

Optimal Design of Novel Ceramic Hollow-Fiber Membrane Units for Pre-centrifuged Lager Clarification

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In this work, pre-centrifuged rough lager was submitted to several clarification tests using a bench-top plant equipped with 0.8- μm ceramic hollow-fiber (HF) membranes. The experimental quasi-steady state permeation flux (J_{ss}) was found to be dependent on the transmembrane pressure difference (TMP) and feed crossflow velocity (v_S) in the ranges of 0.5-4.5 bar, and 0.5-6 m s^{-1} , respectively. Once J_{ss} had been empirically modelled, it was used to design appropriately the membrane clarification unit. By referring to a medium-sized brewery, a rough-grade feasibility study allowed the operating costs (c_o) of such a unit to be minimized at $\sim 0.50 \text{ € hL}^{-1}$ by setting v_S at 4 m s^{-1} and P_F at 5 bar. By operating at $v_S=2.5 \text{ m s}^{-1}$ and $P_F=3.5$ bar with periodic CO_2 backflushing (i.e., the same conditions used to pilot 0.45- μm polyethersulphone HF modules at the Heineken brewery in Zoeterwoude, NL), c_o reduced to 0.47 € hL^{-1} . Finally, by combining an enzymatic treatment with rough beer membrane clarification and stabilization at room temperature, c_o was further lessened to 0.4 € hL^{-1} .

1. Introduction

Beer clarification is a downstream operation directed to remove yeast and haze-responsible components from rough beer. The conventional dead-end filtration with filter-aids (Kieselguhr or diatomaceous earth, DE) has been the standard industrial practice for more than 100 years despite its sanitary and environmental concerns (Fillaudeau et al., 2006). It might be replaced by microfiltration (MF) systems. Unfortunately, the average beer permeation flux through polyethersulphone (PES) hollow-fiber (HF) membrane modules is about one fifth of that (250-500 $\text{L m}^{-2} \text{ h}^{-1}$) attainable with DE filters (Buttrick, 2010). About two decades ago, a few industrial plants have started to run with polymeric membrane systems proposed by Norit Membrane Technology/Heineken Technical Service, Alfa-Laval AB/Sartorius AG, or Pall Food & Beverage/Westfalia Food Tech (Buttrick, 2010). Owing to their short lifespan of just two years, several attempts have been carried out to assess the performance of novel ceramic tubular (Burrell et al., 1994; Cimini et al., 2014; Cimini and Moresi, 2014, 2015a, 2018a; Doleček and Cakl, 1998; Fillaudeau and Lalande, 1998; Gan et al., 1997, 1999;) or HF (Cimini and Moresi, 2015b, 2016ab, 2018b) membrane modules. A novel combined process, entailing pre-centrifugation, Polyvinylpoly-pyrrolidone (PVPP) stabilization, cartridge filtration and MF of rough pale lager, was tested. Its overall operating costs and global warming potential were about one third (Cimini and Moresi, 2015a) of those associated with the DE-filtration and regenerable PVPP stabilization procedures presently used in the great majority of industrial breweries (Cimini and Moresi, 2016c, 2018c).

Aim of this work was to model the effect of the main operating variables (TMP, v_S) on the permeation flux of pre-centrifuged rough pale lager when using a 0.8- μm ceramic HF membrane module in order to design and optimize the MF unit needed for rough beer clarification in a medium-sized brewery.

2. Materials and Methods

The rough pale lager used in this work was withdrawn from a maturation tank of the Italian brewery Birra Peroni Srl (Rome, Italy) and stored at $\sim 0 \text{ °C}$. Before any test, it was centrifuged using a Beckman centrifuge mod. J2-21 at 6,000 $\times g$ for 10 min, treated with 0.5 g L^{-1} of regenerable PVPP (Cimini and Moresi, 2015a), diluted with de-ionized water to attain the commercial real extract of $3.4 \pm 0.3 \text{ °P}$. The resulting beer sample

was analyzed in compliance with the European Brewing Convention (2010) as follows: pH 4.30 ± 0.01 ; density (ρ_B) of $1008 \pm 1 \text{ kg m}^{-3}$; viscosity (η) of $1.42 \pm 0.01 \text{ mPas}$; turbidity at 20 and 0 °C of 1.16 ± 0.02 and 1.77 ± 0.08 EBC unit; color 7.7 ± 0.5 EBC unit; β -glucan $9 \pm 2 \text{ mg L}^{-1}$; real and original extract of 3.9 ± 0.04 and 13.2 ± 0.01 °P; and alcohol content of 5.00 ± 0.02 % v/v. Several total recycle clarification tests were performed at 10 °C using the bench-top MF plant previously described (Cimini and Moresi, 2014, 2016b). It was equipped with an α -Al₂O₃ HF InoCep® membrane module type MM04 (Hyflux Membrane Manufacturing, 2015) consisting of $n_{HF} (=40)$ HF's with nominal pore size of 0.8 μm , 3-mm inside diameter (d_{HF}), 200-mm overall length (L_{HF}), and 0.04-m² effective membrane surface area (a_m). TMP and v_S were varied from 0.5 to 4.5 bar, and 0.5 to 2.5 m s⁻¹, respectively. To increase v_S up to 6 m s⁻¹, 36 out of 40 HF's were sealed with a silicone adhesive (Cimini and Moresi, 2015b, 2016a). The CO₂ backflushing and membrane cleaning program was previously detailed (Cimini and Moresi, 2014, 2015a). The resulting permeate turbidity at 20 and 0 °C reduced to 0.21 ± 0.01 and 0.44 ± 0.06 EBC unit, respectively. All clarification tests were at least duplicated to assess the average coefficient of variation in the estimated permeation flux (J_v). Regression analysis of the multiple non-linear models used in this work was carried out using the built-in regression functions of the software Excel (Microsoft, Redmond, CA, USA).

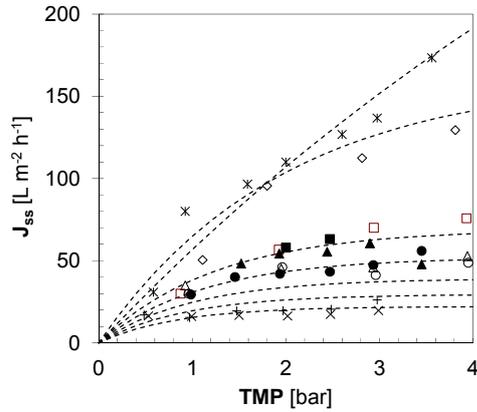


Figure 1: Effect of TMP on J_{ss} when using the 0.8- μm ceramic whole (closed, x, and + symbols) or partitioned (open and * symbols) HF membrane module at 10 °C and different v_S values: x, 0.5 m s⁻¹; +, 1.0 m s⁻¹; O, ●, 1.5 m s⁻¹; △, ▲, 2.0 m s⁻¹; □, ■, 2.5 m s⁻¹; ◇, 4.0 m s⁻¹; *, 6.0 m s⁻¹. The broken lines were calculated using Eq.s (1)-(3) together with empirical coefficients reported in the text.

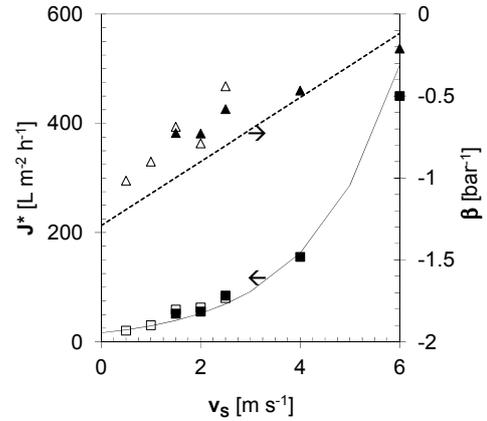


Figure 2: Effect of v_S on the limiting permeation flux (J^* : □, ■) and empiric coefficient (β : △, ▲) of Eq. (1) when using the 0.8- μm ceramic whole (open symbols) or partitioned (closed symbols) HF membrane module at 10 °C. The continuous and broken lines were calculated using Eq. (2) or (3) together with the empirical coefficients reported in the text.

3. Results and Discussion

3.1 Effect of TMP and v_S on permeation flux

Under the total recycling mode, P_F and v_S were initially set at about 1.5 bar and 0.5 m s⁻¹, respectively. Owing to the formation of a concentrated layer at the membrane surface, the overall membrane resistance tended to increase with time, this leading to the so-called *quasi-steady state permeation flux* (J_{ss}). Thereafter, v_S was in sequence increased to 1.0, 1.5, and 2.0 m s⁻¹. This procedure was reiterated by increasing P_F from ~1.5 to 3.5 bar, step 0.5 bar, while resetting v_S at 0.5 m s⁻¹. Totally, J_{ss} was found to range from 19 ± 2 to $173 \pm 7 \text{ L m}^{-2} \text{ h}^{-1}$. Fig. 1 shows J_{ss} vs. TMP at $v_S = \text{const}$ and exhibits a limiting flux (J^*), this tending to increase with v_S . Moreover, J_{ss} appeared to be an exponential function of TMP whatever the v_S value, and was empirically fitted as:

$$J_{ss} = J^* [1 - \exp(-\beta \text{ TMP})] \quad (1)$$

where β is an empiric coefficient. A series of J^* and β values was derived by fitting the experimental data via Eq. (1) in conjunction with the least squares method. As shown in Fig. 2, J^* and β appeared to be an exponential or linear function of v_S , respectively. Thus, they were expressed as follows:

$$J^* = \exp(\varphi_0 + \varphi_1 v_S) \quad (2)$$

$$\beta = \beta_0 + \beta_1 v_S \quad (3)$$

By resorting to a nonlinear regression method, it was possible to determine the following set of optimal empiric coefficient values:

$$\varphi_0 = 2.81; \varphi_1 = 0.57 \text{ m}^{-1} \text{ s}; \beta_0 = -1.29 \text{ bar}^{-1}; \beta_1 = 0.195 \text{ bar}^{-1} \text{ m}^{-1} \text{ s}.$$

These parameters allowed the mean percentage error among all the experimental and calculated J_{ssi} values to be minimized to 19 %. The broken lines shown in Fig. 1 exhibit quite a satisfactorily reconstruction of the experimental J_{ss} values. Moreover, J^* appeared to be quite insensitive to v_S up to 1.0 m s^{-1} , while J^* tended to increase almost linearly with TMP at $v_S=6 \text{ m s}^{-1}$.

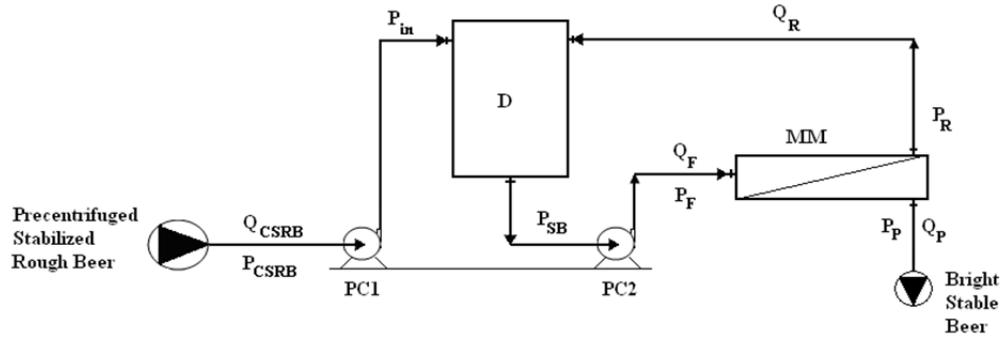


Figure 3: Schematic diagram of the MF unit equipped with ceramic HF membrane modules. Equipment identification items: D, storage tank; MM, membrane module; PC1, feed centrifugal pump; PC2, recycle centrifugal pump. All other operating variables are listed in the text.

3.2 Optimal design and operation of a beer clarification unit using ceramic HF membrane modules

The optimal operating conditions for the MF unit shown in Fig. 3 were assessed by performing a rough-grade feasibility study as referred to a medium-sized brewery working for $\Delta\tau_a=300$ days per annum in three shifts per day ($\Delta\tau_d=20 \text{ h day}^{-1}$, including the membrane cleaning-in-place procedure), its total pale lager capacity (Q_B) being about $1.1 \times 10^5 \text{ hL yr}^{-1}$. The unit throughput (Q_{CSRb}), amounting to $\sim 18.3 \text{ hL h}^{-1}$, was displaced by the centrifugal pump (PC1) to the storage tank (D), where its top pressure (P_{in}) was kept practically coincident with the output pressure (P_R) of the retentate exiting from the membrane module MM. The recycle centrifugal pump (PC2) allowed the pale lager to be recycled across MM. For each given set of v_S and P_F values, the total dynamic head of PC1 or PC2 was estimated by accounting for the theoretical (ΔP_{teo}) and effective ($\sim 3 \Delta P_{teo}$) pressure drops across MM using the Darcy equation with the Fanning friction factor (f) evaluated as suggested by Toledo (2007). Once TMP had been estimated, J_{ss} was predicted via Eq.s (1)-(3), this allowing the estimation of the permeate (Q_P), retentate (Q_R) and feed (Q_F) flow rates together with the overall membrane surface area (A_m) of MM using a 10% correction factor for safety oversize. The bare investment costs for the ceramic HF membrane insert (C_m) and stainless steel housing (C_h) were supplied by Ng (2014), as referred to the membrane modules supplied by Hyflux Membrane Manufacturing (2015), and then correlated to A_m . Those for the stainless steel centrifugal pumps, including the base plates, driving couplings and electric drivers, were derived from Corripio et al. (1982) and updated using the actual Chemical Engineering (CE) Indices for pumps and compressors and electrical equipment (Anon., 2018). Then, the auxiliary costs (e.g., instruments, piping and valves, painting and insulation, civil work, electrical, and installation) needed to install such equipment were evaluated using the total module cost factors suggested by Guthrie (1969). The overall operating costs of the MF unit comprised the investment-related (C_{io}), utility (C_{uo}), and labor (C_{Lo}) costs. C_{uo} included the electric power costs only, the specific electrical power cost (c_{ep}) coinciding with the average one ($0.1872 \text{ € kWh}^{-1}$) in Italy in 2018 (Confartigianato Imprese, 2018). C_{Lo} was evaluated by assigning one fourth of skilled worker per shift to drive the MF unit at 30 k€ per year on three (plus a replacement) shifts per day. Finally, the ancillary material costs (as due to the consumption of tap water and cleaning-in-place solutions to clean and rinse the membrane modules, and energy for CIP solution cooling, heating and pumping) were for the sake of simplicity disregarded with respect to C_{uo} . All the equations and parameters used to evaluate the investment (C_i) and specific operating (c_o) costs of such a MF unit are listed in Table 1.

Table 1: Evaluation of the operating costs of a clarification unit equipped with 0.8- μm ceramic HF membrane modules type MM5 ($a_m=5 \text{ m}^2$; $d_{HF}=3 \text{ mm}$; $L_{HF}=0.439 \text{ m}$; $n_{HF}=1,800$: Hyflux Membrane Manufacturing, 2015).

Parameter	Equation or Value
Theoretical pressure drop (Pa)	$\Delta P_{teo}=2 f \rho_R (v_s)^2 L_{HF}/d_{HF}$
Reynolds number (-)	$Re = \rho_B v_s d_{HF}/\eta$
Fanning friction factor (-)	$f=16/Re$ for $Re \leq 2,100$ $f=0.193 Re^{-0.35}$ for $3 \times 10^3 < Re < 10^4$ $f=0.048 Re^{-0.20}$ for $10^4 < Re < 10^5$
PC1 dynamic head (m)	$H_{PC1}=(P_R-P_{atm})/(\rho_B g)$
PC2 dynamic head (m)	$H_{PC2}=\frac{1}{2} v_s^2/g + (P_F-P_{atm}+3 \Delta P_{teo})/(\rho_B g)$
Pressure at the retentate port (Pa)	$P_R=P_F-1.5 \Delta P_{teo}$
Permeate flow rate (L h^{-1})	$Q_P=J_{ss} A_m$
Retentate flow rate (L h^{-1})	$Q_R=(3.6 \times 10^6) (\pi/4) (d_{HF})^2 n_{HF} v_s$
Feed flow rate (L h^{-1})	$Q_F=Q_P+Q_R$
Overall membrane surface area (m^2)	$A_m=1.1 (Q_{CSR}/J_{ss})$
Investment costs	
HF membrane insert cost (€)	$C_m = e^{6.9} (A_m)^{0.3}$ $0.04 \leq A_m \leq 5 \text{ m}^2$
Membrane insert housing cost (€)	$C_h = e^{6.3} (A_m)^{0.5}$ $0.04 \leq A_m \leq 5 \text{ m}^2$
Bare membrane unit cost (€)	$C_{MM}=C_m+C_h$
J-th brake horsepower (W)	$P_{Bj}=Q_j P_j/\eta_P$
Pump efficiency (-)	$\eta_P=0.885+0.00824 \ln(Q_j)-0.012 [\ln(Q_j)]^2$ for $0.0012 \leq Q_j \leq 0.32 \text{ m}^3 \text{ s}^{-1}$
J-th electric motor power (W)	$P_{Mj}=P_{Bj}/\eta_M$
Electric motor efficiency (-)	$\eta_M=0.5094+0.056 \ln(P_{Bj})-0.00182 [\ln(P_{Bj})]^2$ for $746 \leq P_{Mj} \leq 3.8 \times 10^5 \text{ W}$
Overall j-th pump cost (US\$)	$C_{PCj}=(I_{CEPC}/270) \beta_m \beta_T C^*_{PCj}+(I_{CEE}/175.5) C^*_{Mj}$
Material cost correction factor (-)	$\beta_m=2$
Size parameter ($\text{m}^4 \text{ s}^{-2}$)	$\psi_j=Q_j (g H_{Pj})^{1/2}$
Design-type cost factor (-)	$\beta_T=\exp\{0.7147-0.051 \ln(\psi_j)+0.0102 [\ln(\psi_j)]^2\}$
Bare j-th pump cost (US\$)	$C^*_{PCj}=\exp\{7.223+0.3451 \ln(\psi_j)+0.0519 [\ln(\psi_j)]^2\}$
Bare j-th electric motor cost (US\$)	$C^*_{Mj}=\exp\{5.129+0.1234 \ln(P_{Mj})+0.154 [\ln(P_{Mj})]^2\}$ for $0.75 \leq P_{Mj} \leq 5.6 \text{ kW}$ $C^*_{Mj}=\exp\{4.10+0.8472 \ln(P_{Mj})+0.024 [\ln(P_{Mj})]^2\}$ for $5.6 < P_{Mj} < 186 \text{ kW}$
CE pump and compressor index (-)	$I_{CEPC}=985$
CE electrical equipment index (-)	$I_{CEE}=521.9$
Overall investment cost (€)	$C_i= \zeta_{MM} (C_m+C_h)+\zeta_{PC} (C_{PC1}+C_{PC2})$
Guthrie's module cost factor for MM (-)	$\zeta_{MM}=2.53$
Guthrie's module cost factor for PC _j (-)	$\zeta_{PC}=3.38$
Overall operating costs	
Depreciation cost (€ yr^{-1})	$C_d=(C_m+C_h)/n_{MM}+(C_{PC1}+C_{PC2})/n_{PC}$
MM unit useful life (yr)	$n_{MM}=10$
PC _j useful life (yr)	$n_{PC}=5$
Maintenance cost (€ yr^{-1})	$C_{main}=0.03 C_i+C_m/n_{MM}$
Investment-related cost (€ yr^{-1})	$C_{io}=C_d+C_{main}$
Utility cost (€ yr^{-1})	$C_{Uo}=(P_{B1}+P_{B2}) \Delta \tau_a \Delta \tau_d C_{ep}$
Electrical power cost (€ kWh^{-1})	$C_{ep}=0.1872$
Labor cost (€ yr^{-1})	$C_{Lo}=30,000$
Overall operating costs (€ yr^{-1})	$C_o=C_{io}+C_{Uo}+C_{Lo}$
Specific operating costs (€ hL^{-1})	$c_o=C_o/Q_B$

Figure 4 shows the specific operating costs of the MF unit (c_o) as a function of the pressure (P_F) at the inlet port of the membrane module at different crossflow velocities (v_s).

As P_F was increased from 2 to 4 bar, c_o tended to decrease whatever the v_s value preset (1.5-6.0 m s^{-1}). Despite the highest J_{ss} value ($\sim 178 \text{ L m}^{-2} \text{ h}^{-1}$) achievable at $v_s=6 \text{ m s}^{-1}$ and $P_F=5 \text{ bar}$ enabled the MF system to be composed of just two MM5 InoCep® membrane modules (Hyflux Membrane Manufacturing, 2015), the investment-related costs for PC1 and PC2, as well as the brake power consumed to assure such flow conditions in each hollow fiber, had the greatest contribution to c_o , that was about 0.63 € hL^{-1} (Figure 4). By setting v_s at 4 m s^{-1} and P_F at 5 bar, J_{ss} reduced to about $138 \text{ L m}^{-2} \text{ h}^{-1}$. The MF system was still constituted by two MM5 InoCep® membrane modules, but the specific operating costs reached the minimum value of $\sim 0.51 \text{ € hL}^{-1}$, the 53% of which being the contribution of labor costs. Such specific operating costs were in line

with those (i.e., US\$0.28 or €0.44 hL⁻¹) reported by Gaub (2014) or Fillaudeau et al. (2006), respectively, even if no information was given about the cost items included in such estimates.

By referring to the operating conditions ($v_S=2.5$ m s⁻¹, $P_F=3.5$ bar) applied in the validation test performed previously (Cimini and Moresi, 2016a), J_{ss} lessened to 59 L m⁻² h⁻¹. This made the MF unit consisting of six MM5 InoCep® membrane modules with c_o slightly increasing to 0.54 € hL⁻¹, the 50 % of which being represented by labor costs. Owing to the ±30 % level of accuracy for this rough-grade feasibility study (Westney, 1997), the difference between the above c_o values was regarded as statistically insignificant. Thus, the operation of the 0.8- μ m ceramic HF membrane module at $v_S=2.5$ m s⁻¹ and $P_F=3.5$ bar, appeared to be quite close to the optimal one. By accounting for the positive effect of the CO₂ backflushing procedure on J_{ss} , that increased to 128±33 L m⁻² h⁻¹ (see Table 3 in Cimini and Moresi, 2016a), the number of MM5 InoCep® modules reduced to 3 and c_o was cut by ~13% to 0.47 € hL⁻¹. Such a working condition paralleled that used to run the 0.45- μ m PES HF modules at the Heineken brewery (Zoeterwoude, NL), where 10-min periods of back-flushing every 2 h during filtration kept the permeation flux almost steady at 80-100 L m⁻² h⁻¹, and beer permeate turbidity at approximately 0.6 EBC unit (Noordman et al., 2001).

Finally, a clear and stable beer ready-to-be aseptically packed was obtained by using the novel Kieselguhr- and PVPP-free beer conditioning process recently proposed by Cimini and Moresi (2018b). It combined a proline-specific proteinase pretreatment (Brewers Clarex®, DSM Food Specialities, Delft, NL) with membrane clarification using a 1.4- μ m ceramic HF membrane module under constant TMP (2.5 bar), v_S (2.5 m s⁻¹), and temperature (30 °C). In the circumstances, owing to the quite high J_{ss} (1382±83 L m⁻² h⁻¹), just two MM080 InoCep® modules (each one with $a_m=0.8$ m², $n_{HF}=300$, and $L_{HF}=0.439$ m) were needed, this lessening c_o to 0.4 € hL⁻¹.

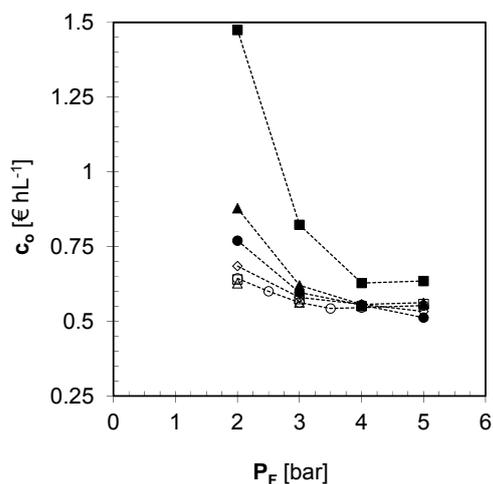


Figure 4: Specific operating costs (c_o) of the MF unit shown in Figure 3 against the pressure (P_F) at the inlet port of the membrane module under different crossflow velocities (v_S : □, 1.5 m s⁻¹; △, 2.0 m s⁻¹; ○, 2.5 m s⁻¹; ◇, 3.0 m s⁻¹; ●, 4 m s⁻¹; ▲, 5.0 m s⁻¹; ■, 6.0 m s⁻¹).

4. Conclusions

In total recycle MF trials, that were used to simulate pale lager clarification in the continuous mode, the quasi-steady state permeation flux (J_{ss}) exhibited the typical phenomenon of concentration polarization with a limiting flux (J^*) increasing with the crossflow velocity (v_S). The empirical model of J_{ss} was used to design a MF unit equipped with ceramic HF membrane modules. By referring to a medium-sized brewery, a rough-grade feasibility study allowed the operating costs (c_o) of the MF unit to be minimized at ~0.50 € hL⁻¹ by setting v_S at 4 m s⁻¹ and P_F at 5 bar. The operation at $v_S=2.5$ m s⁻¹ and $P_F=3.5$ bar with periodic CO₂ backflushing, reduced c_o to 0.47 € hL⁻¹. Use of proline-specific proteinases and 1.4- μ m ceramic HF membranes operating at room temperatures further lessened c_o to 0.4 € hL⁻¹. Thus, this novel process might be really regarded as the most effective alternative to the current beer conditioning techniques based on DE filtration and PVPP treatment.

Acknowledgments

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