rejected by the negative interface. So, the rejections of small salts increased as the concentration profile of proteins in the vicinity of the membrane increased. The same results were obtained by van der Horst et al. [17] during the NF of model solutions and UF whey permeate.

Opposite results were observed for lactose, since the concentration in the permeate increased when J/τ_w increased. The fouled membrane seemed to be more permeable to lactose. Losses of lactose in the permeate were also reported by Kelly and Kelly [11] using NF organic membranes. It is reasonable to assume that lactose leakage in the permeate was based either on diffusion through the membrane or on convection in the largest pores of the membrane, depending on permeate flux. Whatever the transport mechanism involved, the higher the

gradient of lactose concentration across the membrane and hence, the higher the CP, the greater the amount of lactose transferred in the permeate. Moreover, as assumed before, the presumed loose structure of CP layer under these conditions, would increase the diffusivity of lactose within the boundary layer. So, high values of J/τ_w would have serious repercussions on the polluting power of the nanofiltration process. Indeed, the COD of the nanofiltrate is directly related to the lactose transmission [18]. Losses of organic solids from whey in terms of COD, were similar to those reported by Kelly and Kelly [12] during the NF of acid whey.

To conclude with, the synthesis of these results leads to optimal hydrodynamic operating conditions at VRR 2 and 18 °C: for a permeate flux equal to $30 \text{ L}\cdot\text{h}^{-1}\cdot\text{m}^{-2}$, the optimal τ_w stands in the range of 6.0–10.0 Pa, corresponding to pressure drops from 0.30×10^5 to 0.50×10^5 Pa per element. Under these conditions, longer operating times, lower permeate COD and higher demineralisation can be expected at lower processing costs.

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List of symbols

A	Total membrane area (m^2)	PC	Plant cost ($\$/m^3 \cdot h$)
CC	Capital costs ($\$/m^3$)	P_p	Installation power (W)
CF	Capital factor (yr^{-1})	R	Solute retention (%)
C_n	Nanofiltrate solute concentration ($g \cdot kg^{-1}$)	R_f	Overall fouling hydraulic resistance (m^{-1})
COD	Chemical oxygen demand ($mg \cdot kg^{-1}$)	R_{if}	Irreversible fouling hydraulic resistance (m^{-1})
CP	Concentration polarization	R_m	Clean membrane hydraulic resistance (m^{-1})
C_r	Retentate solute concentration ($g \cdot kg^{-1}$)	R_{rf}	Reversible fouling hydraulic resistance (m^{-1})
d	Hydraulic diameter of the membrane (m)	t	Total operating time ($h \cdot yr^{-1}$)
e	Electricity cost ($0.05 \$/kWh^{-1}$)	TP	Transmembrane pressure (Pa)
E_C	Cooling plant energy consumption per permeate volume ($J \cdot m^{-3}$)	VRR	Volume reduction ratio
E_p	Pump energy consumption per permeate volume ($J \cdot m^{-3}$)	\dot{V}_F	Positive feed pump flow rate ($m^3 \cdot s^{-1}$)
FV	Filtered volume ($L \cdot m^{-2}$)	\dot{V}_P	Permeate flow rate ($m^3 \cdot s^{-1}$)
J	Permeation flux ($m^3 \cdot s^{-1} \cdot m^{-2}$)	\dot{V}_R	Recirculating pump flow rate ($m^3 \cdot s^{-1}$)
l	Module length (m)	ξ_C	Cooling plant global efficiency
NPN	Non-protein nitrogen ($g \cdot kg^{-1}$)	ξ_E	Pump engine efficiency
OC	Total operating costs ($\$/m^3$)	ξ_F	Feed pump efficiency
ΔP	Pressure drop across the membrane (Pa)	ξ_R	Recirculating pump efficiency
		τ_w	Wall shear stress (Pa)