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Rough Beer Filterability in Industrial Powder-Filters and Lab-Scale Dead-End and Crossflow Filtration Apparatuses

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Rough beer clarification in large-sized breweries is mainly carried out using powder filters. In this work, it was attempted to explain why two rough beer samples yielded quite different filtrate volumes (~965 vs. 2,068 hL) from industrial-scale powder filters coated with equal doses of filter-aids of the same nominal permeability, but of different lots. By resorting to a bench-top dead-end filtration apparatus, the above 50% reduction in beer filterability was attributed to the lots used, the permeability of the corresponding filter cakes slightly varying from ~0.19 to 0.25 Darcy. When using a bench-top plant equipped with a 0.8-μm ceramic tubular membrane operating under constant values of the transmembrane pressure difference (3.73 bar), feed superficial velocity (6 $m.s^{-1}$), and temperature (1.5 °C), the volumes of permeate recovered, as well as the limiting and average permeation fluxes, were not statistically different. Thus, membrane clarification of rough beer may overcome the intrinsic variation in the particle size distribution of commercially available filter-aids of the same nominal permeability.

1. Introduction

Beer is the alcoholic beverage most widely consumed in the world, totaling ~1.9 billion hectoliters in 2019 (Statista, 2021). Generally, the consumer asks for a brilliant product with persistent qualitative traits till its consumption. The turbidity of beer may derive from biological factors (i.e., growth of bacteria or wild yeasts) and chemical interactions among the polypeptides and polyphenols constitutive of barley and hop (Siebert et al., 1996). The biological stability is currently achieved with the aid of pasteurization and microfiltration, while there is no other way to assure beer brilliance except through the dead-end filtration with filter-aids (such as Kieselguhr or diatomaceous earth, DE) or the crossflow microfiltration process (CMFT). The former is still used by the preponderance of industrial-sized breweries thanks to its high filtration rates and low operating costs notwithstanding its classification as *hazardous waste* before and after filtration by the World Health Organization as the crystalline silica is a cause of silicosis (Fillaudeau et al., 2006), and its responsibility for arsenic contamination of beer (ACS, 2013). This process may also give rise to unexpected reductions in its performance. On the other hand, the CMFT process using 0.45-μm polyethersulfone hollow-fiber modules (Noordman et al., 2001, Buttrick, 2010) asks for no filter aids and no thermal pasteurization of beer (Cimini and Moresi, 2020). The primary aim of this work was to ascertain why two rough lager beers produced in a large-sized brewery using malts of slightly different origin and the standard brewing process (Eßlinger, 2009) yielded quite differently filtrate volumes (i.e., ~965 vs. 2068 hL) from industrial candle filters coated with equal amounts of diatomaceous earth of different lots, while the secondary one was to compare the filtration performances of such rough beers in laboratory-scale Kieselguhr and 0.8-μm ceramic tubular membrane filters.

2. Materials and methods

Two rough beer (RB) samples BP1 and BP2 were produced in a large-sized brewery (Birra Peroni Srl, Rome, Italy) using two malts no. 53 and 56 (Malteria Saplo Spa, Pomezia, Italy) with practically the same physico-chemical characteristics and negligible differences in color (6.0 vs. 6.3 EBC unit, EBC-U), and raw protein content (0.098 vs. 0.096 g g^{-1}). Such RB samples were collected from two different maturation tanks and kept

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in stainless steel barrels at (0.0±0.5) °C till their use. Before being filtered, each RB sample was diluted with deionized water in a volumetric ratio of 1:0.35 $L.L^{-1}$ to obtain the commercial Plato gravity (~13 °Plato). Rough beer clarification was carried out in precoat candle filters, each one with a nominal filtering capacity of 500-600 $hL.h^{-1}$ and area of ~140 m^2. Their filter precoat was formed using a perlite-rich powder (Fibroxcel® 10, FC), while their filter coat by a mixture of two different DE powders labelled DIA1 and DIA2 in a weight ratio of 1:9 $g.g^{-1}$, respectively. Such powders were composed of SiO_2 (86-89% w/w), Al_2O_3 (5.0-7.5% w/w), and Fe_2O_3 (1.0-2.5% w/w) and characterized by similar values of electric conductivity (0.2 $mS.cm^{-1}$), bulk density (140-170 $kg.m^{-3}$), and pH but differed for their nominal hydraulic permeability (k) range (0.09-0.16 vs. 0.16-0.3 Darcy) and percentage of residues left over a 325-mesh screen (8% vs. 6% w/w). Such filter-aids of two different lots were provided by Deref Spa (Castiglione in Teverina, Viterbo, Italy), and labelled A and B. All filter precoating (i.e., FC-A, FC-B) and coating (i.e., DE-A, DE-B) materials were tested in the lab-scale dead-end filtration apparatus shown in Fig, 1. It consisted of a 3-L stainless steel chamber (S1) with an upper screw-capped opening (A1), a bottom opening provided with a manually operated ball valve (V3), and an inlet port equipped with a pressure regulating valve (V1) to inject compressed air, a discharge valve (V2) and a pressure gauge (M1) to monitor the pressure inside the chamber. In the inferior chamber (CF) a metal filter septum (FF) with large meshes, topped by a fine mesh net, was housed. The filtrate outflow was controlled by another manually operated ball valve (V4) and collected in a glass beaker (S2). This was placed on a technical scale (K2) mod. Europe 4000 AR (Gilbertini Elettronica Srl, Novate, MI, Italy) (full scale: 4 kg; sensitivity: 0.1 g), interfaced to the computer (PC) via RS-232 serial port to monitor the time (t) course of the filtrate discharged.

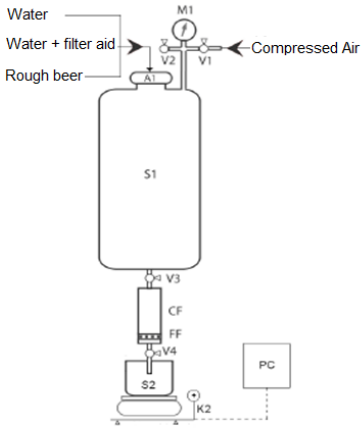

Figure 1: Block diagram of the bench-top dead-end DE filtration apparatus used in this work. All item symbols are reported in the text.

The septum permeability was controlled by depositing a first layer of FC (pre-coat), and a secondary one of DE (coat). A slurry consisting of 1.5 g of FC-A or FC-B thoroughly dispersed in 1 L of deionized water at (20±0.5) °C was firstly poured into S1. Once V2 and A1 had been closed, V1 was opened to inject compressed air up to set an internal pressure (P_{in}) of 2 bar. The liquid exiting the filter through V4 was collected in a beaker and recycled back into S1 until its turbidity was about 0.02 Nephelometric Turbidity Unit (NTU), as measured using a turbidity meter model HD 25.2 (Delta OHM Srl, Caselle di Selvazzano, PD, Italy). As S1 was empty, V4 was closed to allow S1 to be filled with deionized water at (20±0.5) °C and then pressurized at 2 bar. Once the valve V4 had been opened, the filtrate started to be collected into S2, its mass being monitored versus time at time intervals of 0.5 s. Such filtration tests were repeated at least three times and allowed the permeability of the precoat to be assessed. Such procedure was also used to form a DE coat by dispersing 1.9 $g.L^{-1}$ of DE-A or DE-B in 1 L of deionized water to measure the permeability of the corresponding filter cake. Once both layers had been formed, the chamber S1 was filled with 1 L of deionized water or rough beer sample (BP1) at (20±0.5) °C to perform a series of filtration tests. At the end of each filtration test, the filter cake was removed from the chamber CF to measure its diameter (28 mm) and the thickness of the precoat (10±1 mm), coat (7.0±1.5 mm), and overall filter cake (17.0±1.5 mm). The filtration tests were described by means of the Darcy's law (Ripperger et al., 2012), which relates the filtrate mass flow rate (Q_m) to the operating characteristics of the filter:

$$Q_m = \frac{dM}{dt} = k \frac{TMP}{\nu \delta} S \quad (1)$$

where dM/dt is the first derivative of the filtrate mass (M) with respect to time (t), S the filter surface, TMP the pressure difference across the filter medium, ν the kinematic viscosity of the filtrate, δ the thickness of the filter cake, and k the permeability of the filter cake. The latter is expressed in m^2 in the International System of Units or conventionally in Darcy. Such a unit denotes the permeability of a filter medium yielding 1 $cm^3.s^{-1}$ of a liquid with a viscosity of 1 cp upon the application of a pressure gradient of 1 $atm.cm^{-1}$ on 1-cm^2 filtering surface, this being equivalent to ~1 $(\mu m)^2$. By plotting M-vs.-t data, an almost linear relationship was generally observed, this allowing the average mass filtration rate (dM/dt) and cake permeability (k) to be estimated via the least squares method and Eq. (1), respectively.

The bench-top CFMF plant previously described (Cimini and Moresi, 2014) was used. It was equipped with a 0.8-μm ceramic tubular module in ZrO_2 and TiO_2 (Beijing Lianfaxingushun Wire Mesh Trading Co, Fengtai, China), this having an internal diameter of 6 mm, a length of 500 mm, and an effective surface area (A_m) of 94.2 cm^2. A Beckman J2-21 floor model centrifuge operating at 6000×g and ~4 °C for 10 min was used to remove suspended solids and thus standardize the initial permanent turbidity of each sample. After having charged the beer tank with circa 5 L of the above pre-treated samples, a few CFMF trials were performed in the recirculation mode under the following conditions: feed superficial velocity (v_S) of 6 $m.s^{-1}$, temperature of 10 or 1.5 °C, pressure at the permeate exit port of ~1 bar, and transmembrane pressure difference of 3.75 bar (Cimini and Moresi, 2015). The instantaneous permeation flux (J_V) was calculated as:

$$J_V(t) = \frac{Q_P(t)}{A_m} \quad (2)$$

where Q_P is the instantaneous permeate volumetric flow rate. The average permeation flux (J_{Va}) was calculated by means of the Simpson's rule of integration between the starting and end (t_f) times of each CFMF trial with a time increment of 1 min as:

$$J_{Va} = \frac{\int_0^{t_f} J_V(t)\,dt}{t_f} \quad (3)$$

As J_V declined up to reach nearly a constant value (its coefficient of variation being smaller than 5%) for 15–20 min, such a value was recorded as the limiting permeation flux J* (Cheryan, 1998).

To assess the dead-end filterability of both RB samples, the bench-top apparatus shown in Figure 1 was used. All beer samples were characterized via the analytical methods of the European Brewery Convention (EBC, 2010). Their density, viscosity, and ethanol volumetric fraction were respectively determined with the DMA™ 4500 M density meter, rotating ball viscometer Lovis 2000 M/ME, and Alcolyzer Wine M/ME, al being provided by Anton Paar Italia Srl (Rivoli, Italy). The beer turbidity at 20 °C or 1.5 °C, color, and ß-glucan content were determined in accordance with the EBC methods no. 9.29, 9.6, and 9.31.1, respectively (EBC, 2010).

All trials were repeated three times to estimate the mean value and standard deviation of each variable monitored. The Tukey test was applied for the statistical comparison of means at the probability level (p) of 0.05.

Table 1: Main physico-chemical characteristics of pre-centrifuged RB samples BP1 and BP2, as such (TQ) and permeated (P) across a 0.8-μm ceramic tubular membrane under the operating conditions given in the text.

Rough beer sample	**BP1**		**BP2**		**Unit**
Parameter	**TQ**	**P**	**TQ**	**P**	
pH	4.33±0.0	4.33±0.0	4.30±0.0	4.30±0.0	-
Color	7.4±0.1	7.2±0.1	7.7±0.1	7.3±0.1	EBC-U
Real extract	3.96±0.01	3.64±0.01	3.89±0.04	3.47±0.04	°Plato
Apparent extract	13.4±0.1	13.4±0.1	13.1±0.0	13.1±0.0	°Plato
Polyphenols	150.1±1.2	148.0±0.6	152.9±5.2	148.0±1.7	$mg.L^{-1}$
ß-glucans	9.1±1.6	9.1±1.6	8.8±0.0	8.8±1.6	$mg.L^{-1}$
Density	1005.0±0.1	1005.0±0.2	1006.0±0.2	1005.0±0.2	$kg.m^{-3}$
Viscosity @ 1.5 °C	2.642±0.003	2.636±0.003	2.647±0.069	2.669±0.002	mPa.s
Viscosity @ 20.0 °C	1.421±0.003	1.405±0.001	1.435±0.002	1.416±0.002	mPa.s
Alcohol	5.00±0.05	5.00±0.05	5.00±0.01	5.00±0.01	% (v/v)
Turbidity @ 20.0 °C	1.29±0.01	0.13±0.06	1.57±0.06	0.22±0.06	EBC-U

3. Results and Discussion

3.1 Physico-chemical characteristics of rough beer

The main physico-chemical properties of the pre-centrifuged and diluted rough beer samples BP1 and BP2 are reported in Table 1. Although such RB samples had been produced from slightly different malts, they exhibited almost the same characteristics and thus should similarly perform during membrane clarification.

3.2 Performance of industrial candle filters

Such rough beers were clarified in industrial-scale powder filters. Their precoat was made of FC, while their coat of DE powders DIA1 and DIA2 in a weight ratio of 1:9 $g.g^{-1}$. During the filtration process the operating pressure in the filter chamber (P_{in}) was gradually increased up to 8 bar to keep the filtrate flow rate (Q_F) approximately equal to 500 $hL.h^{-1}$, as shown in Figure 2. Despite both filter cakes had been prepared under constant consumption of DE, water, and electricity, the filtration of BP1 was completed in about 120 min, having the pressure in the filter chamber reached its maximum value of 8 bar, and yielded an overall filtrate volume of ~965 hL (Figure 2). When feeding BP2, the maximum P_{in} value was reached after ~270 min, and the filtrate volume recovered was about the double (2068 hL) of that recovered with BP1 (Figure 2).

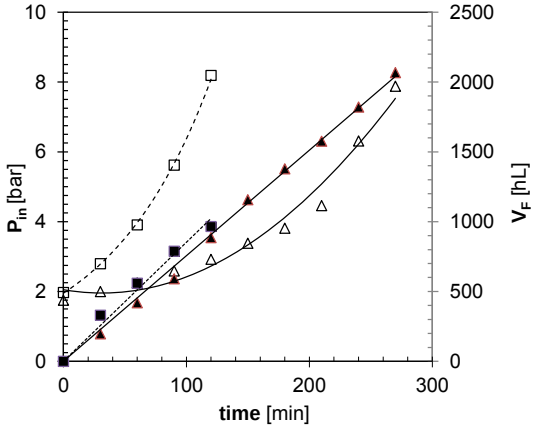

Figure 2: Time course of the operating pressure (P_{in}: open symbols) and volume of filtrate collected (V_F: closed symbols) from an industrial DE filter during the clarification of rough beer samples BP1 (□,■) and BP2 (△,▲).

3.3 Permeability of different filter cakes

The filter septum of the apparatus shown in Figure 1 was covered with a precoat made of FC and a coat based on DIA2 or a mixture of DIA1 and DIA2 in the same weight ratio (1:9 $g.g^{-1}$) used in the industrial brewery. As the mass of the filtrate was plotted against time and correlated via the least squares method, the estimated weight filtration rate (dM/dt) was used to calculate the permeability k of each filter cake using Eq. (1).

Table 2: Average values and standard deviations of the permeability (k) of different filter cakes made of FC and DE of lot A or B to water or rough beer BP1 at 20 °C, as related to triplicated dead-end filtration tests.

Feed	**Filter-aid**	**k** [Darcy]
H_2O	FC-A	1.51 ± 0.04 [a]
H_2O	FC-B	1.52 ± 0.21 [a]
H_2O	FC-A + DIA2-A	0.42 ± 0.02 [b]
H_2O	FC-B + DIA2-B	0.38 ± 0.01 [b]
H_2O	FC-A + DIA1/DIA2-A	0.25 ± 0.02 [c]
H_2O	FC-B + DIA1/DIA2-B	0.19 ± 0.01 [d]
BP1	FC-A + DIA1/DIA2-A	0.11 ± 0.01 [e]
BP1	FC-B + DIA1/DIA2-B	0.08 ± 0.01 [f]

Different lowercase letters indicate statistically significant difference between the k values for filter aids of different lots at the probability level of 0.05.

As shown in Table 2, the k values for the precoat made of Fibroxcel® or overall filter cake made of FC and DIA2 of each lot A or B were approximately equal to 1.51 or 0.40 Darcy, and independent of the lot used at a confidence level of 95%. The permeability of the coating made of FC and above mixture of DIA1 and DIA2 varied from 0.25 Darcy to 0.19 Darcy (Table 2). Even if such hydraulic permeabilities fell within the nominal

ones of the DE powders mentioned above, such a variability seemed to be responsible for the 50% variation in the filtrate recovery shown in Figure 2. The filterability of such filter coatings was further tested using the rough beer (BP1) which had given the worst filtrate recovery yield (Figure 2). The M-vs.-t data plotted in Figure 3 were typical of the so-called blocking filtration (Ripperger et al., 2012). As the open pores within the filter medium were blocked by the particles suspended in rough beer, (dM/dt) declined, TMP being kept constant at 1 bar. By referring to a short time interval of 210 s, the beer permeabilities of the filter coatings made of filter-aids of the lot A or B were statistically different and amounted to 0.11 or 0.08 Darcy, respectively (Table 2). Despite both the resulting filtrates exhibited the same turbidity at 20 °C (0.53±0.3 EBC-U), after a 39-min filtration test their masses amounted to circa 1.0 and 0.43 kg, respectively (Figure 3). The above 58% reduction in the mass of filtrate recovered from the filter precoated with filter-aids of lot B was highly likely attributable to the mixture of DE powders used. Therefore, the performance of conventional DE filtration of rough beer is strongly dependent on small variations in the particle size distribution of the filter-aids used, this intrinsic variability being in principle enlarged by inappropriate dosages due to inexperience or distraction on the part of the filter operators.

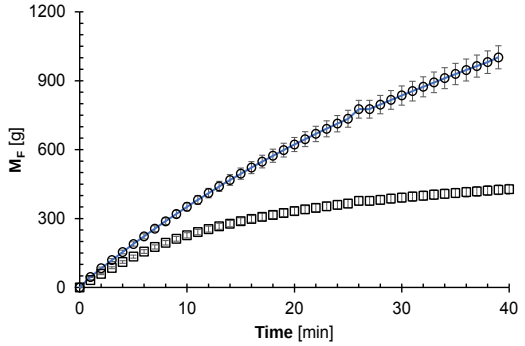

Figure 3: Time course of the mass of filtrate (M_F) from a porous filter cake made of FC and a mixture of DE powders DIA1 and DIA2 (weight ratio of 1:9 g.g^{-1}) of lot A (○) or B (□) when feeding BP1 at 20 °C.

3.4 Membrane clarification of rough beer

Both pre-centrifuged samples BP1 and BP2 were clarified by CFMT under the same operating conditions (TMP= 3.75 bar; v_s=6 m.s^{-1}; and T=10 °C or 1.5 °C) previously used (Cimini and Moresi, 2015). Figure 4 shows the time course of the instantaneous volumetric flux (J_v) and permeated beer mass (P) collected during both tests.

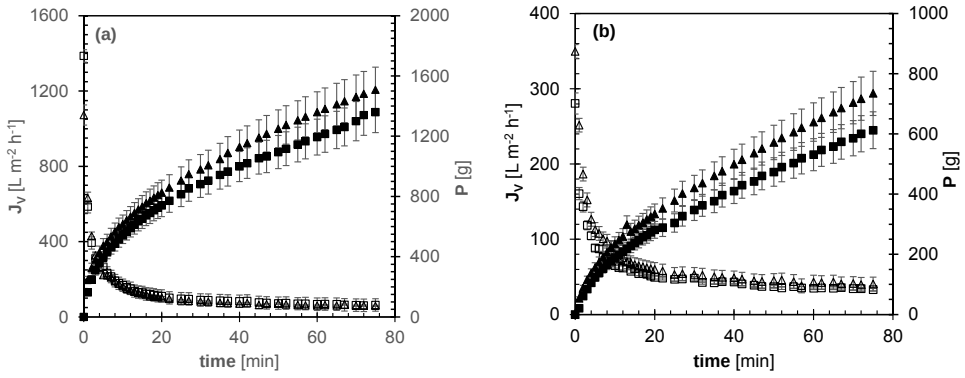

Figure 4: Time course of the instantaneous permeation flux (J_V, open symbols) and permeate mass (P, closed symbols) recovered during the CFMF of rough beer samples BP1 (□, ■) and BP2 (△, ▲) either pre-centrifuged at T=10 °C (a) or used as such at T=1.5 °C (b), TMP=3.75 bar, and v_s=6 m.s^{-1}.

Table 3 shows that the CFMF performance of the two RB samples was similar in terms of the limiting (J*) and average (J_{Va}) permeation fluxes, as well as overall amount of permeated beer collected (P_f). There was no statistically significant difference between their ß-glucan contents, while their viscosities at 1.5 °C significantly increased (Table 1), this reducing J* and J_{Va} (Table 3). Also, their final permeates had practically the same characteristics (Table 1). Thus, it was impossible to detect a clear source for the great difference in the filtrate volumes recovered from the industrial pre-coat candle filters. In this way, it would be possible to get rid of the such processing uncertainties by resorting to rough beer pre-centrifuging, enzymatic treatment, primary

clarification via 1.4-µm ceramic hollow-fiber membrane, and secondary sterilization step via a 0.45-µm polyethylene terephthalate cartridge filter to yield a beer ready for aseptic packaging at a process throughput of 500-2000 $L.m^{-2}.h^{-1}$ (Cimini and Moresi, 2020).

Table 3: Main results of triplicated CFMT tests performed with rough beer samples BP1 and BP2, as such or pre-centrifuged, at different temperatures (T), limiting permeation flux (J), average permeation flow (J_{Va}), and overall amount of permeate collected (P_f).*

Rough beer		**BP1**	**BP2**	**BP1**	**BP2**	**BP1**	**BP2**
Pre-centrifugation	**T** [°C]	**J*** [L m^{-2} h^{-1}]	**J*** [L m^{-2} h^{-1}]	**J_{Va}** [L m^{-2} h^{-1}]	**J_{Va}** [L m^{-2} h^{-1}]	**P_f** [g]	**P_f** [g]
Yes	10	63 ± 1 ^{A}	61 ± 2 ^{A}	120 ± 6 ^{a}	112 ± 3 ^{b}	1377 ± 61 $^{\alpha}$	1285 ± 60 $^{\alpha}$
No	1.5	34 ± 2 ^{B}	40 ± 1 ^{C}	52 ± 4 ^{c}	62 ± 4 ^{d}	621 ± 23 $^{\beta}$	734 ± 45 $^{\gamma}$
Yes	1.5	51 ± 1 ^{D}	51 ± 1 ^{D}	78 ± 3 ^{e}	78 ± 4 ^{e}	956 ± 51 $^{\delta}$	1012 ± 50 $^{\delta}$

Different Latin uppercase and lowercase, or Greek lowercase, letters indicate statistically significant difference between the J^*, J_{Va}, and P_f values at the probability level of 0.05, respectively.

4. Conclusions

By combining lab-scale dead-end and crossflow filtration tests, it was possible to identify the most probable cause for the great variation observed in the industrial-scale filterability of two rough beers made of malts of slightly different origin. These exhibited practically the same physico-chemical properties and, upon preliminary centrifugation, their membrane clarification at 1.5 °C was characterized by similar limiting and average permeation fluxes and permeate recovery yields. Use of a bench-top dead-end filtration apparatus allowed such a different filterability to be attributed to the different particle size distribution of the DE powder lots used to coat the industrial candle filters. A small reduction in the initial filter cake permeability from 0.11 to 0.08 Darcy resulted in as much as 58% smaller recovery of filtered beer. Such sensitivity, as well as the safety problems associated to DE handling and disposal of exhausted particles, exposes the entire beer production process to the risk of an uncontrollable increase in the processing costs and times. By contrast, appropriate downstream processing of rough beer in conjunction with CFMF might solve such processing uncertainties.

Acknowledgments

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