

Examination of Whey Degreasing by Modified Membrane Filtration

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Abstract: The largest amount of dairy by-products, especially the whey, comes from the manufacture of cheese. The whey proteins are used in several different industry technologies. The forage production is used for animal feeding in the forms of various flours mixed in feeds, and the food industry uses whey proteins as human nutrition, such as different dry soups, infant formulas and supplements. The fat components of whey may inhibit the efficient processing and might impair the use of whey in these technologies. Thus, the aim of the experiment was to investigate a cheap and economical separation of the lipid fraction of whey. This separation method was made by microfiltration, which is an inexpensive, effective and energy efficient method for this task. During the measurements, 0.2 μm and 0.45 μm microfiltration membranes were used in a laboratory tubular membrane filtration module, and the membrane separation method was combined and modified by using astatic mixer and/or air insufflation. The same pore size membranes were used in a vibrating membrane filtration equipment (VSEP), too. The two different membrane filtration devices allowed the comparison of the effect of vibration and the effect of the static mixer and/or air insufflation. The flux values above 0.2 MPa transmembrane pressures strongly decreased on using the tubular membrane. Therefore, it can be determined that the use of the lower transmembrane pressures gave better flux combined with air insufflation and the use of static mixer. The flux values increased three times higher with using vibration during the microfiltration process than that without vibration. Comparing these methods, it can be concluded that the separation made on tubular membrane (0.2 μm) combined with static mixer gave sufficient result according to the degreasing, retentions and flux values of the other components.

Key words: Degreasing, membrane filtration, vibrating membrane filtration equipment, air insufflation, static mixer.

1. Introduction

The whey contains a number of several components, such as, lactose, vitamins, proteins, minerals and lipids. Whey cream is more salty, tangy and “cheesy” than “sweet” cream skimmed from milk, and this is the first reason to be used to make whey butter. The second reason of use of degreasing is the further processing of whey for dry powder or nutritional supplement for food industry. The degreasing method made by membrane filtration is a completely new technology. This new process of degreasing with a serious problem to be solved is the low flux and high resistances values during the separation proceedings

[1]. These effects could be reduced by using different methods combined with the filtration process, i.e., using static mixer and/or air insufflation, or vibration [2].

Newtonian fluids, such as, an aqueous solution, are being turbulent flow in most industrial applications, but within small diameter tubes or narrow niches, the turbulence is not high enough to develop adequate shear force to build adequate flux [3, 4].

The use of static mixers or air insufflation was better efficient during the experiments than increasing the speed of turbulence or increasing the pressure [5, 6]. The flux is increased when these new methods were used, and the operating costs are decreased at tubular membrane filtration equipment [7].

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The rapid fouling of the membranes is the biggest factor that prohibits the filtering procedures. In the near future, it will be the most important issue in the membrane separation processes to be solved [8]. The fouling of the membrane is possible to decrease with using different pre-treatments, such as, insufflation of a gas into the liquid [9]. The insufflation of a specific gas-air was used in the present work directly into the fluid, and it created a two-phase gas/liquid flow before the membrane module [10]. The efficiency of the filtration process is influenced by the direction of the flow (up or down) and the position of the membrane (vertical or horizontal location) [11]. The air insufflation method is limited by the gas distribution, the composition of gas, the parameters of equipment and the management of this process [12]. The vibratory shear enhanced process (VSEP) is a rather new technology of membrane filtration processes. The VSEP technology could be another possibility to decrease the total membrane filtration resistance [13]. The particle displacement at the membrane surface increased or decreased by varying the frequency of vibratory motor of membrane module [14]. The lifetime and the usage time of filtration membrane can be increased by using VSEP, and the operational costs can be decreased, too [15]. The polarization layer, the resistance values and the fouling were measured by the effect of vibration, and the changes of retention values were measured by the effect of increasing the vibrational amplitude [16]. The aim of this study was to see which type of combined membrane processes gave the best results for the separation of the lipid fraction of whey, and also to investigate the cheapest and most economical way of combined membrane process.

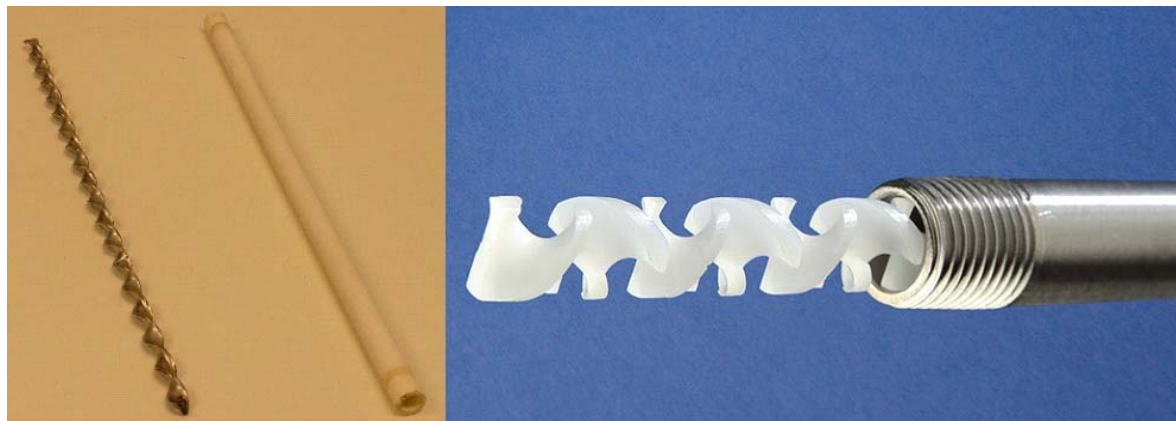
2. Materials and Methods

In this study, sweet cheese whey was used for the experiments, which base material came from the Soma Budapest Ltd. Dairy Industry Company. The sweet whey's basic analytical parameters were measured by

Bentley B150 milk analyzer device, and data showed that on the weight base, the fat content was 0.18%, the protein content was 0.33%, the milk sugar content was 2.61%, the dry materials were 3.72% and the total protein content was 0.47%.

The air insufflation and the static mixing method were implemented separately or complementing each other by tubular and hollow fiber membranes. 0.45 μm and 0.2 μm cut-off value membranes were used in the laboratory tubular filtration equipment. The membrane was 0.25 m length, and one tube which has an internal diameter of 0.007 m was included in it. The applied static mixer was a 0.25 m length Helix type helical ribbon screw static mixer (made by metal material and produced by StaMixCo Ltd.), with a pitch of 0.006 m and an inner radius of the mixer of 0.0035 m (Kenics™, Helix) (Fig. 1). The Kenics™ type helical twist bowtie static mixer (made by plastic material) was used also with a length of 0.241 m, and a thickness of 0.001 m and a diameter of 0.00635 m. The Kenics™ mixer has more different mixing elements than the Helix type that are configured by helically twisted and rigid bowtie plates. These bowtie shaped mixing elements are fixed up tightly one after another. The blades are twisted 180° in both directions. The filtration device was a specially customized equipment. Where the tubular membranes were used with or without the static mixers, the air insufflation was introduced into the equipment before the membrane module. The air insufflation was performed on 50, 100, and 150 L/h feed recirculation flow rate, 0.2 MPa transmembrane pressure and 20 L/h air insufflation rate. The initial amount of the feed material was 2 L of sweet whey in every measurement. The temperature was 30 °C during the tests. The airflow was blowing into the fluid flow before the membrane module.

The VSEP set was produced by New Logic International Corporation. The equipment can be used in two different modes—L-mode (laboratory methods) and P-mode (pilot methods). The device was used at



(a) Helix type helical ribbon screw static mixer

(b) Kenics™ helical twist bowtie static mixer

Fig. 1 The Helix type helical ribbon screw static mixer from StaMixCo Ltd. and the Kenics™ helical twist bowtie static mixer.

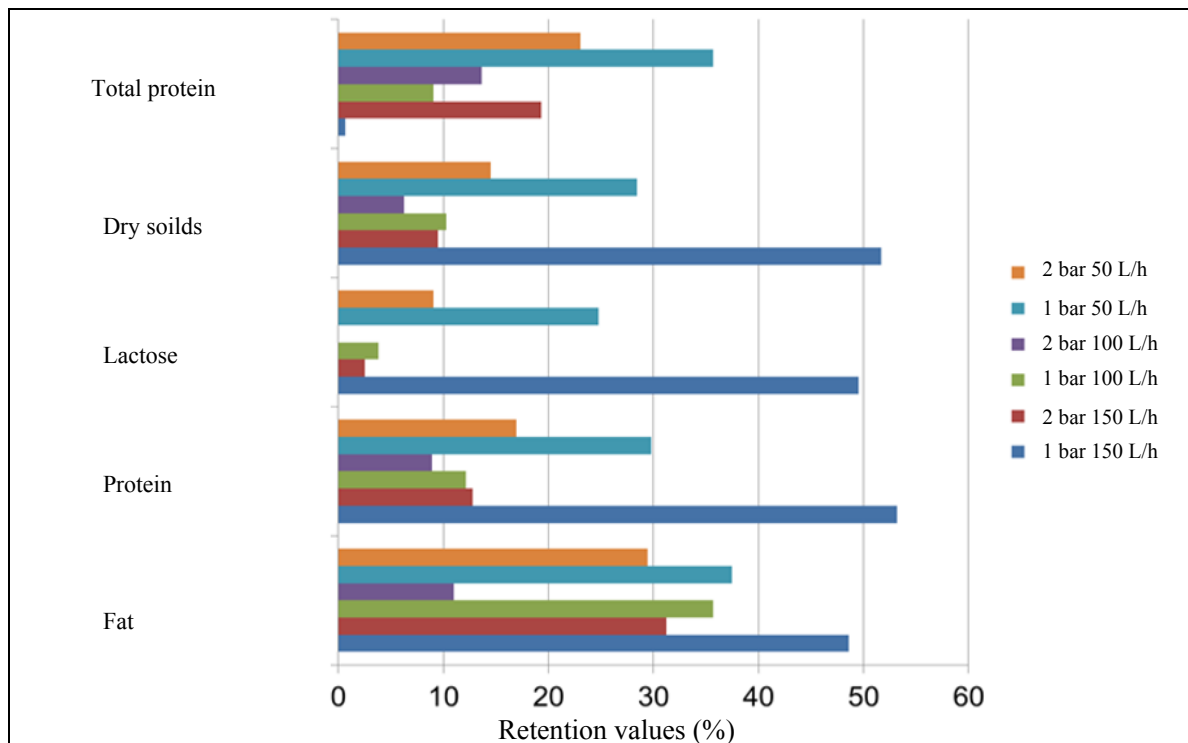


Fig. 2 The retention values (%) of different components measured at the different transmembrane pressure (bar) and flow rate (L/h) on 0.45 μm cut-off value tubular membrane.

L-mode, which comprises one disk-shaped membrane with an active filter surface 503 cm^2 . The VSEP system's disk-shaped flat-sheet membrane (produced by NewLogic Corp.) was placed in the filtration module, which is attached to a central shaft. This central shaft can be rotated in a short distance between at a frequency of 50-54 Hz. In this study, the 54 Hz frequency value was used with 0.2 μm cut-off value

membrane (made of polyethersulfone). During the measurements, the transmembrane pressure was at 0.3 MPa. In this equipment, the initial amount of feed was 10 L of sweet whey.

3. Results and Discussion

In the tubular membrane filtration equipment, the retention values were measured at different

recirculation flow rates and transmembrane pressures by a 0.45 μm cut-off value membrane (Fig. 2). The most important goal was to hold back the fat molecules as much as possible and the other particles released onto the concentrate.

When 0.1 MPa transmembrane pressure and 150 L/h recirculation flow rate were used, then the maximum amount of fat micelles was retained. The other components, such as the proteins, lactose and dry solids, were restrained also at the maximum amount, when the same filtering parameters were used. These measured results were not sufficient to solely degrease the feed material, because the rate of degreasing was less than 50%, so the 0.45 μm pore size's membrane was too large for this task.

The work was continued with 0.2 μm cut-off value tubular and capillary type membranes, where better retention values were measured at using a lower transmembrane pressure value (0.1 MPa). The capillary type membranes gave better flux values than the tubular membranes, but the retention values of different components were also important,

therefore these experiments were continued with the tubular type membranes. As we realized, the use of higher transmembrane pressure value with high level of recirculation flow rate were made the worst retention values. The trend was similar in both membranes. The retention values of the other components were increased also at this pore size membrane. Fig. 3 presents that the static mixer has a decreasing effect on the retention of protein, lactose and dry solids. It also showed that only the fat retention values were increased by using the static mixer during the filtration process. The higher amount of fat retention values was obtained at using 0.2 MPa transmembrane pressure and recirculation flow rate (q_v): 100 L/h. The tubular membranes gave better fat retention values than the capillary membranes. In view of the goal to minimize the fat content and keep the other components in the concentrate, the data clearly demonstrate that the 0.2 μm tubular membrane combined with static mixer gives the best result compared to the other procedures.

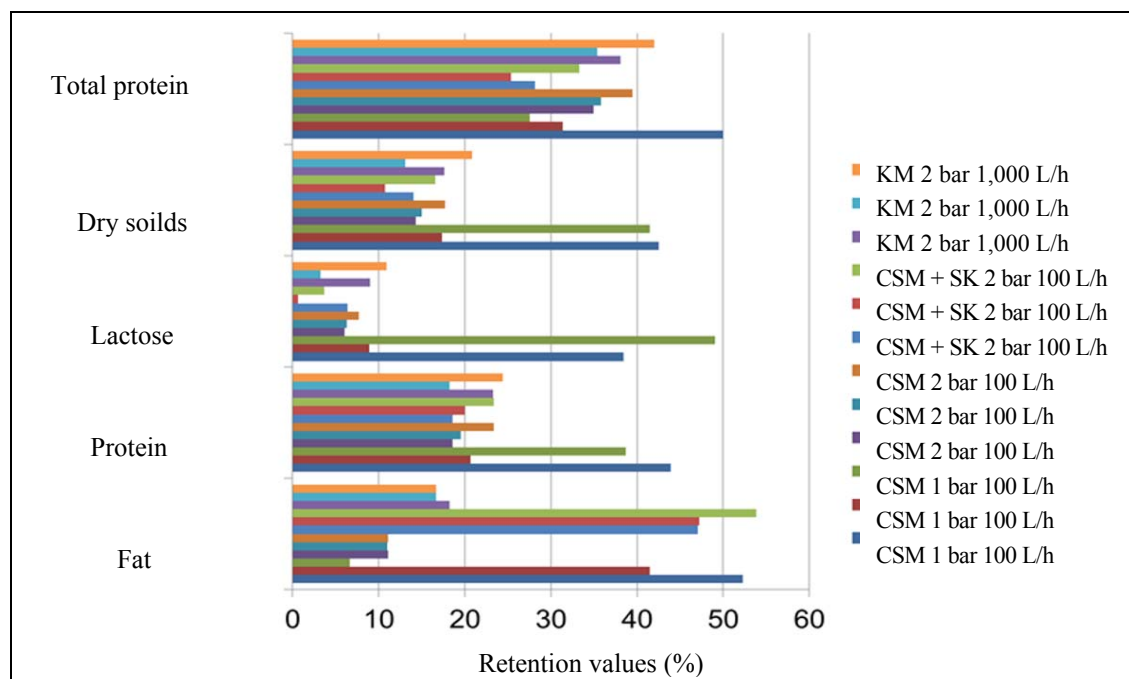


Fig. 3 The retention values (%) of the different components measured in 0.2 μm cut-off value tubular membrane with CSM + SK and without static mixer.

KM: capillary membrane; CSM: tubular membrane; SK: static mixer.

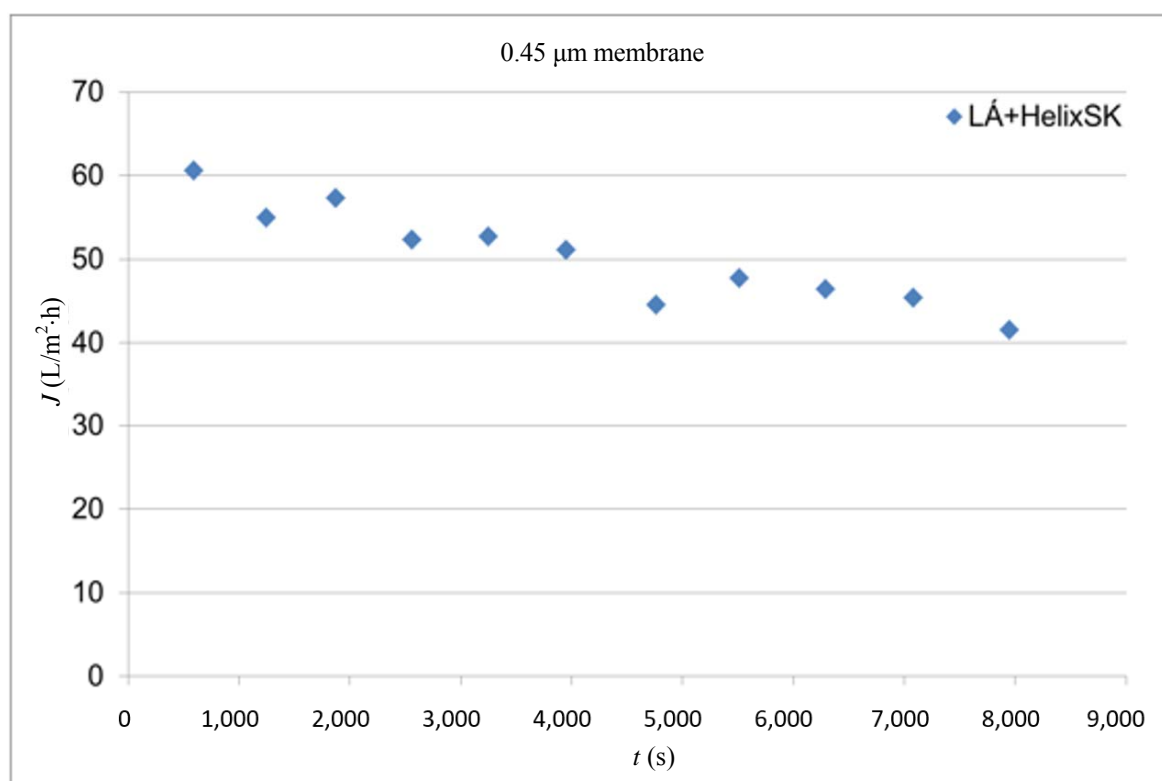


Fig. 4 The flux values (J) as a function of time (t) on the 0.45 μm cut-off value membrane with air insufflation and static mixer. LÁ: air insufflation; HelixSK: static mixer.

The experiment started by a 0.45 μm tubular membrane combined with air insufflation and Helix type static mixer. The use of the higher pore size membrane resulted in higher flux values. Fig. 4 presents the result of filtering method combined with air insufflation and the use of static mixer. The flux values started at 60 $\text{L}/\text{m}^2\cdot\text{h}$ and these values were presented a slow decrease. Comparison of the two flux values got from two different pore size membranes, it showed that the higher pore size membranes produced higher flux values than the lower pore size membrane.

In the end of the basic measurements, the optimal operation parameters of the filtration were 0.2 MPa transmembrane pressure at 100 L/h recirculation flow rate on 0.2 μm tubular membrane. The good flux values are also very important during the experiments as well as the good fat retention values. Fig. 5 presents the changes in flux values at 0.2 MPa transmembrane pressure, and at q_v : 100 L/h on a 0.2 μm tubular

membrane, with or without using air insufflation and with or without using different static mixers. The basic flux values were 17-18 $\text{L}/\text{m}^2\cdot\text{h}$ during the filtration process. The flux was decreased slightly during the filtering with combined air insufflations, which indicates that the filtering in the optimal operation parameters combined with air insufflation did not give better results than the normal filtering. The flux value increased up to 30 $\text{L}/\text{m}^2\cdot\text{h}$ when the filtration device was used together with Helix type ribbon screw static mixer. When the Helix type static mixer was combined with air insufflation during filtration, the flux values were increased two times more, up to 40 $\text{L}/\text{m}^2\cdot\text{h}$ (Fig. 5).

In the case of retention, the effect of increasing pressure did not give increasing flux values. The increase of transmembrane pressures increased the flux values until 0.2 MPa, but on the higher pressure values, the flux values presented constant data or suddenly showed a strong decrease. When the

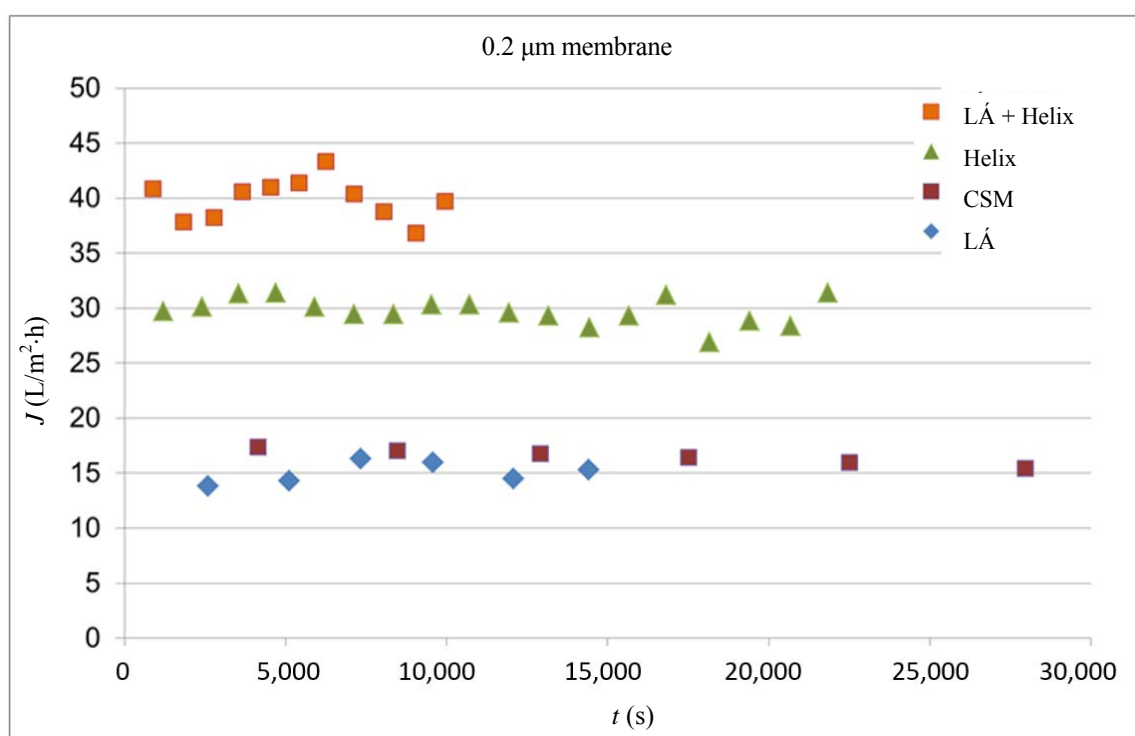
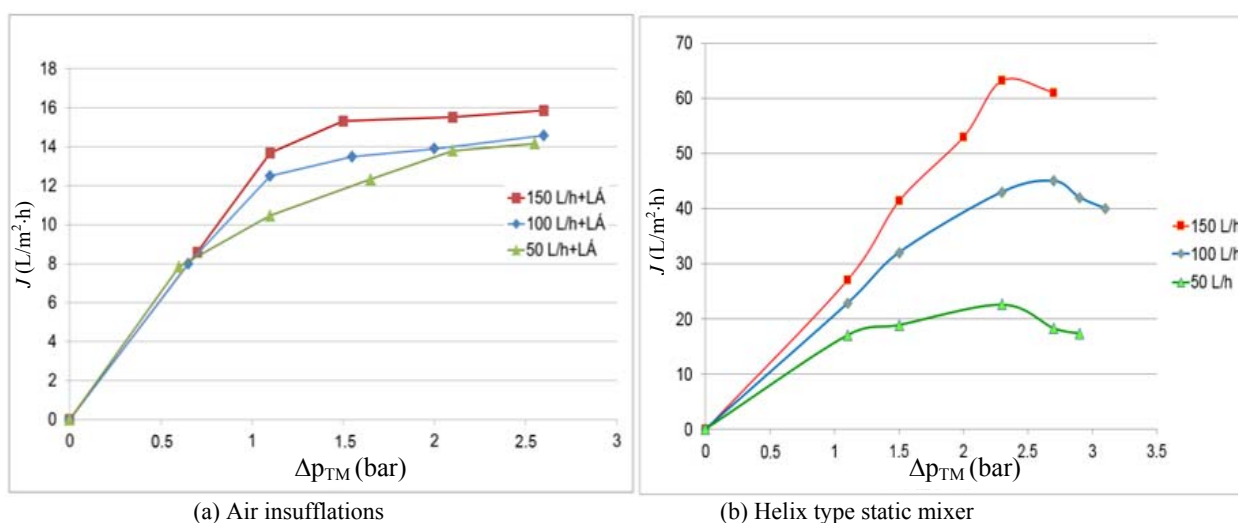


Fig. 5 The flux values (J) as a function of time (t) on the 0.2 μm cut-off value membrane.

LÁ: air insufflation; Helix: static mixer; CSM: tubular membrane.



(a) Air insufflations

(b) Helix type static mixer

Fig. 6 The changes of whey flux (J) as a function of transmembrane pressure at different recirculation flow rate by air insufflations (a) and Helix type static mixer (b).

two different pre-treatment were used alone, under the same filtering parameters, the flux values remained very low in the air insufflation process (Fig. 6a), therefore the air insufflation process itself is not suitable for degreasing. When the Helix type static mixer was used only, the flux presented higher values than the measurements with air insufflations (Fig. 6b)

[17]. These flux values above 0.2 MPa transmembrane pressures were strongly decreased; therefore, it can be determined that the use of the lower transmembrane pressures gave better flux results.

Although the experiments with the static mixer were made with the 0.2 μm in the same operation parameters, the Helix type static mixer was changed to

a Kenics™ type helical twist bowtie static mixer. The flux values were increased also with using the Kenics™ type static mixer (45 L/m²·h) during the filtration, but the increasing rate of these values was not as high as using the Helix type static mixer (53 L/m²·h) [18].

The resistance parameters of the filtrating explained the measured differences in the case of different arrangements (Fig. 7).

The different resistance values, such as the membrane resistance (R_m), the fouling resistance (R_f) and the gel layer resistance (R_g) are an order of magnitude less than using a static mixer. The experiments do not present measurable value of the gel resistance in the use of static mixer.

The vibratory shear enhanced membrane filtration was examined by a 0.2 μm pore size polyethersulfone microfiltration membrane on 0.3 MPa transmembrane pressures at 0 Hz and 54 Hz frequency. The measurements showed that the flux was increased three times higher by using vibration frequency (54 Hz) compared to normal filtration (Fig. 8). The very high permeate flux ratio has been kept during the

separation process, while the viscosity of the concentrate increased [19].

All the retention values were decreased by using the vibration, therefore the fat molecules of the whey were allowed to pass through the membrane pores (Fig. 9).

The retention values of the small components were increased due to fouling the membrane pores. This low fat and high protein retention can be explained by that without using the vibration, the flexible fat molecules were moved into the capillaries of the membrane under pressure, and due to their sizes (3.5 μm), these components get stuck inside the membrane capillaries. The fouled pores could increase the retention values of the fat and the protein components [20].

It can be realized during the examinations of the resistance values that there was no significant difference between the gel layer and the membrane resistance values in the case of vibrated and non vibrated systems as illustrated below in Fig. 10. It was also realized that there is a big difference in the fouling resistance [21].

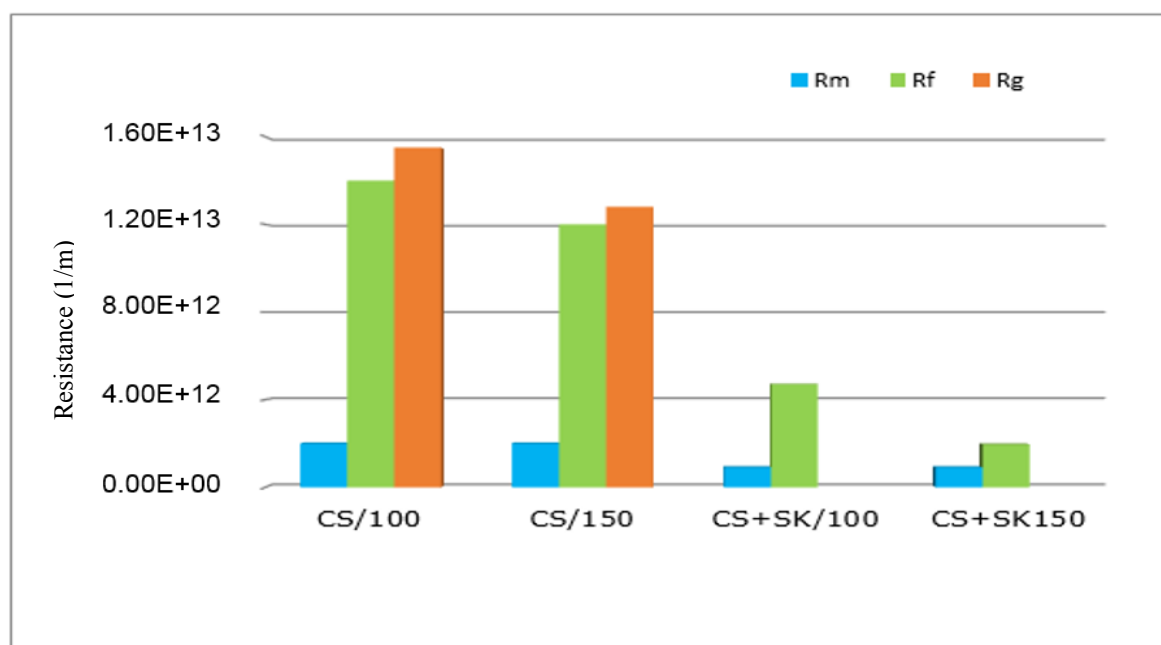


Fig. 7 The different resistances of the tubular membrane (CS) separation with and without statics mixer (SK) at different recirculation flow rates (100 L/h, 150 L/h) on whey separation.

Rg: gel layer resistance; Rf: fouling resistance; Rm: membrane resistance.

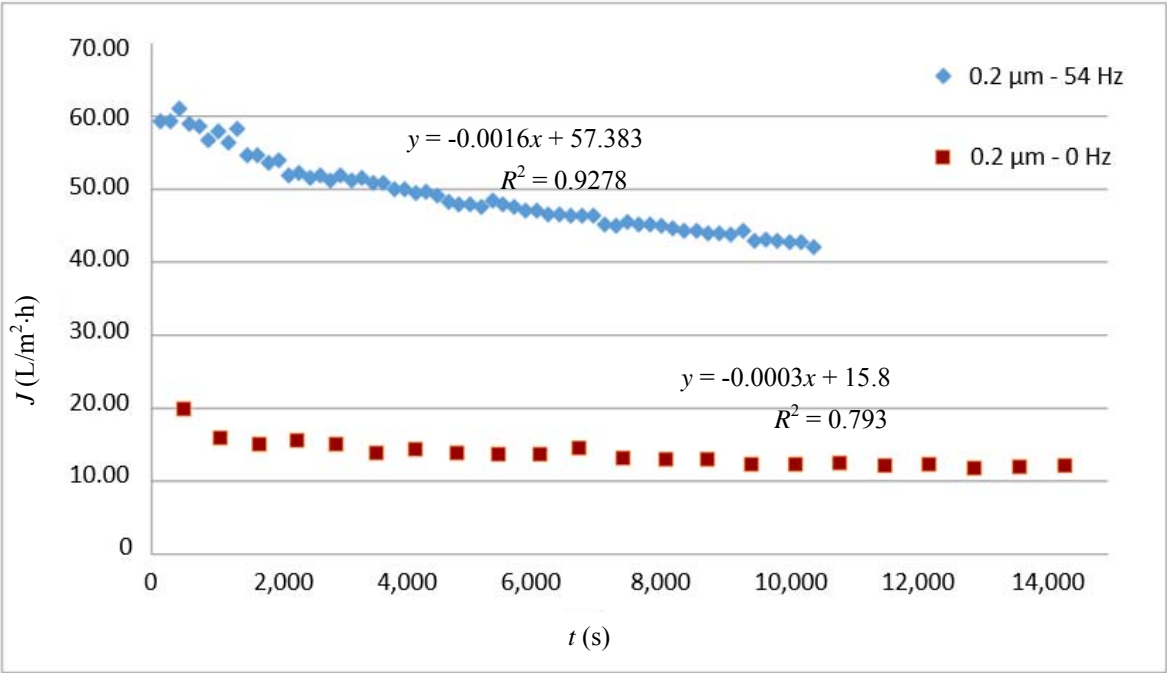


Fig. 8 The flux values (J) as a function of time (t) by vibrated and non vibrated methods.

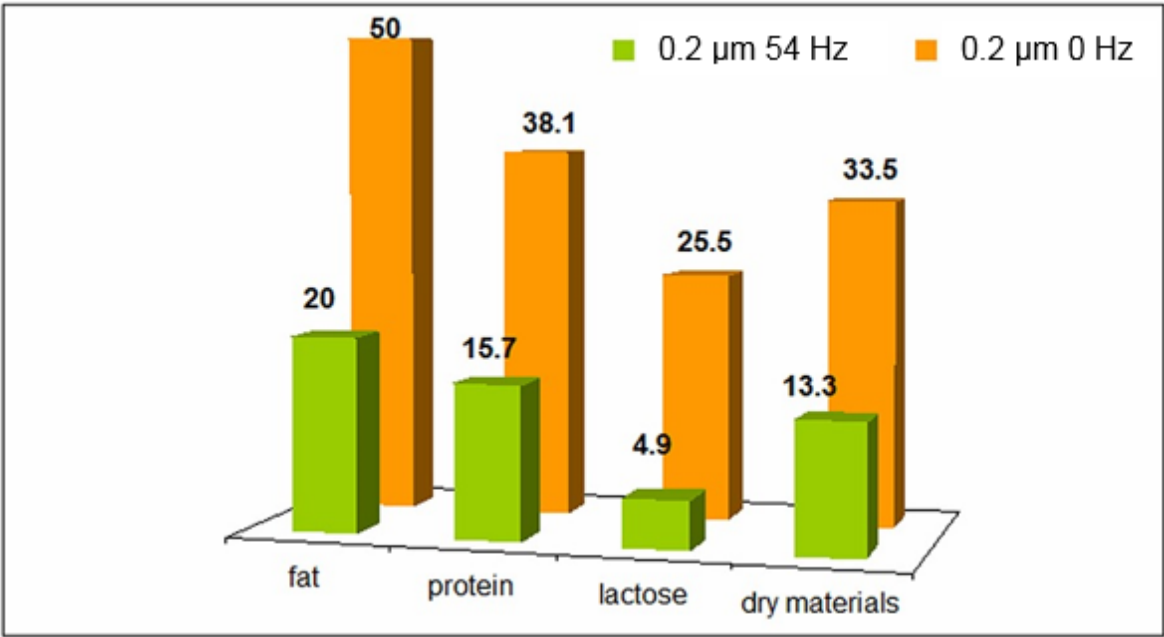


Fig. 9 The retention value of the most important components of whey measured in $0.2\ \mu\text{m}$ cut-off value flat sheet membrane.

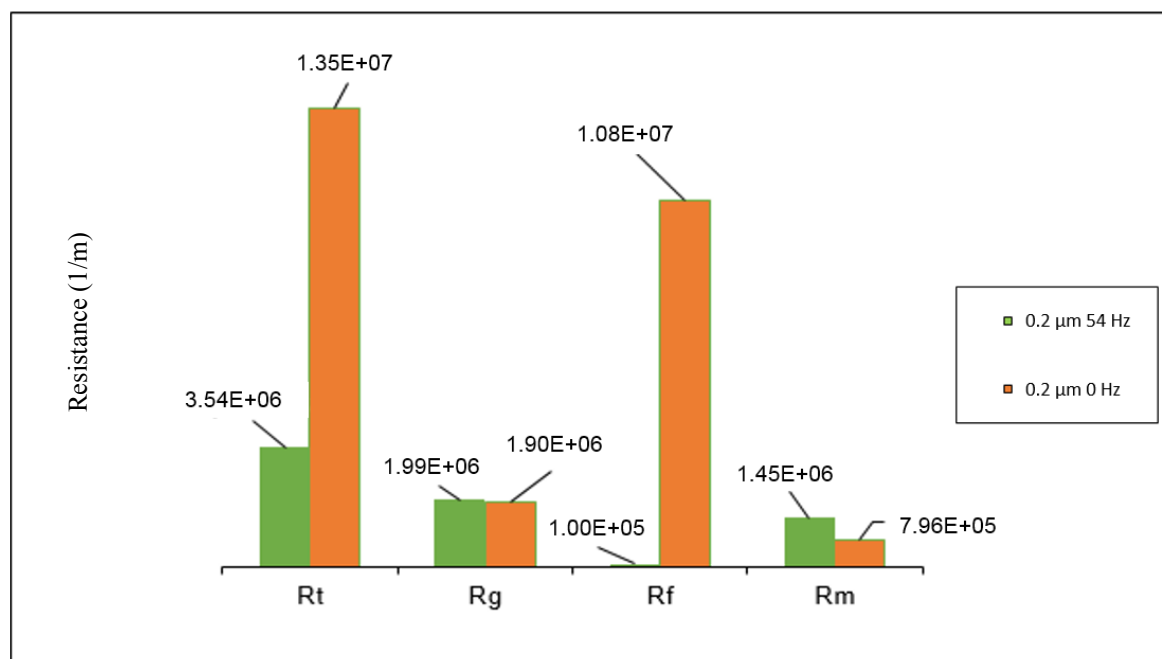


Fig. 10 The differences of the resistance values of the vibrated and non vibrated whey microfiltration.

In non-vibrating mode, not only the total resistance value was different, but the ratio of different resistances as well. The flux values in the non-vibrating mode showed four times lower values compared to the vibrating mode and the total resistance was one order of magnitude higher. The fouling resistance values presented two orders of magnitude higher values compared to the vibrating mode.

4. Conclusions

Comparing the filtration methods, it was obtained that the 0.2 μm pore size tubular membrane combined with static mixer gave sufficient result according to the degreasing, the retentions and the flux values of the other components.

The measurements, which were presented on different pore size tubular membrane (0.45 μm), could only slightly hold back the fat molecules from the feed material, which means that this pore size membrane is insufficient to reduce the fat content of whey. 45% higher flux values was produced by the use of Helix type static mixer than the simple tubular membrane filtration process and 20% higher flux values than the

KenicsTM type static mixer, and a synergetic effect occurred by using both air insufflation and static mixer but the fat retention did not show this rising tendency. The air insufflation could not produce a high quantity of flux values during the filtration process, and it means that this process might be useful for degreasing only combined with other treatments, such as the use of static mixer.

The VSEP technology showed that without using vibration, the retention values of the fat content and the other elements all increased. The measured flux values were three times higher by using 54 Hz vibration than without vibration. This shows clearly that the vibration can increase the efficiency of the filtration process, and it can increase the lifetime of the membrane, too.

Finally, it can be concluded that the combination of a tubular membrane with static mixer could be the cheapest and economical solution to separate fat molecules from whey, and the best solution to separate the maximum quantity of fat molecules from whey. This could be a good solution for the food industry to recover maximum value of whey protein from dairy by-products.

Acknowledgments

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References

- [1] Byrde, O., and Sawley, M. L. 1999. “Optimization of a Kenics Static Mixer for Non-creeping Flow Conditions.” *Chemical Engineering Journal* 72 (2): 163-9.
- [2] Ahmadun, F. R., Pendashteh, A., Abdullah, L. C., Biak, D. R. A., Madaeni, S. S., and Abidin, Z. Z. 2009. “Review of Technologies for Oil and Gas Produced Water Treatment.” *Journal of Hazardous Materials* 170 (2-3): 530-51.
- [3] Krstić, D. M., Tekić, M. N., Carić, M. D., and Milanović, S. D. 2002. “The Effect of Turbulence Promoter on Cross-Flow Microfiltration of Skim Milk.” *Journal of Membrane Science* 208 (1-2): 303-14.
- [4] Scott, K., and Lobato, J. 2002. “Mass Transfer Characteristics of Cross-Corrugated Membranes.” *Desalination* 146: 255-8.
- [5] Ahmad, A. L., and Mariadas, A. 2004. “Baffled Microfiltration Membrane and Its Fouling Control for Feed Water of Desalination.” *Desalination* 168: 223-30.
- [6] Bellhouse, B. J., Costigan, G., Abhinava, K., and Merry, A. 2001. “The Performance of Helical Screw-Thread Inserts in Tubular Membranes.” *Separation and Purification Technology* 22-23: 89-113.
- [7] Costigan, G., Bellhouse, B. J., and Picard, C. 2002. “Flux Enhancement in Microfiltration by Corkscrew Vortices Formed in Helical Flow Passages.” *Journal of Membrane Science* 206: 179-88.
- [8] Cui, Z. F., and Wright, K. I. T. 1996. “Flux Enhancements with Gas Sparging in Downwards Cross-Flow Ultrafiltration: Performances and Mechanisms.” *Journal of Membrane Science* 117: 109-16.
- [9] Laborie, S., Cabassud, C., Durand-Bourlier, L., and Lainé, J. M. 1998. “Fouling Control by Air Sparging inside Hollow Fibre Membranes: Effect on Energy Consumption.” *Desalination* 118: 189-96.
- [10] Vatai, G. N., Krstić, D. M., Höflinger, W., Koris, A. K., and Tekić, M. N. 2007. “Combining Air Sparging and the Use of a Static Mixer in Cross-Flow Ultrafiltration of Oil/Water Emulsion.” *Desalination* 204: 255-64.
- [11] Cheryan, M. 1998. *Ultrafiltration and Microfiltration Handbook*. USA: Techomic Publication Co. Inc..
- [12] Derradji, A. F., Bernabeu-Madico, A., Taha, S., and Dorange, G. 2000. “The Effect of a Static Mixer on the Ultrafiltration of a Two-Phase Flow.” *Desalination* 128 (3): 223-30.
- [13] Frappart, M., Jaffrin, M. Y., Ding, L. H., and Espina, V. 2008. “Effect of Vibration Frequency and Membrane Shear Rate on Nanofiltration of Diluted Milk, Using a Vibratory Dynamic Filtration System.” *Separation and Purification Technology* 62 (1): 212-21.
- [14] Jaffrin, M. Y., Frappart, M., and Ding, L. H. 2008. “Reverse Osmosis of Diluted Skim Milk: Comparison of Results Obtained from Vibratory and Rotating Disk Modules.” *Separation and Purification Technology* 60 (3): 321-9.
- [15] Rautenbach, R. 1997. *Membrane Processes: Fundamentals of Module and System Design*. Berlin: Springer-Verlag, 60-77. (in German)
- [16] Kertész, S., Törteli, J., László, Z., Kovács-Róbertné, V. P., Szabó, G., and Hodúr, C. 2010. “Dairy Waste Water Purification by Vibratory Shear-Enhanced Process.” In *Proceedings of the PERMEA Conference 2010*, 48-58.
- [17] Krstić, D. M., Tekić, M. N., Carić, M. D., and Milanović, S. D. 2003. “Kenics Static Mixer as Turbulence Promoter in Cross-Flow Microfiltration of Skim Milk.” *Separation Science and Technology* 38: 1549-60.
- [18] Cabassud, C., Laborie, S., Durand-Bourlier, L., and Lainé, J. M. 2001. “Air Sparging in Ultrafiltration Hollow Fibers: Relationship between Flux Enhancement, Cake Characteristics and Hydrodynamic Parameters.” *Journal of Membrane Science* 181 (1): 57-69.
- [19] Hodúr, C., Kertész, S., Csanádi, J., Szabó, G., and László, Z. 2009. “Investigation of Vibratory Shear-Enhanced Processing System.” *Progress in Agricultural Engineering Sciences* 5 (1): 97-110.
- [20] Kertész, S., Erbası, E., László, Z., Hovorka-Horváth, Z., Szabó, G., and Hodúr, C. 2010. “Oily Wastewaters Separation by Ultrafiltration.” In *Proceedings of IWA Regional Conference and Exhibition on Membrane Technology & Water Reuse*, 351-5.
- [21] Meyer, P., Meyer, A., and Kulozik, U. 2015. “High Concentration of Skim Milk Proteins by Ultrafiltration: Characterisation of a Dynamic Membrane System with a Rotating Membrane in Comparison with a Spiral Wound Membrane.” *International Dairy Journal* 51: 75-83.