ULTRAFILTRATION OF LABANEH WHEY: EFFECT OF OPERATING PARAMETERS

Hassan A. Arafat
Chemical Engineering Department
University of Cincinnati
PO Box 210171, Cincinnati, OH 45221-0171, USA

and

Ibrahim M. Abu-Reesh*
Chemical Engineering Department
King Fahd University of Petroleum & Minerals
Dhahran, Saudi Arabia

الخلاصـة :

ندرس في هذا البحث تأثير متغيرات التشغيل على الترشيح الدقيق لسائل شُرش اللبّنة بواسطة الأغشية الليفية المفرغة . لقد تم استخدام أغشية ليفية من مادة البولي سلفون ذات أوزان جزيئية أسمية مقدارها خمسة و ٣٠ و ١٠٠ كيلو دالتون في عملية الترشيح الدقيق . لقد تم دراسة تأثير متغيرات التشغيل المختلفة على عملية الترشيح مثل درجات الحرارة والرقم الهيدروجيني وتركيز كلٍّ من البروتين والمواد الصلبة وفرق الضغط عبر الغشاء وسرعة التدفق وكذلك المعالجة المبدئية لسائل شُرش اللبّنة .

بدا لنا في هذا البحث أن التدفق يزداد بزيادة الضغط عبر الغشاء بالإضافة لزيادة درجة الحرارة وسرعة التدفق وهذا يساعد على تقليل المقاومة للسريان على سطح الغشاء . ولقد وجد أن التدفق يقل بزيادة الرقم الهيدروجيني حيث إنَّ الرقم الهيدروجيني العالي يساعد على تَكَوُّن المادة الغروية فسفات ثلاثي الكالسيوم وهذا يؤدي إلى زيادة معدل انسداد الغشاء .

ولقد وجُد أن المسامات الكبيرة للغشاء تعمل على زيادة التدفق بسبب قلة المقاومة الذاتية له . وكذلك تمت دراسة كفاءة عملية الترشيح الدقيق لسائل شرش اللبنة بهدف تركيز البروتين . ووجِدَ أن العملية فعّالة عند استعمال الغشاء (٣٠ كيلو دالتون) . ولكن عند استعمال الغشاء (١٠٠ كيلو دالتون) فإن عملية التركيز غير فعّالة بسبب مرور البروتين خلال سطح الغشاء.

*Address for correspondence:
KFUPM Box 969
King Fahd University of Petroleum & Minerals
Dhahran 31261, Saudi Arabia
e-mail: abureesh@kfupm.edu.sa

ABSTRACT

In this work, the effect of operating parameters on the ultrafiltration of 'labaneh' whey in hollow-fiber membranes was investigated.

Polysulfonic, hollow-fiber membranes, with nominal molecular weight cut-off (NMWCO) of 5, 30, and 100 kDalton, were used in the ultrafiltration process. Different operating conditions of temperature, pH, protein and total solid concentration, trans-membrane pressure, flow rates, and pre-treatment were studied.

It was found that flux increased with increasing trans-membrane pressure, temperature, and flow rate. This was attributed to the drop in the resistance on the membrane surface. Flux was found to decrease when the pH was increased. Higher pH was found to be responsible for tricalcium phosphate colloid formation, which led to a higher fouling rate in the membrane. Larger pore size was found to enhance the flux by decreasing the membrane intrinsic resistance.

The efficiency of the ultrafiltration (UF) process in concentrating protein in labaneh whey was also studied. The UF process was found efficient in concentrating protein in labaneh whey when a 30 kDa membrane was used. It was found, however, that the 100 kDa membrane was inefficient for concentrating protein in labaneh whey. In this case, protein was found to pass through the membrane's surface.

Key words and phrases: Ultrafiltration, Labaneh whey, hollow fiber membrane, membrane flux

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INTRODUCTION

Ultrafiltration (UF) is a pressure-driven membrane separation process. It is considered as an effective method for concentrating protein solutions. UF has been widely used to replace many conventional separation methods in the pharmaceutical, chemical, and food industries [1]. The UF process is used to produce whey protein concentrate and permeate (deproteinized whey) which may be used for animal feeding, microbial fermentation or lactose separation by crystallization [1]. Two major problems that limit the use of UF are membrane fouling and concentration polarization, which reduce the flux. The economy of the UF process depends on the maximization of the flux. The flux obtained in UF of whey depends on the operation parameters and the type of whey, since different fluxes have been reported for different whey types [2].

"Labaneh" is a popular type of fermented milk product in the eastern Mediterranean countries. Labaneh whey is the yellow–greenish aqueous portion of milk that separates from the curds during conventional labaneh manufacturing. It retains about 55% of the milk nutrients. The disposal of labaneh whey is considered a serious pollution problem facing the dairy industry.

There is little information published about labaneh whey. Batshon [3] studied the physical and chemical properties of labaneh whey. Table 1 shows some of these properties for fresh liquid labaneh whey collected from the Jordan Dairy Company (Al-Zarka / Jordan). Typical pH of labaneh whey is about 3.5, which is even lower than acid or sweet whey. There is more calcium, magnesium, phosphorus, and lactic acid but less lactose in labaneh whey, compared to sweet or acid whey [4]. This composition difference results in a different permeation behavior and different optimum operating conditions for labaneh whey during ultrafiltration. Proteins and calcium phosphates play an important role in membrane fouling and, consequently, membrane permeability [5].

The main fractions of typical whey proteins are β-lactoglobulin (60 %) with a molecular weight of 18 000 and β-lactalbumin (20%) with a molecular weight of 14 000.

There is little or no information available about the permeation behavior of labaneh whey in UF membranes. The objective of the present work is to investigate the effect of processing parameters on the permeation properties of labaneh whey in UF membranes.

Table 1. Chemical and Physical Analysis of Labaneh Whey from Jordan Dairy Company [3].

pH	3.4
Lactic Acid (w %)	1.568
Specific Gravity	1.03
Total Solid (w %)	6.2
Total Ash (w %)	0.9
Glucose (w/v %)	1.009
Lactose (w/v %)	1.234
Total Nitrogen (w %)	0.298
Protein (w %)	1.862
Fat (w/v %)	0.683

Our specific objectives were to determine the effect of temperature, pH, trans-membrane pressure, flow rate, total solid concentration, and pre-treatment of whey on the membrane performance (presented as flux) during UF of labaneh whey in polysulfonic hollow-fiber membranes. The effect of membrane nominal molecular weight cut-off (NMWCO) on flux is also investigated.

THEORY

The performance of UF membranes is dependent on a number of factors. Some of these factors are related to the membrane itself, such as the NMWCO of the membrane [6], or, in other words, its pore size. Operating parameters, on the other hand, influence the flux of whey permeate through UF membranes. Temperature [7–11], pH [2, 7, 9, 10], trans-membrane pressure [11–13], flow rate [11, 12], protein and solids concentration in whey [7, 8, 13, 14], and pre-treatment of whey [5, 15], are all factors that have been found to affect the flux of permeate, for both acid and sweet whey, in UF membranes.

Effect of Trans-membrane Pressure

The trans-membrane pressure can be calculated as [12]:

$$\Delta P_T = 0.5\,(P_i + P_o) - P_p \tag{1}$$

where ΔP_T is trans-membrane pressure, P_i is inlet pressure to the membrane, P_o is outlet pressure from the membrane, and P_p is permeate backpressure. Using the resistance in series model, the permeate flux from the membrane surface is related to trans-membrane pressure as [11]:

$$J = \frac{\Delta P_T}{R_m + R_P}\,, \tag{2}$$

where J is the permeate flux, R_m is the membrane's intrinsic resistance and R_p is the resistance due to the gel polarization layer. In case of pure water flux (J_w), there is no gel polarization. Consequently, $R_p = 0$ and Equation (2) becomes:

$$J_w = \frac{\Delta P_T}{R_m}\,. \tag{3}$$

R_p is proportional to ΔP_T and can be expressed as [11]:

$$R_p = \phi\,\Delta P_T\,, \tag{4}$$

where ϕ is a constant for a particular membrane-solute system under fixed operating conditions [11]. Substituting for R_p from Equation (4) into Equation (2) and dividing both the numerator and denominator of Equation (2) by ΔP_T. This equation can be written as:

$$J = \frac{1}{R_m / \Delta P_T + \phi}\,. \tag{5}$$

Effect of Protein and Total Solid Concentration

As the protein and total solid concentration increases in whey, the UF flux decreases with time. This is due to increased fouling during operation. Merin and Cheryan [16] expressed the change in flux (J) during the concentration process as:

$$J = J_o\,V^{-b}\,, \tag{6}$$

where J_o is flux at time = 0, when no fouling has taken place yet. V is cumulative permeate volume and b is a constant. Mass balance implies:

$$\frac{dV}{dt} = A\,J\,, \tag{7}$$

where A is the membrane surface area. It can be shown from Equations (6) and (7) that:

$$J = K\, t^{\frac{-b}{b+1}}, \tag{8}$$

where K is a constant. Equation (8) is particularly interesting because it can be applied to obtain empirical values of b and J_o from experimental data. This can be accomplished by plotting log (J) *vs.* log (t), then using slope to obtain (b), and intercept for J_o.

Permeate flux (J) is related to protein concentration by the so-called concentration polarization model. The relation takes the form:

$$J = k \ln\frac{C_m}{C_b}, \tag{9}$$

where k is the mass transfer coefficient through the membrane surface and C_m and C_b are protein concentrations at the membrane surface and the bulk fluid, respectively.

Effect of Flow Rate

Permeate flux through the membrane surface is directly related to the mass transfer coefficient (k) across the surface. K is generally related through the Sherwood number (N_{Sh}) to the Reynolds number (N_{Re}) and Schmidt number (N_{Sc}). Blatt *et al.* [17] suggest the following relationship:

$$N_{Sh} = \alpha\, N_{Re}^{\beta}\, N_{Sc}^{1/3}, \tag{10}$$

where α, β are constants. Experimental values of β are found in literature as 0.5 for laminar flow and 1.0 for turbulent flow [17–19]. Consequently, k is proportional to $v^{0.5}$ for laminar flow, and to v for turbulent flow, where v is the fluid velocity through the membrane.

The above equations will be used to qualitatively describe the experimental results of labaneh whey ultrafiltration.

EXPERIMENTAL

Apparatus

Three Hollow-Fiber Ultrafiltration membranes made from polysulfonic material were used. The NMWCO of these membranes were 5, 30, and 100 kDa. The membranes were bought from A/G Tech. Corp. (Needham, MA, USA). The specifications of the membranes are listed in Table 2.

Table 2. Specifications For Polysulfonic UF Membranes Used.

Cartridge Model	*NMWCO (Daltons)*	Fiber Internal Diameter (mm)	Surface Area (m^2)	Cartridge Housing Diameter (cm)	Cartridge Length (cm)
UFP-5-E-4A	5 000	1	0.032	1.9	36.2
UFP-30-E-4A	30 000	1	0.032	1.9	36.2
UFP-100-D-4	100 000	0.75	0.046	1.9	36.2

As shown in Figure 1, the membrane was connected to the whey feed tank through a peristaltic pump. Inlet and outlet pressures of the membrane were monitored and controlled using a pressure gauge and a control valve respectively. The permeate from the membrane was collected in a graduated flask, measured, then mixed with retentate, and returned to the feed tank to maintain fixed concentration of whey, except for concentration-effect experiments. In this case permeate was disposed and retentate from membrane was returned to the feed tank, allowing protein concentration to increase.

Whey's temperature was controlled with a heating coil immersed in the feed tank with stirring applied to ensure homogeneity. Temperature was monitored with two thermometers one placed on the feed tank and another on the feed pipe.

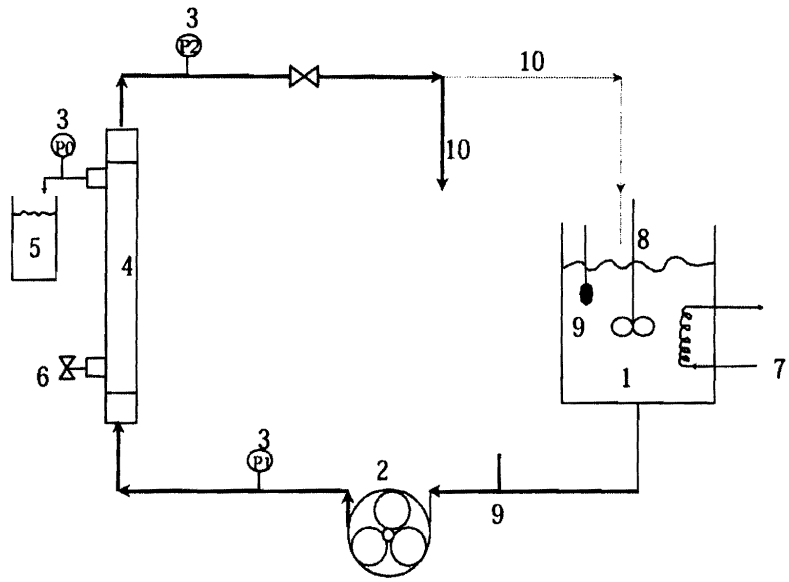

Figure 1. Schematic diagram of ultrafiltration membrane apparatus.

1. Feed tank *2. Peristaltic pump* *3. Pressure gauge* *4. Hollow-fiber membrane cartridge*
5. Permeate tank *6. Hose clamp, closed* *7. Heating coil* *8. Stirrer*
9. Thermometer *10. Retentate line: returned or disposed*

Materials

Labaneh whey was obtained from the dairy factory at the College of Agriculture/University of Jordan. The whey was obtained at pH slightly less than 3.5, with a green–yellowish color. The pH was adjusted to 3.5 using concentrated sodium hydroxide solution (approx. 2M) which was also added to the whey to raise its pH during pH-effect experiments. Distilled water, 50 mg/l sodium hypochlorite solution, and dilute phosphoric acid solution were used to flush and clean the membranes after each use. Cleaning continued until the water flux was about 80–90 % of the new membrane flux.

Procedure

Whey Pretreatment

Whey was pasteurized at 55 °C for 30 minutes and fat was removed from the surface by decanting. Additional fat removal was accomplished by suction filtration using Whatman-41 filter paper. In experiments where the goal was to study UF without pretreatment, whey was only pasteurized without fat decanting or pre-filtration.

Flux Measurements

In all the experiments, flux (l m^{-2} h^{-1}) was measured as an indicator of membrane performance. Flux was calculated by measuring the amount of permeate collected in a graduated flask over a known period of time.

Total Solids and Protein Concentration

Total solids was measured by taking samples from the tank (about 4 ml), and drying it in an oven at 96 °C for 5 hours. Solid content was obtained by measuring the weight of residual solids. Protein concentration was measured by the Kjeldahl analytical method [20].

RESULTS AND DISCUSSION

Effect of Trans-Membrane Pressure and Flow Rate

30 and 100 kDa membranes were used to study the effect of trans-membrane pressure (ΔP_T). The results are shown in Figures 2 and 3 for 30 and 100 kDa membranes respectively.

At very low values of ΔP_T ($\Delta P_T \cong 0$), the flux is independent of flow rate, varies linearly with ΔP_T, and is closer to pure water flux. This agrees with theory. When $\Delta P_T \rightarrow 0$ Equation (5) reduces to Equation (3). This indicates that in this ΔP_T region, membrane resistance is controlling and the gel layer is negligible. At higher ΔP_T, the effect of the gel layer is more significant and the curve levels off, because the gel layer resistance (R_p) becomes comparable to membrane's resistance (R_m), as can be interpreted using Equation (5).

It is observed from Figures 2 and 3 that increasing the flow rate increases the permeate flux. This can be interpreted as an enhancement in the mass transfer coefficient across the membrane wall, due to the increased flow rate, as suggested by Equation (10). Similar observations were found in the literature for skim milk [11] and acid whey [12].

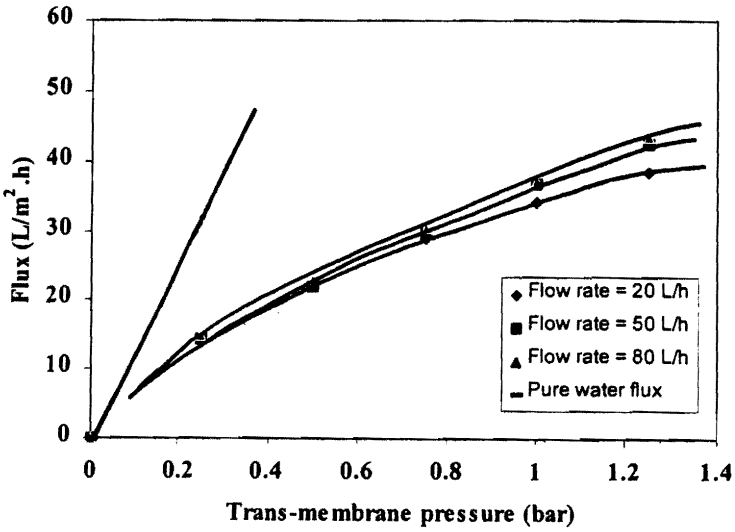

Figure 2. Effect of trans-membrane pressure on labaneh whey flux in a 30 kDa NMWCO membrane at T = 30°C and pH = 3.5.

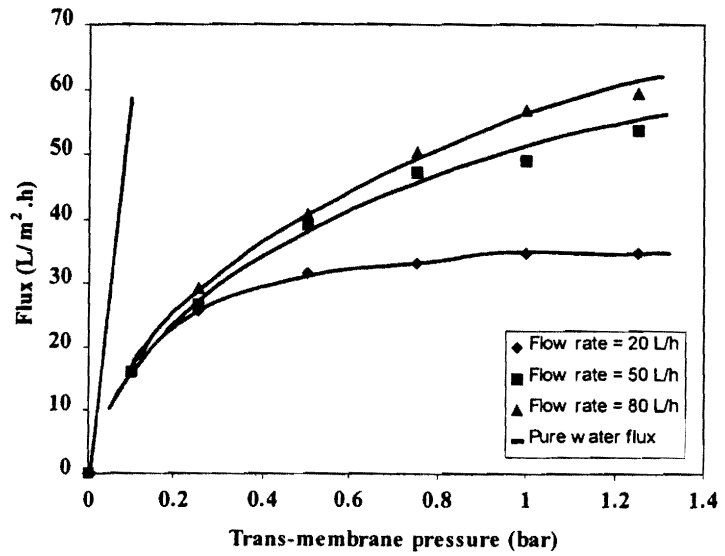

Figure 3. Effect of trans-membrane pressure on labaneh whey flux in a 100 kDa NMWCO membrane at T = 50°C and pH = 3.5.

Effect of Membrane's Pore Size and Pre-treatment of Whey

Figure 4 shows a comparison between the performance of 30 and 100 kDa membranes at whey flow rates of 20 and 80 l/h through the membrane. It is observed that larger membrane's pore size results in higher permeate flux. Larger pore size permits more water and other soluble contents to pass through the membrane by lowering membrane's intrinsic resistance. This leads to higher flux, and more efficient operation of the membrane. This conclusion about membrane efficiency could not be generalized, however, for larger pore sizes. It was observed during these experiments that the efficiency of the 100 kDa membrane in concentrating protein was very low. It is anticipated that the large pore size was behind the loss of protein to the permeate stream. Garoutte *et al.* [6] observed an increase in flux and a decrease in the yield of protein with increasing the pore size of the membrane.

Figure 5 shows the effect of pre-treatment of labaneh whey, on the performance of a 30 kDa membrane at 30 °C and flow rates of 20 and 50 l/h. An increase in permeate flux is observed after pre-treatment. Pre-treatment, which includes suction filtration, removes a considerable amount of fat and suspended solids from whey. This lowers the gel layer resistance at high ΔP_T and improves membrane performance. At low ΔP_T, the membrane's intrinsic resistance is controlling over the gel layer resistance, and pre-treatment does not improve the performance significantly. Delaney *et al.* [21] observed an improvement in membrane's performance when cheese whey was pre-treated. Other similar observations on cheese whey are reported elsewhere [15].

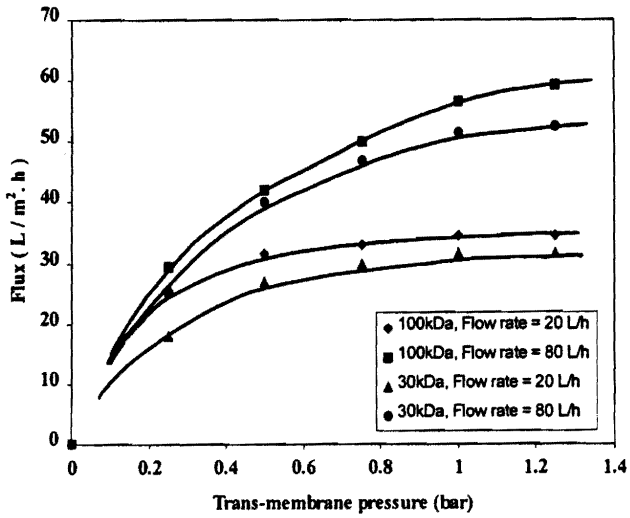

Figure 4. Effect of pore size on labaneh whey flux at T = 50°C and pH = 3.5.

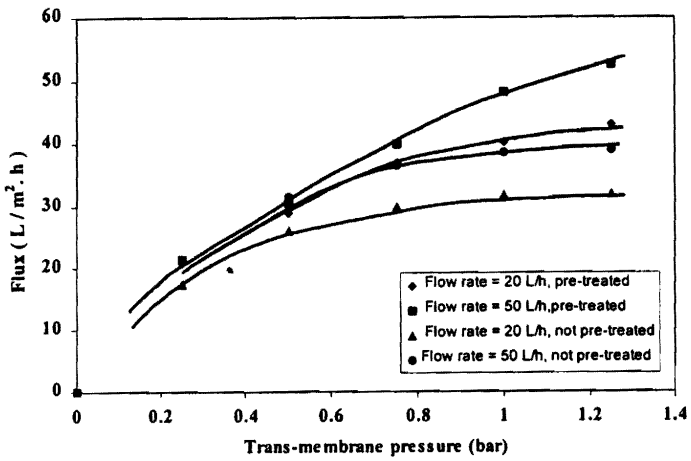

Figure 5. Effect of pre-treatment on labaneh whey flux in a 30 kDa membrane at T = 30°C and pH = 3.5.

Effect of pH

Figure 6 shows the flux change with pH for a 30 kDa membrane at 50 °C and 0.5 bar trans-membrane pressure. As can be seen, labaneh whey flux decreases when pH increases. Labaneh whey contains larger amounts of calcium, mainly present as tri-calcium-phosphate (TCP), than sweet or acid whey [4]. TCP is less soluble at high pH, and tends to form colloids. These colloids plug the membrane pores and cause fouling. Flow rate, as Figure 6 illustrates, has a less significant effect at higher pH, because fouling by TCP becomes severe and dominant.

It is reported in the literature [2] that the pH of cheese whey has affected the distribution of calcium and phosphorus content in the permeate. A decreasing flux as pH increases is also reported in the literature for acid whey [7]. On the other hand, an increasing flux as pH increases is reported for skim milk [10].

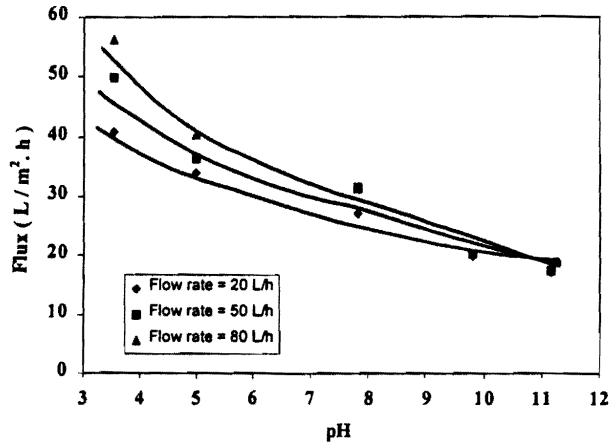

Figure 6. Effect of pH on labaneh whey flux in a 30 kDa membrane at T = 50°C and transmembrane pressure = 0.5 bar.

Effect of Temperature

Raising the temperature was found to increase the flux for all flow rates studied; 20, 50, and 80 l/h, as illustrated in Figure 7. The main reason for this trend is the reduction in viscosity with temperature increase. As viscosity decreases, the mass transfer through the membrane surface is enhanced. This can be interpreted from Equation (10). Lower viscosity leads to higher Reynolds number and lower Schmidt number. The net effect of these changes is an increase in (k) as the viscosity drops. Figure 7 also indicates that flow rate has a strong influence on flux at higher temperatures. Similar trend was found in the literature for acid whey [7] and for skim milk [9–11].

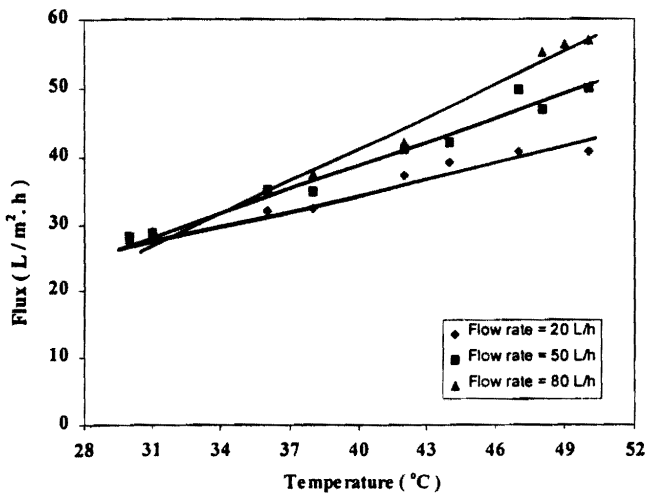

Figure 7. Effect of temperature on labaneh whey flux in a 30 kDa membrane at pH = 3.5 and transmembrane pressure = 0.5 bar.

Effect of Protein Concentration

When retentate from the UF membrane was returned to the feed tank, and permeate was disposed, protein and total solids are concentrated in whey, as expected. Figure 8 shows how the flux from 5, 30, and 100 kDa membranes changed with time during a concentration experiment. Labaneh whey at 50 °C was used in these experiments with flow rate of 80 l/h and 1.125 bar trans-membrane pressure. As Figure 8 indicates, flux decline is clear for the 30 and 100 kDa membranes, while the small pore size membrane (5 kDa) gives low flux value with very small decline over 15 hours experiment. It is observed, however, that the decline in flux is more pronounced at initial time intervals. This is clearer in the 30 kDa membrane. In this case, for example, the flux was 36% of its initial value after 5 hours, and 14% of its initial value after 15 hours. During the initial period of time, a gel layer forms on the membrane surface, and this layer becomes thicker as the protein and ash concentrate in whey. This causes a rapid decline in flux. After the initial period, the gel layer attains a fixed thickness and the resistance of this layer becomes essentially constant. Consequently, the flux levels off during the later time period. This trend is described by Equation (8).

The change in flux *versus* the change in protein level, which accompanies the concentration process, is plotted in Figure 9 on a semi-logarithmic scale. As protein concentration increases, flux decreases, which indicates that protein in whey contributes to the gel layer formed and to membrane fouling.

The trend line of the data presented in Figure 9 indicates that the flux varies linearly with log (C_b), which means that the concentration polarization model presented by Equation (9) applies for labaneh whey.

Declining flux with time was reported in the literature [7, 13, 14] for different types of whey and dairy products. Other literature stated the decline of flux with increasing protein concentration in acid whey [8] and cheese whey [21].

In a batch concentration experiment, with permeate rejection and retentate recycling the concentrations of protein and total solid in labaneh whey increase as the whey volume drops (Figure 10). It is observed that protein and total solid concentration increases almost linearly with percentage of volume reduction (up to 70 %). At higher volume reductions, the concentration of protein and solids increases, membrane fouling increases, resulting in even higher rejection of protein and solids by the membrane surface. Hence, faster concentration of protein and total solids takes place (Data not shown). May *et al.* observed a similar trend with cheese whey [8].

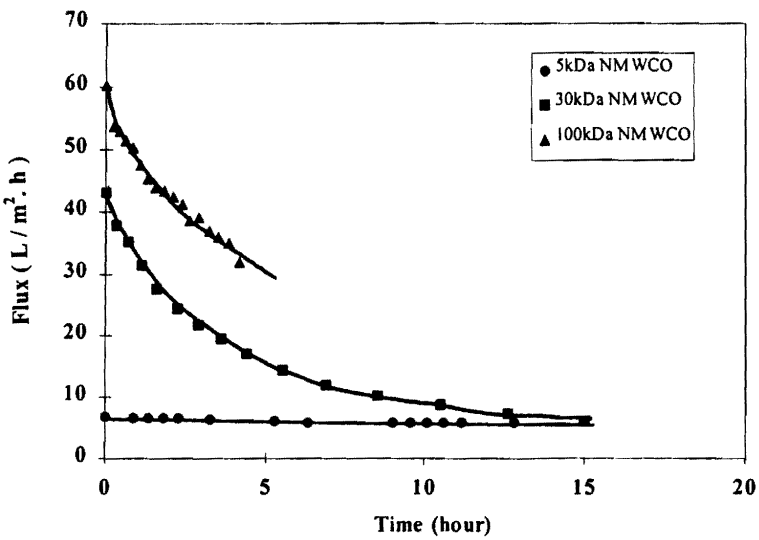

Figure 8. Change of labaneh whey flux with time during a protein concentration experiment at pH = 3.5, T = 50°C, flow rate = 80 l/h, and transmembrane pressure = 1.125 bar.

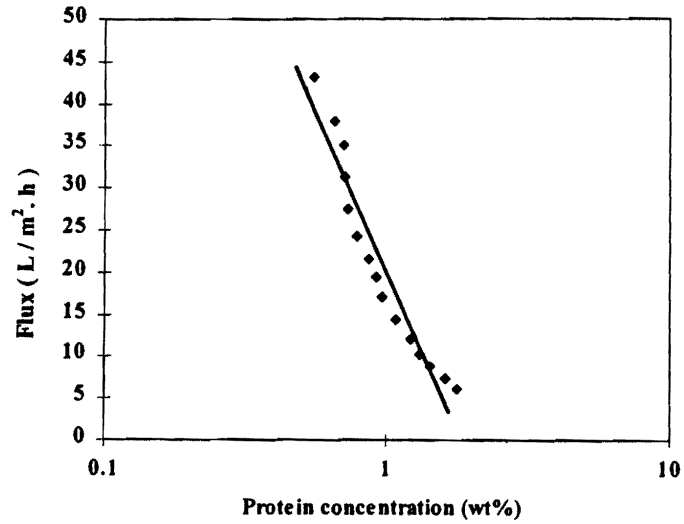

Figure 9. Flux change with protein concentration in a 30 kDa NMWCO membrane at T = 50°C, pH = 3.5, flow rate = 80 l/h, and transmembrane pressure = 1.125 bar.

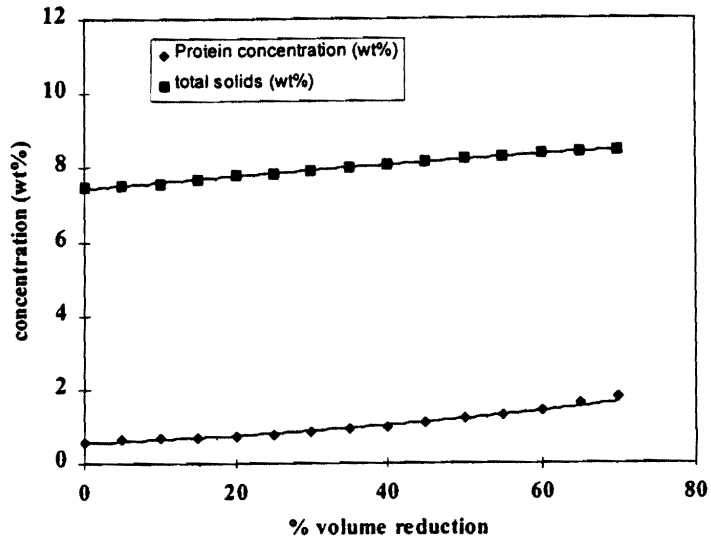

Figure 10. Change of protein and total solid concentration in labaneh whey vs. *volume reduction during a concentration experiment. 30 kDa membrane at T = 50°C, pH = 3.5, flow rate = 80 l/h, and P = 1.125 bar.*

CONCLUSIONS

The effect of operating parameters on the performance of the UF process for labaneh whey in hollow-fiber membranes was investigated. It was found that increasing the temperature, flow rate, trans-membrane pressure, and NMWCO of the membrane has enhanced the permeate flux. Increasing the pH, on the other hand, has reduced the permeate flux. This was attributed to the TCP-colloids formation, which plug the membrane and cause fouling. This phenomenon is particularly important in labaneh whey, in which calcium and magnesium contents are higher than those in acid and sweet whey. It was found that the conventional resistance in series model established in the literature could describe the effect of operating parameters on labaneh whey flux. Pre-treatment of labaneh whey by removing fat was also found to increase the flux of the permeate significantly.

The UF process was found to be successful in concentrating protein in labaneh whey when a 30 kDa membrane was used. This provides a potential for utilizing UF in producing labaneh whey protein concentrate. However, the 100 kDa membrane was found to be inefficient in concentrating protein due to its permeability for protein.

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NOMENCLATURE

A	membrane surface area (m^2)
b	exponent in Equation (6)
C_b	protein concentration in bulk fluid (wt%)
C_m	protein concentration on the membrane surface (wt%)
J	flux from membrane surface (l m^{-2} h^{-1})
J_o	flux at time = 0 (l m^{-2} h^{-1})
J_w	pure water flux (l m^{-2} h^{-1})
k	mass transfer coefficient through the membrane surface (mole m^{-2} s^{-1})
K	constant factor in Equation (8)
NMWCO	nominal molecular weight cut-off
N_{Re}	Reynolds number
N_{Sc}	Schmidt number
N_{Sh}	Sherwood number
P_i	inlet pressure to the membrane (bar)
P_o	outlet pressure from the membrane (bar)
P_p	permeate back pressure (bar)
ΔP_T	trans-membrane pressure (bar)
R_m	membrane's intrinsic resistance
R_P	gel layer resistance
t	time (h)
TCP	tri-calcium-phosphate
UF	ultrafiltration
V	cumulative permeate volume (m^3)
v	fluid velocity through the membrane tubes (m s^{-1})

Greek Symbols

α	constant factor in Equation (10)
β	exponent in Equation (10)
ϕ	factor in Equation (4)

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