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Enhancement of ultrafiltration of milk proteins by application of twisted tapes: A sensitivity analysis using a response surface methodology

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Abstract: This paper presents intensification of the ultrafiltration of milk proteins by application of twisted tapes as turbulence promoters to minimize membrane fouling. The aim was to examine the influence of operating conditions and twisted tape dimensions on the alleviation of flux and the consumption of specific energy. A twisted tape was inserted in the ultrafiltration membrane (50 nm pore size) to alleviate turbulence and minimize fouling. The response surface methodology was used for the sensitivity analysis of the effects of operating conditions on the responses. The analysis showed that the linear effect of the aspect ratio of the twisted tape has a dominant significant effect on flux improvement. The linear effect of cross-flow rate has a positive dominant effect on the specific energy consumption. The linear effect of concentration and the mutual effect of aspect ratio and transmembrane pressure are statistically significant for both responses. By properly adjusting the operating conditions, a high flux improvement of 300 % could be reached with a specific energy consumption below 1.0 kW h m^{-3} using a twisted tape of an aspect ratio of 1.0 and imposing low transmembrane pressure.

Keywords: turbulence promoter; fouling minimization; membrane filtration; response surface methodology; sensitivity analysis.

INTRODUCTION

Ultrafiltration is commonly employed in the dairy industry for concentrating and fractionating proteins.[1] Even though the separation characteristics of ultrafiltration are excellent, the fouling of a membrane poses a problem in terms of process efficiency. The fouling by proteins is severe and difficult to mitigate and thus, the frequent cleaning of membranes is required causing membrane deterior-

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ation and consequently their frequent replacement. The fouling occurs because of accumulation and adsorption of the proteins on the membrane surface and/or in the pores of the membrane.[2,3] The permeation flux declines during the filtration process resulting in lower process efficiency due to the appearance of both, concentration polarization and fouling. Concentration polarization and surface fouling can be controlled by applying hydrodynamic methods.

Among others, the simplest hydrodynamic method of fouling minimization is an acceleration of fluid, *i.e.*, an increase in the cross-flow velocity of the fluid. In this way, the turbulence in the fluid is increased and fouling particles are taken away from the membrane surface by the turbulent fluid flow. The hydrodynamic technique proved highly efficient is the insertion of motionless turbulence promoters in the membrane module. Besides the acceleration, the main impact of turbulence promoters is the formation of secondary flows additionally contributing to the disruption of the boundary layer and an improvement of mass transport. The effect of the geometry of the turbulence promoter on the flow field in the membrane channel and fouling minimization have been widely presented.[4–6] Turbulence promoters of diverse geometries were studied in the microfiltration of various mixtures, such as yeast,[7,8] skimmed milk,[4,6] bentonite,[9] *etc*. where excellent flux improvements were gained. Furthermore, they have been studied in several ultrafiltration processes, such as the ultrafiltration of oily wastewaters,[10,11] milk,[12] dispersion with colloidal SiO_2[13] and natural organic matter.[14] The ultrafiltration of milk was enhanced by a cork-screw thread turbulence promoter, which causes a high pressure drop along the membrane and operation under high transmembrane pressure,[12] which is energetically inefficient.

Generally, the drawback of the application of a turbulence promoter is an increase in pressure loss across the membrane module. However, it depends on geometrical characteristics and dimensions that influence the flux improvement greatly. Thus, it is necessary to find a compromise between flux improvement and the energy consumed per the cubic meter of the produced permeate. Other operating conditions cannot be disregarded and their influence has to be analysed as well.

Response surface methodology (RSM) is a simple and widely used tool for detecting and quantifying influences of the chosen independent variables (factors) on the dependent variables (responses) and for process optimization. The RSM approach is multivariate so the responses of the system are analysed by varying the multiple independent variables simultaneously. For analysis, the experimental measurement has to be organized using an appropriate design of experiments (DOE). The factorial design of the experiments allows wider and more differentiated information on the system and greatly usable conclusions.[15] There are various designs of experiments used for the planning of experiments. The RSM has been successfully used in studies on the membrane processes, to examine the influence of the operating conditions and thereby to find the optimal process solutions.[7,11]

In previous studies, the focus was on the application of various geometries of turbulence promoters in the microfiltration of partially skimmed milk to prove the concept and to analyse the fluid dynamics in the membrane with a promoter.[5,6] Given that membrane fouling is considerably affected by the characteristics of filtered feed, case studies are needed to detect the influences of all possible factors.

This study was designed to identify the significance of particular operating conditions for intensification of the ultrafiltration of milk proteins. Twisted tapes as low-pressure loss turbulence promoters were for the first time used for fouling mitigation, thereby enhancement of the ultrafiltration of milk proteins. The sensitivity of the flux improvement and specific energy consumption to the dimension of twisted tape (aspect ratio), cross-flow rate, transmembrane pressure and the concentration of proteins were examined.

EXPERIMENTAL

Materials and methods

Ultrafiltration of reconstituted skimmed milk was performed using the microfiltration/ /ultrafiltration set-up described in details elsewhere.[6] The transmembrane pressure (*TMP*) was measured by digital pressure gauges (0–10 bar accuracy ±1 %, Cerebar M, Endress+Hauser, Germany) and the flow rate was measured using a rotameter. All experiments were realised at 50±0.5 °C. Ultrafiltration was operated completely recycling both streams, permeate and retentate, maintaining the concentration of the feed at a constant value. Each experiment took 90 min to assure the achievement of a steady-state flux. The permeate was gathered in a beaker placed on a digital balance. The mass of permeate with time was measured while the data were transferred to a PC unit and used for the calculation of the flux.

A single-channel ceramic membrane with a zirconium filtering layer on an α-alumina support was used for the ultrafiltration. The dimensions of the membrane were as follows: an average pore size of 50 nm, length of 250 mm and *OD*/*ID* 10 mm/6.8 mm (GEA, Germany). The active filtering area of a membrane was 46.2 cm^2.

Custom-made stainless-steel twisted tapes (Inox Bravarija, Bački Petrovac, Serbia) were used as turbulence promoters. An illustration of a membrane module with a twisted tape and the streamlines of fluid flow is shown in Fig. 1. The characteristics of the twisted tapes are given in Table I. For comparing various geometries and dimensions of turbulence promoters, the aspect ratio (*AR*) has usually been used. The *AR* is the ratio of the pitch length (L_e), to its diameter (D_{TP}).

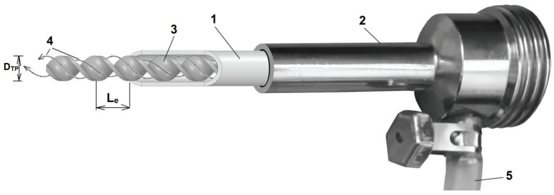

Fig. 1. An illustration of the membrane module with a twisted tape: 1 – membrane module, 2 – housing, 3 – twisted tape, 4 – streamlines in the retentate flow field and 5 – permeate.

TABLE I. Characteristics of the twisted tapes

Annotation	D_{TP} / mm	L_{TP} / mm	δ_{TP} / mm	L_e / mm	N_{twists}	AR
TT1.0	6.5	241	1.2	6.5	37	1.0
TT2.5	6.5	238	1.2	16.2	15	2.5
TT4.0	6.5	250	1.2	26	9.6	4.0

Reconstituted skimmed milk was used as the feed. The composition of the skimmed milk powder (Subotica Dairy Industry, Serbia) according to the manufacturer was as follows in mass %: proteins 33.55, fat 1.1, lactose 52.7, ash 7.8 and moisture 4.85. A batch of seven kilograms of milk was prepared a day before the filtration experiment by mixing the skimmed milk, anti-microbial agent (NaN_3 0.2 g L^{-1}), and deionised water. Firstly, a given mass of the skimmed milk was reconstituted to obtain 1 kg of the concentrated emulsion by stirring on a magnetic stirrer at 500 rpm for 10 min. Then it was mixed with the rest amount of water using an overhead stirrer at 1000 rpm for 15 min. The reconstituted milk was stored overnight in a refrigerator at 2–4 °C. Before the experiment, the reconstituted milk was heated to 50 °C. The pH of the feed was measured before and after each filtration experiment and it was stable at a value of 6.8±0.1. After each ultrafiltration experiment, the set-up unit was thoroughly cleaned and rinsed. The acid-based cleaning-in-place procedure was performed according to the membrane manufacturer recommendation. The membrane was considered as clean if 95 % of the new membrane water flux was restored.

Calculations

Response surface methodology (RSM) is a statistical method that allows a multifactorial analysis of data.[16] Using this approach, it is possible to identify the order of the effects of factors on the responses, and whether there are mutual effects of the factors. The experimental data are usually fitted with a second-order polynomial equation. The effects of the factors on the responses are analysed by subjecting the coefficients in the model equation to significance testing using an analysis of variance method (ANOVA). The level of confidence was set at 95 % (two-sided) and the probability expressed as a *p*-value at the significance level of 0.05. Box–Behnken experimental design (BBD) with four factors on three levels, –1, 0 and 1, was chosen (Table II). The concentration of milk proteins (*c*), cross-flow rate (*CFR*), transmembrane pressure (*TMP*) and aspect ratio (*AR*) of the twisted tape (TT) were chosen as the factors. The flux improvement, *FI,* and specific energy consumption, *E*, were chosen as responses. The flux improvement is the most important from the productivity perspective for membrane processes. The specific energy consumption is important from an economic point of view. The BB runs and the obtained responses are presented in Table S-I of the Supplementary material to this paper. The same experimental plan was used to obtain fluxes without twisted tape to calculate the flux improvement, %:

$$FI=100\frac{J_{TT}-J_{NTP}}{J_{TT}} \quad (1)$$

where J_{TT} is the steady-state flux with a twisted tape, L m^{-2} h^{-1}, and J_{NTP} is the steady-state flux without the twisted tape, L m^{-2} h^{-1}.

For fitting experimental data, a second-order polynomial model equation was employed:

$$Y=b_0+\sum_{i=1}^{k}b_iX_i+\sum_{i=1}^{k}b_{ii}X_i^2+\sum_{1\le i\le j}^{k}b_{ij}X_iX_j \quad (2)$$

where Y is the process response, X_i are the independent variables and b polynomial coefficients: b_0 the intercept, b_i the linear, b_{ii} the quadratic and b_{ij} the mutual effect of the independent variables.

TABLE II. Levels of factors and characteristics of the Box–Behnken design

No.	Factors	Low (–1)	Centre (0)	High (1)	Box–Behnken design	
1	c / %	2.0	2.5	3.0	Total runs	27
2	CFR, L min^{-1}	1.0	2.0	3.0	Centre points	3
3	AR	1.0	2.5	4.0	–	–
4	TMP, kPa	50	100	150	–	–

The specific energy consumption in kW h m^{-3} is suitable for comparing the energy consumption in the empty module and the module with an inserted twisted tape and can be calculated from[4]:

$$E=\frac{CFR\,\Delta P}{V_{\mathrm{p}}} \tag{3}$$

where CFR is the cross-flow rate, m^3 s^{-1}, ΔP is the pressure loss, Pa, and V_{p} is the volumetric flow of a permeate, m^3 s^{-1}.

Software Statistica 13 was used for the calculations. The least square method was chosen as the calculation method that should enable residual error near zero. The results of the ANOVA method were used for the sensitivity analysis of the responses to the variables.

RESULTS AND DISCUSSION

The time dependency of flux obtained during ultrafiltration with and without twisted tapes had the shape typically indicating the occurrence of concentration polarization with stable steady-state fluxes after an initial period of fouling (Fig. S-1 of the Supplementary material). The internal fouling of pores is more pronounced in ultrafiltration than in microfiltration of milk and thus, it has an influence on the decrease in the flux particularly when high TMPs are imposed. Yet, in this study, besides concentration polarization, the internal fouling was controlled due to the improved hydrodynamics by using a twisted tape. Therefore, the obtained steady-state fluxes are considerably higher than in conventional operation. Since the effects of cross-flow rate and twisted tapes on hydrodynamics are similar, statistical analysis has to be performed to confirm which variable is more effective.

The second-order polynomial equation in RSM was used for modelling the experimental data a presented in Table S-I of the Supplementary material. The equations obtained for the flux improvement and specific energy consumption are given in the Supplementary material (Eqs. (S-1) and (S-2), respectively). To validate the hypothesis that H$_0$: $b_i = b_{ii} = b_{ij}\cdots b_k = 0$, H$_1$: $b_i \neq 0$, the ANOVA test of the analysis of variance was performed. The model is proved to fit the experimental data when the regression coefficient is significant, and a lack of fit is non-significant within the confidence interval.

The results of the analysis of variance (ANOVA) and the lack-of-fit test for the flux improvement and specific energy consumption are given in Tables III and IV, respectively. The lack-of-fit test for both responses confirms that the null hypotheses could be rejected as the *p*-values are 0.74 and 0.81 (>0.05) for the flux improvement and specific energy consumption, respectively. The proposed model fits the experimental data very well over the entire experimental range. For the flux improvement, the coefficient of determination (R^2) and the adjusted coefficient of determination (R^2_{adj}) are 0.969 and 0.940, respectively. This means that less than 6 % of the variation in the flux improvement could not be explained by the model. For the specific energy consumption, the coefficient of determination (R^2) and the adjusted coefficient of determination (R^2_{adj}) are 0.996 and 0.992, respectively. Further, given that the *F*-value is 13.46 and 243.1 ($>F_{crit(\alpha/2,14,12)}$ = = 2.51), for the flux improvement and specific energy consumption, respectively, with *p*-value < 0.00001 confirms the consistency and robustness of both models.

TABLE III. Results of ANOVA analysis for flux improvement

Factor	Sum of squares	Degrees of freedom	Mean square	*F*-Ratio	*p*-Value
Model	59760.83	12	4268.63	13.46	0.0000[a]
c	3605.33	1	3605.33	11.36	0.0055[a]
c^2	31.15	1	31.15	0.098	0.7594
CFR	40.33	1	40.33	0.127	0.7276
CFR^2	1526.26	1	1526.26	4.811	0.0487[a]
AR	50052.08	1	50052.08	157.8	0.0000[a]
AR^2	28.01	1	28.01	0.088	0.7714
TMP	690.08	1	690.08	2.175	0.1659
TMP^2	158.90	1	158.90	0.500	0.4926
c×*CFR*	210.25	1	210.25	0.663	0.4314
c×*AR*	2.25	1	2.25	0.007	0.9343
c×*TMP*	961.00	1	961.00	3.029	0.1073
CFR×*AR*	30.25	1	30.25	0.095	0.7628
CFR×*TMP*	9.00	1	9.00	0.028	0.8690
AR×*TMP*	1892.25	1	1892.25	5.965	0.0310[a]
Error	3806.58	12	317.22	–	–
Total SS	63567.41	26	–	–	–
Lack-of-fit	2905.92	10	290.59	0.645	0.7407
Pure error	900.7	2	450.35	–	–

[a]Significant if *p*-value < 0.05

The response surfaces of flux improvement are shown in Fig. 2 for pairs of the aspect ratio of TT (*AR*) with the each of operating conditions: *CFR* (a), the concentration of proteins (b) and *TMP* (c). The effects of *AR* and *CFR* on the flux are shown in Fig. 2a for a concentration of proteins of 2.5 mass % and a TMP of 100 kPa (central point). It could be observed that flux improvement increases with decreasing *AR*. In the dependency of flux improvement on cross-flow rate, a small maximum could be observed. It could be noticed that the flux improvement slightly increases with increasing cross-flow rate and then slightly decreases with further increasing in *CFR*. The fluxes are significantly higher when twisted tapes were used compared to the conventional operation. However, under higher *CFR*s, operation without promoters delivers higher fluxes and thus, the flux improvement as a relative value decreases slightly.

TABLE IV. Results of ANOVA analysis for specific energy consumption

Factor	Sum of squares	Degrees of freedom	Mean square	*F*-Ratio	*p*-Value
Model	7.02	12	0.50	243.1	0.0000[a]
c	0.16	1	0.16	79.1	0.0000[a]
c^2	0.003	1	0.003	1.53	0.2398
CFR	6.53	1	6.53	3165.4	0.0000[a]
CFR^2	0.12	1	0.12	59.5	0.0000[a]
AR	0.08	1	0.08	39.17	0.0000[a]
AR^2	0.047	1	0.05	22.93	0.0004[a]
TMP	0.012	1	0.012	6.1	0.0297[a]
TMP^2	0.000	1	0.000	0.000	0.9834
$c\times CFR$	0.039	1	0.04	18.9	0.0009[a]
$c\times AR$	0.005	1	0.006	2.8	0.1224
$c\times TMP$	0.000	1	0.000	0.004	0.9484
$\mathrm{CFR}\times AR$	0.008	1	0.008	3.924	0.0709
$\mathrm{CFR}\times TMP$	0.002	1	0.002	1.211	0.2927
$AR\times TMP$	0.016	1	0.016	7.569	0.0310[a]
Error	0.025	12	0.002	–	–
Total SS	7.05	26	–	–	–
Lack-of-fit	0.018	10	0.0018	0.51	0.8104
Pure error	0.007	2	0.0035	–	–

[a]Significant if *p*-value < 0.05

The effects of the *AR* and concentration of proteins on the flux improvement are shown in Fig. 2b for a *CFR* of 2.0 L min^{-1} and a *TMP* of 100 kPa (central point). The flux improvement increases with increasing the concentration of proteins for all the tested aspect ratios. Since the fouling is severe when the concentration of proteins is high, operation without promoters is very ineffective and so the flux improvement as a relative value increases with increasing concentration of proteins.

The effects of *AR* and *TMP* on the flux improvement are shown in Fig. 2c for a concentration of proteins of 2.5 mass % and a *CFR* of 2.0 L min^{-1} (central point). The influence of *TMP* decreases with decreasing aspect ratio. The flux improvement increases with an increase in *TMP* for the twisted tape of the aspect ratio 4.0. Yet, the influence of *TMP* can be neglected for the twisted tape of aspect ratio 1.0. Generally, the flux should increase with increasing *TMP* as the driving force but that could cause a build-up of fouling matters in the pores.

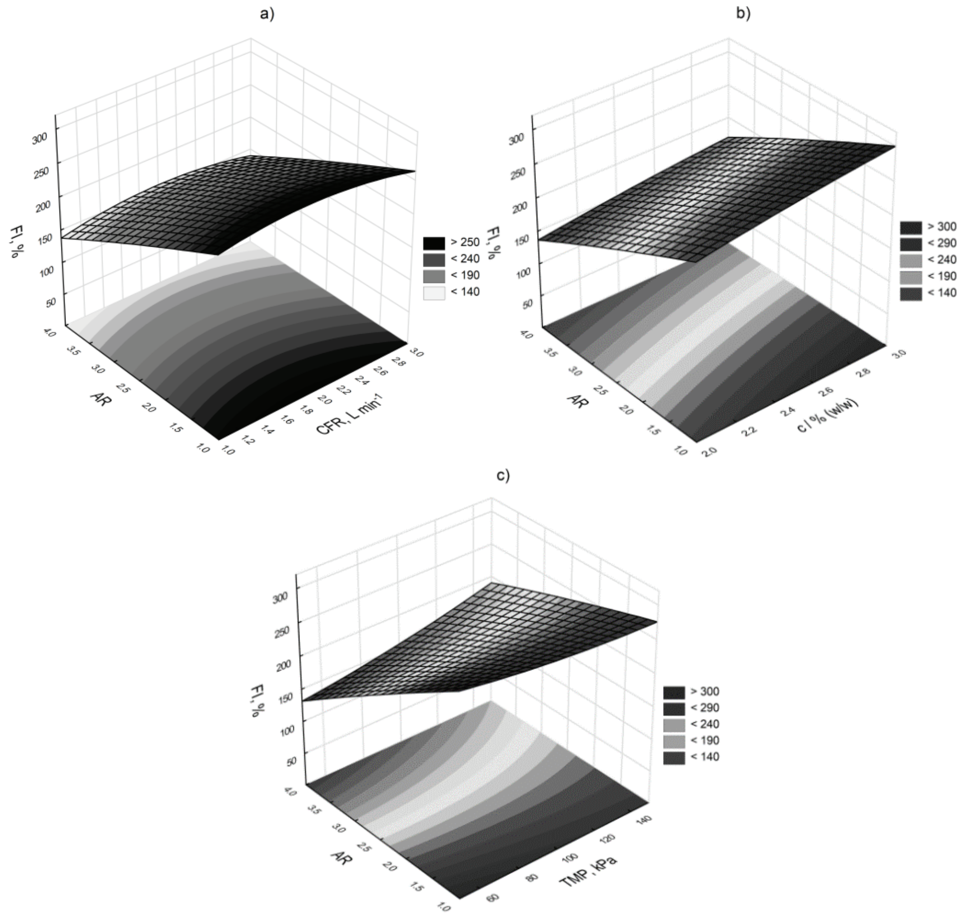

Fig. 2. Response surfaces for the flux improvement for pairs of the aspect ratio of TT with each of operating conditions: a) cross-flow rate, b) the concentration of proteins and c) *TMP*.

However, this also depends on imposed CFR. As the cross-flow velocity is higher for the twisted tape of *AR* 1.0 and both concentration polarization and in-pore fouling are minimized, the influence of *TMP* is shifted to higher values, beyond 150 kPa. The insertion of a twisted tape in the membrane module causes an acceleration of the fluid and changes in the flow field such as the, alternation of streamlines from straight to helical.[6,17] The acceleration of the fluid causes an increase in shear stress. An increase in the shear stress alleviates the detachment of the fouling matter from the membrane surface and its removal back to the bulk of the feed. In this way, the fouling boundary layer is disrupted and fouling is controlled by shear stress at the membrane surface. In microfiltration, fouling is predominantly controlled by shear stress but the *TMP* has no significant influence.[6]

The efficiency of a twisted tape is proven by the remarkably high flux improvements obtained under low *TMP*s. For instance, the flux improvement can reach 300 % for a concentration of proteins of 3 % under a *CFR* of 2.0 L min^{-1}, a *TMP* of 100 kPa and the smallest *AR* of 1.0 (Fig. 2b). However, it should be noted that for other concentrations of proteins, there is a combination of *CFR*, *TMP* and *AR* that can deliver very high flux improvements even beyond 300 %. Still, the flux improvement as the response has to be analysed alongside the specific energy consumption in decision making concerning the operating conditions that should be imposed.

The significance of effects for flux improvement was determined by analysing the ANOVA results presented in Table III. All factors with a *p*-value below 0.05 are statistically significant. Here, for flux improvement, the linear effects of concentration and *AR*, the squared effect of *CFR* and the mutual effect of *AR* and *TMP* are significant. The sensitivity of the response to the significant factors can be explained from a Pareto chart of the standardized effects presented in Fig. S-2 of the Supplementary material. The absolute values of the standardized effects are presented in descending order from the largest to the smallest one. Flux improvement is most sensitive to a linear variation in *AR* and the concentration, followed by the mutual effect of *AR* and *TMP*, and the quadratic effect of the *CFR*. The standardized effect of *AR* has a negative value –12.56 because the flux improvement decreases with increasing *AR*. The standardized effect of concentration is less influential than that of *AR* and has a positive value of 3.37, meaning that the flux improvement increases with increasing concentration of proteins. This confirms that the application of twisted tapes is more effective when the concentration of proteins is high. The standardized mutual effect of *AR* and *TMP* has a positive value of 2.44. Although the value is small, it shows that a simultaneous change in *TMP* and *AR* affects the flux improvement. In addition, the standardized quadratic effect of *CFR* is positive because the flux improvement increases with increasing *CFR*. However, its value of 2.19 indicates the low sensitivity of flux improvement. Based on the absolute values of standardized effects, the flux improvement is the most sensitive to the AR of turbulence promoter.

The response surfaces of specific energy consumption for the pairs of the *AR* of TT with each of the operating conditions are shown in Fig. 3, *CFR* (a), the concentration of proteins (b) and *TMP* (c). Fig. 3a shows the effects of *AR* and *CFR* on the specific energy consumption for a concentration of proteins of 2.5 mass % and a *TMP* of 100 kPa (central point). The specific energy consumption increases sharply with increasing cross-flow rate for all aspect ratios. A minor influence of *AR* on increasing specific energy consumption could be observed under higher *CFR* values.

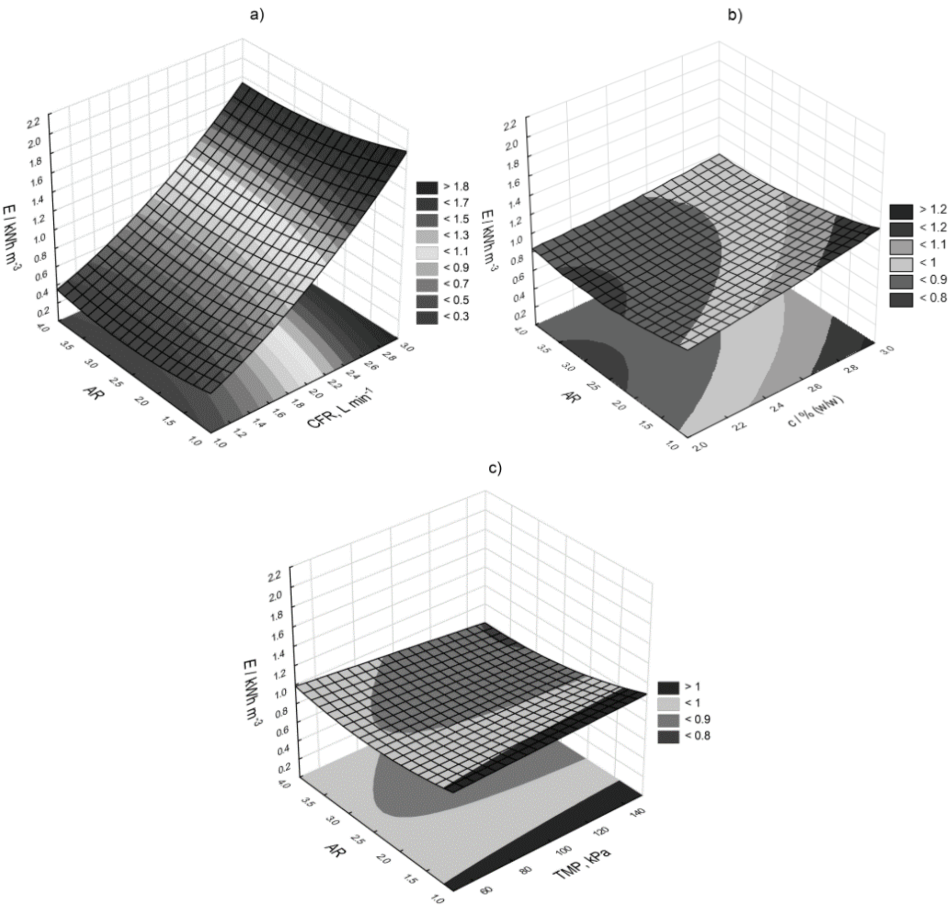

Fig. 3. Response surfaces for the specific energy consumption for pairs of the aspect ratio of TT with the operating conditions: a) cross-flow rate, b) the concentration of proteins and c) *TMP*.

The effects of *AR* and concentration of proteins on the specific energy consumption are shown in Fig. 3b for a *CFR* of 2.0 L min^{-1} and a *TMP* of 100 kPa (central point). The specific energy consumption increases slightly with increasing concentration of proteins for all tested *AR*s. This is because with a higher concentration of proteins, the membrane is more fouled and consequently the flux is lower.

The effects of *AR* and *TMP* on the specific energy consumption are shown in Fig. 3c for a concentration of proteins of 2.5 mass % and a *CFR* 2.0 L min^{-1} (central point). A significant influence of *TMP* on the specific energy consumption could not be observed. Only a slight decrease in the specific energy consumption could be noticed with increasing *TMP* when the *AR* of twisted tape was 4.0. This is due to low pressure loss in the case of this aspect ratio.

The significance of effects for the specific energy consumption was determined by analysing the ANOVA results presented in Table IV. For the specific energy consumption, the linear effects of concentration, cross-flow rate, aspect ratio and *TMP*, squared effects of cross-flow rate and aspect ratio, and the mutual effects of concentration and cross-flow rate, and aspect ratio and *TMP* are significant. The sensitivity of the response to the significant effects could be analysed from the Pareto chart of the standardized effects presented in Fig. S-3 of the Supplementary material. The specific energy consumption is the most sensitive to the linear variation in *CFR* and concentration, followed by the quadratic variation in *CFR*, the linear and the quadratic variation in *AR*, and the mutual variation in *AR* and *TMP*. The linear effect of *CFR* has a positive value of 56.26, indicating that the specific energy consumption increases the most with increasing *CFR*. The standardized effect of concentration is less influential and has a positive value of 8.89, meaning that it is significantly less influential. The linear and the quadratic effect of *AR* are negative with values of –7.71 and –6.26, respectively, showing that the energy consumption decreases with increasing *AR*. The standardized effect of the mutual effect of concentration and *CFR* is positive (4.35), meaning that a simultaneous change in both factors increases the specific energy consumption. Given that twisted tapes cause pressure loss in a membrane module, it could be suggested the aspect ratio as the most significant effect for specific energy consumption. However, this analysis shows that the specific energy consumption is extremely more sensitive to the linear effect of *CFR* than to that of *AR*.

The consumption of specific energy can be low (below 1.0 kWh m^{-3}) in the enhanced ultrafiltration of proteins if the operating conditions are chosen appropriately. This applies especially to the choice of *CFR* as one of the most influential effects.

The analysis showed that energy consumption is not significantly sensitive to *AR* despite the pressure loss caused by the twisted tape. On the other hand, the flux improvement is highly sensitive to *AR*. Furthermore, the twisted tape of the smallest aspect ratio of 1.0 is proven to be the most efficient and suitable for the application. However, the efficiency of the process also depends on the concentration of proteins. In practice, concentrations above 3 mass % of proteins are expected and higher energy consumption should be expected due to operation under higher *CFR*. For conventional operation without a promoter, the specific energy consumption can reach 10 kWh m^{-3}. In the dairy industry, long membrane modules are used, typically 1.2 m, with a big pressure loss. Generally, the energy consumption is high due to the pressure loss, larger than in a short membrane with a promoter, and due to high *TMP*s applied in the ultrafiltration of milk. Operation under high *TMP*s can be unbeneficial because the high *TMP*s worsen in-pore fouling. In the present study, however, it was shown that operation under low *TMP*s is possible despite the pressure loss caused by the twisted tape. Moreover, it was proved that the pressure loss and energy consumption dominantly depend on the imposed cross-flow rate. In commercial ultrafiltration of milk, high cross-flow velocities (4–10 m s^{-1}) and *TMP*s (300–700 kPa) are imposed to obtain fluxes ranging up to 50 L m^{-2} h^{-1},[18] and thus energy consumption is very high. Here, it should be noted that the fluxes could be slightly higher depending on the milk content and pre-treatment. When milk is skimmed and thermally treated, the milk proteins become partially denatured and they are more prone to fouling of the membrane. An increase in certain operating conditions, such as the concentration of proteins and transmembrane pressure, can exacerbate fouling in the ultrafiltration making it more challenging to mitigate. However, this is not the case with fouling in microfiltration. In previous studies on the microfiltration of partially skimmed milk, very high flux improvements were achieved with twisted tapes ranging from 400 to 600 %.[6] Flux improvements in the ultrafiltration are lower due to the different mechanisms of fouling compared to microfiltration. This is especially the case with the ultrafiltration of skimmed milk with the high content of proteins where in-pore fouling can be predominant under certain operating conditions, such as higher *TMP*s and lower cross-flow rates.

However, this study provides enhanced ultrafiltration of skimmed milk with operating fluxes above 70 L m^{-2} h^{-1} and low specific energy consumption below 1.0 kW h m^{-3}. Moreover, the overall energy consumption is lower due to *TMP* as low as 50 kPa. Generally, this study confirms the feasibility and sustainability of the application of twisted tapes as low-pressure turbulence promoters for the enhancement of ultrafiltration of milk proteins.

CONCLUSIONS

A sensitivity analysis of enhanced ultrafiltration of milk proteins has been presented in this study. For the enhancement of ultrafiltration, twisted tapes as low-pressure loss turbulence promoters were used. The effects of the operating conditions and twisted tape dimensions on flux improvement and specific energy consumption were analysed by application of response surface methodology. The fluxes are remarkably alleviated using twisted tapes due to minimisation of both concentration polarization and in-pore fouling of the membrane. The results of the goodness of fit justified the application of a second-order polynomial model for predicting both the flux improvement and the specific energy consumption. The analysis of variance (ANOVA) test showed that the aspect ratio has a dominant linear effect on flux improvement while the cross-flow rate has a dominant positive linear effect on the specific energy consumption. The linear effect of concentration and the mutual effect of aspect ratio and *TMP* are significant for both responses. By proper adjustment of the operating conditions, a high flux improvement of 300 % could be attained with specific energy consumption below 1.0 kW h m^{-3} using a twisted tape with an aspect ratio of 1.0 and imposing a low transmembrane pressure.

SUPPLEMENTARY MATERIAL

Additional data are available electronically at the pages of journal website: https:// //www.shd-pub.org.rs/index.php/JSCS/index, or from the corresponding author on request.

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ИЗВОД

УНАПРЕЂЕЊЕ УЛТРАФИЛТРАЦИЈЕ ПРОТЕИНА МЛЕКА ПРИМЕНОМ УВИЈЕНИХ ТРАКА: ИСПИТИВАЊЕ ОСЕТЉИВОСТИ ПРИМЕНОМ МЕТОДЕ ОДЗИВНЕ ПОВРШИНЕ

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У овом раду је презентована анализа утицаја радних услова на ултрафилтрацију протеина млека унапређену применом увијених трака као промотора турбуленције како би се смањило прљање, побољшао флукс и смањила потрошња енергије. Циљ рада је да се испита осетљивост унапређеног процеса на промене радних услова и димензије увијене траке. Увијена трака је уметнута у ултрафилтрациону мембрану (величина пора 50 nm) како би се повећала турбуленција и смањило прљање. Метода одзивне површине примењена је за моделовање и анализу утицаја радних услова и карактеристичне димензије увијене траке. Анализа је показала да карактеристична дименизија увијене траке има доминантан линеаран утицај на повећање флукса. Проток напојне смеше има доминантан линеаран утицај на специфичну потрошњу енергије. Линеаран утицај концентрације и заједнички утицај карактеристичне димензије и трансмембранског притиска су значајни за оба одзива. Погодним подешавањем радних услова, постиже се релативно велико повећање флукса од 300 % при специфичној потрошњи енергије мањој од 1,0 kW h m^{-3} применом увијене траке карактеристичне димензије 1,0 при ниском трансмембранском притиску.

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REFERENCES

1. Y. Pouliot, *Int. Dairy J.* **18** (2008) 735 (https://dx.doi.org/10.1016/j.idairyj.2008.03.005)
2. B. Antoine, Q. U. Peng, B. Patrice, G. Geneviève, *Langmuir* **30** (2013) 1 (https://dx.doi.org/https://doi.org/10.1021/la402865p)
3. G. Brans, C. G. P. H. Schroën, R. G. M. Van Der Sman, R. M. Boom, *J. Memb. Sci.* **243** (2004) 263 (https://dx.doi.org/10.1016/j.memsci.2004.06.029)
4. D. M. Krstić, M. N. Tekić, M. Đ. Carić, S. D. Milanović, *J. Memb. Sci.* **208** (2002) 303 (https://dx.doi.org/10.1016/S0376-7388(02)00308-3)

5. S. Popović, D. Jovičević, M. Muhadinović, S. Milanović, M. N. Tekić, *J. Memb. Sci.* **425–426** (2013) 113 (https://dx.doi.org/10.1016/j.memsci.2012.09.032)
6. S. Popović, M. Tekić, *J. Memb. Sci.* **384** (2011) 97 (https://dx.doi.org/10.1016/j.memsci.2011.09.016)
7. A. Jokić, Z. Zavargo, Z. Šereš, M. Tekić, *J. Memb. Sci.* **350** (2010) 269 (https://dx.doi.org/10.1016/j.memsci.2009.12.037)
8. B. B. Gupta, J. A. Howell, D. Wu, R. W. Field, *J. Memb. Sci.* **102** (1995) 31 (https://dx.doi.org/10.1016/0376-7388(94)00241-P)
9. N. Hilal, O. O. Ogunbiyi, N. J. Miles, *Sep. Sci. Technol.* **43** (2008) 286 (https://dx.doi.org/10.1080/01496390701787438)
10. D. M. Krstić, W. Höflinger, A. K. Koris, G. N. Vatai, *Sep. Purif. Technol.* **57** (2007) 134 (https://dx.doi.org/10.1016/j.seppur.2007.03.023)
11. S. Popović, M. Karadžić, J. Cakl, *J. Clean. Prod.* **231** (2019) 320 (https://dx.doi.org/10.1016/j.jclepro.2019.05.184)
12. B. Bellhouse, G. Costigan, K. Abhinava, A. Merry, *Sep. Purif. Technol.* **22–23** (2001) 89 (https://dx.doi.org/10.1016/S1383-5866(00)00145-3)
13. H. Y. Tsai, A. Huang, J. F. Soesanto, Y. L. Luo, T. Y. Hsu, C. H. Chen, K. J. Hwang, C. D. Ho, K. L. Tung, *J. Memb. Sci.* **573** (2019) 647 (https://dx.doi.org/10.1016/j.memsci.2018.11.081)
14. S. Armbruster, O. Cheong, J. Lölsberg, S. Popovic, S. Yüce, M. Wessling, *J. Memb. Sci.* **554** (2018) 156 (https://dx.doi.org/10.1016/j.memsci.2018.02.015)
15. Ž. R. Lazić, *Design of Experiments in Chemical Engineering*, Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim, 2004
16. M. C. Martí-Calatayud, M. C. Vincent-Vela, S. Álvarez-Blanco, J. Lora-García, E. Bergantiños-Rodríguez, *Chem. Eng. J.* **156** (2010) 337 (https://dx.doi.org/10.1016/j.cej.2009.10.031)
17. Gaspar, P. Tekic, A. Koris, A. Krisztina, S. Popovic, G. Vatai, *Hem. Ind.* **69** (2015) 713 (https://dx.doi.org/10.2298/HEMIND140312001G)
18. M. Leu, A. Marciniak, J. Chamberland, Y. Pouliot, L. Bazinet, A. Doyen, *J. Dairy Sci.* **100** (2017) 7071 (https://dx.doi.org/10.3168/jds.2017-12774).