



Modeling of beer membrane filtration

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ABSTRACT

The scope of this study is the modeling of beer membrane filtration, focusing on fouling mechanisms. Standardized lager beer was produced for the crossflow microfiltration investigations. Pall Membralox T1-70 tubular ceramic membrane with 0.5 μm pore size was used for filtrations. 2^p full factorial experimental design was applied, the three factors were the following: silica gel concentration (SGC): 0, 40, 80 g hL⁻¹; transmembrane pressure (TMP): 0.4, 0.8, and 1.2 bar, and retentate flow rate (Q): 50, 125, and 200 L h⁻¹. Steady-state fouling layer resistance ($R_{f,ss}$) was considered as the response. Analytical parameters of the rough beer and the dynamic viscosity values of the permeate samples were measured. The hydrodynamic parameters of the filtrations were determined. The parameters of the objective function were estimated, and the effect sizes were calculated. The global minimum of the objective function was found. The effect sizes of the significant parameters were the following: Q: -0.48; TMP: 0.81. The optimal values of the factors amounted to, respectively, TMP = 0.4 bar, Q = 200 L h⁻¹. The predicted $R_{f,ss}$ under the above condition was $1.925677776404 \times 10^{12} \text{ m}^{-1}$. The detailed method in this study can be implemented by membrane researchers and breweries.

Keywords: Beer membrane filtration; Brewing; Clarification; Crossflow microfiltration; Full factorial experimental design; Membrane fouling; Modeling; Optimization; Silica gel; Tubular ceramic membrane

1. Introduction

Beer is one of the most popular beverages all over the world [1] and it contains more than 90% water [2]. Brewing is water-consuming [3], thus optimization and modeling of the processes are important.

The purpose of beer membrane filtration (BMF) is to eliminate yeast and colloidal particles responsible for haze. Furthermore, BMF should ensure the microbiological stability of beer [4]. The alternative process to conventional clarification with Kieselguhr is BMF because of higher product quality, less environmental issues, less health and safety concerns, simplicity, flexibility, and lower cost [5]. However, one of the main problems of the ordinary application of BMF is fouling

mechanisms. The reasons of the fouling (flux decline during BMF) are the following: (i) concentration polarization, (ii) compact cake layer formation by yeast cells, debris, and coagulated materials on membrane surface, (iii) partial or complete plugging of pore entrances by suspended particles, and (iv) adsorption of macromolecules onto the pore walls which causes the membrane pore narrowing [6]. Unfortunately, high fouling resistance always leads to high operation costs, which restrict the application of microfiltration technology [7]. Thus, it is essential to reduce membrane fouling during BMF. Optimization of operating parameters can be a solution for reducing membrane fouling. Fortunately, full factorial experimental design can be used successfully to optimize the operating parameters of membrane filtration and study the process [8,9] with a minimal number of experiments [10].

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Generally, polymeric membranes are used for industrial BMF (e.g., Pentair’s beer membrane filtration system—BMF [11]), but ceramic membranes are suitable to be used in extreme conditions which could not be achieved by traditional polymer membranes [12]. The advantages of ceramic membranes include high chemical, microbial, physical, and thermal stability, insensitivity to swelling and ease of cleaning [12,13].

Application of filtration aids, for example, silica gel (SG), can also be a solution for reducing membrane fouling. However, the effect of SG is questionable. In one case, silica had an interactive or little effect on the normalized fouling rate during dead-end microfiltration of synthetic mixtures [14]. In another case, SG had a mainly positive effect on filtration rate during conventional beer filtration [15]. The properties and mechanisms of SG are discussed below. SG has a very large surface area containing a network of pores and this surface of SG is covered in silanol groups which form interactions with proline residues in haze-active proteins [16]. The mechanism of action of SG is via hydrogen bonding of protein carbonyl groups to hydroxyl groups on SG [17].

The scope of this study is to investigate the physical and mathematical modeling of BMF, focusing on fouling mechanisms.

The goals of the present investigation are: (i) to determine the analytical parameters of rough beer and permeate samples (dynamic viscosity values for the physical modeling), (ii) to determine the hydrodynamic parameters of the membrane filtrations for the response (physical modeling) of the experimental design, (iii) to analyze the experimental design (mathematical modeling) of the membrane filtrations (parameter and effect size estimation), and (iv) to optimize the objective function (the mathematical model) extracted from the analysis of the experimental design.

2. Materials and methods

2.1. Brewing

Thirty-three liters of standardized lager beer, “2A. International Pale Lager” from Beer Judge Certification Program (BJCP) [18] was brewed for the filtration investigations in the pilot-scale brewery of Department of Brewing and Distilling, Szent István University (Budapest, Hungary).

The BJCP vital statistics of 2A. International Pale Lager and the measured analytical parameters of the rough beer (feed) are shown in Table 1.

The brewing process is as follows. Eleven kilograms of Extra Pale Pilsner Malt from Weyermann, Germany and 40 L water with 12°dH hardness were used during mashing-in. The multi-step mashing program was the following with 1°C/min temperature increases and ±0.5°C temperature accuracy: 20 min at 50°C, 40 min at 63°C, 20 min at 72°C, and 1 min at 78°C. Lautering was carried out in a lauter tun. Sparging water with a temperature of 78°C was added in such a way to reach a final wort volume before boiling of 65 L. Twenty-eight grams of Hallertauer Magnum pellet hops with 14.6% (w/w) alpha acid content from HVG, Germany were added at the start of 90 min boiling, aiming for 22 IBU. After boiling, the hot trub was separated from bitter wort by whirlpool in 20 min. Then the wort was cooled to 12°C and oxygenated. The third generation lager yeast (Dreher, Hungary) was pitched at the rate of 15 million cells/mL. The fermentation was carried out at 11°C ± 1°C for 8 d, followed by a maturation at 4°C ± 1°C under 0.5 bar overpressure for 14 d.

2.2. Membrane filtration

The filtration experiments were carried out with bench scale in-house developed crossflow microfiltration (CFMF) equipment (Fig. 1).

Membralox T1–70 tubular ceramic membrane (Pall, USA) with active layer of aluminum oxide, 0.5 µm pore size, 7 mm channel diameter, 250 mm length, and 0.005 m² active surface was used for filtration purpose.

Filtration experiments were performed according to the experimental design (Table 3) discussed in Section 2.7.1. The three factors were silica gel concentration (SGC), transmembrane pressure (TMP), and retentate flow rate (Q). The used SG (Stabifix Brauerei-Technik, Germany) was a hydrogel with moisture content up to 65% (w/w) and designed for filtration with polysulfone based membranes.

Before each filtration experiment, water flux was measured at a given temperature and TMP. Following the water flux measurement, to avoid the dilution of rough beer with water, the water from CFMF equipment was drained with the

Table 1
BJCP vital statistics of 2A. International Pale Lager and measured analytical parameters of the rough beer (feed)

Name of parameter	BJCP vital statistics	Rough beer (feed)
Alcohol content (V/V %)	4.6–6.0	4.74
Original real extract (w/w %)	10.5–12.5	11.58
Final real extract (w/w %)	ND	4.10
Final apparent extract (w/w %)	2–3	2.37
Bitterness (IBU)	18–25	24
Color (EBC)	3.9–11.8	5.25
pH	ND	4.63
Turbidity at 20°C (EBC)	ND	18.0
Dynamic viscosity at 20°C (mPas)	ND	4.82

ND, no data.

purpose of the water flux measurement was to check the degree of membrane cleanliness [19]. Water flux is affected by temperature and TMP [20]. Thus, the water flux measurement has to be performed with given temperature and TMP values (the same values as the values of the water flux measurement before the filtration) to get comparable results.

The above-mentioned membrane cleaning procedure was applied based on the literature of cleaning after BMF [21].

2.4. Analytical parameters

Alcohol, real extract and apparent extract contents of rough beer were measured with AlcoLyzer Plus (Anton-Paar, Austria). The bitterness (concentrations of iso-alpha acids in ppm) and color of the rough beer were measured according to “Analytica European Brewery Convention (EBC) | Beer | 9.8—bitterness of beer (IM)” [22] and “Analytica EBC | Beer | 9.6—Color of Beer: spectrophotometric method (IM)” [23]. Absorbances were measured with DR 6000 spectrophotometer (Hach, USA) and Heraeus Megafuge 16R Centrifuge (Thermo Fisher Scientific, USA) was used for the separation of samples of bitterness measurements. Hydrogen chloride and Isooctane for bitterness measurements were purchased from Reanal, Hungary. The pH value of the rough beer was determined with 1100 H pH meter (VWR, USA). The turbidity of the rough beer was measured at a temperature of 20°C (permanent haze) with 2100P Turbidimeter (Hach, USA) in NTU and converted to EBC [24]. Dynamic viscosity values of rough beer and permeate samples were measured with Physica MCR 51 Rheometer (Anton-Paar Hungary, Hungary) with DG27 double gap concentric cylinder measurement system. Data were acquired and analyzed using Rheoplus/32 software [25]. Flow curves of samples were measured by increasing the shear rate from 500 to 1,000 s⁻¹ at temperatures of 0°C, 5°C, 10°C, 15°C, and 20°C. Dynamic viscosity values of samples were calculated based on the Herschel–Bulkley model [26] fitted to measured data of flow curve (shear stress in the function of shear rate).

2.5. Hydrodynamic parameters

Water and beer fluxes were determined with Eq. (1) [27]:

$$J = \frac{V}{A_m \times t_i} \tag{1}$$

where J (m³ m⁻² s⁻¹ = 3.6 × 10⁶ L m⁻² h⁻¹) is the flux, V (m³ = 10³ L) is the permeate volume, A_m (m²) is the membrane active surface area and t_i (s = 2.7777 × 10⁻⁴ h) is the time interval.

To describe the permeate flux during the filtration process a mathematical model [Eq. (2)] was used [28]:

$$J_b = J_{b0} + (J_{bss} - J_{b0}) \times (1 - e^{-K \times t}) \tag{2}$$

where J_b (m³ m⁻² s⁻¹ = 3.6 × 10⁶ L m⁻² h⁻¹) is the beer flux at any time, J_{b0} (m³ m⁻² s⁻¹ = 3.6 × 10⁶ L m⁻² h⁻¹) is the initial beer flux, J_{bss} (m³ m⁻² s⁻¹ = 3.6 × 10⁶ L m⁻² h⁻¹) is the steady-state beer flux, K (s⁻¹ = 3.6 × 10³ h⁻¹) is the flux decline coefficient and t (s = 2.7777 × 10⁻⁴ h) is the time.

TMPs were determined with Eq. (3) [29]:

$$\text{TMP} = \frac{p_1 + p_2}{2} - p_0 \tag{3}$$

where TMP (Pa = 10⁻⁵ bar) is the TMP, p_1 (Pa = 10⁻⁵ bar) is the inlet pressure, p_2 (Pa = 10⁻⁵ bar) is the outlet pressure and p_0 (Pa = 10⁻⁵ bar) is the pressure of the permeate.

Then intrinsic resistances of the clean membrane before filtration were determined with Eq. (4) [29]:

$$J_{w0} = \frac{\text{TMP}}{\mu_w \times R_m} \tag{4}$$

where J_{w0} (m³ m⁻² s⁻¹ = 3.6 × 10⁶ L m⁻² h⁻¹) is the water flux before filtration, μ_w (Pas) is the dynamic viscosity of water at given temperature and R_m (m⁻¹) is the intrinsic resistance of the clean membrane. Then total resistances were determined with Eq. (5) [29]:

$$J_b = \frac{\text{TMP}}{\mu_b \times R_t} \tag{5}$$

where μ_b (Pas) is the dynamic viscosity of the permeate of beer at a given temperature and R_t (m⁻¹) is the total resistance. Then, fouling layer resistances were determined with the following Eq. (6) [29]:

$$R_t = R_m + R_f \tag{6}$$

where R_f (m⁻¹) is the fouling layer resistance. For each filtration, R_{f0} (m⁻¹) initial fouling layer resistance and R_{fss} (m⁻¹) steady-state fouling layer resistance values were calculated with J_{b0} and J_{bss} values from Eq. (2).

2.6. Nonlinear regression

Based on Eq. (2) and time–flux data, J_{b0} , J_{bss} , and K -values of nine individual filtrations were determined with iterations by using IBM SPSS Statistics software [30]. Significances of parameter estimates, F -values and determination coefficients (R^2) of the models were evaluated. Normality of the residuals was accepted by the absolute values of their skewness and kurtosis as they all were below 1 [31].

2.7. Modeling

2.7.1. Experimental design

Filtration experiments were performed according to 2^p full factorial experimental design [32] because the application of experimental design minimizes the required number of experiments [33]. The aims of the application of the experimental design were the following: formulation of an objective function that describes the relationship between the factors and the response, determination of the significant parameters and the effect sizes.

The general mathematical model for a 2^3 full factorial experimental design (three factors, each at two levels) [Eq. (7)] is the following [32]:

$$Y = b_0 + \sum_{i=1}^3 b_i \times x_i + \sum_{i=1}^3 \sum_{j=1, i \neq j}^3 b_{ij} \times x_i \times x_j + b_{123} \times x_1 \times x_2 \times x_3 \quad (7)$$

where Y is the response; b_0 is the constant; b_i ($i = 1, 2, 3$) are the regression coefficients of the main factor effects; b_{ij} ($i = 1, 2, 3$; $j = 1, 2, 3$; $i \neq j$) and b_{123} are the regression coefficients of the interactions and x_i ($i = 1, 2, 3$) are the coded factors.

The factors and levels of the 2^p full factorial experimental design are shown in Table 2. $J_{b_{ss}}$ is the most important hydrodynamic parameter because generally, most of the time of the filtration run is operated with this flux value or when it is achieved permeate backflow techniques are applied. But the $R_{f_{ss}}$ describe more accurately the fouling characteristics than the $J_{b_{ss}}$ (Section 3.2). Thus, $R_{f_{ss}}$ was considered as the response of the 2^p full factorial experimental design.

The design matrix of the 2^p full factorial experimental design was generated in Statistica software [34] and it is shown in Table 3. The experiments were run in random order to reduce the potential for bias.

2.7.2. Analysis of the experimental design

The results of the experimental design were analyzed in various steps.

First, the parameters of the objective function were estimated (the non-significant parameters were eliminated), and model accuracy and determination coefficients were evaluated in R software [35] using RcmdrPlugin.DoE package [36].

Secondly, after the standardization of the response values, the effect sizes of the significant parameters were calculated (linear regression without the constant), and model accuracy and determination coefficients were evaluated in R software [35] using RcmdrPlugin.DoE package [36].

Finally, the normality of the residuals was checked by the Shapiro–Wilk normality test in RStudio software [37]. According to Shapiro–Wilk normality test, the normality of residuals of the objective function and function for effect size determination was accepted ($p = 0.23$).

2.7.3. Optimization

It was essential to find the global minimum of the objective function because the lower $R_{f_{ss}}$ is better from the technological point of view. Global optimization method “Grid Search” [38] was used for this purpose. Aspects and comments about the Grid Search optimization method applied for response surface objective function are shown in Table 4. Based on the literature [39], the Grid Search algorithm was implemented in Scilab software [40]. Furthermore, the response surface of the effects of significant parameters for $R_{f_{ss}}$ was plotted in Scilab software [40].

2.8. Limitations

Filtration experiments were conducted as single trials because in a pilot-scale brewery small amount of rough beer can be produced compared to the demand of multiple trials, and the same product quality between different batches of rough beer cannot be guaranteed. However, based on literature [24,41–43], some measurements were replicated for studying the reproducibility potential of the process (error variance for all the experimental campaign). The average coefficient of variation (10%) in the estimated beer flux values within the data population was appropriate and this value is very similar to the value in the literature [24,41–43].

3. Results and discussion

3.1. Analytical parameters

As it can be seen in Table 1, the analytical parameters of the rough beer that was brewed for the investigations correspond to BJCP vital statistics of 2A. International Pale Lager.

Because of the high apparent attenuation (79%) of the used lager yeast, the final apparent extract is low. Generally lower final extract content could lead to lower fouling resistances.

The bitterness of beer comes from a group of substances that are extracted components of hops during wort boiling [44]. The bitterness of the rough beer is not so high, because the wort was hopped moderately. About 20% of phenolic compounds present in beer are derived from hops [45] and polyphenols are membrane foulants [46].

Table 4
Aspects and comments about Grid Search optimization method applied for response surface objective function

Method	Comments	Conclusion
Response surface method	<ul style="list-style-type: none"> - The objective function is continuous. - Analytical optimization of the objective function results in a parameter set that does not necessarily fit to the parameter settings available for membrane filtration. 	Using Grid Search optimization of response surface objective function can provide us an optimal parameter set which can be directly applied in membrane filtration.
Grid Search optimization method	<ul style="list-style-type: none"> - It is a numerical method with brute force (exhaustive) search (global optimization method on a grid). - It does not get stuck at a local optimum. - The set of optimization grid points can be adjusted to the resolution of the parameter ranges available for membrane filtration. 	

The color of the rough beer was light because Extra Pale Pilsner Malt had been used for the brewing.

The pH of the rough beer was slightly higher than the normal pH values (4.2–4.4) of lager beers at the end of the fermentation [47], but this small pH difference has no significant effect on beer membrane filtration.

According to the EBC standard [48], the rough beer was very hazy (>8.0 EBC). It appeared that the reason for high fouling resistances was the high turbidity in the rough beer.

The dynamic viscosity values of rough beer and permeate samples at the filtration temperature are shown in Table 5.

The dynamic viscosity values are slightly high, but the reasons for this phenomenon are discussed below. The rotary viscometer was chosen because it provides rapid measurement of the flow curve of the sample tested with high reproducibility. The shear rate used in the test was rather high (when compared to shear rate occurring in a falling ball or capillary viscometer) and therefore shear stress values were also higher. However, all of the samples proved to show Newtonian behavior (linear flow curve). Furthermore, at a lower temperature, the dynamic viscosity values of beer samples are higher [49]. Therefore, the measured viscosity values (~5.5 mPas) are appropriate values and are in the proper range (10⁻³ Pas).

3.2. Hydrodynamic parameters

Fig. 2 shows the hydrodynamic parameters of the filtrations.

Unfortunately, the membrane resistance changes in time [50] because of membrane aging [51] and membrane cleaning efficiency [50]. Thus, the $R_{f,0}$ and $R_{f,ss}$ described more accurately the fouling characteristics than the $J_{b,0}$ and $J_{b,ss}$ values, because during the determination of the fouling layer resistances the actual intrinsic resistance of clean membrane was taken into consideration [Eq. (6)].

The lower flux decline coefficient is better from the technological point of view because if it is lower, the $J_{b,ss}$ is reached later.

Table 5
Dynamic viscosity values of rough beer and permeate samples at the filtration temperature

Sample	Dynamic viscosity at 10°C (mPas)
Rough beer (feed)	5.57 ± 0.01
1	5.55 ± 0.19
2	5.23 ± 0.03
3	6.11 ± 0.11
4	5.69 ± 0.12
Standard order number (permeate)	5.66 ± 0.09
5	5.60 ± 0.06
6	5.31 ± 0.05
7	5.30 ± 0.20
8	5.48 ± 0.43
9	

3.3. Nonlinear regression

According to the Student’s *t*-test, the parameter estimates were all significant ($p < 0.05$). Similarly, *F*-values and *R*²-values [$F(3,8) > 99.4$; $p < 0.001$; $R^2 > 0.9$; $p < 0.05$] of the models were also significant. There were two exceptions when a bootstrapping was necessary with 60 samples. In the case of setting “Standard order number 3,” the estimation of the coefficient of $J_{b,ss}$ was close to significant ($p = 0.06$), while for “Standard order number 7,” *R*² was as low as 0.51, though still significant ($p < 0.05$). Having such a low number of observations, it can be considered as very good results of fit.

3.4. Modeling

3.4.1. Analysis of the experimental design

Parameter estimates and effect size estimates of the significant parameters of the objective function are shown in Table 6.

SGC had no significant effect on $R_{f,ss}$. Furthermore, there were no significant interactions between the factors. From the final model, we omitted SGC and the interaction terms while the significant coefficients of TMP and Q are represented in Table 6. Model accuracy and determination coefficients of the objective function were also significant [$F(2,6) = 23.22$; $p < 0.01$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$]. The objective function [Eq. (8)] which exactly included the parameters determined as significant in Table 6 was the following:

$$R_{f,ss} = 7267766337848 + 3338343371150 \times x_{TMP} - 2003745190294 \times x_Q \tag{8}$$

The linear model which includes merely two factors (TMP and Q) is quite simple and accurate at the same time.

A positive sign of the effect size indicates an interactive effect of the factors, while a negative sign of the effect size indicates an antagonistic effect of the factors. Thus, TMP had an interactive effect and Q had an antagonistic effect on $R_{f,ss}$. The possible reasons for these phenomena are discussed below.

Firstly, TMP is the driving force of membrane filtration. It appears, that TMP pressed the foulants on the membrane surface and into the membrane pores. Maybe higher TMP pressed more the foulants. Therefore, higher TMP led to higher $R_{f,ss}$.

Secondly, Q determines directly the crossflow velocity and turbulence of the feed in the flow channel of the membrane. It appears that flowing feed could sweep the membrane. Maybe feed with higher crossflow velocity swept more the foulants. Therefore, a higher Q led to lower $R_{f,ss}$. Furthermore, the absolute value of the effect size of the TMP was higher than the absolute value of the effect size of the Q. This implied that TMP had a higher effect on $R_{f,ss}$ than Q had.

Model accuracy and determination coefficients of the effect size estimation were significant [$F(2,7) = 27.09$; $p < 0.001$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$].

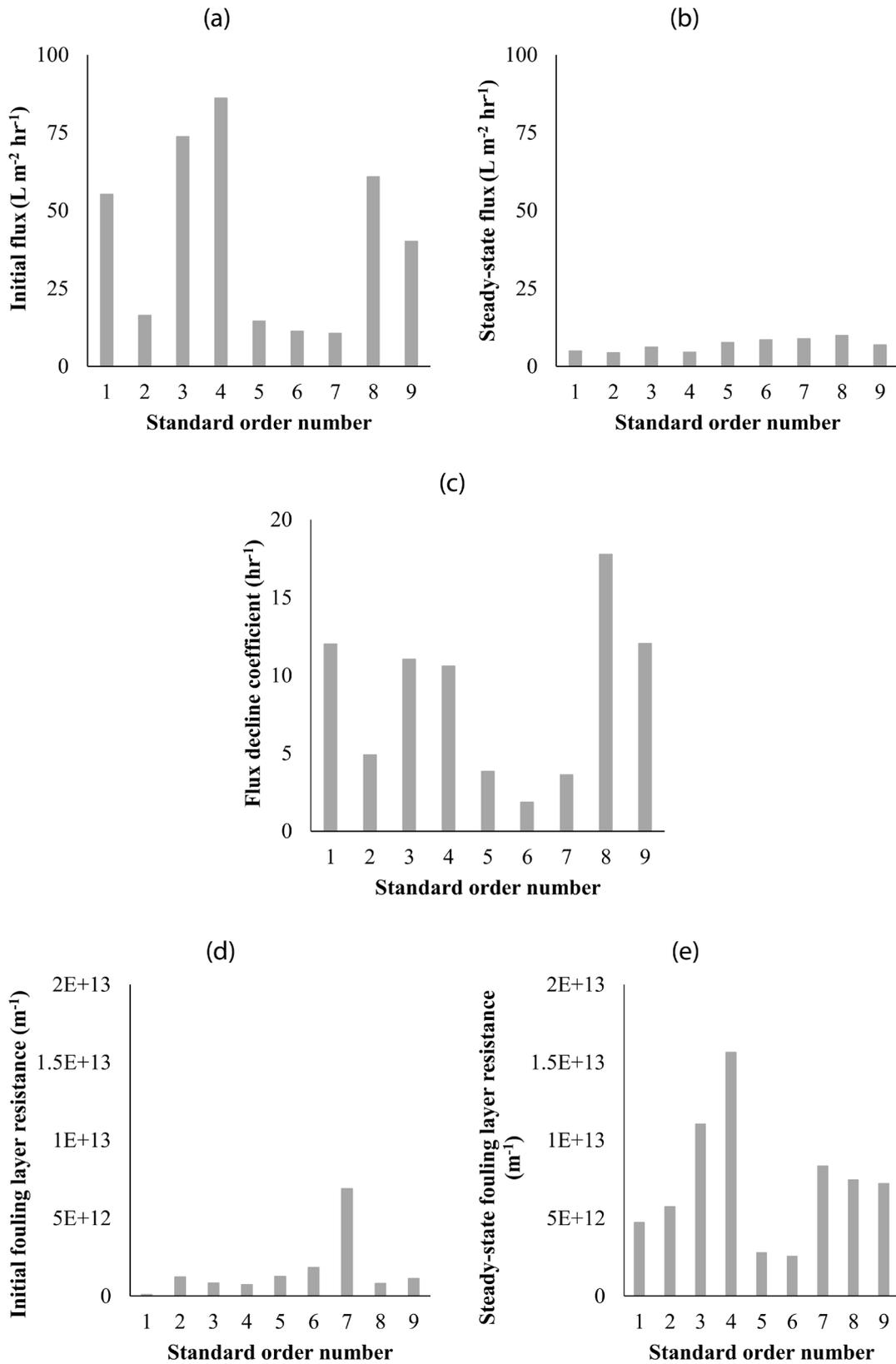


Fig. 2. Hydrodynamic parameters of the filtrations. (a) Initial beer flux, (b) steady-state beer flux, (c) flux decline coefficient, (d) $R_{f,0}$ and (e) $R_{f,ss}$.

Table 6
Parameter (coefficient) estimates and effect size estimates of the significant parameters of the objective function

Term	Estimate	Coefficient			Effect size				
		Standard error	<i>t</i>	Pr(> <i>t</i>)	Parameter	Estimate	Standard error	<i>t</i>	Pr(> <i>t</i>)
b_0	7,267,766,337,848	538,651,945,026	13.4925	***	–	–	–	–	–
b_{TMP}	3,338,343,371,150	571,326,664,541	5.8431	**	TMP	0.8069	0.1278	6.311	***
b_Q	-2,003,745,190,294	571,326,664,541	-3.5072	*	Q	-0.4843	0.1278	-3.788	**

Response: $R_{f,ss}$
Significance codes: ***0.001, **0.01, *0.05.

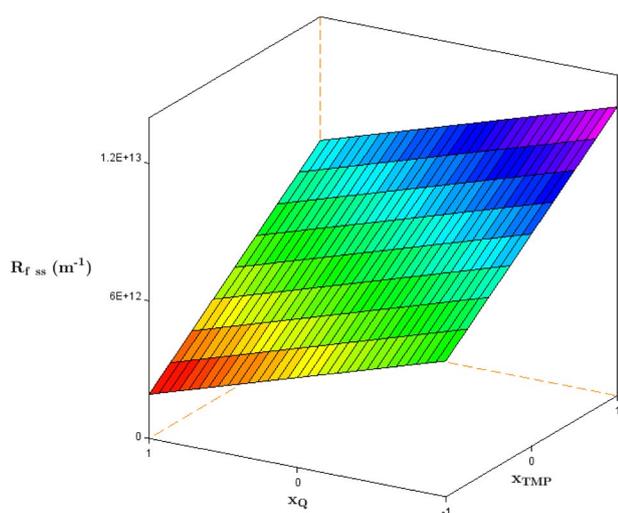


Fig. 3. Response surface of the effects of significant parameters (x_{TMP} , x_Q) for $R_{f,ss}$.

3.4.2. Optimization

Fig. 3 shows the response surface of the effects of significant parameters (x_{TMP} , x_Q) for $R_{f,ss}$.

The optimal values of the factors amounted to, respectively, $\text{TMP} = 0.4 \text{ bar}$, $Q = 200 \text{ L h}^{-1}$. The predicted $R_{f,ss}$ under the above condition was $1.925677776404 \times 10^{12} \text{ m}^{-1}$. Therefore, lowest $R_{f,ss}$ could be achieved with the lowest TMP and the highest Q.

4. Conclusions

All of the goals of the present investigation mentioned in Introduction section (Section 1.) have been completely achieved: (i) valuable information for membrane filtrations was gained from determined analytical parameters of rough beer and viscosity values of permeate samples could be used for the physical modeling, (ii) the determined values of hydrodynamic parameters of the membrane filtrations could be used for the physical modeling and the experimental design, (iii) the experimental design was analyzed, parameters of the objective function and effect sizes were estimated, and (iv) the global minimum of the objective function was successfully found and the results of the optimization can be applied in practice.

The most important findings of this research paper are summarized, and conclusions are drawn below.

According to the analysis of the experimental design, TMP and Q had a significant effect, while SGC had no significant effect on $R_{f,ss}$ with the given parameters. Furthermore, there were no significant interactions between the factors. This means that the commercial breweries should only focus on the optimization of TMP and Q, and SG free BMF can be performed. The SG free BMF is important because of environmental issues. However, filtration aids other than SG can be developed and tested to intensify BMF.

TMP had an interactive effect and Q had an antagonistic effect on $R_{f,ss}$. Furthermore, the effect size of TMP is higher than the effect size of Q.

Based on the results of the optimization the lowest $R_{f,ss}$ could be achieved with the lowest TMP and the highest Q. Thus, commercial breweries should set the operating parameters at these levels.

The laboratory measurements, modeling, and optimization methods that were detailed in this research paper can be implemented by membrane researchers and commercial breweries during product and technology development because of the simplicity and relatively low resource demand.

Acknowledgments



EMBERI ERŐFORRÁSOK
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Symbols

μ_b	—	Dynamic viscosity of the permeate of beer at a given temperature, Pas
μ_w	—	Dynamic viscosity of water at a given temperature, Pas
A_m	—	Membrane active surface area, m ²
b_i	—	Regression coefficients of the main factor effects
b_{ij}, b_{123}	—	Regression coefficients of the interactions
J	—	Flux, L m ⁻² h ⁻¹
J_b	—	Beer flux at any time, L m ⁻² h ⁻¹
J_{b0}	—	Initial beer flux, L m ⁻² h ⁻¹
J_{bss}	—	Steady-state beer flux, L m ⁻² h ⁻¹
J_{w0}	—	Water flux before filtration, L m ⁻² h ⁻¹
K	—	Flux decline coefficient, h ⁻¹
p_0	—	Pressure of the permeate, bar
p_1	—	Inlet pressure, bar
p_2	—	Outlet pressure, bar
Q	—	Retentate Flow Rate, L h ⁻¹
R_f	—	Fouling layer resistance, m ⁻¹
R_{f0}	—	Initial fouling layer resistance, m ⁻¹
R_{fss}	—	Steady-state fouling layer resistance, m ⁻¹
R_m	—	Intrinsic resistance of clean membrane, m ⁻¹
R_t	—	Total resistance, m ⁻¹
SGC	—	Silica gel concentration, g hL ⁻¹
t	—	Time, h
t_i	—	Time interval, h
TMP	—	Transmembrane pressure, bar, Pa
V	—	Permeate volume, L
x_i	—	Coded factors
Y	—	Response

References

- [1] S. Wunderlich, W. Back, In: V.R. Preedy, Ed., Beer in Health and Disease Prevention, Academic Press, Cambridge, 2008, pp. 3–16.
- [2] V.N. Okafor, A.N. Eboatu, R.I. Anyalebechi, U.W. Okafor, Comparative studies of the physicochemical properties of beers brewed with hop extracts and extracts from four selected tropical plants, *J. Adv. Chem. Sci.*, 2 (2016) 382–386.
- [3] L. Fillaudeau, P. Blanpain-Avet, G. Daufin, Water, wastewater and waste management in brewing industries, *J. Clean. Prod.*, 14 (2006) 463–471.
- [4] G. Daufin, J.-P. Escudier, H. Carrère, S. Bérot, L. Fillaudeau, M. Decloux, Recent and emerging applications of membrane processes in the food and dairy industry, *Food Bioprod. Process. Trans. Inst. Chem. Eng. Part C*, 79 (2001) 89–102.
- [5] A. Ambrosi, N.S.M. Cardozo, I.C. Tessaro, Membrane separation processes for the beer industry: a review and state of the art, *Food Bioprocess Technol.*, 7 (2014) 921–936.
- [6] M.A. Kazemi, M. Soltanieh, M. Yazdanshenas, L. Fillaudeau, Influence of crossflow microfiltration on ceramic membrane fouling and beer quality, *Desalin. Water Treat.*, 51 (2013) 4302–4312.
- [7] Y. Sun, Z. Qin, L. Zhao, Q. Chen, Q. Hou, H. Lin, L. Jiang, J. Liu, Z. Du, Membrane fouling mechanisms and permeate flux decline model in soy sauce microfiltration, *J. Food Process Eng.*, 41 (2018) e12599.
- [8] H. Azizi Namaghi, S.M. Mousavi, Factorial experimental design for treatment of an industrial wastewater using micellar-enhanced ultrafiltration, *Desalin. Water Treat.*, 57 (2016) 5416–5424.
- [9] S. Banvolgyi, K.S. Bahceci, G. Vatai, S. Bekassy, E. Bekassy-Molnar, Partial dealcoholization of red wine by nanofiltration and its effect on anthocyanin and resveratrol levels, *Food Sci. Technol. Int.*, 22 (2016) 677–687.
- [10] L. Hamdi, L. Boumehti Toumi, Z. Salem, K. Allia, Full factorial experimental design applied to methylene blue adsorption onto Alfa stems, *Desalin. Water Treat.*, 57 (2016) 6098–6105.
- [11] Pentair - Beer Membrane Filtration System - Beverage Filtration Solutions | Pentair Food and Beverage Process Solutions. Available at: <https://foodandbeverage.pentair.com/en/products/beverage-filtration-solutions-bmf-18>
- [12] Y.-L. Elaine Fung, H. Wang, Investigation of reinforcement of porous alumina by nickel aluminate spinel for its use as ceramic membrane, *J. Membr. Sci.*, 444 (2013) 252–258.
- [13] N. Das, H.S. Maiti, Formation of pore structure in tape-cast alumina membranes – effects of binder content and firing temperature, *J. Membr. Sci.*, 140 (1998) 205–212.
- [14] Y.-J. Kim, J. Jung, S. Lee, J. Sohn, Modeling fouling of hollow fiber membrane using response surface methodology, *Desalin. Water Treat.*, 54 (2015) 966–972.
- [15] K.A. Leiper, R. Duszansky, G.G. Stewart, Premixing of isinglass and silica gel to obtain improved beer stability, *J. Inst. Brew.*, 108 (2002) 28–31.
- [16] E.I. Benítez, M.R. Acquisgrana, N.M. Peruchena, G.L. Sosa, J.E. Lozano, Effects of silica gel on reduction in gluten during several beer brewing stages, *Int. J. Food Sci. Technol.*, 51 (2016) 920–928.
- [17] D.S. Ryder, J. Power, In: F.G. Priest, G.G. Stewart, Eds., Handbook of Brewing, CRC Press, Boca Raton, FL, 2006, pp. 333–382.
- [18] Beer Judge Certification Program 2015 Style Guidelines. Available at: http://bjcp.org/docs/2015_Guidelines_Beer.pdf
- [19] P. Blanpain-Avet, J.F. Migdal, T. Bénézech, The effect of multiple fouling and cleaning cycles on a tubular ceramic microfiltration membrane fouled with a whey protein concentrate. Membrane performance and cleaning efficiency, *Food Bioprod. Process.*, 82 (2004) 231–243.
- [20] I.H. Huisman, B. Dutré, K.M. Persson, G. Trägårdh, Water permeability in ultrafiltration and microfiltration: viscous and electroviscous effects, *Desalination*, 113 (1997) 95–103.
- [21] Á. Varga, I. Gáspár, R. Juhász, M. Ladányi, B. Hegyes-Vecseri, Z. Kókai, E. Márki, Beer microfiltration with static turbulence promoter: sum of ranking differences comparison, *J. Food Process Eng.*, 42 (2018) e12941.
- [22] Analytica EBC | Beer | 9.8 - Bitterness of Beer (IM). Available at: <https://brewup.eu/ebc-analytica/beer/bitterness-of-beer-im/9.8>
- [23] Analytica EBC | Beer | 9.6 - Colour of Beer: Spectrophotometric Method (IM). Available at: <https://brewup.eu/ebc-analytica/beer/colour-of-beer-spectrophotometric-method-im/9.6>.
- [24] A. Cimini, M. Moresi, Beer clarification using ceramic tubular membranes, *Food Bioprocess Technol.*, 7 (2014) 2694–2710.
- [25] Rheoplus/32 v.3.40, Anton-Paar, Austria, 2008.
- [26] T.G. Mezger, The Rheology Handbook, Vincentz Network, Hannover, 2006, pp. 57.
- [27] I. Gáspár, A. Koris, Z. Bertalan, G. Vatai, Comparison of ceramic capillary membrane and ceramic tubular membrane with inserted static mixer, *Chem. Pap.*, 65 (2011) 596–602.
- [28] Á. Varga, E. Márki, Microfiltration: a novel technology for removal of trub from hopped wort, *J. Food Process Eng.*, 42 (2019) e13200.
- [29] I. Ben Hassan, M. Ennouri, C. Lafforgue, P. Schmitz, A. Ayadi, Experimental study of membrane fouling during crossflow microfiltration of yeast and bacteria suspensions: towards an analysis at the microscopic level, *Membranes*, 3 (2013) 44–68.
- [30] SPSS Statistics 25.0, IBM, USA, 2017.
- [31] B.G. Tabachnick, L.S. Fidell, Using Multivariate Statistics, Pearson, London, 2013, p. 79.
- [32] S. Kemény, Design and Analysis of Experiments (in Hungarian), Tankönyvkiadó Vállalat, Budapest, 1985, pp. 137–142.
- [33] B. Akcal Comoglu, C. Filik Iscen, S. Ilhan, The anaerobic treatment of pharmaceutical industry wastewater in an anaerobic batch and upflow packed-bed reactor, *Desalin. Water Treat.*, 57 (2016) 6278–6289.
- [34] Statistica 12.0, StatSoft, USA, 2012.
- [35] R Core Team, R-3.5.1, R Foundation for Statistical Computing, Vienna, Austria, 2018.
- [36] U. Groemping, J. Fox, RcmdrPlugin.DoE 0.12-3, R Foundation for Statistical Computing, Vienna, Austria, 2014.

- [37] RStudio Team, RStudio 1.2.1335, Integrated Development for R. RStudio, Inc, Boston, MA, USA, 2015.
- [38] Global optimization (in Hungarian). Available at: http://math.bme.hu/~tuzcsaba/doc/globopt_hun.pdf
- [39] Grid Search in MATLAB. Available at: <https://www.andriiparkhomenko.net/files/Grid%20Search%20in%20Matlab.pdf>
- [40] Scilab 6.0.1, ESI Group, Paris, France, 2018.
- [41] A. Cimini, M. Moresi, Assessment of the optimal operating conditions for pale lager clarification using novel ceramic hollow-fiber membranes, *J. Food Eng.*, 173 (2016) 132–142.
- [42] A. Cimini, M. Moresi, Novel cold sterilization and stabilization process applied to a pale lager, *J. Food Eng.*, 145 (2015) 1–9.
- [43] A. Cimini, O. Marconi, G. Perretti, M. Moresi, Novel procedure for lager beer clarification and stabilization using sequential enzymatic, centrifugal, regenerable PVPP and crossflow microfiltration processing, *Food Bioprocess Technol.*, 7 (2014) 3156–3165.
- [44] V. Popescu, A. Soceanu, S. Dobrin, G. Stanciu, A study of beer bitterness loss during the various stages of the Romanian beer production process, *J. Inst. Brew.*, 119 (2013) 111–115.
- [45] A. Fărcaș, M. Tofana, S. Socaci, S. Scrob, L.-C. Salanta, B. Andrei, preliminary study on antioxidant activity and polyphenols content in discharged waste from beer production, *J. Agroaliment. Process. Technol.*, 19 (2013) 319–324.
- [46] J. Stopka, Š. Schlosser, Z. Dömény, D. Šmogrovičová, Flux decline in microfiltration of beer and related solutions of model foulants through ceramic membranes, *Pol. J. Environ. Stud.*, 9 (2000) 65–69.
- [47] H. Kaneda, M. Takashio, T. Tamaki, T. Osawa, Influence of pH on flavour staling during beer storage, *J. Inst. Brew.*, 103 (1997) 21–23.
- [48] HI-847492-02 Haze Meter for Beer Quality Analysis - Turbidity – Parameters. Available at: <https://www.hannainstruments.co.uk/asbc-standard-haze-meter-for-beer-analysis.html#specifications>
- [49] L. Severa, J. Los, On the influence of temperature on dynamic viscosity of dark beer, *Acta Univ. Agric. Silvic. Mendelianae Brun.*, 56 (2008) 303–307.
- [50] D. Jeison, *Anaerobic Membrane Bioreactors for Wastewater Treatment: Feasibility and Potential Applications*, Wageningen University, Wageningen, Netherlands, 2007.
- [51] S. Robinson, S.Z. Abdullah, P. Berube, P. Le-Clech, Ageing of membranes for water treatment: linking changes to performance, *J. Membr. Sci.*, 503 (2016) 177–187.