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Investigation of microfiltration for pretreatment of whey concentration

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ABSTRACT

Whey, which contains large amount of food protein, is the liquid residue of cheese and casein production. Direct discharge of whey means nutrition waste and environmental pollution. Recently, the membrane technology has great applications in milk industry field. The current studies are focused on the study of the concentration process applying ultrafiltration (UF) membranes for whey recovery. How to pre-treat whey effluent is key point for whey preparation by the UF method. In this study, the application of microfiltration for pre-treatment of whey protein concentration was studied in details. Two types of microfiltration hollow fibre membranes, polyethersulfones (PES) and Polyvinylidene Fluoride (PVDF), were investigated. It was found that the application of microfiltration helped to obtain an enhanced flux for whey ultrafiltration process. No fat and microorganisms were found in the permeate of microfiltration. The filtration characteristics were obviously influenced by the operation parameters, such as pressure, temperature, recycling flow rate, pH and concentration factor. In addition, a preferable cleaning method was proposed. The cleaning method with order of 0.1% NaOH, 200 ppm NaClO and 0.5% NaOH was more efficient for the PES membrane and PVDF membrane.

Keywords: Whey protein; Pre-treatment; Microfiltration; PES; PVDF; Membrane cleaning

1. Introduction

Whey, the liquid residue of cheese production, contains large amount of food protein. Despite of the shortage of protein in the world, most of the whey is charged directly or used as animal feed. How to use the whey has been a focus topic in the dairy industrial field [1]. Developments in microfiltration (MF) technique have created the opportunity for many applications in food industry. In the milk and dairy industry, bacteria removal and selective fractionation of milk fat and removal of whey fat, cheese brine purification using microfiltration are reported [2]. With lower pressure, MF can entirely replace the centrifugal separations as the pre-treatment of cheese whey. Significant improvement was achieved both for the recovery of butter fat as well as increasing permeate fluxes of UF membrane in the whey concentration process [3–5].

Fouling is defined as the organic and inorganic deposited on the membrane surface, which increases the membrane resistance and causes a decrease in the permeate flux. To overcome this problem, a proper cleaning process must be investigated. The washing agents should have the characteristics such as chemical stability, safety, low cost and easy to clean [6]. These cleaning agents also must be able to dissolve most of the precipitated materials and take them away from the surface while they should not damage the membrane surface [7].

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The purpose of this study is to discuss the utilization of microfiltration for pre-treatment of whey protein concentration. The influence of microfiltration on whey ultrafiltration process was investigated. The effects of operation parameters and a preferable cleaning method on the performance recovery of the membranes were observed.

2. Materials and methods

2.1. Whey composition

Model whey feed was prepared as follow: Whey powder was dissolved in diluted water in appropriate proportion (60 g dry powder per litre of water) to obtain the normal composition of 6 g protein per liter of typical whey. Model whey powder was produced by James Farrell & Co., USA. Protein content was calculated by Bradford Method [8]. Lactose concentration was determined according to the Chinese standard GB/T5413.5-1997, and fats were measured according to Chinese standard GB/T5413.3-1997. The pH of solutions was determined by pH meter.

2.2. Membranes

Two different microfiltration membranes and one ultrafiltration membrane have been investigated. The polyethersulfones (PES) and polyvinylidene fluoride (PVDF) membranes, with the membrane pore size of 0.1μm were tested. The effective area of every membrane sample was between 150 cm^2 and 250 cm^2. The polysulfone (PS) membrane with the MWCO of 20000Da was used for concentrating the whey protein from whey solution.

2.3. Microfiltration of whey

The experiments were carried out in a lab unit. The temperature of feed (20–45°C) was maintained by using a thermostat bath. The pressure (0.02–0.1 MPa) was controlled by a regulation valve and the recycling flow rate (80–160 l/h) was controlled by controlling the rotation speed of the pump. In addition, the other parameter, concentration factor was studied. Permeate flux, protein permeation and protein content in the permeate were measured during the experimental runs.

The measured filtration characteristics were defined as follows:

- Protein permeation (P):

  $P = (C_P / C_R) 100\%$

  where C_P—solute concentration in the permeate (g/l); C_R—solute concentration in the whey (g/l).

- Concentration factor (F):

  $F = V_F / V_R$

  where V_F—volume of feed (L); V_R—volume of retentate (L).

2.4. Membrane cleaning

The cleaning of the fouled membrane after filtration experiments was directly carried out in the same apparatus. Nine types of cleaning agents were tested, as shown in Table 1. The cleaning time was firstly 15 min by deionized water and then 30 min by cleaning agents.

3. Results and discussion

3.1. Effect of microfiltration

The influence of microfiltration on the whey composition is shown in Table 2. The results show that the most of the protein and almost 100% of the lactose were permeated. The reduction of protein is due to the unavoidable adsorption by microfiltration membrane in the process. No fat was found in the permeate of microfiltration. It demonstrates that the microfiltration process had significant effect on removing fat.

Table 1
Cleaning agents

Number	Solutions
1	deionized water
2	0.5% NaOH
3	1.0% NaOH
4	200 ppm NaClO
5	0.1%NaOH+(200 ppm NaClO + 0.5% NaOH)*
6	0.5% HCl
7	1.0% HCl
8	1.0% HCl + 1.0% NaOH
9	200 ppm H_2O_2

*The membranes were flushed by 0.1% NaOH, and then immersed by 200 ppm NaClO and 0.5% NaOH.

Table 2
Whey composition of model whey and MF whey

Parameters	Feed	PES permeate	PVDF permeate
protein (g/l)	5.98	4.82	5.03
Lactose (g/l)	38.5	38.3	38.3
Fats (g/l)	0.12	No detected	No detected
pH	6.75	6.73	6.72

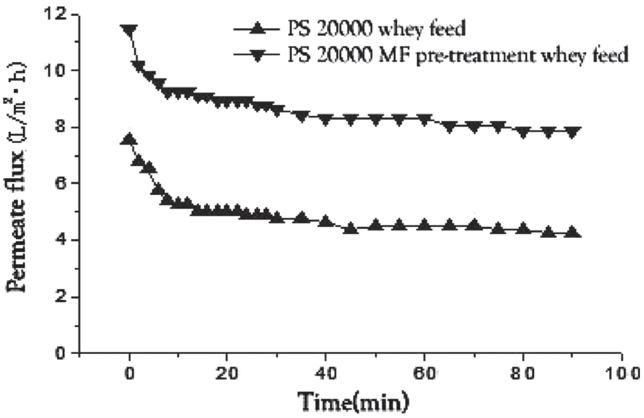


Fig. 1. Effect of MF pre-treatment of whey feed on ultrafiltation performance (0.04 MPa, 30°C, 120 l/h).

The effect of microfiltration on permeate fluxes of UF for model whey solution is shown in Fig. 1, which shows that the permeate fluxes of MF whey are approximately double to that of the whey feed fluxes. It can be seen that microfiltration may reduce the flux decline of subsequent UF process resulting from membrane fouling as well as concentration polarization.

3.2. *Microfiltration of whey*

3.2.1. *Effect of trans-membrane pressure*

The influence of the pressure on the permeate flux and protein permeation performance is shown in Fig. 2. It can be seen from Fig. 2(a) that the permeate flux increased with trans-membrane pressure in the range of 0.02–0.1 MPa, but did not have significantly influence. As shown in Fig. 2(b), the protein content increases with an increase in the pressure and the the protein permeation of PES and PVDF membrane are up to 87.1% and 92.7%, respectively. The protein permeation performance of PVDF membrane was better than the PES membrane.

3.2.2. *Effect of temperature*

The effect of temperature on the permeate flux is illustrated in Fig. 3. The permeate flux increases with an increase in temperature. It can be believed that an increase in the temperature results in a decrease in the viscosity of whey solution. High temperature increases the solute diffusivity and the transport rate of the solutes from the membrane surface into the stream. The higher operating temperature resulted in an increase in the permeate flux until 40°C, where the viscosity of the processed whey reaches its minimum value and here after temperature increase can cause heat denaturation of the whey proteins.

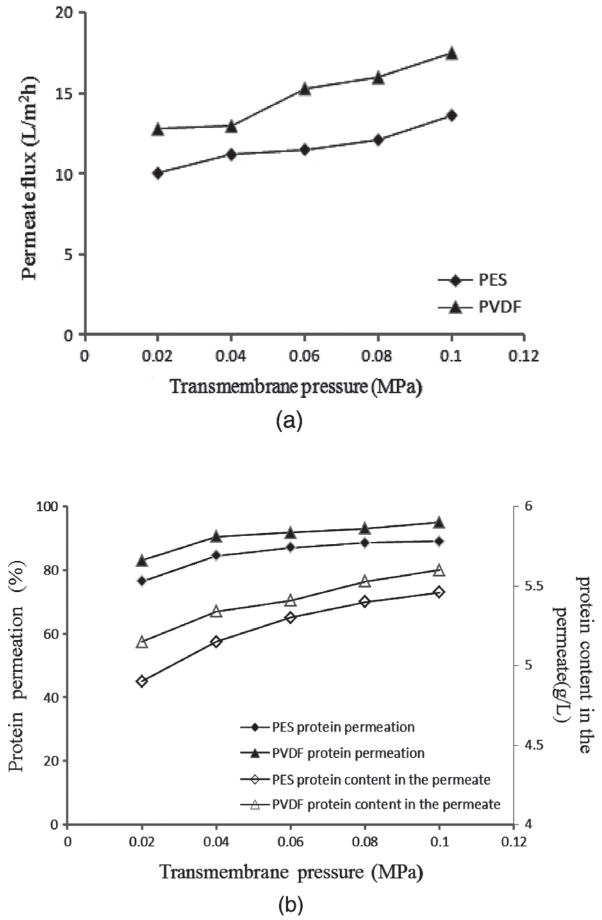


Fig. 2. Effect of trans-membrane pressure on the permeate flux and protein permeation performance (30°C, 120 l/h): (a) Effect on the permeate flux (b) Effect on protein permeation performance.

3.3. *Effect of recycling flow rate*

The effect of recycling flow rate on the permeation is shown in Fig. 4. The flux increased linearly with recycling flow rate within the tested range. The deposition of the membrane surface can be removed continuously when a higher recycling flow rate is applied, so that the hydraulic resistance of the fouling layer is reduced. But continued gain in flux is limited by energy and the protein content in the permeate did not significantly increase with recycling flow rate.

3.4. *Effect of pH of whey feed*

The pH value of the solution changes the electric charge of protein molecules. At the isoelectric point of 5.3, whey protein aggregates and reaches its lowest solubility. As a result, protein deposits easily on the surface of the membrane and forms a fouling layer, which causes a decrease on the permeate flux (Fig. 5). Above

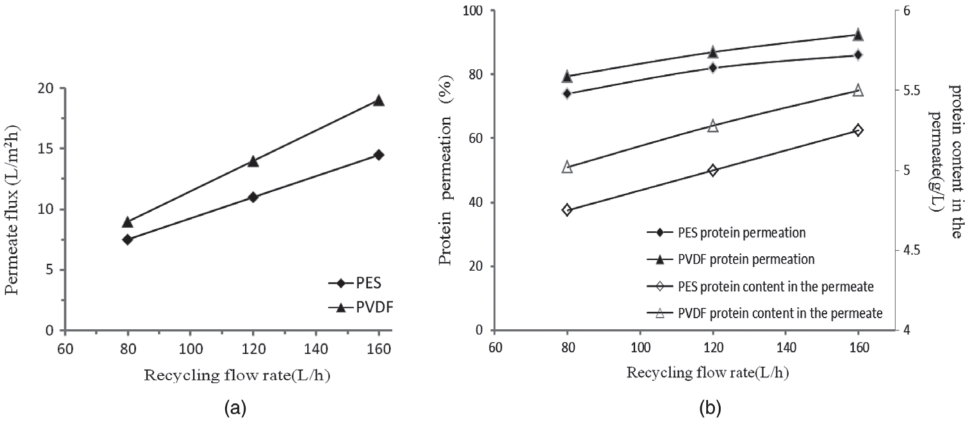


Fig. 3. Effect of temperature on the permeate flux and protein permeation performance (0.04 MPa, 120 l/h): (a) Effect on the permeate flux (b) Effect on protein permeation performance.

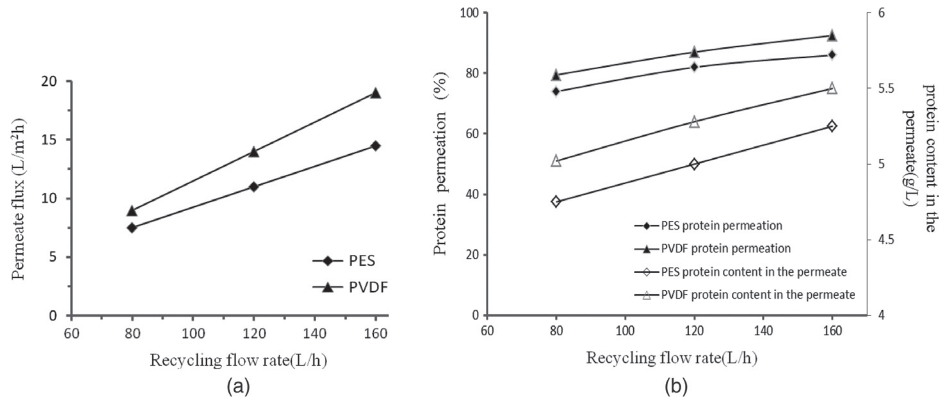


Fig. 4. Effect of recycling flow rate on the permeate flux and protein permeation performance (0.04 MPa, 40°C): (a) Effect on the permeate flux (b) Effect on protein permeation performance.

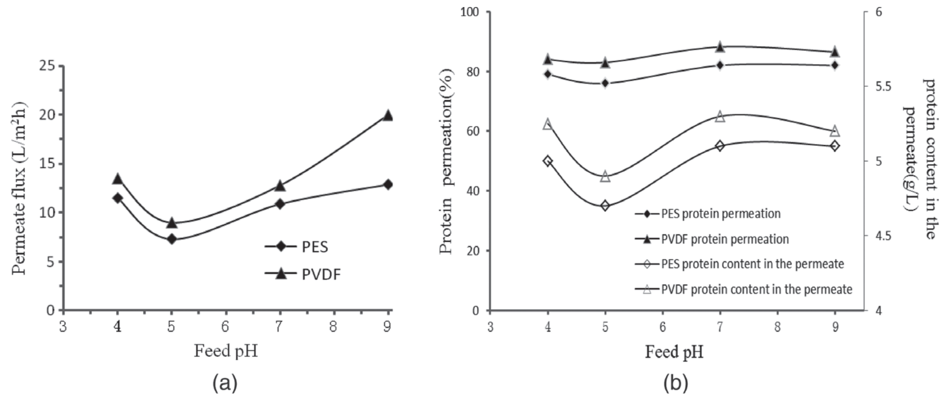
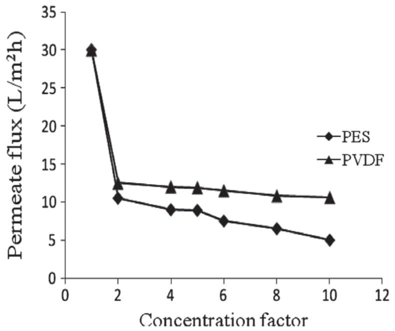


Fig. 5. Effect of feed pH on the permeate flux and protein permeation performance (0.04 MPa, 40°C, 120 l/h): (a) Effect on the permeate flux (b) Effect on protein permeation performance.

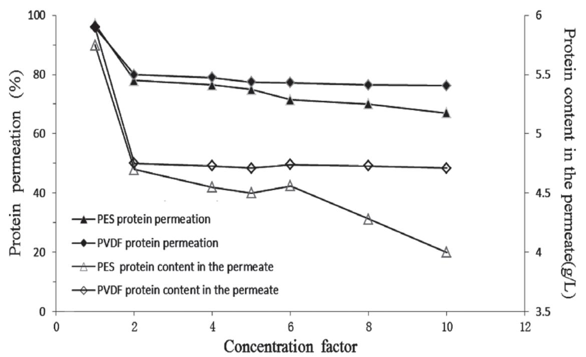
the isoelectric point, protein shows electronegative and the same electric charge prevents the adsorption to the membrane. Below the isoelectric point, protein is electropositive which inclines to adsorb on the membrane, and as a result the permeate flux is lower than that of feed at high pH. The pH higher than 9.3 has not been investigated because it might cause the denaturation of whey protein.

3.5. Effect of concentration factor

The influence of the concentration factor on the permeate is shown Fig. 6. It can be observed that as the concentration of protein increased the permeate flux decreased. At concentration factor higher than 2, the flux gradually approaches a steady state. This fact can be explained as a consequence of the concentration polarization layer formation on the membrane surface. At the same condition, the PVDF membrane had a higher permeate flux and better protein permeation performance than the PES membrane.



(a)



(b)

Fig. 6. Effect of concentration factor on the permeate flux and protein permeation performance (40°C, 120 l/h): (a) Effect on the permeate flux (b) Effect on protein permeation performance.

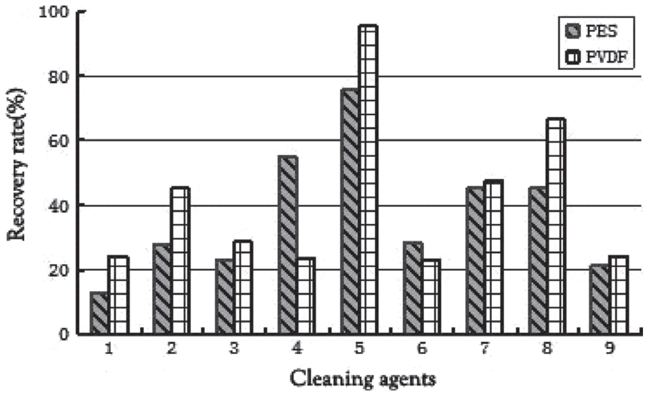


Fig. 7. Recovery rate of membrane performance.

3.6. Membrane cleaning

As an important step in membrane process, cleaning method was also investigated. To compare the cleaning effects, the fouled membranes under the same condition were cleaned with different methods. HCl is often used to wash away the inorganic substance while NaOH dissolves and removes the protein. NaClO and H_2O_2 have better effect on the striping of organic substance from the membrane. As shown in Fig. 7, the cleaning method with order of 0.1% NaOH, 200 ppm NaClO and 0.5% NaOH was more efficient for the PES membrane and PVDF membrane.

4. Conclusions

It is concluded that microfiltration method as a pretreatment before ultrafiltration concentration of whey can be successfully used.

The application of microfiltration as pre-treatment had an important impact on protein concentration. It helped to obtain an enhanced flux for whey ultrafiltration process and had significant effect of removing fat. The permeate fluxes of MF whey are approximately double the whey feed fluxes. The protein permeation of PES and PVDF membrane are over 85% and 90%, respectively.

The best operating conditions for the process were 0.04 MPa, 40°C and the recycling flow rate was 120 l/h. At the protein isoelectric point, the flux was in low value. High pH was benefit to whey permeation. An increase of concentration factor in the feed decreases the permeate flux and protein permeation. The cleaning method with order of 0.1%NaOH, 200 ppm NaClO and 0.5% NaOH was more efficient for the PES membrane and PVDF membrane.

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