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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 (Jss) was found to be dependent on the transmembrane pressure difference (TMP) and feed crossflow velocity (vS) in the ranges of 0.5-4.5 bar, and 0.5-6 m s-1, respectively. Once Jss 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 (co) of such a unit to be minimized at ~0.50 € hL-1 by setting vS at 4 m s-1 and PF at 5 bar. By operating at vS=2.5 m s-1 and PF=3.5 bar with periodic CO2 backflushing (i.e., the same conditions used to pilot 0.45-µm polyethersulphone HF modules at the Heineken brewery in Zoeterwoude, NL), co reduced to 0.47 € hL-1. Finally, by combining an enzymatic treatment with rough beer membrane clarification and stabilization at room temperature, co 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 L m-2 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, vS) 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.

* 1. 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 ~0 °C. Before any test, it was centrifuged using a Beckman centrifuge mod. J2-21 at 6,000x*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±0.3 °P. The resulting beer sample was analyzed in compliance with the European Brewing Convention (2010) as follows: pH 4.30±0.01; density (ρB) of 1008±1 kg m-3; viscosity (η) of 1.42±0.01 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±2 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 nHF(=40) HFs with nominal pore size of 0.8 µm, 3-mm inside diameter (dHF), 200-mm overall length (LHF), and 0.04-m2 effective membrane surface area (am). TMP and vS were varied from 0.5 to 4.5 bar, and 0.5 to 2.5 m s-1, respectively. To increase vS up to 6 m s-1, 36 out of 40 HFs 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 (Jv). 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 Jss when using the 0.8-µm ceramic whole (closed, , and  symbols) or partitioned (open and \* symbols) HF membrane module at 10 °C and different vS values: , 0.5 m s-1; , 1.0 m s-1; , , 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 vS on the limiting permeation flux (J\*: 🞏, ◼) and empiric coefficient (:, ) 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.

* 1. Results and Discussion
     1. Effect of TMP and vS on permeation flux

Under the total recycling mode, PF and vS 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* (Jss). Thereafter, vS was in sequence increased to 1.0, 1.5, and 2.0 m s-1. This procedure was reiterated by increasing PF from ~1.5 to 3.5 bar, step 0.5 bar, while resetting vS at 0.5 m s-1. Totally, Jss was found to range from 19±2 to 173±7 L m-2 h-1.

Fig. 1 shows Jss vs. TMP at vS=const and exhibits a limiting flux (J\*), this tending to increase with vS. Moreover, Jss appeared to be an exponential function of TMP whatever the vS value, and was empirically fitted as:

*Jss = J\* [1-exp(-β TMP)]* ( 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  appeared to be an exponential or linear function of vS, respectively. Thus, they were expressed as follows:

*J\*exp(φφvS)* ( 2)

*βββvS* ( 3)

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

φ0= 2.81; φ1 = 0.57 m-1 s; β0= -1.29 bar-1; β1 = 0.195 bar-1 m-1 s.

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

* + 1. 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 Δa=300 days per annum in three shifts per day (Δd=20 h day-1, including the membrane cleaning-in-place procedure), its total pale lager capacity (QB) being about 1.1x105 hL yr-1. The unit throughput (QCSRB), amounting to ~18.3 hL h-1, was displaced by the centrifugal pump (PC1) to the storage tank (D), where its top pressure (Pin) was kept practically coincident with the output pressure (PR) 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 vs and PF values, the total dynamic head of PC1 or PC2 was estimated by accounting for the theoretical (ΔPteo) and effective (~3 ΔPteo) 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, Jss was predicted via Eq.s (1)-(3), this allowing the estimation of the permeate (QP), retentate (QR) and feed (QF) flow rates together with the overall membrane surface area (Am) of MM using a 10% correction factor for safety overdesign. The bare investment costs for the ceramic HF membrane insert (Cm) and stainless steel housing (Ch) were supplied by Ng (2014), as referred to the membrane modules supplied by Hyflux Membrane Manufacturing (2015), and then correlated to Am. 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 (CIo), utility (CUo), and labor (CLo) costs. CUo included the electric power costs only, the specific electrical power cost (cep) coinciding with the average one (0.1872 € kWh-1) in Italy in 2018 (Confartigianato Imprese, 2018). CLo 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 CUo. All the equations and parameters used to evaluate the investment (CI) and specific operating (co) 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 (am=5 m2; dHF=3 mm; LHF=0.439 m; nHF=1,800: Hyflux Membrane Manufacturing, 2015).

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| **Parameter** | **Equation or Value** |
| Theoretical pressure drop (Pa) | ΔPteo=2 f R (vS)2 LHF/dHF |
| Reynolds number (-) | Re = ρB vS dHF/η |
| Fanning friction factor (-) | f=16/Re for Re2,100 |
|  | f=0.193 Re−0.35 for 3x103<Re<104 |
|  | f=0.048 Re-0.20 for 104<Re<106 |
| PC1 dynamic head (m) | HPC1=(PR-Patm)/(ρB g) |
| PC2 dynamic head (m) | HPC2=½ vs2/g + (PF-Patm+3 ΔPteo)/(ρB g) |
| Pressure at the retentate port (Pa) | PR=PF-1.5 ΔPteo |
| Permeate flow rate (L h-1) | QP=Jss Am |
| Retentate flow rate (L h-1) | QR=(3.6x106) (π (dHF)2 nHF vS |
| Feed flow rate (L h-1) | QF=QP+QR |
| Overall membrane surface area (m2) | Am= 1.1 (QCSRB/Jss) |
| *Investment costs* | |
| HF membrane insert cost (€) | Cm = e6.9 (Am)0.3 0.04≤Am≤5 m2 |
| Membrane insert housing cost (€) | Ch = e6.3 (Am)0.5 0.04≤Am≤5 m2 |
| Bare membrane unit cost (€) | CMM=Cm+Ch |
| J-th brake horsepower (W) | PBj= Qj Pj/ηP |
| Pump efficiency (-) | ηP=0.885+0.00824 ln (Qj)-0.012 [ln(Qj)]2 for0.0012≤Qj≤0.32 m3 s-1 |
| J-th electric motor power (W) | PMj= PBj/ηM |
| Electric motor efficiency (-) | ηM=0.5094+0.056 ln(PBj)-0.00182 [ln(PBj)]2 for746≤PMj≤3.8x105 W |
| Overall j-th pump cost (US$) | CPCj=(ICEPC/270) βm βT C\*PCj+(ICEE/175.5) C\*Mj |
| Material cost correction factor (-) | βm=2 |
| Size parameter (m4 s-2) | ψj=Qj (g HPj)½ |
| Design-type cost factor (-) | βT=exp{0.7147-0.051 ln(ψj)+0.0102 [ln(ψj)]2} |
| Bare j-th pump cost (US$) | C\*PCj=exp{7.223+0.3451 ln(ψj)+0.0519 [ln(ψj)]2} |
| Bare j-th electric motor cost (US$) | C\*Mj=exp{5.129+0.1234 ln(PMj)+0.154 [ln(PMj)]2} for 0.75≤PMj≤5.6 kW |
|  | C\*Mj=exp{4.10+0.8472 ln(PMj)+0.024 [ln(PMj)]2} for 5.6<PMj<186 kW |
| CE pump and compressor index (-) | ICEPC=985 |
| CE electrical equipment index (-) | ICEE=521.9 |
| Overall investment cost (€) | CI= ςMM (Cm+Ch)+ςPC (CPC1+CPC2) |
| Guthrie’s module cost factor for MM (-) | ςMM=2.53 |
| Guthrie’s module cost factor for PCj (-) | ςPC=3.38 |
| *Overall operating costs* | |
| Depreciation cost (€ yr-1) | Cd=(Cm+Ch)/nMM+(CPC1+CPC2)/nPC |
| MM unit useful life (yr) | nMM=10 |
| PCj useful life (yr) | nPC=5 |
| Maintenance cost (€ yr-1) | Cmain=0.03 CI+Cm/nMM |
| Investment-related cost (€ yr-1) | CIo=Cd+Cmain |
| Utility cost (€ yr-1) | CUo=(PB1+PB2) Δa Δd cep |
| Electrical power cost (€ kWh-1) | cep=0.1872 |
| Labor cost (€ yr-1) | CLo=30,000 |
| Overall operating costs (€ yr-1) | Co=CIo+CUo+CLo |
| Specific operating costs (€ hL-1) | co=Co/QB |

Fig. 4 shows the specific operating costs of the MF unit (co) as a function of the pressure (PF) at the inlet port of the membrane module at different crossflow velocities (vs).

As PF was increased from 2 to 4 bar, co tended to decrease whatever the vs value preset (1.5-6.0 m s-1).

Despite the highest Jss value (~178 L m-2 h-1) achievable at vs=6 m s-1 and PF=5 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 co, that was about 0.63 € hL-1 (Fig. 4).

By setting vS at 4 m s-1 and PF at 5 bar, Jss reduced to about 138 L m-2 h-1. The MF system was still constituted by two MM5 InoCep® membrane modules, but the specific operating costs reached the minimum value of ~0.51 € 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 (vS=2.5 m s-1, PF=3.5 bar) applied in the validation test performed previously (Cimini and Moresi, 2016a), Jss lessened to 59 L m-2 h-1. This made the MF unit consisting of six MM5 InoCep® membrane modules with co 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 co values was regarded as statistically insignificant. Thus, the operation of the 0.8-µm ceramic HF membrane module at vS=2.5 m s-1 and PF=3.5 bar, appeared to be quite close to the optimal one. By accounting for the positive effect of the CO2 backflushing procedure on Jss, 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 co was cut by ~13% to 0.47 € hL-1. Such a working condition paralleled that used to run the 0.45-µm 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-µm ceramic HF membrane module under constant TMP (2.5 bar), vS (2.5 m s-1), and temperature (30 °C). In the circumstances, owing to the quite high Jss (1382±83 L m-2 h-1), just two MM080 InoCep® modules (each one with am=0.8 m2, nHF=300, and LHF=0.439 m) were needed, this lessening co to 0.4 € hL-1.



*Figure 4: Specific operating costs (co) of the MF unit shown in Fig. 3 against the pressure (PF) at the inlet port of the membrane module under different crossflow velocities (vs:* 🞏*, 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).*

* 1. Conclusions

In total recycle MF trials, that were used to simulate pale lager clarification in the continuous mode, the quasi-steady state permeation flux (Jss) exhibited the typical phenomenon of concentration polarization with a limiting flux (J\*) increasing with the crossflow velocity (vS). The empirical model of Jss 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 (co) of the MF unit to be minimized at ~0.50 € hL-1 by setting vS at 4 m s-1 and PF at 5 bar. The operation at vS=2.5 m s-1 and PF=3.5 bar with periodic CO2 backflushing, reduced co to 0.47 € hL-1. Use of proline-specific proteinases and 1.4-µm ceramic HF membranes operating at room temperatures further lessened co 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.

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