



Assessment of the optimal operating conditions for pale lager clarification using novel ceramic hollow-fiber membranes



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ABSTRACT

In this work, the lager beer clarification and stabilization process previously developed was further tested by replacing the 0.8- μm ceramic single-tube membrane module with a novel ceramic hollow-fiber membrane module having the same pore size to offset the known ineffectiveness of back-flushing cleaning techniques in ceramic multi-channel monolithic modules.

In total recycle crossflow microfiltration (CFMF) trials, the quasi-steady state permeation flux (J_{ss}) tended to a limiting flux (J^*), that was found to increase with the crossflow velocity (v_s) in the range of 0.5–6.0 m s^{-1} . The ideal hydraulic pump energy consumption per unit liter of permeate recovered was practically independent of the aforementioned operating variables and of the order of $(66.5 \pm 0.5) \text{ W h L}^{-1}$. Nevertheless, to obtain a quasi-steady state permeation flux greater than the target permeation flux (i.e., 100 $\text{L m}^{-2} \text{ h}^{-1}$) for rough beer clarification via membrane processing in the absence of CO_2 backpulsing, TMP had to be greater than 2 bar and v_s to vary from 4 to 6 m s^{-1} . Not only was the performance of the ceramic hollow-fiber membrane module at 10 $^\circ\text{C}$, $v_s = 2.5 \text{ m s}^{-1}$, and $\text{TMP} = 2.4$ bar with 2-min periods of CO_2 back-flushing applied every 50–60 min superior to that of the polymeric hollow-fiber membrane process patented by Heineken and Norit Membrane Technology, but also the use of ceramic hollow-fiber membrane systems would extend the membrane life span up to ten years, thus reducing the contribution of the annual membrane replacement to the overall operating costs of beer clarification.

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1. Introduction

Despite its ancient tradition, the brewing industry has to perform within the concurrent constraints of product quality, process safety, economic viability, and limited environmental damage. In particular, the Beverage Industry Environmental Roundtable has scrutinized a series of strategies to minimize the environmental impact of brewing by selecting properly the packaging format and materials, distribution logistics, recycling rates, etc. in either Europe or North America (BIER, 2012). Also the environmental and safety concerns associated with filter-aid handling and spent filter sludge disposal makes the beer industry potentially eager to substitute the conventional diatomaceous earth (DE) filters with crossflow microfiltration (CFMF) systems.

Since the year 2000, rough beer clarification may be carried out by resorting to three different membrane systems, namely those

proposed by Norit Membrane Technology/Heineken Technical Service (Buttrick, 2007, 2010; Noordman et al., 2001), Alfa-Laval AB/Sartorius AG (Borremans and Modrok, 2003; Buttrick, 2007), and Pall Food & Beverage/Westfalia Food Tech (Denniger and Gaub, 2004; Gaub, 2014; Höflinger and Graf, 2006; Buttrick, 2007, 2010). Whereas the Norit/Heineken or Pall Food & Beverage CFMF units consist of polyethersulfone (PES) hollow-fiber modules with pore size of 0.50 or 0.65 μm , respectively, the Alfa-Laval/Sartorius CFMF units are made of PES flat-sheet cassettes with pore size of 0.60 μm (Buttrick, 2007). It is still arguable whether the pre-centrifugation stage is effectively expedient to minimize membrane fouling by yeast cells and larger aggregates as recommended by the Pall and Alfa Laval processes (Buttrick, 2007, 2010). Regrettably, the average beer permeation flux through such membrane modules is about one fifth of that (250–500 $\text{L m}^{-2} \text{ h}^{-1}$) attained with DE filters (Buttrick, 2007; Fillaudeau et al., 2006).

Recently, a novel combined process consisting of sequential pre-centrifugation, PVPP stabilization, cartridge filtration and CFMF of pale lager has been regarded as technically and economically

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feasible, its overall operating costs and global warming potential being as low as one third of those associated with the DE-filtration and regenerable PVPP stabilization procedures presently used in the great majority of industrial breweries (Cimini and Moresi, 2015a). In particular, the efficacy of such a combined clarification process mainly derived from the use of a ceramic single-tube membrane module with nominal pore size of 0.8 μm , fed with pre-centrifuged and PVPP-stabilized rough pale lager at a crossflow velocity (v_s) of 6 m s^{-1} under a transmembrane pressure difference (TMP) of 3–4 bar, temperature (T) of 10 $^\circ\text{C}$, and periodic CO_2 back-flushing. Such a process was tested on three different types of beer, that was produced in a laboratory- (Cimini and Moresi, 2014), a pilot- (Cimini et al., 2014), or an industrial- (Cimini and Moresi, 2015a) scale plant, respectively. In the circumstances, it was possible to achieve an average permeation flux in the range of 250–385 $\text{L m}^{-2} \text{h}^{-1}$, this falling within the operating limits of conventional DE-filters (Buttrick, 2007), as well as to reduce the permeate chill haze to less than 0.5 EBC unit, as recommended by the European Brewery Convention (2010), while keeping the main characteristics (i.e., pH, color, total phenolics, real extract, and alcohol content) of the commercial beer.

The scaling-up of the aforementioned operating conditions and CO_2 back-flushing program from a ceramic 6-mm single-tube membrane to any commercial multi-channel monolithic module would be a priori hindered, because it is practically impossible to guarantee the same CO_2 back-flushing flow rate across all the channels of the monolith, as shown by Doleček and Cakl (1998). Thus, the inner the channel of the monolith the lower the CO_2 flow rate is, this resulting in permeation fluxes most likely by far lower than those achieved previously in a ceramic single-tube membrane.

To scale up the aforementioned process, it would be more advantageous to rely on hollow-fiber membrane modules, such as those used by the Norit and Pall processes (Buttrick, 2007), for several reasons: i) the high packing density, ii) the relatively low-power consumption, and iii) the capacity to withstand back-flushing procedures. Unfortunately, their membrane life span is as short as two years (Buttrick, 2010). In order to extend the membrane life span up to ten years, Hyflux Membrane Manufacturing (2010) has started manufacturing novel ceramic hollow-fiber membrane modules consisting of 40–1800 tubular elements with inside and outside diameters of 3 and 4 mm, and length of 200 or 439 mm, all of them being aligned with the ends of the fiber bundle and sealed in resin to separate permeate and retentate streams. According to Smith (2013), the difference between hollow-fiber and tubular designs is the diameter of the membrane element. Moreover, hollow fibers generally have inside diameters of 0.04–3 mm compared with tubular elements with inside diameters of 5–25 mm.

The first aim of this work was to assess the effect of the main operating variables (TMP, v_s) on the permeation flux of pre-centrifuged, PVPP-stabilized, and cartridge-filtered rough pale lager when using a ceramic hollow-fiber membrane module having the same nominal pore size (0.8 μm) of the ceramic tubular one previously used (Cimini and Moresi, 2015a). The second one was to carry out a few batch tests to validate the CFMF performance of the operating variables selected in combination with a CO_2 back-flushing program and thus minimize the ideal hydraulic pump energy consumption per unit volume of beer permeate recovered.

2. Materials and methods

2.1. Raw materials

The rough pale lager used here was made of malted barley, maize grits and hop pellets, and was produced from the Italian

brewery Birra Peroni Srl (Rome, Italy). It was withdrawn from the maturation tank and stored at 0.0 ± 0.5 $^\circ\text{C}$. Before CFMF testing, the rough lager samples were clarified using a laboratory centrifuge (Beckman mod. J2-21) at $6000 \times g$ and about 4 $^\circ\text{C}$ for 10 min, and then diluted with de-ionized water as recommended by the brewmaster to approach the commercial real extract (i.e., 3.4 ± 0.2 $^\circ\text{P}$).

2.2. Equipment

Beer clarification was carried out using the bench-top CFMF plant, previously described (Cimini and Moresi, 2014), its process flow sheet being shown in Fig. 1. It was equipped with an $\alpha\text{-Al}_2\text{O}_3$ hollow-fiber InoCep[®] membrane module type MM04 (Hyflux Ltd, Singapore; <http://www.hyfluxmembranes.com/inocep-ceramic-hollow-fibre-membrane.html>). Such a module was composed of 40 hollow-fibers having nominal pore size of 0.8 μm (type M800). Each hollow fiber had inside (d_{HF}) and outside diameters of 3 and 4 mm, and an overall length (L_{HF}) of 200 mm. As claimed by the manufacturer, the effective membrane surface area (A_m) of the module amounted to 0.04 m^2 , while the nominal water permeability (L_w) was about 2500 $\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$ at 25 $^\circ\text{C}$. Fig. 2a shows the front view of the membrane module used.

Digital pressure transducers (Inmsystem, Cagliari, Italy) and Bourdon manometers (OMET di Ceresa Srl, Pessano con Bornago, Milan, Italy) with a maximum pressure of 6 bar were attached at the feed inlet, and retentate and permeate outlets of the membrane module (MM). The process temperature was monitored by a digital temperature indicator (TI) and controlled by a thermostat (type LTD6, Grant Instrument Ltd., Cambridge, UK), this regulating automatically the flow rate of the cooling fluid (cf), consisting of a mixture of water and ethylene glycol, through a stainless-steel plate heat exchanger (E1) having an overall heat transfer surface area of 0.36 m^2 . The flowmeter FI01 (type E5-2800/H, ASA Srl, Sesto San Giovanni, Italy) was used to measure the feed volumetric flow rate (Q_F) in the range of (0.1–2.6) $\text{m}^3 \text{h}^{-1}$, while the rotameter FI02 (type E5-2600/H, ASA Srl, Sesto San Giovanni, Italy) allowed the permeate flow rate to be determined in the range (2–40) L h^{-1} . The retentate flow rate was measured by the digital flowmeter transducer FI03 in the range of (0.1–2.6) $\text{m}^3 \text{h}^{-1}$. The permeate flow rate was also assessed by using two technical-grade scales depending on the CFMF test performed. In particular, K1 or K2 was the type PCE-TS 150 (PCE Italia Srl, Gagnano, Lucca, Italy) or Europe 4000 AR (Gibertini, Elettronica Srl, Novate, Milan, I), its accuracy and maximum capacity being \pm (20.0 or 0.01) g and (150 or 4) kg, respectively. Both scales were interfaced to a personal computer (PC) via RS-232 serial ports.

When the 25-L AISI 304 storage tank D1 had been charged with about 5 L of rough beer, the centrifugal pump G1 (type HMS, maximum volumetric flow rate of 4.2 $\text{m}^3 \text{h}^{-1}$, head of 40 m of water and power of 0.45 kW; Lowara, Montecchio Maggiore, Italy) was switched on. To assure simultaneous setting of Q_F and TMP, the manual ball valve (V7) was regulated while varying the frequency of the electric voltage applied to the asynchronous motor piloting G1 by means of a frequency inverter type Commander SK 0.75 k (Control Techniques, Powys, UK). All the other stainless steel ball valves shown in Fig. 1 allowed the feed to be charged (V8); the retentate to be discharged (V2); the permeate to be recycled back into D1 (V10), accumulated into D2 (V11 and V12), discharged (V9) or sampled via V13, as well as a series of other ancillary operations (such as valve, membrane module, or pump replacement) to be performed.

A 4-kg liquid CO_2 bottle (CB) at an average pressure of 200 bar was used to ensure an inert atmosphere in both tanks D1 and D2, as well as in the permeate circuit, and minimize O_2 pick-up. By means

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