Hungarian University of Agriculture and Life Sciences

Studying the Application of Membrane Separation Processes in the Brewing Industry

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Background of the work and its aims

The scope of this thesis is studying the application of membrane separation processes (MSPs) in the brewing industry. Since MSPs are cleaner technology with efficient separation capability and generally mild operating conditions, and still an emerging technology in the brewing industry, the above-mentioned topic of the thesis is essential and relevant.

After the detailed literature review, four main research gaps were examined in this study: wort membrane filtration (WMF), beer membrane filtration (BMF) with static turbulence promoter (STP), beer membrane filtration (BMF) with silica gel (SG) and beer dealcoholization (BDA) by reverse osmosis (RO).

The aims of wort membrane filtration (WMF) were the following: complete removal of hot trub and cold trub from pale hopped wort by membrane filtration, studying the effect of membrane filtration on the changes in analytical parameters, determination of permeate flux values.

The aims of beer membrane filtration (BMF) with static turbulence promoter (STP) were the following: developing a model to describe the BMF with STP process, determination of the effect sizes of the significant parameters of the model, finding the optimum and the optimal values of the significant parameters of the model.

The aims of beer membrane filtration (BMF) with silica gel (SG) were the following: developing a model to describe the BMF with SG process, determination of the effect sizes of the significant parameters of the model, finding the optimum and the optimal

values of the significant parameters of the model.

The aims of membrane cleaning for BMF were the following: recovering the initial intrinsic resistance of the microfiltration membrane, developing a novel and efficient membrane cleaning method for beer membrane filtration.

The aims of beer dealcoholization (BDA) by reverse osmosis (RO) were the following: developing a model to describe the BDA by RO process, determination of the effect sizes of the significant parameters of the model, finding the optimum and the optimal values of the significant parameters of the model.

The aims of membrane cleaning for beer dealcoholization by reverse osmosis were the following: recovering the initial intrinsic resistance of the reverse osmosis membrane, developing a novel and efficient membrane cleaning method for beer dealcoholization by reverse osmosis.

Materials and methods

Materials

The following materials was used for the experiments: brewing water (13°dH total hardness) for WMF and BMFs; malts: Pilsner Malt (Boortmalt, Hungary) for WMF, Extra Pale Premium Pilsner Malt (Weyermann, Germany) for BMFs; hops: Hallertauer Tradition T90 hop pellets (HVG, Germany) for WMF; Hallertauer Magnum T90 hop pellets (HVG, Germany) for BMFs; yeast: liquid lager yeast (Cara Technology, United Kingdom) for BMFs; beers: 0.5 L canned Soproni Klasszikus pale lager bright beers (HEINEKEN Hungária, Hungary) with 4.5% (V/V) ethanol

content for BDA by RO; static turbulence promoter: SPIRAL LD2 STP (Inox, Serbia) for BMF with STP; silica gel: Stabifix W MF (Stabifix Brauerei-Technik, Germany) for BMF with SG.

The characteristics of the applied membranes for the membrane separation experiments are shown in *Table 1*.

Table 1: The characteristics of the applied membranes for the membrane separation experiments

Application	**Manufacturer**	**Type**	**Material**	**Pore size**
WMF	Pall, United States of America	Membralox T1-70	ceramic	0.2 µm
BMF with STP	Pall, United States of America	Membralox T1-70	ceramic	0.5 µm
BMF with SG	Pall, United States of America	Membralox T1-70	ceramic	0.5 µm
BDA by RO	Alfa Laval, Sweden	RO99	polyester	[a]

[a] The reverse osmosis membrane is not characterised according to pore size, as the selective layer is dense. $R_{NaCl} \geq 98$ %, measured on 2000 ppm NaCl, 16 bar, 25 °C.

Equipment

50 L pilot-scale brewery (HBH, Hungary) was used for wort and rough beer productions. Bench scale in-house developed crossflow microfiltration (CFMF) equipment was used for WMF and BMF experiments, and bench scale "HF-528/08." crossflow reverse osmosis equipment (CFRO) (Hidrofilt, Hungary) was

used for BDA experiments.

Methods

Wort was produced for WMF experiments and rough beer was brewed for BMF experiments. The rough beer recipe was designed based on "2A. International Pale Lager" from Beer Judge Certification Program (BJCP).

Operating parameters of the examined MSPs in this study are shown in *Table 2*.

Table 2: Operating parameters of the examined MSPs in this study

Experiment	**TMP**	**Q**	**STP**	**SGC**
WMF	0.4 bar	50 L h^{-1}	-	-
BMF with STP	0.4, 0.8, 1.2 bar	50, 125, 200 L h^{-1}	no, yes	-
BMF with SG	0.4, 0.8, 1.2 bar	50, 125, 200 L h^{-1}	-	0, 40, 80 g hL^{-1}
BDA by RO	10, 20, 30 bar	120, 180, 240 L h^{-1}	-	-

TMP = transmembrane pressure, Q = retentate flow rate, STP = static turbulence promoter, SGC = silica gel concentration

Membrane separation experiments were performed at 10 ± 1 °C, but BDA by RO was performed at 15 ± 1 °C. The feed volumes of WMF, BMF with STP, BMF with SG and BDA by RO were 3 L, 3 L, 3 L and 5 L respectively. The volume concentration factor (VCF) values of WMF, BMF with STP, BMF with SG and BDA by RO were 1.04, 1.1, 1.1 and 1.06 respectively.

The applied cleaning method of MF membranes was the following: deionized water for 5 min at 25 °C; 1 % (w/w) Sodium

hydroxide for 60 min at 60 °C; deionized water for 10 min at 25 °C; 1 % (w/w) Hydrogen peroxide for 60 min at 25 °C; deionized water for 10 min at 25 °C; TMP = 0.2 bar, Q = 50 L hr^{-1}.

The applied cleaning method of RO membrane was the following: deionized water for 10 min at 25 °C; 0.2 % (w/w) Sodium hydroxide for 60 min at 25 °C; deionized water for 10 min at 25 °C; TMP = 6 bar; Q = 240 L hr^{-1}.

Different analytical parameters of feed and permeate samples were measured: ethanol content, extract content, bitterness, colour, dynamic viscosity, free amino nitrogen content (FAN), particle size distribution, pH, total polyphenol content (TPC), turbidity, β-glucan content. Furthermore, separation characteristic parameters (retention) of different components were calculated.

Different hydrodynamic parameters of the MSPs were determined: initial flux, steady-state flux, flux decline coefficient, initial fouling layer resistance, steady-state fouling layer resistance, initial ethanol flux.

For checking the cleaning efficiency of the applied membrane cleaning methods, flux recoveries were determined.

In case of WMF and BMF experiments, initial flux, steady-state flux and flux decline coefficient values were determined with nonlinear regression (iterations by using *SPSS Statistics 25.0*, 2017).

In case of BDA by RO experiments, initial flux and flux decline coefficient values were determined with linear regression

(iterations by using *SPSS Statistics 25.0*, 2017).

Different models were created to describe the membrane separation processes. 2^p full factorial experimental designs were applied. Factors of BMF with STP experiments were the following: static turbulence promoter (STP), TMP, Q. Factors of BMF with SG experiments were the following: silica gel concentration (SGC), TMP, Q. Responses of BMF experiments were steady-state fouling layer resistance ($R_{f\,ss}$). Factors of BDA by RO experiments were the following: TMP, Q. Response of BDA experiments was initial ethanol flux ($J_{EtOH\,0}$). The experimental designs were analysed, parameters of the objective functions were estimated, and effect sizes of the significant parameters were calculated in *R-3.5.1*, 2018 software using *RcmdrPlugin.DoE 0.12-3*, 2014 package. The objective functions of the models were optimized with Grid Search optimisation method in *Scilab 6.1.0*, 2020 software with self-developed codes. In case of BMF with STP and BMF with SG, it was essential to find the global minima of the objective functions, because the lower steady-state fouling layer resistance ($R_{f\,ss}$) is better from technological point of view. In case of BDA by RO, it was essential to find the global maximum of the objective function, because the higher initial ethanol flux ($J_{EtOH\,0}$) is better from technological point of view.

Results and discussion

It has been proven that hot trub and cold trub can be completely removed by WMF and the changes in the analytical parameters are appropriate. The bitterness decreased by approximately 5 unit,

TPC decreased by approximately 5.6 %, retention of β-glucan was 40.17 % and free amino nitrogen content did not change. The initial wort flux and the steady-state wort flux of WMF were 16.75 L m^{-2} h^{-1} and 4.89 L m^{-2} h^{-1}, respectively. These values are quite low, because of fouling mechanism. Higher flux values, stable fluxes and better permeate quality can be achieved with optimisation of the process and pre-treatment of the wort.

In case of BMF with STP, the model (*Equation* 1) was the following:

$$\begin{aligned} R_{f\,ss} = {} & 4.4630 \times 10^{12} - 1.7662 \times 10^{12} \times x_{STP} \\ & + 1.5702 \times 10^{12} \times x_{TMP} \\ & - 1.5166 \times 10^{12} \times x_{Q} \\ & - 6.9648 \times 10^{11} \times x_{STP} \times x_{TMP} \\ & + 4.6600 \times 10^{11} \times x_{STP} \times x_{Q} \\ & - 4.3718 \times 10^{11} \times x_{TMP} \times x_{Q} \end{aligned} \qquad \text{Equation 1}$$

where $R_{f\,ss}$ (m^{-1}) is the steady-state fouling layer resistance; x_{STP} is the coded factor for static turbulence promoter (STP) with the factor values: -1, +1; x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1 and x_Q is the coded factor for retentate flow rate (Q) with the factor interval: -1 – +1. The range of validity: STP = no or yes; TMP = 0.4 – 1.2 bar, Q = 50 - 200 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(6;3) = 203.7$; $p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$). The effect sizes of the significant parameters were the following: STP = -0.61, TMP = 0.54, Q = -0.52, STP:TMP = -0.24, STP:Q = 0.16 and TMP:Q = -0.15. Model

accuracy and determination coefficients of the effect size estimation were significant ($F(6;4) = 271.6$; $p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$). I have found the optimum (global minimum) of the objective function. The optimal values of the factors amounted to respectively STP = yes, TMP = 0.4 bar, Q = 200 L h^{-1}. The predicted $R_{f\,ss}$ under the above condition was 1.2097×10^{12} m^{-1}. Therefore, lowest steady-state fouling layer resistance ($R_{f\,ss}$) could be achieved with the usage of turbulence promoter (STP), the lowest transmembrane pressure (TMP) and the highest retentate flow rate (Q).

In case of BMF with SG, the model (*Equation 2*) was the following:

$$R_{f\,ss} = 7.2678 \times 10^{12} + 3.3383 \times 10^{12} \times x_{TMP} - 2.0038 \times 10^{12} \times x_Q \qquad \text{Equation 2}$$

where $R_{f\,ss}$ (m^{-1}) is the steady-state fouling layer resistance, x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1 and x_Q is the coded factor for retentate flow rate (Q) with the factor interval: -1 – +1. The range of validity: silica gel concentration (SGC) = 0 – 80 g hL^{-1}; TMP = 0.4 – 1.2 bar, Q = 50 - 200 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(2;6) = 23.22$; $p < 0.01$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$). The effect sizes of the significant parameters were the following: TMP = 0.81, Q = -0.48. 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 $R^2 = 0.85$). I have found the optimum (global minimum) of the objective function. 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 1.9257×10^{12} m^{-1}. Therefore, lowest steady-state fouling layer resistance ($R_{f\ ss}$) could be achieved with the lowest transmembrane pressure (TMP) and the highest retentate flow rate (Q). Furthermore, silica gel free BMF can be performed.

In case of BDA by RO, the model (*Equation 3*) was the following:

$$J_{EtOH\ 0} = 80.871 + 41.094 \times x_{TMP} \qquad \text{Equation 3}$$

where $J_{EtOH\ 0}$ (g m^{-2} h^{-1}) is the initial ethanol flux and x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1. The range of validity: TMP = 10 – 30 bar, Q = 120 - 240 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(1;5) = 143.1$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$). The effect size of the significant parameter was the following: TMP = 1.20. Model accuracy and determination coefficients of the effect size estimation were significant ($F(1;6) = 171.7$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$). I have found the optimum (global maximum) of the objective function. The optimal value of the factor amounted to TMP = 30 bar. The predicted $J_{EtOH\ 0}$ under the above condition was 121.965 g m^{-2} h^{-1}. Therefore, highest initial ethanol flux ($J_{EtOH\ 0}$) could be achieved with the highest transmembrane pressure (TMP) and the lowest retentate flow rate (Q) can be applied.

In addition, a novel and efficient membrane cleaning methods were developed and applied to recover the initial intrinsic

resistances of MF and RO membranes. In case of MF membranes, the average of flux recoveries was higher than 97 %. In case of RO membranes, the average of flux recoveries was 109 %.

Conclusion and recommendations

In case of WMF, the changes in the analytical parameters could be improved by the optimisation of operating parameters (e.g. TMP and crossflow velocity) and application of permeate backflow techniques, enzymes, filtration aids, flow pulsation, gas sparging, static turbulence promoter (STP), Vibratory Shear Enhanced Process (VSEP) etc. Fluxes could be enhanced by the above-mentioned optimisations and applications.

In case of BMF with STP, the commercial breweries should focus on the optimisation of usage of STP, TMP and Q too. In this study, a turbulence promoter (STP) with a specific geometry was tested. However, in a later exercise, a wider range of operating parameters and several STPs with different geometries could be tested with the aid of lowering fouling layer resistances.

In case of BMF with SG, the commercial breweries should focus on the optimisation of TMP and Q, and silica gel (SG) free BMF can be performed. The SG free BMF is important because of environmental issues. However, filtration aids other than silica gel (SG) can be developed and tested to intensify BMF.

In case of BDA by RO, the commercial breweries should focus on the optimisation of transmembrane pressure (TMP). BDA by RO can be performed with lowest required retentate flow rate (Q), which results in lower energy consumption. The lower energy consumption is important because of environmental and economic

issues. In a later exercise, beers with different alcohol and extract content could be dealcoholized by RO.

New scientific results

1.

I have proven that hot trub (d = 30 – 80 μm) and cold trub (d = ~0.5 μm) can be completely removed from pale hopped wort (extract content = 11.16 ± 0.01 w/w %, bitterness = 49 ± 4 IBU, turbidity at 20 °C = 106.75 ± 5.50 EBC) by microfiltration with the application of Membralox T1-70 tubular ceramic membrane (Pall, USA; 0.2 μm pore size and 7 mm channel diameter) and with the following operating parameters: T = 10 ± 1 °C, transmembrane pressure (TMP) = 0.4 bar, retentate flowrate (Q) = 50 L h^{-1}.

According to the results of the analytical measurements, the changes in analytical parameters were appropriate: the bitterness decreased by approximately 5 unit, TPC decreased by approximately 5.6 %, retention of β-glucan was 40.17 % and free amino nitrogen content did not change.

The initial and steady-state fluxes, with the above-mentioned conditions, were 16.75 and 4.89 L m^{-2} h^{-1}, respectively.

2.

I have developed a model that describes rough beer ("2A. International Pale Lager" (BJCP); alcohol content = 4.58 V/V %, final real extract content = 4.48 w/w %, bitterness = 18 IBU, turbidity at 20 °C = 2.50) membrane filtration at a temperature of 10 ± 1 °C with static turbulence promoter (SPIRAL LD2 STP from Inox, Serbia) and Membralox T1-70 tubular ceramic

membrane (Pall, USA; 0.5 μm pore size and 7 mm channel diameter).

The model (objective function) (*Equation 4*) was the following:

$$\begin{aligned} R_{f\,ss} = {} & 4.4630 \times 10^{12} - 1.7662 \times 10^{12} \times x_{STP} \\ & + 1.5702 \times 10^{12} \times x_{TMP} \\ & - 1.5166 \times 10^{12} \times x_{Q} \\ & - 6.9648 \times 10^{11} \times x_{STP} \times x_{TMP} \\ & + 4.6600 \times 10^{11} \times x_{STP} \times x_{Q} \\ & - 4.3718 \times 10^{11} \times x_{TMP} \times x_{Q} \end{aligned} \qquad \text{Equation 4}$$

where $R_{f\,ss}$ (m^{-1}) is the steady-state fouling layer resistance; x_{STP} is the coded factor for static turbulence promoter (STP) with the factor values: -1, +1; x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1 and x_Q is the coded factor for retentate flow rate (Q) with the factor interval: -1 – +1. The range of validity: STP = no or yes; TMP = 0.4 – 1.2 bar, Q = 50 - 200 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(6;3) = 203.7$; $p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$).

I have determined the effect sizes of the significant parameters and they were the following: STP = -0.61, TMP = 0.54, Q = -0.52, STP:TMP = -0.24, STP:Q = 0.16 and TMP:Q = -0.15. Model accuracy and determination coefficients of the effect size estimation were significant ($F(6;4) = 271.6$; $p < 0.001$; Multiple $R^2 > 0.9$; Adjusted $R^2 > 0.9$).

I have found the optimum (global minimum) of the objective

function. The optimal values of the factors amounted to respectively STP = yes, TMP = 0.4 bar, Q = 200 L h^{-1}. The predicted $R_{f\,ss}$ under the above condition was 1.2097×10^{12} m^{-1}.

3.

I have developed a model that describes rough beer ("2A. International Pale Lager" (BJCP); alcohol content = 4.74 V/V %, final real extract content = 4.10 w/w %, bitterness = 24 IBU, turbidity at 20 °C = 18.00) membrane filtration at a temperature of 10 ± 1 °C with silica gel (Stabifix W MF from Stabifix Brauerei-Technik, Germany) and Membralox T1-70 tubular ceramic membrane (Pall, USA; 0.5 µm pore size and 7 mm channel diameter).

The model (objective function) (*Equation 5*) was the following:

$$R_{f\,ss} = 7.2678 \times 10^{12} + 3.3383 \times 10^{12} \times x_{TMP} - 2.0038 \times 10^{12} \times x_Q \qquad \text{Equation 5}$$

where $R_{f\,ss}$ (m^{-1}) is the steady-state fouling layer resistance, x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1 and x_Q is the coded factor for retentate flow rate (Q) with the factor interval: -1 – +1. The range of validity: silica gel concentration (SGC) = 0 – 80 g hL^{-1}; TMP = 0.4 – 1.2 bar, Q = 50 - 200 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(2;6) = 23.22$; $p < 0.01$; Multiple $R^2 = 0.89$; Adjusted $R^2 = 0.85$).

I have determined the effect sizes of the significant parameters and they were the following: TMP = 0.81, Q = -0.48. 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 $R^2 = 0.85$).

I have found the optimum (global minimum) of the objective function. 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 1.9257×10^{12} m^{-1}.

4.

I have developed a model that describes pale lager bright beer (Soproni Klasszikus from HEINEKEN Hungária, Hungary; alcohol content = 4.34 V/V %, final real extract content = 3.63 w/w %, bitterness = 12 IBU, turbidity at 20 °C = 0.48) dealcoholization by reverse osmosis at a temperature of 15 ± 1 °C with RO99 flat sheet polyester membrane (Alfa Laval, Sweden; $R_{NaCl} \geq 98$ %).

The model (objective function) (*Equation 6*) was the following:

$$J_{EtOH\ 0} = 80.871 + 41.094 \times x_{TMP} \qquad \text{Equation 6}$$

where $J_{EtOH\ 0}$ (g m^{-2} h^{-1}) is the initial ethanol flux and x_{TMP} is the coded factor for transmembrane pressure (TMP) with the factor interval: -1 – +1. The range of validity: TMP = 10 – 30 bar, Q = 120 - 240 L h^{-1}. Model accuracy and determination coefficients of the objective function were significant ($F(1;5) = 143.1$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$).

I have determined the effect size of the significant parameter and it was the following: TMP = 1.20. Model accuracy and

determination coefficients of the effect size estimation were significant ($F(1;6) = 171.7$; $p < 0.001$; Multiple $R^2 = 0.97$; Adjusted $R^2 = 0.96$).

I have found the optimum (global maximum) of the objective function. The optimal value of the factor amounted to TMP = 30 bar. The predicted $J_{EtOH\,0}$ under the above condition was 121.965 g m^{-2} h^{-1}.

5.

I have developed a novel and efficient (average of flux recoveries > 97 %) membrane cleaning method for "2A. International Pale Lager" (BJCP) rough beer (alcohol content = 4.58 V/V %, final real extract content = 4.48 w/w %, bitterness = 18 IBU, turbidity at 20 °C = 2.50) membrane filtration at a temperature of 10 ± 1 °C with Membralox T1-70 tubular ceramic membrane (Pall, USA; 0.5 μm pore size and 7 mm channel diameter).

The developed membrane cleaning method is detailed below. After the membrane filtration experiment, the used membrane was cleaned thoroughly by deionized water for 5 min at a temperature of 25 °C and then by 1 % (w/w) Sodium hydroxide (Reanal, Hungary) for 60 min at a temperature of 60 °C. After cleaning by alkali, the membrane was rinsed again by deionized water for 10 min at a temperature of 25 °C followed by cleaning with 1 % (w/w) Hydrogen peroxide (Hungaro Chemicals, Hungary) for 60 min at a temperature of 25 °C. Finally, the membrane was cleaned thoroughly with deionized water for 10 min at a temperature of 25 °C. In all cases transmembrane pressure (TMP)

and retentate flow rate (Q) were maintained at 0.2 bar and 50 L h^{-1}, respectively.

I have developed a novel and efficient (average of flux recoveries = 109 %) membrane cleaning method for pale lager bright beer (Soproni Klasszikus from HEINEKEN Hungária, Hungary; alcohol content = 4.34 V/V %, final real extract content = 3.63 w/w %, bitterness = 12 IBU, turbidity at 20 °C = 0.48) dealcoholization by reverse osmosis at a temperature of 15 ± 1 °C with RO99 flat sheet polyester membrane (Alfa Laval, Sweden; $R_{NaCl} \geq 98$ %).

The developed membrane cleaning method is detailed below. After the dealcoholization experiment, the used membrane was cleaned thoroughly by deionized water for 10 min at a temperature of 25 °C and then by 0.2 % (w/w) Sodium hydroxide (Reanal, Hungary) for 60 min at a temperature of 25 °C. After cleaning by alkali, the membrane was rinsed again by deionized water for 10 min at a temperature of 25 °C. In all cases transmembrane pressure (TMP) and retentate flow rate (Q) were maintained at 6 bar and 240 L h^{-1}, respectively.

The publications of the author in the field of studies

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