



Effect of soluble and insoluble gas bubbling methods on ultrafiltration fouling control

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ABSTRACT

Fouling phenomenon is well known as a main obstacle in membrane separation technology. In this study, the effects of bubbling with various hydrodynamic factors and gas–liquid solubility were evaluated on fouling control and permeation flux in ultrafiltration of skimmed milk solution. Direct gas injection and carbonated feed, as a new bubbling method, were used for bubbling. In the direct gas injection technique, various two-phase flow patterns (slug and bubble), gas flow rates, and bubbling modes (continuous and intermittent) were investigated during a cross-flow ultrafiltration. The results showed that the both of gas bubbling methods improved the permeation flux during 30-min filtration. The permeation flux was enhanced up to 72 and 40% by direct injecting of N₂ and CO₂, respectively, while it was only enhanced up to 58% with carbonated feed. The evaluation of hydrodynamic resistance of membranes indicated that the gas bubbling by carbonated feed was more effective on the fouling resistance, while the cake resistance was affected by the gas injection during infiltration. In addition, the slug flow pattern was more effective than the bubble flow pattern on decline of membrane fouling. In the slug flow pattern, the permeate flux increased when medium flow rate was applied. Furthermore, ultrafiltration performance was improved using the intermittent gas bubbling mode. This study indicated that slug flow pattern with insoluble gas has a higher performance than the other ones in preventing fouling and flux enhancement during ultrafiltration processing.

Keywords: Ultrafiltration; Fouling; Gas bubbling; Two-phase flow pattern

1. Introduction

Membrane filtration is considered as a green technology in industrial applications. However, the development of membrane technology faces fouling as a serious obstacle [1,2]. Concentration polarization can be

considered the reason for the limitation of the membrane filtration due to its negative effect on the permeate flux. Fouling is often a result of concentration polarization, but it can also be founded by other reasons [3]. Many novel physical cleaning techniques have been developed to overcome the fouling problems. Various systems of dynamic filtration also called shear-enhanced filtration, such as rotating disk [4,5] or

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rotating membranes [6,7] or membrane vibration [8,9], have been used to control the fouling, which create the sufficient shear rate to maintain the filtration [10,11]. In addition, it has been well known that the use of low frequencies of ultrasound has a good effect on flux recovery and reduction of fouling [12–15]. One of the common strategies for membrane fouling control is application of gas/liquid two-phase flow which improves the performance of filtration in some membrane processing [16]. Many studies have been carried out to investigate the effects of gas bubbling on membrane fouling during membrane filtration [7,17–21]. Ndinisa et al. studied the application of gas–liquid two-phase flow as a fouling control mechanism in submerged flat sheet membrane bioreactors [20]. They showed that the permeation flux in submerged flat sheet membranes improved with increasing of nozzle size and airflow rate. The effect of gas bubbling combined with intermittent filtration was investigated on membrane fouling by Cerón-Vivas et al. [18]. They reported that the intermittent filtration combined with gas bubbling in order to minimize membrane fouling was an effective procedure. The fraction time of relaxation period was the strategic target. Chen et al. found that incorporation of gas bubbling into close contact of membrane surface enhanced the permeation flux by average 26% during membrane distillation [19]. Javadi et al. optimized the effect of gas sparging on microfiltration of microbial suspension [22]. They showed that gas sparging technique was more efficient in low-concentration microalgae microfiltration, in which up to 60% enhancement was achieved in slug flow pattern. Jiang et al. investigated the influences of air bubbling parameters on mitigation of fouling in immersed hollow-fiber (HF) membrane for ultrafiltration of river water. They concluded that the small size of bubbles and continuous air bubbling were more effective than large and intermittent bubbling procedure on mitigation of membrane fouling [7]. Further applications of gas bubbling may be found in food technology and biotechnology in order to improve filtration performance [16,17,20,23]. The consumption of energy in order to create the bubbles is an important parameter and intermittent bubbling is a conventional method in membrane filtration process [20]. In spite of many investigations on application of gas bubbling to control fouling, few information were reported on some characteristics such as hydrodynamic parameters particularly in gas solubility on feed during ultrafiltration. This study is the first research about bubbling by carbonated feed. In this study, the effects of soluble and insoluble gas bubbling, and also different methods of bubbling have been evaluated on flux enhancement during the flat sheet ultrafiltration membrane processing.

2. Materials and methods

2.1. Materials

Skimmed milk powder was purchased from the local market and a 1% (weight percentage) “solution” of skim milk powder was prepared and used as a liquid feed. The physicochemical properties of feed solution were shown in Table 1. The temperature of the feed was fixed at $20 \pm 2^\circ\text{C}$ during separation process.

Flat sheet polyethersulfone (PES) ultrafiltration membrane (Sepro Company USA), with 10 KD molecular weight cut-off (MWCO) was used in Minitan S (Millipore Inc.) system. The effective membrane area was 112 cm^2 . The membrane was placed between two large perforated silicon rubbers in order to create a series of linear cross-flow channels. Two acrylic manifolds of thickness 2.3 cm were placed in upper and lower sides of membrane which were covered by two stainless steel plates of 1.1 cm thickness. Both liquid N_2 and CO_2 with purity of 99.97% purchased from Tous Gas Company, Mashhad (Iran), were used separately for each treatment.

2.2. Methods

2.2.1. Ultrafiltration

The schematic diagram of experimental setup is shown in Fig. 1. The system consists of a flat sheet membrane and a feed tank. A peristaltic pump supplied sufficient and constant pressure for the feed flow. All of experimental tests were carried out for 30 min under fixed 3 bar inlet pressure and $20 \pm 2^\circ\text{C}$ temperature. We experimentally found that this time duration is enough to obtain the constant value of permeate flux. The membrane renewed for each experiment. The differences between inlet and outlet pressure of feed (ΔP) were measured by two pressure gages before and after the feed flow. Also, one pressure gage was inserted into permeate flux outlet. During the fouling process, permeate and retentate were recycled to the feed tank to maintain the feed concentration. In order to find out the damage probability of membranes, the integrity test (pressure decay test) was carried out for each new membrane [24]. The viscosity of permeate was measured by Brookfield viscometer Tokimec model BL.

2.2.2. Gas bubbling

2.2.2.1. *Bubbling by direct gas injection.* In order to generate the bubbles by gas injection, N_2 and CO_2 were injected directly into feed inlet (Fig. 1). The gas bubbling was supplied in two bubble and slug patterns of

Table 1
Physicochemical properties of feed solution

Ash (kg/100 kg)	Lactose (kg/100 kg)	Protein (kg/100 kg)	Density (kg/m ³)	Viscosity (Pa s)	Conductivity (S/m)	Brix (%)	TDS (ppm)	pH	Particle size of powder (m)
0.00721	0.04657	0.03035	1,032	1.47×10^{-3}	9.1×10^{-2}	1.11	460	6.93	$0.2\text{--}2.5 \times 10^{-4}$

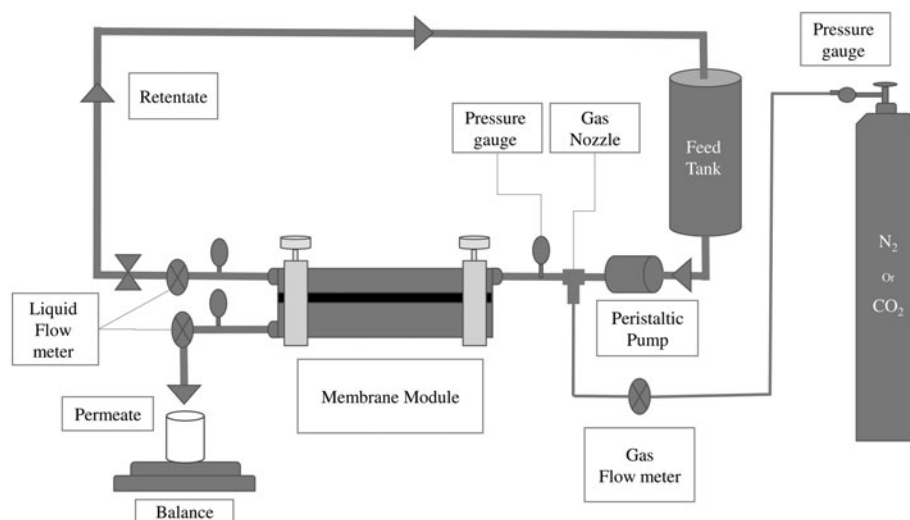


Fig. 1. Experimental setup of cross-flow ultrafiltration with gas injector.

two-phase flow (Fig. 2). In bubble pattern, the diameter of gas bubbles were less than (e.g. <60%) of the channel size during all treatments. The slug flow (also called plug flow) occurred when the gas flows as large bullet-shaped bubbles approaching the diameter of the channel size [17]. The gas–liquid two-phase flow pattern depends on the gas injection factor (r) which equals to $U_g/(U_g + U_l)$. U_g and U_l are the superficial gas and liquid flow rate or flow velocity, respectively. The two-phase flow pattern changes from bubble flow ($0 < r < 0.2$) over slug flow ($0.2 < r < 0.9$) to annular flow ($0.9 < r < 1.0$) [25]. The gas flow was set at three flow rates, 0.1, 0.2, and 0.3 L min⁻¹ for bubble

two-phase flow pattern and 0.5, 1, and 1.5 L min⁻¹ for slug two-phase pattern.

In addition, two types of gas bubbling—continuous and intermittent—injection modes were studied during ultrafiltration. The intermittent bubbling was conducted with the time sequence of 1 min “on” and 5 min “off” alternatively, which was in match with the filtration mode of 5 min on/1 min off [16]. During the non-filtration period, the pressure pump is switched off and the transmembrane pressure (TMP) was dropped to zero. The bubbling treatment was performed by direct injection of N₂ in slug and bubble two-phase flow patterns and medium flow rate.

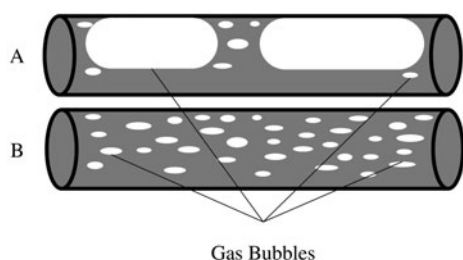


Fig. 2. Gas bubbling two-phase flow patterns: (A) slug pattern and (B) bubble pattern.

2.2.2.2. *Bubbling by carbonated feed.* In order to provide bubbling by soluble gas in feed, the 1% milk solution was carbonated in a stainless steel chamber using circulating CO₂ in 4 bar pressure at 4°C for 15 min. The dissolved CO₂ in feed (carbonated feed) was released by applying degas mode of ultrasonic cleaning bath model type of the Elmasonic, Germany, in 80 kHz frequency and 300 watt of power intensity. Degassing of carbonated feed was carried out in the vicinity of active zone of membrane surface module using ultrasonic waves. Here, this method is named second method of bubbling.

2.2.3. Calculations

Transmembrane pressure was calculated by Eq. (1):

$$\Delta p = [(P_{fe} + P_r) \times 2^{-1}] - P_p \quad (1)$$

where (ΔP) (Pa) is the transmembrane pressure; P_f (Pa) is the feed pressure; P_r (Pa) is the retentate pressure, and P_p (Pa) is the permeate pressure.

The permeate flux was measured by Eq. (2) [26]:

$$J = (W_{ti} - W_{ti-1}) \cdot (d \cdot \Delta t)^{-1} \quad (2)$$

where J ($\text{m}^3 \text{m}^{-2} \text{s}^{-1}$) is the permeate flux; W_{ti} (kg) is the permeate weight at time i ; W_{ti-1} (kg) is the permeate weight at time $i - 1$; d (kg m^{-3}) is the density of permeate, and Δt is the time interval.

The hydrodynamic resistance was calculated by Eq. (3):

$$R_H = \Delta P \cdot (\mu \cdot J)^{-1} \quad (3)$$

where R_H (1 m^{-1}) is the hydrodynamic resistance; ΔP is the steady-state system pressure; μ (Pa s) is the permeate viscosity; and J is the permeate flux.

The resistance of cake layer was calculated by determination of different amounts of membrane resistance before and after the removal of cake layer [27]. The total hydrodynamic resistance was calculated by Eq. (4):

$$R_t = R_m + R_c + R_f \quad (4)$$

where R_t is the total hydrodynamic resistance; R_m is the new membrane resistance; R_c is the cake resistance, and R_f is the fouling resistance.

The effective factor on permeation flux was calculated and defined as following Eq. (5):

$$\text{EF} \% = \left[\left(\frac{J_{gb} - J}{J} \right) \cdot J^{-1} \right] \times 100 \quad (5)$$

where J_{gb} is the permeate flux with gas bubbling treatments; J is the permeate flux without treatment.

The difference between flux of deionized water before and after membrane fouling per time unit was represented as fouling percent and calculated using Eq. (6):

$$\text{Fouling} \% = \left[1 - \left(\frac{J_{wp}}{J_{w-1}} \right) \right] \times 100 \quad (6)$$

where J_{wp} and J_w are the distilled water flux of membrane after and before fouling, respectively.

2.2.4. Statistical analysis

Each treatment was carried out at least three times. The obtained data were statistically analyzed using multifactor design in ANOVA table. The least significant differences (LSD) were calculated and the obtained means were evaluated by Duncan Multiple Range Test. Statistical analysis was performed using SigmaStat 3.1 and Microsoft EXCEL software.

3. Results and discussions

3.1. Effect of bubbling

3.1.1. Bubbling by gas injection

As can be seen in Fig. 3, the bubbling treatment significantly increased the permeation flux ($p < 0.05$). Disturbing the mass transfer of boundary layer near the membrane wall is the key factor for improving the performance of membrane filtration. An increase in cross-flow velocity is a simple and practical method to enhance the mass transfer. However, increasing the cross-flow velocity to obtain more turbulent flow is not always efficient and also has drawbacks such as increasing energy consumption. A practical method to increase turbulence is the injection of gas bubbles into the feed flow [17]. The concentration polarization as an important phenomenon in membrane fouling is significantly decreased by an increase in the turbulence. Decreasing the concentration polarization causes higher flux [28,29]. Using surface shear is a major procedure to control fouling phenomenon. The surface shear can also be enhanced by two-phase flow [30]. In the two-phase flow, wakes and vortex mechanisms appear which cause secondary flow and increase the surface shear [17]. The surface shear could remove the deposited foulants during filtration.

3.1.2. Bubbling by carbonated feed

We tested the effect of ultrasound in the presence of soluble gas by an aluminum foil first (cavitation effect test) which was exposed to ultrasonic waves in the presence of carbonated water. In this test, no effect was observed on surface of foil due to dissipation of cavitation energy in soluble gas. The aluminum foil affected the ultrasound when it was submerged in pure water (with absence of any gas) in the second test [31]. Although ultrasound degassed the feed, no cleaning effect was observed in the first test. We can

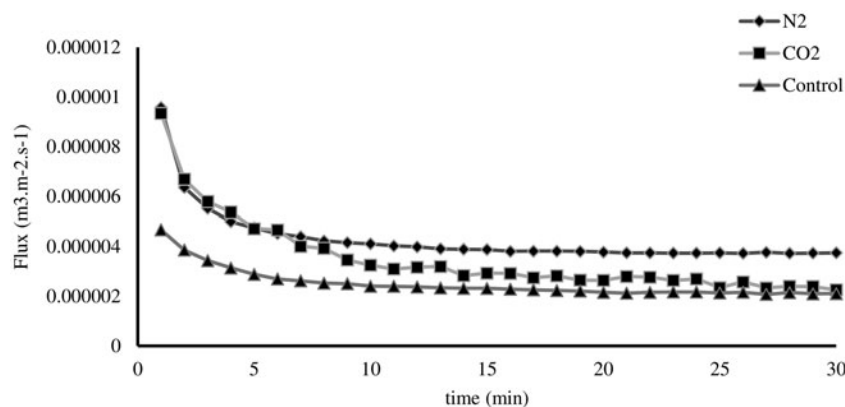


Fig. 3. Permeation flux under bubbling by N₂ and CO₂ direct injection treatments compared to control during 30-min ultrafiltration.

conclude that the cleaning effect of this method can be attributed to gas bubbles generated by ultrasound degassing. On the other hand, the ultrasound energy was consumed more for degassing and less for cavitation created. Furthermore, the obtained results showed that bubbling by gas releasing from carbonated feed led to a significant enhancement in the permeation flux up to 58% compared to the control ($p < 0.05$). The bubble released from carbonated feed under ultrasound degassing could increase the turbulence in the vicinity of membrane surface; therefore, the concentration polarization was decreased [32]. Our results showed that the amount of carbonate was enhanced by increasing the carbonating time and pressure; however, there was no significant difference in flux enhancement. In Fig. 4, the permeation flux was compared under two methods of bubbling. As can be seen, the performance of bubbling by N₂ injection was higher than the bubbling by carbonated feed. The generated bubbles by releasing gas from carbonated feed (the second method of bubbling) were not as strong as N₂ bubbles generated during gas injection (the first method of bubbling). Thus, the total applied shear force was not considerable in the second method of bubbling. Presumably, the cleaning effect of these bubbles was related to small size of carbonated feed bubble. We predicted that these bubbles could penetrate inside the membrane pores and remove the internal membrane fouling and that undoubtedly needs further research. By carbonating, pH of feed decreased from 6.8 to 6.2. Hydrophilic membranes in the lower pH level have better performance as reducing the pH below 6.0 increases the flux [33]. In this study, the effect of pH change about 0.6 was not significant compared to the bubbling effect.

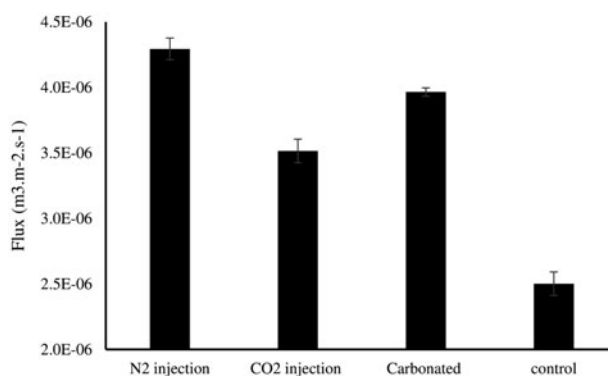


Fig. 4. Mean of permeation flux under bubbling with injection of different gas and carbonated feed treatments compared to control after 30-min ultrafiltration.

3.2. Effect of soluble and insoluble gas

The results showed that the injected insoluble gas (N₂) had a higher effect on flux recovery than the soluble gas (CO₂) (Fig. 3). The permeation flux was enhanced up to 72 and 40% by feeding N₂ and CO₂, respectively. As mentioned [34], it might be because of that a considerable part of CO₂ became dissolved after injecting into the membrane module. Therefore, the number of soluble bubbles was reduced and the shearing effect of soluble gas decreased, as compared to insoluble gas.

The skimmed milk feed solution can be considered as a protein solution. The ultrafiltration of protein solutions is characterized by a progressive decline in flux with time (e.g. Fig. 3). The initial flux drop in ultrafiltration is due to local convective deposition of protein molecules close to or in the pores, a process completes in less than a few seconds. Flux then

continues to drop slowly due to the adsorption of a protein monolayer at the membrane surface, which is attributed to the reversible polymerization of protein to gel layer [35].

We observed that the permeation flux rate with insoluble gas was obviously higher than soluble gas, especially after 7 min of filtration. In this case, the cake layer which deposited on membrane surface was significantly increased. On the other hand, the cleaning effect of injected CO₂, in the same gas flow rate and same two-phase flow pattern, was weaker than N₂, so the permeation flux under CO₂ bubble injection reduced faster than N₂ bubble injection.

3.3. Effect of phase flow pattern

The effect of various two-phase flow patterns on fouling percentage was indicated in Fig. 5. The slug pattern was more effective than the bubble pattern, while there were no significant differences ($p \geq 0.05$) between the slug and bubble patterns for CO₂ injection. Furthermore, the larger bubbles or slug patterns are more effective than the smaller one in promoting local mixing because of larger wake regions and stronger secondary flows [17]. It seems that when the CO₂ is injected as slug pattern, the larger part of slug bubble would be dissolved and it acts as bubble pattern. Ndinisa et al. reported that in submerged flat sheet membranes, fouling reduction improved with an increase in nozzle size of gas injection system [20]. Javadi et al. found that slug pattern was more effective than the bubble and churn patterns in microfiltration of microbial suspension [22].

3.4. Effect of gas flow rate

In Fig. 6, we can observe that the permeate flux was improved with an increase in gas flow rate until

medium flow rate point, in slug pattern, and became worse after this point. The presented data in Fig. 6 indicate the gas flow rate about 1 L min⁻¹ might be an optimal gas flow rate for this system. The shear intensity which is linked to gas bubbling improved with increasing gas flow rate. High shear forces may result in foulant removal from membrane surface. Thus, a higher gas flow rate creates two-phase flow which is a more beneficial effect on fouling control than the lower one [16,23].

Qaisrani and Samhaber explained this phenomenon with bubble size and air flow rate relationship. Bubble size is directly proportional to air flow rate. Accordingly, bubble diameter increased with increasing air flow rate. When the air flow rate became more than optimum, it seems that the size of the bubbles became so great. Large bubbles hinder the liquid to reach the membrane surface. Here, the bubbles act as cushions along the membrane surface. So the permeate flux decreased with an increase in air flow rate [30]. In the optimum point of gas bubbling (with N₂ bubbles in slug pattern and 1 L min⁻¹ gas flow rate), the permeation flux was enhanced up to 84.4% compared to the control.

3.5. Effect of gas bubbling mode

The use of gas bubbling treatment can be very effective on fouling control and most recent studies have confirmed this results [16,18]; however, it can be energy costly if not operated under an appropriate condition.

The use of intermittent filtration is a simple method to reduce energy consumption. In Fig. 7, the mean of permeation flux was shown for intermittent and continuous ultrafiltration under various hydrodynamic conditions. During non-filtration period, the

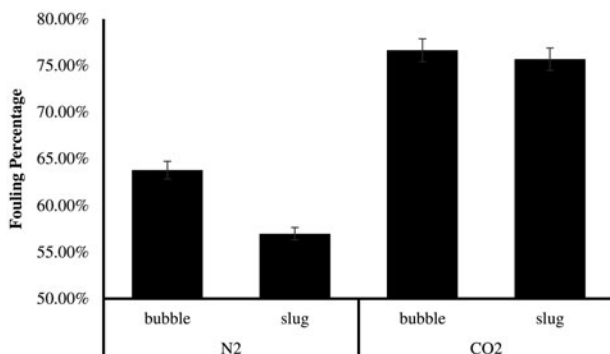


Fig. 5. Fouling percentage under different gases and two-phase flow patterns after 30-min ultrafiltration.

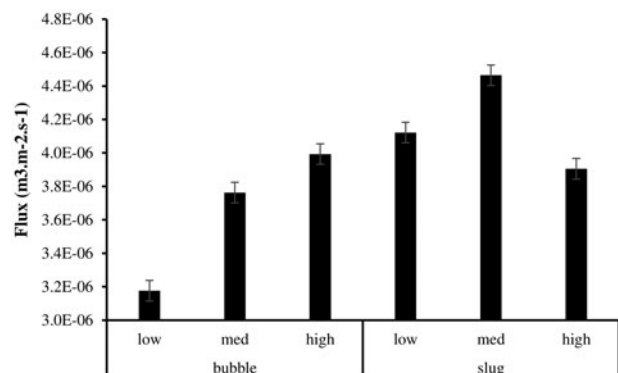


Fig. 6. Mean of permeate flux for different levels of gas injecting rate in various two-phase flow patterns.

bubbling treatment was only applied by direct injection of N_2 in slug and bubble two-phase flow patterns and medium flow rate. When the filtration is suspended, the shearing of gas bubbling on the membrane surface is continued and compression of the cake layer is reduced and better permeability is obtained [20,36]. In the condition of simultaneous presence of shear stress and absence of pressure force, the deposited particles are removed from the membrane surface easily. When the filtration period is resumed again, the membrane surface is relatively clean compared to the time that filtration period was stopped [20].

3.6. Hydrodynamic resistance

In Fig. 8, the practical hydrodynamic resistance was shown for different methods. The hydrodynamic resistance after 30 min under N_2 injection was significantly lower than CO_2 injection and carbonated feed ones ($p < 0.05$). As mentioned before, the performance of ultrafiltration with N_2 injection was more than CO_2 one which approves the results of hydrodynamic resistance. By considering Fig. 8, when ultrafiltration was performed with N_2 injection, the fouling resistance was more effective on the total resistance compared to cake resistance. However, the difference between fouling resistances under N_2 and CO_2 injection treatments was not significant; the difference was remarkable between their cake resistances ($p < 0.05$). It can be concluded that the higher performance of ultrafiltration was related to the higher ability of N_2 injection treatment to remove cake layer during ultrafiltration. This ability was attributed to shearing effect [20,32]. With respect to the results, the fouling resistance under bubbling by carbonated feed is lower

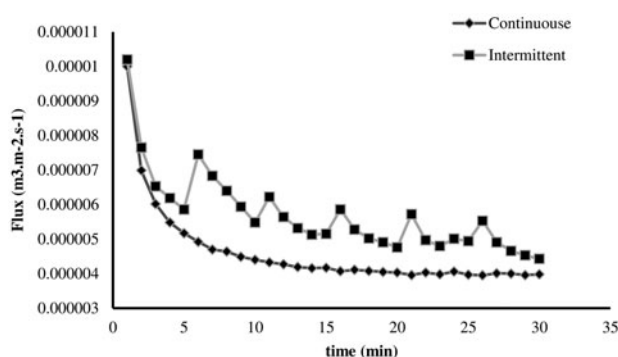


Fig. 7. Mean of permeation flux for intermittent and continuous filtration modes, during 30-min ultrafiltration, under various gas bubbling treatments.

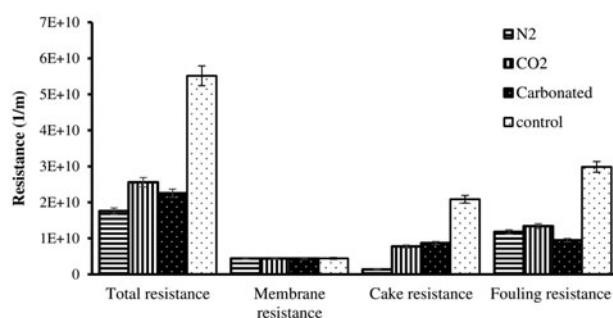


Fig. 8. Mean of hydrodynamic resistances for bubbling with injection of different gases and carbonated feed treatments compared to control after 30-min ultrafiltration.

than the bubbling by gas injection. This can be due to the more cleaning effect of carbonated feed on the pores than the surface of membrane. This effect is due to nucleation sites for small bubbles to form and shear off foulants. This mechanism was reported by Partlan, and Ngene et al. tried to clean fouled membrane using carbonated feed [37,38]. In general, the bubbles generated during bubbling by carbonated feed could remove or prevent from fouling resistance compared to cake resistance. These results showed that carbonated feed could be applied to clean the fouled pores of membrane and could not produce considerable shear force.

4. Conclusion

The effect of gas bubbling using N_2 and CO_2 , as insoluble and soluble gas, respectively, was investigated on fouling control of cross-flow ultrafiltration. The results showed that the gas bubble injection led to improve the permeation flux during 30-min ultrafiltration; however, insoluble gas had a higher effect on fouling control compared to the soluble gas up to 72%. The permeation flux was enhanced up to 58% by CO_2 injection and bubbling by carbonated feed. The fouling percentage for slug pattern was lower than the bubble pattern, indicating the slug pattern was more effective than the bubble one. However, the permeate flux improved with an increase in the gas flow rate until medium flow rate in slug pattern, as an optimum point. Ultrafiltration performance was increased under the intermittent gas bubbling and filtration. The obtained results proved that the difference between hydrodynamic resistances of two kinds of gases is concerned to the difference between cake resistances and fouling resistance. It can be concluded that use of insoluble gas led to an improvement in ultrafiltration performance.

Acknowledgments

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Symbols

EF	—	enhancement factor (%)
d	—	density (kg m^{-3})
J	—	permeate flux ($\text{m}^3 \text{m}^{-2} \text{s}^{-1}$)
R	—	resistance (1/m)
r	—	gas injection factor
t	—	time (s)
ΔP (TMP)	—	transmembrane pressure (Pa)
U	—	flow velocity (m/s)
W	—	weight (kg)
μ	—	viscosity (Pa s)

Subscript

c	—	cake
f	—	fouling
fe	—	feed
g	—	gas
gb	—	gas bubbling
H	—	hydrodynamic
l	—	liquid
m	—	membrane
p	—	permeate
r	—	retentate
t	—	total
w	—	before fouling
wp	—	after fouling

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