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Concentration of skim milk using a hybrid system of osmotic distillation and membrane distillation

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ABSTRACT

For several dairy processes, concentration of milk by removing its water content at certain proportions is a principal step. In this study, milk is concentrated using osmotic distillation. The effect of operation parameters including draw solution concentration, temperature, feed and draw solution rates on flux was assessed. The experimental data were compared with the simulated data and it was shown that theoretical results were coherent with the experimental ones. To avoid brine dilution during osmotic distillation, a hybrid process of osmotic distillation and direct contact membrane distillation was proposed to concentrate milk and brine simultaneously. Final brine concentrations and consequently fluxes obtained with hybrid process were higher than the fluxes obtained with osmotic distillation. The proposed osmotic distillation–membrane distillation hybrid system is promising for concentration of liquid food and dairy products such as milk and whey.

Keywords: Osmotic distillation; Membrane distillation; Milk concentration; Membrane contactors; Brine recovery

1. Introduction

Milk is conventionally concentrated using multi-stage evaporators to provide microbiological and chemical stability. For the evaporated milk production the dry matter content in milk should be concentrated from 8%–12% to 20%–25% [1]. In dairy industry, milk concentration is an inevitable process to produce several dairy products such as cheese, milk powder, yogurt, etc. In these processes milk should be concentrated by removing its water content to a certain concentration depended on the product. For example, in milk powder process the water in milk must be eliminated until 50% dry matter content as a pretreatment step prior to spray drying [2]. Ramirez et al. [3] reported that in dairy industry the most energy intensive operation is the milk concentration which involves drying and evaporation. Generally milk concentration is achieved by thermal treatments. However, as well as being an energy demanding process, conventional thermal evaporation may also have negative impacts on nutritious components, sensory parameters and technological properties such as loss of aroma compounds, taste and color degradation [4,5]. Hence non-thermal technologies such as membrane technologies for food processing have been attracting attention by food industry recently. Among the membrane processes, forward osmosis (FO) in an interesting application for concentrating foods, power generation, desalination and wastewater treatment in which the driving force is achieved by the difference between water activities across the membrane. FO is the process of the diffusion of water through a semi-permeable membrane from a solution with higher water concentration to an osmotic agent, while this process is usually named as osmotic distillation (OD) in particular when the process utilizes hydrophobic membrane contactors. OD includes a phase change step during the water transfer through the membrane because of the hydrophobic structure of the membrane contactor utilized [6]. The main advantage of the osmosis-based systems such as FO or OD is the low pressure requirement for the operation. This

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reduces the costs of high pressure equipment and electrical energy as well as membrane fouling phenomena. When it is compared with reverse osmosis, OD can treat the feed solutions until high solid contents whereas reverse osmosis can be used economically only for pre-concentration steps.

In the literature, OD has been reported for various fruit/vegetable juice concentration applications [4–14] including juices such as tomato [11], cactus pear [12], broccoli [13], cranberries [14], etc.; however milk concentration by means of OD was not investigated by the researchers. In literature, milk processing is usually achieved by membrane techniques such as reverse osmosis [15], ultrafiltration [16] and microfiltration [17,18]. The main disadvantages of these pressure-driven membrane filtration processes are the fouling and the requirement of high pressure equipment. On the other hand, lower pumping energy due to low hydraulic pressures, high solid retention and low membrane fouling tendency are the main advantages of osmotic-driven processes compared with pressure-driven membrane filtration systems. Although milk concentration by OD was not encountered in the literature specifically, there are few works about dairy product or whey processing by means of FO using hydrophilic membranes [19–21] and membrane distillation (MD) [22–24] using membrane contactors. MD is another application of membrane contactors relatively similar to OD. In MD, the driving force for the water transfer is provided by the temperature difference applied across the membrane while this driving force is the activity difference in OD. Hausmann et al. [22–24] investigated the performance of MD for milk and whey processing. They concentrated skim milk and whey in MD process and reported that dissolved solid retention was almost 100%. Moreover several studies in literature report that OD has several advantages over MD in terms of producing less temperature polarization and maintaining the nutrient content and volatile compounds in feed [9,25]. It may be more advantageous to use the OD for food concentration applications to preserve the nutrients while MD for applications such as desalination and water treatment. The main drawback of osmosis-based systems is the dilution of the brine stream in time because of the continuous water transfer to this solution. Since the brine concentration decreases in time, the activity difference across the membrane also decreases and as a result fluxes decrease [26]. To avoid this dilution, brine solution can be concentrated by a secondary process simultaneously during FO runs and thus FO can be operated more efficiently.

Osmosis-based systems can be integrated with MD to improve the performance of the concentration process and reduce the recovery costs of the brine stream. In this hybrid system, osmotic driven process (FO or OD) is used to concentrate the feed solution whereas MD is employed to concentrate the diluted brine solution simultaneously. The re-concentrated draw solution left the MD module is sent back to the OD/FO module, thus concentration of the feed and brine can be achieved at the same time by removing the water from the brine solution continuously. This configuration is very advantageous as it allows working at higher driving force in FO and allows treating the brine at the same time. FO–MD hybrid system is a newly establishing process coupling type and only a limited number of applications of this system were reported so far. In the literature, this configuration is applied for the treatment of human urine to produce fresh water [27], wastewater treatment containing acid dye [28], wastewater treatment containing arsenic [29], sustainable wastewater reuse [30], concentration of protein solutions [31], clean water production from sewer mining [32] and from digested sludge [33]. In all these applications, the authors indicated that with the coupling of MD to FO unit, the process became more efficient allowing the system to work at constant osmotic solution activity. The reports on the use of FO–MD hybrid system only include environmental or pharmaceutical applications although this configuration has a great potential in food applications. Furthermore the reported studies on the hybrid system involve FO process with hydrophilic membranes and the use of OD with hydrophobic membranes has not been investigated yet. In this paper, OD–MD hybrid system was applied for liquid food concentration, namely for milk processing. The preliminary experimental results obtained with OD and their comparison with MD was reported previously where the concentration of milk was found to be more advantageous in OD than in MD [34,35]. In the present work, a theoretical model was developed for mass transfer in OD and the use of OD–MD hybrid process was evaluated using several module type configurations.

2. Experimental

2.1. Materials

Milk was prepared using 9% wt. of milk powder purchased from local brand of Pınar Süt Mamülleri (Turkey). To avoid bacterial growth, 0.1% NaN_3 of dry matter was added in the milk solution which is purchased from Fisher Scientific (USA). Brine solution was prepared using dehydrated granular $CaCl_2$ purchased from Carlo Erba (Italy). NaOH used for cleaning membrane modules was purchased from Sigma-Aldrich (USA). Two different membrane contactor modules supplied from Germany (MC-1 and MC-2) were used in the experiments. The brands and the properties of the modules are reported in Table 1.

Table 1
Properties of membrane contactors used in this work

Membrane contactor (MC) modulus	MC-1	MC-2
Model	MEMBRANA Liqui-Cel® minimodule	MICRODYN® – MD 020 CP 2N
Membrane material	Polypropylene	Polypropylene
Number of fibers	7,400	40
Contact area, m^2	0.58	0.1
Outer diameter of fiber, μm	300	2,600
Inner diameter of fiber, μm	220	1,800
Mean pore diameter, d_p, μm	0.2	0.2
Length of fiber, L, m	0.12	0.47
Porosity, ε	0.4	0.7

2.2. Experimental setup

Milk was concentrated until its volume was reduced by half using OD individually. Direct contact membrane distillation (DCMD) was evaluated for brine concentration and after, OD and MD were integrated to form the hybrid process. In this hybrid process, milk was concentrated in OD part while brine was re-concentrated in MD part of the integrated system. Concentration of brine solution was in the range of 2–5 M $CaCl_2$. The temperature of the streams in the experimental work was between 12°C and 36°C.

2.2.1. Osmotic distillation

In OD experiments, feed solution of milk was circulated through the shell-side of the module, and the $CaCl_2$ draw solution was pumped through the lumen-side of PP hollow fiber by peristaltic pumps counter-currently. The temperatures of brine and feed solutions were adjusted using thermostatic baths. The volume reduction of feed was monitored by a volumetric flask every 10 min to estimate the fluxes. The experimental setup of OD process is given in Fig. 1.

2.2.2. Membrane distillation

MD tests were carried out as DCMD process. In DCMD, feed solution was pumped through the shell-side and the chilled water drafted from a chiller was circulated as distillate through the lumen side of hollow fiber module counter-currently. This way, water was transferred to the distillate stream under the temperature difference applied as the driving force. The temperature of the permeate was varied between 12°C and 20°C, while the temperature of feed was varied between 23°C and 36°C. Experimental setup of DCMD is given in Fig. 2. The flux was determined by weight reduction of feed every 10 min using a scale.

2.2.3. Osmotic distillation–membrane distillation coupling

OD was integrated with MD to run the system in hybrid mode. In this way, the diluted brine stream that left the OD module was sent to the MD module, concentrated here and sent back to the OD module. Thus concentration of milk and recovery of brine was achieved simultaneously. The concentration of brine could be controlled in this hybrid mode. The flux of water transferred from milk and from brine was monitored using a balance and volumetric flask. The experimental setup of OD–MD hybrid system is given in Fig. 3.

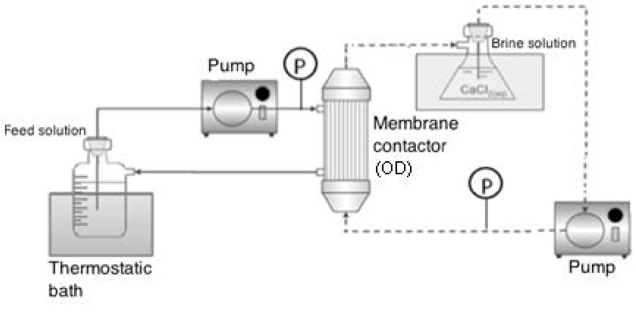

Fig. 1. Experimental setup of OD process.

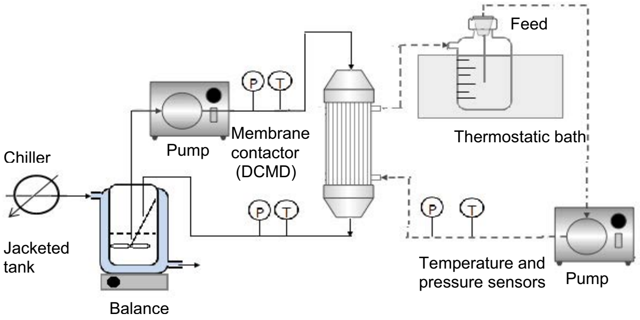

Fig. 2. Experimental setup of DCMD process.

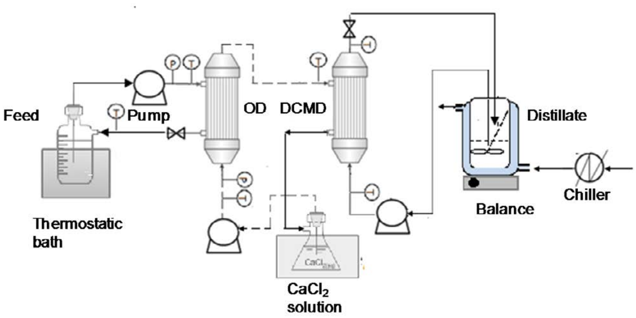

Fig. 3. OD–DCMD hybrid process.

3. Results and discussion

3.1. Mass transfer in osmotic distillation and model validation

In OD process, since the membrane material is hydrophobic, water cannot penetrate the pores in its liquid form and thus an interface forms between liquid and vapor phases at the pore entrance. Thus water vaporizes at the pore entrance, diffuses through the pore, and condenses on the other interface. The driving force is the water vapor pressure difference across the membrane induced by the activity difference, thus water flux through the membrane can be written as in Eq. (1):

$$J_w = k_{\mathrm{mp}} \times \Delta P_{w,m} = k_{\mathrm{mp}} \left(a_f^m \times P_{w,f}^* - a_b^m \times P_{w,b}^* \right) \tag{1}$$

In Eq. (1) $P^*_{w,f}$ and $P^*_{w,b}$ are the water vapor pressures, k_{mp} is the membrane permeability and a_f^m and a_b^m are the water activities of the feed and brine solutions at the membrane interface, respectively. If temperature polarization is neglected, the temperature can be assumed to be equal at both interfaces ($P^*_{w,f} = P^*_{w,b} = P^*$, pure water pressure), then Eq. (1) becomes [36,37]:

$$J_w = k_{\mathrm{mp}} \times P^* \left(a_f^m - a_b^m \right) \tag{2}$$

In the presence of concentration polarization, resistances of boundary layers in feed and brine side should be taken into account. In this case, water first transfers from bulk to the interface, vaporizes at pore entrance and diffuses through the pore, condenses at the other interface and transfers to the bulk solution at the other side. According to the resistance in series model, this transfer of water through the membrane involves three resistances; feed side boundary layer ($1/k_f$), membrane resistance ($1/k_m$) and brine side boundary layer ($1/k_b$) resistances where k_f, k_m and k_b represent the mass transfer coefficient of each step, respectively. The overall mass transfer coefficient, K, can be written as in Eq. (3):

$$K = \left(\frac{1}{k_f} + \frac{1}{k_m} + \frac{1}{k_b} \right)^{-1} \tag{3}$$

The activity profile of water through the membrane and the resistances involved in the mass transfer of water is illustrated in Fig. 4.

The fluxes through the boundary layers and the membrane pores relate with the following equation:

$$J_w = k_f \left(a_f - a_f^m \right) = k_b \left(a_b^m - a_b \right) = k_{\text{mp}} \times P^* \left(a_f^m - a_b^m \right) \tag{4}$$

Considering $k_m = k_{\text{mp}} \times P^*$, the combination of Eqs. (3) and (4) gives Eq. (5):

$$J_w = K \times \left(a_f - a_b \right) \tag{5}$$

According to Eq. (5), the flux through the membrane can be calculated by the product of activity difference across the membrane and the overall mass transfer coefficient. If a mass balance is performed over the boundary layer for the solute of the each solution, i, Eqs. (6) and (7) are obtained for feed and brine side, respectively, which give the concentrations on the membrane surfaces. These equations are governed from the mass balance around the boundary layer as the solute flow to the membrane interface due to the convection will be equal to the sum of solute flux through the membrane and the back diffusive flow from the membrane interface to the bulk [38]:

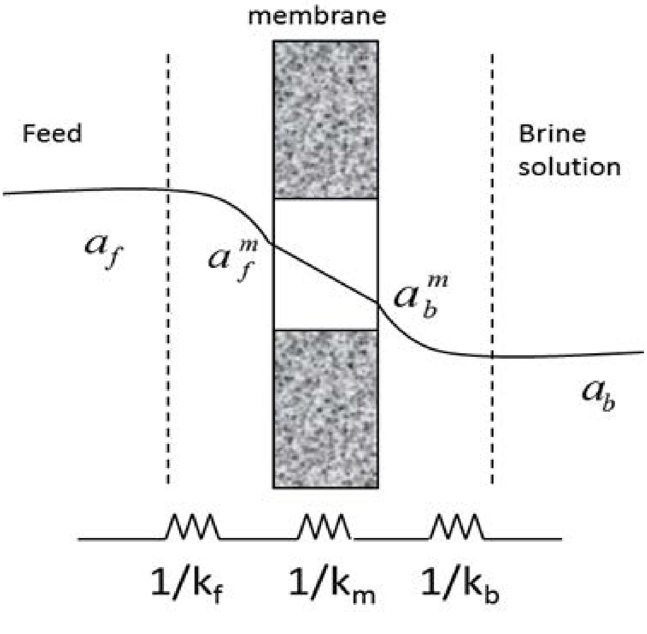

Fig. 4. Activity profile of water and mass transfer resistances in osmotic distillation.

$$C_{i,f}^m = C_{i,f} \times \exp\left(\frac{J}{k_f} \right) \tag{6}$$

$$C_{i,b}^m = C_{i,b} \times \exp\left(\frac{-J}{k_b} \right) \tag{7}$$

The individual mass transfer coefficient related to each boundary layer can be estimated using empirical correlations given for Sh, Re and Sc numbers. In this study, Eq. (8) [39] is used for the feed side which was circulated on shellside where $\text{Sh} = \frac{k \times D_h}{D_{\text{ab}}}$: $\text{Re} = \frac{\upsilon D_h \times \rho}{\mu}$ and $\text{Sc} = \frac{\mu}{\rho} \times D_{\text{ab}}$, D_h is the hydrodynamic diameter, D_{ab} is the water diffusion coefficient, k is the mass transfer coefficient, μ is the viscosity, υ is the velocity of the fluid and ρ is the density.

$$\text{Sh} = \beta \left[D_h \times \frac{1-\phi}{L} \right] \times \text{Re}^{0.6} \times \text{Sc}^{0.33} \tag{8}$$

Eq. (8) is valid for $0 < \text{Re} < 500$ and for the hydrophobic fibers $\beta = 5.8$. ϕ is the packing density given in Eq. (9) and L is the fiber length.

$$\phi = \text{Number of fibers} \left(\frac{\text{outer diameter of fiber}}{\text{inner diameter of the module}} \right)^2 \tag{9}$$

Diffusion coefficient of water in milk was between 1.53×10^{-6} and 1.87×10^{-6} in the temperature range of 25°C–35°C [40] and the viscosity of milk at the desired temperature was calculated using below correlation where F is the milk fat content [41]:

$$\ln \mu = \frac{2{,}721.5}{T} + 0.1 \times F - 8.9 \tag{10}$$

Brine side mass transfer coefficient was estimated using Eq. (11) [42].

$$\text{Sh} = 0.0149 \left(\text{Re}^{0.88} \right) \times \left(\text{Sc}^{1/3} \right) \tag{11}$$

The diffusion coefficient of water in $CaCl_2$ solution at different molarities (M) was calculated using Eq. (12) [43] where h is 0.18 for $CaCl_2$ and $D_{(H_2O)}$ is the self-diffusion coefficient of water of which values at different temperatures were taken from the literature [44].

$$D_{\text{ab}} = D_{(H_2O)} \left(1 - h \times M \right) \tag{12}$$

The viscosity of $CaCl_2$ solutions at different molarities and temperatures was taken from the literature [45]. After the estimation of mass transfer coefficients of each boundary layer using related equations, the mass transfer coefficient of the membrane was estimated. The estimation of mass transfer of water vapor in a porous membrane can be based on several transport models such as ordinary diffusion, Knudsen, Poiseuille or combination of them such as Scholfield and Dusty-Gas model [46]. In this study, mass transfer in the membrane pores was described by both Knudsen and Dusty Gas models to define the best transport model for the transfer. Knudsen diffusion coefficient is expressed as in Eq. (13) where M_w is the water molar mass, R is gas universal gas constant and the membrane mass transfer coefficient can be defined as in Eq. (14) where, ε is the porosity, δ_m is the membrane thickness and τ is the tortuosity.

$$D_{\mathrm{Kn}} = \frac{d_p}{3} \times \left(\frac{8 \times R \times T}{\pi M_w} \right) \tag{13}$$

$$k_m = \frac{D_{\mathrm{Kn}} \times \varepsilon}{\tau \times \delta_m} \tag{14}$$

Dusty-gas model has been described for the water vapor transfer in a porous media based on the kinetic theory of gases. According to Dusty-gas model, the porous media acts as a “dusty gas” component of the gas mixture. Babu et al. [36] gave the membrane mass transfer coefficient according to Dusty-gas model in which simultaneous effects of Knudsen diffusion (D_{kn}) and ordinary diffusion ($D_{w\text{-air}}$) were taken into account [36]:

$$k_m^{\mathrm{DG}} = \frac{1.8 \times 10^{-5} \times P^*}{R \times T \times \delta} \left(\frac{\tau}{\varepsilon \times D_{\mathrm{Kn}}} + \frac{\tau \times P_{\mathrm{air}}}{\varepsilon \times \mathrm{PD}_{w-\mathrm{air}}} \right)^{-1} \tag{15}$$

In Eq. (15), P_{air} is the partial pressure of air, P is the total pressure and the term $\mathrm{PD}_{w\text{-air}}$ for the air–water system can be estimated using the equation below [36]:

$$\mathrm{PD}_{w-\mathrm{air}} = 4.46 \times 10^{-6} T^{2.334} \tag{16}$$

As all the mass transfer coefficients are estimated, overall mass transfer coefficient can be calculated using Eq. (3). The activity difference in Eq. (5) can be calculated by estimating water activity in each solution. For the water activity calculation in milk, Oswald equation was used which gives the water activity in milk in terms of water content and temperature [47]. The activity of water in $\mathrm{CaCl_2}$ solutions was calculated applying modified ASOG contribution method proposed by Correa et al. [48]. The modified ASOG contribution method combines the UNIFAC method and Debye–Hückel theory. For the activity difference estimation, average of initial and final activities of water was taken for each solution (milk and brine) during the concentration process. The model was applied for both membrane contactors and the comparison of model and experimental results are given in Fig. 5.

Fig. 5 shows the comparison of model and experimental results. As can be seen in the figure, the model results are

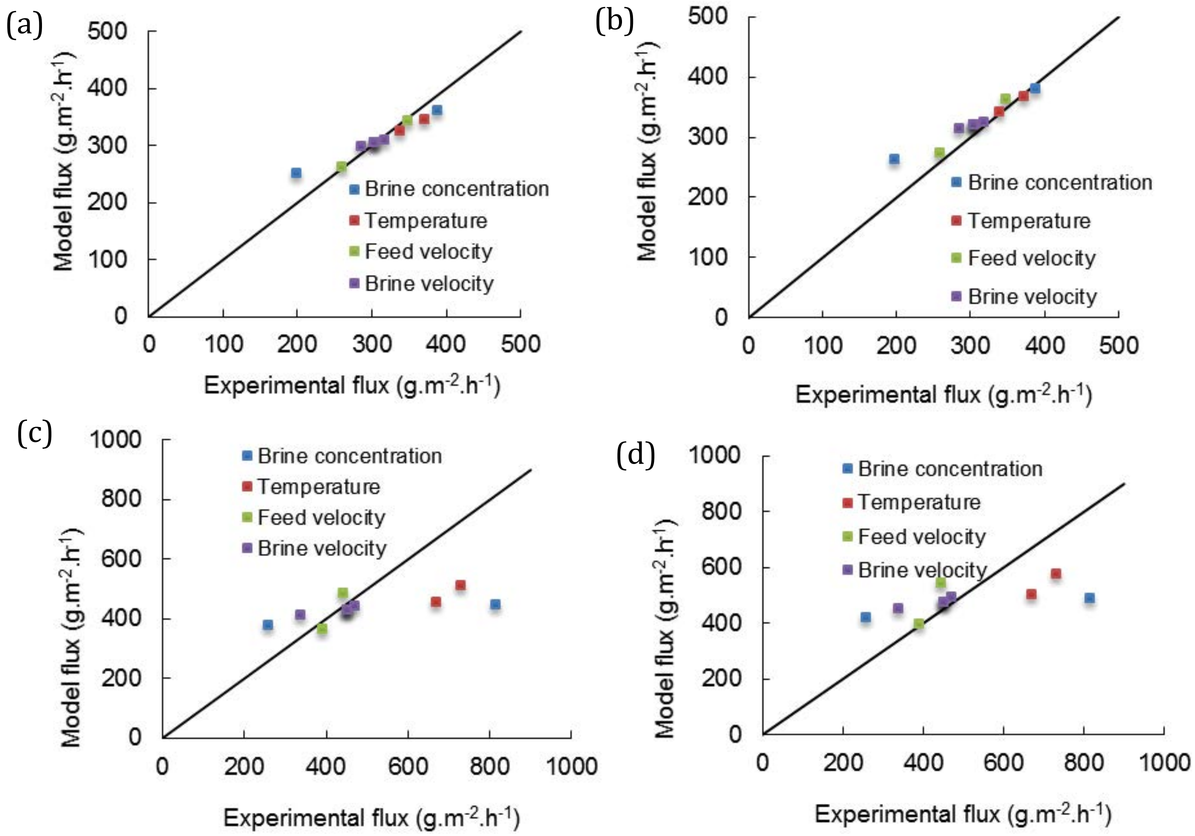

Fig. 5. Comparison of experimental-model results (a) dusty gas model in MC-1, (b) Knudsen model in MC-1, (c) dusty gas model in MC-2, and (d) Knudsen diffusion in MC-2.

coherent with the experimental results. Both Knudsen and Dusty models have a reasonable compatibility with experimental results, a significant difference was not observed between the two predicted results although some parameters such as velocity had better compatibility with Knudsen while some others such as temperature had better compatibility with Dusty-gas model. The model results were more compatible with the experimental results for MC-1 than MC-2. This can be resulted from the different packing densities of each module. The packing density of MC-2 is higher than MC-1 and this can result in deviation in the model results for the given Sh correlations. This deviation could be also attributed to a less uneven pore distribution and tortuosity possibility as well as formation of complex hydrodynamic nature of the viscous boundary layers at higher packing factors [49,50]. As the model predictions at different parameters were examined for MC-2 in Figs. 5(c) and (d), it can be seen that model predicted fluxes at varying feed and brine velocity parameters better than varying temperature and brine concentration parameters. This can be attributed to the fact that temperature and brine concentration have a stronger influence on the solution viscosity than the velocity. With the dramatic change of viscosities, the boundary layers may become more complex and the prediction shows some deviations for these parameters. Although the fluxes obtained in MC-2 were higher than MC-1, water removal rates were higher in MC-1 since the two membrane contactors have different contact areas. In general, MC-1 gave more predictable results and had higher water removal rate than MC-2 in OD.

3.1.1. *Effect of brine concentration*

Experiments were performed at different molarities of $CaCl_2$ solution (3–5 M) to investigate the effect of brine concentration using each module. The brine and feed velocities were 1,600 mL min^{-1} in MC-1 and 500 mL min^{-1} in MC-2. In both modules, water fluxes increased with increasing brine concentrations. The fluxes increased from 258 to 814 g m^{-2} h^{-1} in MC-2 while 197 to 389 g m^{-2} h^{-1} in MC-1 as the brine concentration was varied from 3 to 5 M. Since the magnitude of the driving force across the membrane increases with increasing brine concentration, fluxes increase accordingly. Fig. 6 shows the effect of brine concentration on fluxes in MC-1. As can be seen in Fig. 6, model and experimental results are in good agreement for MC-1. The deviation of model results from the experimental results in MC-2 is greater than in MC-1 (Fig. 5).

3.1.2. *Effect of feed and brine velocity*

Experiments were carried out to study the hydrodynamic conditions in both membrane contactors. The effect of feed and brine velocities on fluxes was investigated in each module by changing the velocity of one stream while keeping the other constant. For MC-1 one stream was kept constant at 1,600 mL min^{-1} while the other stream was varied from 1,200 to 2,000 mL min^{-1}, and for MC-2 one stream was kept constant at 500 mL min^{-1} while the other stream was in the range of 350–650 mL min^{-1}. The results obtained in MC-1 are given in Fig. 7.

In Fig. 7, it is apparent that the fluxes increase with increasing feed and brine velocities. This increase in fluxes

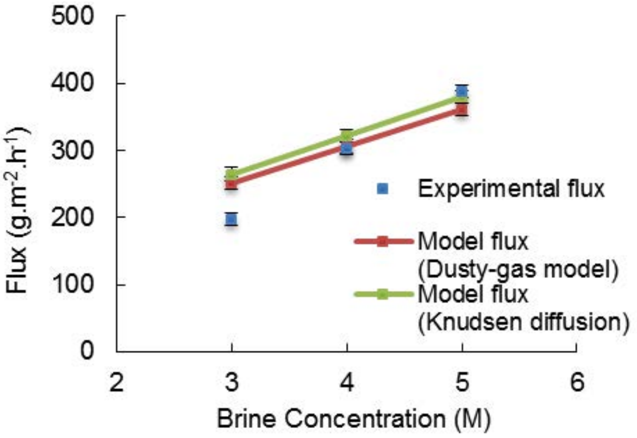

Fig. 6. Effect of brine concentration on fluxes in MC-1 at 25°C. (Q_{feed} = Q_{brine} = 1,600 mL min^{-1}).

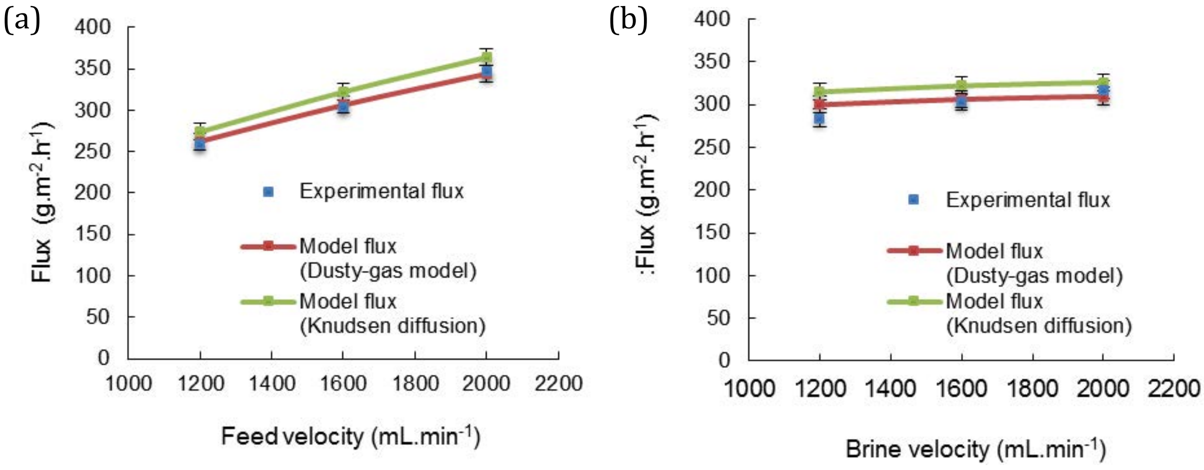

Fig. 7. Effect of brine and feed velocities on fluxes in MC-1 at 25°C. (a) Effect of feed velocity and (b) effect of brine velocity.

is due to the decrease of the boundary layer thicknesses, and consequently the decrease of the mass transfer resistances at higher velocities. As the velocity of the stream increases, Re number increases and the mass transfer coefficient increases accordingly. When the brine velocity was kept constant at 1,600 mL min^{-1} and milk velocity ranged from 1,200 to 1,600 mL min^{-1} in MC-1, Re number of milk stream was between 8 and 13.4 and overall mass transfer coefficients were between 0.85×10^{-7} and 4.1×10^{-7} m s^{-1}. When the effects of feed and brine velocities are compared, it can be seen that feed velocity has much more effect on fluxes than the brine velocity. These results indicate that mass transfer of water is more dominated by feed boundary layer resistance rather than by brine boundary layer resistance. In Fig. 7, it can be seen that predicted results at different velocities are in good agreement with the experimental results obtained in MC-1. For MC-2, similar results were obtained. Water fluxes increased with both feed and brine velocity in MC-2. The fluxes obtained in MC-2 were in the range of 338 to 469 g m^{-2} h^{-1} with varying velocities of feed and brine streams at 30°C. It is worth to note that each membrane contactor module has different contact area thus the comparison between these modules should be done on the basis of water removal rate rather than fluxes. The Re numbers were in the range of 69 and 90 with varying velocities using MC-2. The predicted results show a good agreement with the experimental results for MC-2 (Fig. 5) for varying velocities. The model predicts velocity effect on fluxes better than the brine concentration effect on fluxes for MC-2.

3.1.3. Effect of temperature

Experiments were carried out between 25°C and 35°C to assess the effect of the temperature on water transport using both membrane contactor modules. The brine and feed solution temperatures were changed together keeping both solutions at the same temperature. With increasing temperatures, fluxes increased since the diffusion coefficient and the vapor pressure of the water increased and the viscosity of the solutions decreased. Moreover, the kinetic energy of the water vapor also increases with increasing temperatures and this phenomenon enhances the mass transfer across the membrane. The effect of temperature on fluxes with comparison of model results using MC-1 is shown in Fig. 8. As seen in Fig. 8, model results are in good agreement with experimental results and Knudsen diffusion can describe the mass transfer slightly better than Dusty-gas model for the temperature effect on fluxes. In MC-2, the model fluxes for temperature parameter showed a slight deviation from the experimental values (Fig. 5). In general, temperature and brine concentrations are the strongest parameters that affect fluxes as they change the complexity of the boundary layer dramatically especially at higher packing densities which is the case for MC-2. As a result, the effect of these parameters on fluxes shows much deviation from the model than the velocity parameters on fluxes in particular for MC-2.

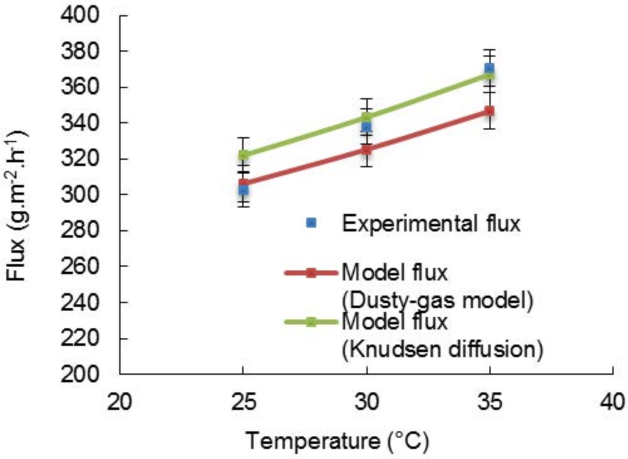

Fig. 8. Effect of temperature on fluxes in MC-1 ($Q_{\text{feed}} = Q_{\text{brine}}$ = 1,600 mL min^{-1}, Brine concentration is 4 M).

3.2. Membrane distillation experiments

MD performances of the membrane contactors were evaluated for further use of the MD process in the hybrid process. At the end of the milk concentration experiments in OD, the brine solution dilutes to 3.12 M from 4 M. This diluted brine was intended to re-concentrate in DCMD using MC-1; however, reverse flux was obtained at several temperature differences applied between feed and distillate. The applied temperature differences were between ΔT = 9.6°C–15.9°C which correspond to brine temperatures between 24.8°C and 38.7°C and distillate temperatures between 15.3°C and 22.8°C. Since the brine osmotic pressure is high, the temperature difference cannot overcome the activity difference as a driving force thus activity difference dominates the mass transfer. As a result, water transfers from the distillate side to the brine side although the temperature of the brine is much higher than the distillate side. The brine concentration at which the activity difference with distillate could not compensate the temperature difference was investigated and finally it was seen that the temperature difference overcame the activity difference when the brine concentration was 0.94 M. Thus the limit brine concentration that could be used in MD in MC-1 was obtained as 0.94 M $CaCl_2$. Under an applied temperature difference between brine and distillate sides, above this limit value water transfers from distillate to brine whereas below this value water transfers from brine to distillate. Higher feed temperatures could be applied to maintain a higher temperature difference, however distillate temperature also increases at higher feed temperatures because of the heat transfer by conduction across the membrane. To maintain the temperature difference higher, other designs of MD such as air gap membrane distillation involving a chilled condensing plate could be used.

After, MD experiments were carried out using MC-2 at different temperature differences of brine and distillate between ΔT = 11°C–13.4°C to recover the diluted brine used in OD experiments. However similar to MC-1, a reverse flux was observed since the activity difference overcame the temperature difference. The limit brine concentration to be used in DCMD was investigated and obtained as 2 M. Thus this limit concentration of MC-2 was used in further hybrid process experiments. The limit concentration of brine to be used in MC-2 was almost two times greater than the one in MC-1. These results show that MC-2 is more favorable for DCMD than MC-1. This may be attributed to the higher wall thickness of the membranes in MC-2. The wall thickness of MC-2 is about 10 times greater than that of MC-1. This thicker wall of the membrane decreases heat conduction through the membrane acting as an insulator and higher temperature differences can be obtained in MC-2 under the same applied set temperatures. The wall thickness of the membrane is an important parameter for an efficient DCMD operation to achieve higher temperature differences between two sides of the membrane. In general, DCMD can provide only mild temperature differences between two sides of the membrane since heat is mostly transferred by conduction. Although use of higher membrane thickness may help, to avoid heat loss by conduction increasing the membrane thickness can also result in higher membrane resistances for mass transfer. To achieve higher temperature differences between two sides of the membrane other configurations such as air gap membrane distillation with a chilled condensing plate can be used.

3.3. Osmotic distillation and membrane distillation coupling

OD and MD coupling was applied using different membrane contactor combinations. First MC-1 was used in both units of the hybrid process. The upper limit of brine concentration that can be used in DCMD in MC-1 was obtained as 0.94 M previously. However this brine concentration yielded in a low flux or a reverse flux especially for higher average temperatures in the hybrid process. Several trials of arrangements for various water activities between two sides of the membrane were applied using this hybrid system but eventually MD fluxes were found to be very low compared with OD fluxes (data not shown). The water flux ratios of OD to MD were in the range of 3.6–4.5 in this hybrid system using two MC-1 units. As this ratio (OD/MD flux ratio) approaches to unity, the operation can be conducted at constant brine concentration and the brine can be completely recovered. Thus, in such a hybrid system design, the focus must be on equalizing the fluxes related to OD and MD parts of the integrated system. Various temperature differences between brine and distillate (Δ*T*) were applied at different feed, brine and distillate temperatures using this configuration but significant enhancement was not observed. MC-1 was not found to be suitable to be employed as a MD unit in the hybrid system. Although MC-1 gave good results in OD, it was not efficient for MD, and thus MC-1 in the MD part was replaced with MC-2.

Hence the next configuration for OD and MD coupling involved the use of MC-1 in OD and MC-2 in MD. In the previous experiments, the upper limit for brine concentration that can be used in MD with MC-2 was obtained as 2 M. Thus this brine concentration was used in the hybrid process. Several temperature differences with different feed and brine temperatures were applied in this configuration and the results are given in Fig. 9.

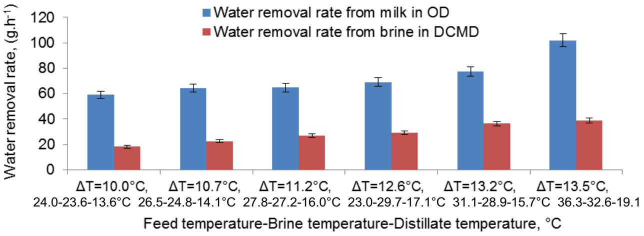

Fig. 9. Water removal rates in OD and DCMD in OD (MC-1) + DCMD(MC-2) hybrid process.

As can be seen in Fig. 9, as the temperature difference increases, flux in MD has a tendency to increase. Various feed, brine and distillate temperatures were applied with small increments or decrements. The ratio of water removal rate from milk to brine decreased from 3.2 to 2.13 when Δ*T*, the temperature difference between brine and distillate, increased from 10°C to 13.2°C. The OD/MD water removal ratio was obtained as 2.1 when Δ*T* = 13.5°C and as 2.6 when Δ*T* = 13.2°C. Although the temperature differences between distillate and brine were close to each other in these two experiments, different water removal ratios were obtained since the feed temperature was relatively higher when Δ*T* = 13.5°C. Also, in order to make a more concrete comparison, the driving force which is the difference of vapor pressures of brine and distillate side should be compared. It is worth to note that the same vapor pressure difference can be created by various temperature differences and the same temperature difference can generate different driving forces. The effect of the solution temperatures on the driving forces in MD is reported by Kujawa and Kujawski [51]. When two systems with equal temperature differences are compared, the one with higher average temperature will have the greater driving force. In the studied case, the vapor pressure difference between brine and distillate for Δ*T* = 13.5°C was greater than the one for Δ*T* = 13.2°C. Besides the feed temperature was higher when Δ*T* = 13.5°C. As a result the OD flux increased in this experiment and the ratio of OD/MD removal rate decreased accordingly. In general this configuration with the combination of MC-1 and MC-2 gave better results than the former configuration with two MC-1. This configuration allows use of higher brine concentration and consequently higher fluxes can be obtained. Nevertheless the water removal ratio of OD/MD in hybrid process or in other words brine recovery rates should justify the use of this process in terms of economic aspects. An enhancement in the process by decreasing the flux ratio of OD/MD in the hybrid process might be needed for the justification of the process. To improve the process, in the next configuration two MC-2 membrane contactors were coupled as the hybrid process. The fluxes in each unit of the hybrid process are shown in Fig. 10.

As can be seen in Fig. 10, when MC-2 was used in both units of the hybrid process, the fluxes obtained in each part are very close to each other which indicates an efficient concentration of brine during the concentration of milk simultaneously. This configuration was the best configuration among the studied hybrid process configurations and can be proposed for an efficient operation of concentration of liquid foods. In general, MC-2 gives better results in MD since it has a higher membrane thickness and this can be an advantage for its use in hybrid process. The ratios of the fluxes of OD to MD were varying between 1.2 and 1.06, reflecting a close final brine concentration to the initial value during the experiment. In Fig. 10, it can be seen that small changes in

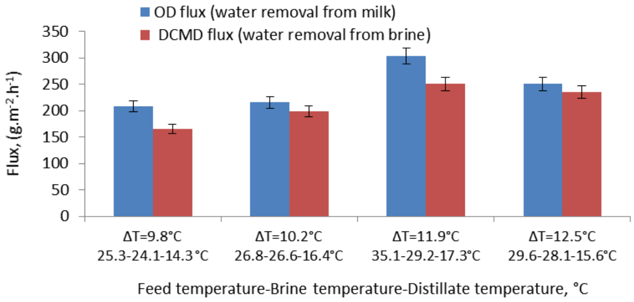

Fig. 10. Fluxes in OD and DCMD in OD(MC-2) + DCMD(MC-2) hybrid process.

the temperature differences can affect the flux considerably. This is due to the change in the driving force through the membrane with varying solution temperatures. The permeate flux in the MD is a function of the driving force where the driving force is the difference in vapor pressure of the solutions between the two sides of the membrane (Eq. (1)). The vapor pressure of the distillate, P_w^d which is pure water can be calculated by Antoine equation given below where A, B and C are the constants and T is the temperature.

$$P_w^d = P_w^* = \exp\left(A - \frac{B}{C+T}\right) \tag{17}$$

For water $A = 23.1964$; $B = 3{,}816.44$ and $C = 46.13$ where T is in Kelvin and pressure is in Pa [51]. The vapor pressure of the water in brine (P_w^b) can be calculated from Eq. (18) where a_w^b is the activity of water in brine solution and P_w^* is the pure water vapor pressure calculated by Eq. (17) at the brine temperature.

$$P_w^b = a_w^b \times P_w^* \tag{18}$$

The water activity in brine solution at different molar concentrations and temperatures can be calculated either from the ASOG contribution method [48] or from the empirical equation given for $CaCl_2$ solutions by Viet Bui et al. [52] as a function of concentration and temperature based on the Norrish model. The driving force, ΔP, is the difference between vapor pressures of the solutions given in Eq. (19):

$$\Delta P = P_w^b - P_w^d \tag{19}$$

The driving forces for the MD part of the hybrid systems were calculated and reported in Table 2.

From the results in Table 2, several conclusions can be drawn. Small changes in the temperature differences can lead to considerable change in driving forces due to the exponential dependence of the vapor pressure on temperature. Moreover, similar temperature differences can lead to different driving forces and similar driving forces can be resulted from different values of feed and distillate temperatures. When the temperature differences are equal or very close for two different MD systems, the driving force is higher for the system with higher average solution temperature. The driving forces were plotted vs. fluxes in MD units of two hybrid systems together and it is shown in Fig. 11. In MD unit, the fluxes increase with increasing driving forces. In the literature activity, energy for the water transport dependent on the temperature is given in some MD applications [51,53]. Since the permeate flux is a temperature-dependent phenomenon, the theory of the rate processes and the concept of activation energy is applied to the calculation of the activation energy of the water transport across the membrane. According to this concept, the relation between flux and feed temperature is presented by an Arrhenius-type relation and then activation energy is estimated for the solvent. The activation energy for water transport in MD was reported as 41 kJ mol^{-1} by Gyrta [53], and as between 43.1 and 58.8 kJ mol^{-1} by Kujawa and Kujawski [51].

Table 2
Driving forces applied in MD units of the hybrid processes at various solution temperatures

Driving force, ΔP (Pa)	T_b (°C)	T_d (°C)	T_{avg} (°C)	ΔT (°C)	Flux ($g\ m^{-2}\ h^{-1}$)
OD (MC-1) + DCMD(MC-2) hybrid process					
933.9	23.6	13.6	18.6	10.0	181.8
1,068.3	24.8	14.1	19.5	10.7	225.1
1,268.4	27.2	16.0	21.6	11.2	270.3
1,618.9	29.7	17.1	23.4	12.6	194.0
1,622.9	28.9	15.7	22.3	13.2	363.0
1,999.3	32.6	19.1	25.9	13.5	388.7
OD (MC-2) + DCMD(MC-2) hybrid process					
Driving force, ΔP (Pa)	T_b (°C)	T_d (°C)	T_{avg} (°C)	ΔT (°C)	Flux ($g\ m^{-2}\ h^{-1}$)
938.1	24.1	14.3	19.2	9.8	165.8
1,115.3	26.6	16.4	21.5	10.2	198.8
1,493.2	29.2	17.3	23.3	11.9	250.9
1,480.0	28.1	15.6	21.9	12.5	234.9

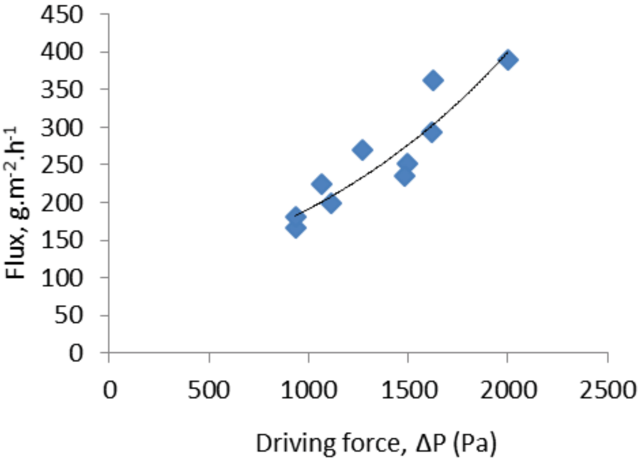

Fig. 11. Fluxes vs. driving force in MD unit of the hybrid systems.

For the OD (MC-2) + DCMD(MC-2) hybrid process, the change of the amount of the removed water from milk and brine concentration with time during the experiments is given in Fig. 12.

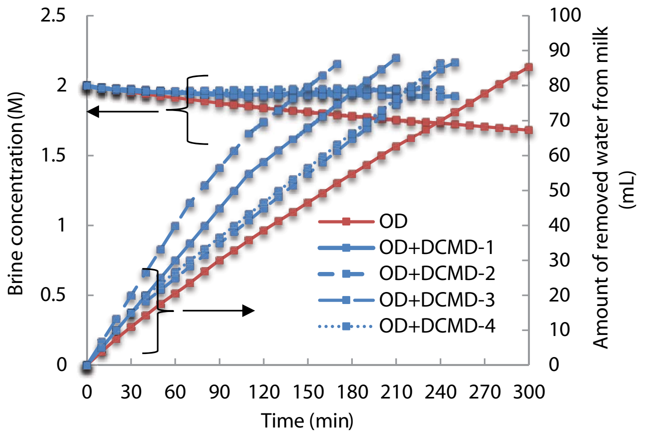

Fig. 12. Change of the amount of removed water from milk and brine concentration with time in hybrid process and OD process. (OD + DCMD-1: ΔT = 12.5°C; OD + DCMD-2: ΔT = 9.8°C; OD + DCMD-3: ΔT = 11.9°C; OD + DCMD-4: ΔT = 10.2°C).

As can be seen from Fig. 12, in hybrid process experiments brine concentration at a given time was higher than the OD during the experiments. This process allows working almost at constant brine concentration.

In the literature on the OD experiments, brine volume used is usually greater than the feed volume to avoid the dilution effects. One of the advantages of the hybrid process is that it allows working volume 1:1 ratio of feed to brine. The dilution amount of brine in an OD process depends on how much volume brine is used in the process. In an OD process, for a half volume reduction of feed using 2 M brine solution, if the feed to brine volume ratio is used as 1:1, 1:2 and 1:3, then the final concentrations of brine will be 1.33, 1.6 and 1.71 M, respectively. In this study, the best OD flux/MD flux ratio was obtained as 1.07 using the OD(MC-2) + DCMD(MC-2) hybrid process where this ratio can be seen as a measure of how close is the final brine concentration to the initial concentration. Using this hybrid process, for a volume reduction of feed by half using 2 M initial brine concentration, the final brine concentrations are estimated as 1.94, 1.97 and 1.98 M using 1:1, 1:2 and 1:3 feed/brine volume ratios, respectively. These values are higher than the values obtained in OD. Thus, with the use of hybrid process, the operation proceeds at almost constant brine concentration. With precise temperature detection in the hybrid process and adequate membrane area arrangement, the conditions that give exactly equal fluxes in OD and DCMD can be determined. When these conditions are used in the hybrid process, the concentration operation can run at constant brine concentration. In the hybrid process, there are several conditions that affect the magnitude of the fluxes in OD and DCMD such as feed and brine velocities, temperature and membrane surface area of each unit. The fluxes can be set to equal by adjusting these parameters accordingly in the design of such a hybrid process.

4. Conclusions

In this study, milk concentration was carried out in OD individually and then the hybrid use of MD and OD processes was evaluated to perform simultaneous milk concentration and brine recovery. Two different hydrophobic membrane contactors were used with polypropylene fibers where the main differences were the module geometry, membrane thickness and porosity. A mathematical model was developed for OD on the basis of both Knudsen diffusion and Dusty-gas model using resistance in series approach. It was seen that both models can describe the mass transfer reasonably. For hybrid process, various configurations of membrane contactors were applied and finally the best configuration was found to be osmotic distillation–direct contact membrane distillation coupling using capillary modules. Using hybrid process, simultaneous milk concentration and brine recovery was achieved. This hybrid process is a promising alternative technology for the concentration of liquid food at moderate temperatures keeping its nutritive and aroma compounds. The integration of MD to OD unit can allow working at constant brine concentration and it prevents additional steps for brine recovery.

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Symbols

a_f^m, a_b^m — Water activities of the feed and brine solutions at the membrane interface, respectively
c — Concentration, wt.%
$D_{(H_2O)}$ — Self-diffusion coefficient of water, $m^2\ s^{-1}$
D_{ab} — Diffusion coefficient, $m^2\ s^{-1}$
D_h — Hydraulic diameter, m
D_{Kn} — Knudsen diffusivity, $m^2\ s^{-1}$
$D_{w\text{-}air}$ — Diffusion coefficient of water in air, $m^2\ s^{-1}$
F — Milk fat content, wt.%
h — Parameter for salts used in Eq. (12)
J — Flux, $m^3\ m^{-2}\ s^{-1}$
J_w — Water flux, $m^3\ m^{-2}\ s^{-1}$
K — Overall mass transfer coefficient, $m\ s^{-1}$
k_b — Brine-side mass transfer coefficient, $m\ s^{-1}$
k_f — Feed-side mass transfer coefficient, $m\ s^{-1}$
k_m — Membrane mass transfer coefficient, $m\ s^{-1}$
k_{mp} — Membrane permeability, $m\ s^{-1}\ Pa^{-1}$
M — Molarity, $mol\ L^{-1}$
M_w — Water molar mass, $kg\ mol^{-1}$
P — Total pressure, Pa
P^*, P_w^* — Pure water vapor pressure, Pa
P_{air} — Partial pressure of air, Pa
$\Delta P_{w,m}$ — Water vapor pressure difference across the membrane, Pa
R — Gas constant, 8,314 $J\ mol^{-1}\ K^{-1}$
Re — Reynolds number
Sc — Schmidt number
Sh — Sherwood number
T — Temperature, K or °C

Greek

μ — Viscosity, Pa s
ρ — Density, $kg\ m^{-3}$

υ — Velocity, m s^{-1}
ϕ — Packing fraction
ε — Porosity
τ — Tortuosity
δ_m — Membrane thickness, m

Subscripts

avg — Average
f — Feed
b — Brine
d — Distillate
w — Water
i — Solute

Superscripts

m — Membrane
DG — Dusty-gas model

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