REMOVAL OF COLD TRUB FROM HOPPED WORT BY CROSSFLOW MICROFILTRATION: ANALYTICAL ASPECTS

Áron Varga[1], Richard Bertok[1,2], Beáta Hegyesné-Vecseri[2], Edit Márki[1]

[1]*Department of Food Engineering, Szent István University, H-1118 Budapest, Ménesi út 44., Hungary*
[2]*Department of Brewing and Distilling, Szent István University, H-1118 Budapest, Ménesi út 45., Hungary*
e-mail: mr.aron.varga@gmail.com

Abstract
Removing cold trub (cold break) from hopped wort is important, because of yeast viability, beer quality and less fouling during fermentation in a Membrane Bioreactor (MBR). Cold trub can be removed from hopped wort by several methods, but Crossflow Microfiltration (CFMF) would be an alternative technology. For the membrane filtration experiment a pale wort was made in pilot scale. The membrane filtration was performed with the following operating parameters: temperature (10±1°C), Transmembrane Pressure (0.4 bar) and Retentate Flow Rate (50 l/h). The size of particles in cold trub is about 0.5 µm, the nominal pore size of the used membrane was 0.2 µm. The Initial Flux and Steady State Flux of the membrane filtration were calculated. Chemical and physical properties (β-glucan content, bitterness, colour, dynamic viscosity, extract content, free amino nitrogen content, pH, turbidity, particle size distribution) of original wort and permeate samples were measured and retentions of different components (β-glucan, iso-alpha acids, extract, free amino nitrogen) were calculated. The Initial Flux and Steady State Flux of the membrane filtration were 16.75 L/(m^{2}h) and 4.89 L/(m^{2}h), respectively. Changing of the analytical parameters are appropriate, for example, retention of β-glucan was 40.17 % and free amino nitrogen content of original wort and permeate were same. According to the particle size distribution measurement cold trub can be completely removed by CFMF. CFMF is an alternative method for removing cold trub from hopped wort based on the results, but the optimization of the technology is needed for purposes of increasing Flux values and improve analytical parameters.

Introduction
The scope of this study is to investigate the analytical aspects of removal of cold trub (cold break) from hopped wort by Crossflow Microfiltration (CFMF).
Hopped wort is an intermediate product of brewing. It is the liquid extracted from the mashing process and boiled with hops [1]. Wort cooling before fermentation leads to the formation of cold trub (composed of proteins, protein-polyphenol complexes and carbohydrates). The size of particles in cold trub is about 0.5 µm and it settles only with great difficulty [2], [3]. Removing at least some cold trub can improve yeast viability and the quality of finished beer [4]. Furthermore, if primary fermentation is performed in a Membrane Bioreactor (MBR), cold trub causes membrane fouling [5]. Centrifugation, diatomaceous earth filtration, flotation and sedimentation are some methods used to promote the removal of cold trub [2], but CFMF would be an alternative technology [6]. In CFMF, the fluid (wort in this case) to be filtered flows parallel to the membrane surface and permeates through the membrane by Transmembrane Pressure [7].

The goals of the present investigation are to determine Flux values of the CFMF with given operating parameters, chemical and physical properties of feed (original wort) and permeate.

Experimental

Wort production

Pale wort was made with multi step mashing in pilot scale at the Department of Brewing and Distilling, Szent István University. Pilsner Malt from Boortmalt, Hungary and Hallertauer Tradition pellet hops from HVG, Germany were used at the brewhouse.

Membrane filtration

The membrane filtration experiment was carried out with bench scale in-house developed CFMF equipment. Membralox T1-70 tubular ceramic membrane (Pall, USA), with active layer of aluminium oxide, 0.2 µm nominal pore size, 7 mm channel diameter, 250 mm length and 0.005 m^2 active surface was used for filtration purpose. During the membrane filtration process temperature, Transmembrane Pressure and Retentate Flow Rate were maintained 10±1°C, 0.4 bar and 50 l/h, respectively. Permeate samples were collected with a constant volume and Flux values were calculated.

After membrane filtration experiment, the membrane was cleaned thoroughly by deionized water for 5 minutes at 25°C and then by 1 % (w/w) Sodium hydroxide for 60 minutes at 60°C. After cleaning by alkali the membrane was rinsed again by deionized water for 10 minutes at 25°C followed by cleaning with 1 % (w/w) Hydrogen peroxide for 60 minutes at 25°C temperature. Finally the membrane was cleaned thoroughly by deionized water for 10 minutes at 25°C. In all cases Transmembrane Pressure and Retentate Flow Rate were maintained 0.4 bar and 50 l/h, respectively. Sodium hydroxide were purchased from Reanal, Hungary and Hydrogen peroxide from Hungaro Chemicals, Hungary.

Chemical and physical measurements

The β-glucan content of the samples were determined by "Analytica EBC 8.13.2 High Molecular Weight ß-Glucan Content of Wort: Fluorimetric Method", 2008. The bitterness (concentrations of iso-alpha acids in ppm) and colour of the samples were measured according to "Analytica EBC 8.8 Bitterness of Wort", 2004 and "Analytica EBC 8.5 Colour of Wort: Spectrophotometric Method (IM)", 2000. Dynamic viscosity of original wort and permeate samples were measured with Physica MCR 51 Rheometer (Anton-Paar Hungary Ltd., Hungary) with DG27 double gap concentric cylinder measurement system. Data were acquired and analysed using Rheoplus/32 v.3.40 software. Flow curve of samples were measured by increasing the shear rate from 500 to 1000 1/s at temperature of 20 °C. Dynamic viscosity of samples were calculated based on Herschel Bulkley model [8] fitted to measured data of flow curve (shear stress in function of shear rate). Extract contents of samples were measured with Alcolyzer Plus (Anton-Paar, Austria). The free amino nitrogen (FAN) content of samples were determined by the "Analytica EBC 8.10.1 Free Amino Nitrogen in Wort by Spectrophotometry – Manual method (IM)", 2017. All of the absorbances were measured with DR 6000 spectrophotometer (Hach, USA).The pH of samples were determined with 1100 H pH meter (VWR, USA). Turbidity of samples were measured at 20°C with 2100P Turbidimeter (Hach, USA) in NTU and converted to EBC. Retentions of β-glucan, iso-alpha acids, extract and FAN were calculated. Particle size distribution was measured with Malvern Zetasizer Nano-ZS. Data were acquired and analysed using Zetasizer 6.32 software.

Results and discussion

Membrane filtration

The Initial Flux and Steady State Flux of the membrane filtration were 16.75 L/(m^2h) and 4.89 L/(m^2h), respectively. These values are quite low, because of fouling mechanism.

Chemical and physical measurements

Analytical parameters of original wort and permeate are shown in Table 1.

Table 1. Analytical parameters of original wort and permeate

Parameter	Original wort	Permeate
β-glucan content (mg/L)	117	70
Bitterness (IBU)	49	44
Colour (EBC)	12.98	8.98
Dynamic viscosity at 20°C (mPas)	5.43	4.95
Extract content % (w/w)	11.16	10.34
FAN content (mg/L)	159	159
pH	6.02	6.42
Turbidity at 20°C (EBC)	106.75	7.88

β-glucan content decreased dramatically that leads to less fouling during fermentation in a MBR. Furthermore, the lower β-glucan content can improve clarification of rough beer (higher filtration throughput and less haze problems in the final product). The bitterness decreased by 5 unit, but this difference can't be evaluated with sensory analysis. Colour became paler, supposedly due to notable retention of polyphenols, carbohydrates and Maillard reaction products. The dynamic viscosity decreased, this is mainly because of the lower β-glucan content. The extract decreased by reason of retention of different compounds (e.g. carbohydrates). FAN content wasn't changed that is essential, because adequate level of FAN (150 - 200 mg/L [9]) in wort ensures efficient yeast cell growth and desirable fermentation performance. The pH increased that negatively affects the microbiological stability of the permeate. The turbidity was decreased by two orders of magnitude, because of removal of cold break. This results in less haze problems in the final product.

Retentions of different components are shown in Table 2.

Table 2. Retentions of different components

Component	Retention (%)
β-glucan	40.17
Iso-alpha acids (bitterness)	10.20
Extract	7.65
FAN	0

From a technological point of view, the retention values of β-glucan and FAN are suitable. The retention values of iso-alpha acids and extract are acceptable.

Original wort sample was too polydisperse for particle size distribution analysis and the average particle size of this sample was larger than upper size analysis limit (10 μm).

Particle size distribution of permeate is shown in Figure 1.

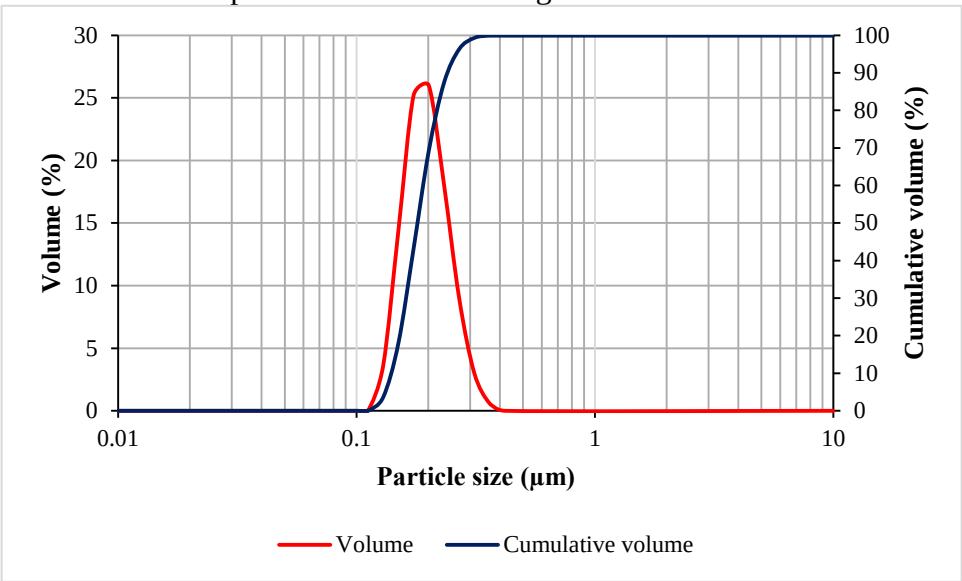

Figure 1. Particle size distribution of permeate

As it can be seen in Figure 1. cold trub (particles about 0.5 µm in diameter) was completely removed by microfiltration. The average particle size of the permeate is around 0.2 µm that corresponds to the nominal pore size of the membrane.

Conclusion

Flux values of the membrane filtration experiment were quite low, but these values could be increased by the optimization of operating parameters (e.g. Transmembrane Pressure and Retentate Flow Rate). It has been proven that the changing of the analytical parameters are appropriate and cold trub can be completely removed by CFMF.

Acknowledgements

This work was supported by the ÚNKP-17-3 the New National Excellence Program of the Ministry of Human Capacities; the Szent István University, Doctoral School of Food Sciences; the European Union and co-financed by the European Social Fund (grant agreement no. EFOP-3.6.3-VEKOP-16-2017-00005). We would like to say thanks to Szent István University, Faculty of Food Science, Department of Brewing and Distilling and Szent István University, Faculty of Food Science, Department of Food Preservation for supporting the measurements.

References

[1] J. Csanádi, 'Sörlékészítés', in *Élelmiszer-technológia mérnököknek*, Szeged, Hungary: Szegedi Tudományegyetem Mérnöki Kar, 2010, pp. 451–454.

[2] R. Barchet, 'Cold Trub: Implications for Finished Beer, and Methods of Removal', *Brewing Techniques*, 1994. [Online]. Available: http://www.morebeer.com/brewingtechniques/library/backissues/issue2.2/barchet.html. [Accessed: 22-Sep-2017].

[3] W. Kunze, 'Formation and optimal remove of cold break', in *Technology Brewing and Malting*, 3rd ed., Berlin, Germany: VLB, 2004, p. 347.
[4] L. Narziß and W. Back, 'Abtrennung des Kühltrubs', in *Die Bierbrauerei - Die Technologie der Würzebereitung*, Weinheim, Germany: WILEY-VCH Verlag GmbH & Co. KGaA, 2009, pp. 700–701.
[5] S. Schildbach and L. Stumpf, 'Enhancing Main Fermentation Velocities in Beer by the Use of a Membrane Bioreactor – Approach and First Results', presented at the BiosysFoodEng 2016, Budapest, 08-Dec-2016.
[6] A. Ambrosi, N. S. M. Cardozo, and I. C. Tessaro, 'Membrane Separation Processes for the Beer Industry: A Review and State of the Art', *Food Bioprocess Technol.*, vol. 7, no. 4, pp. 921–936, 2014.
[7] R. El *et al.*, 'Cross-flow microfiltration applied to oenology: A review', *J. Membr. Sci.*, vol. 382, no. 1–2, pp. 1–19, 2011.
[8] T. G. Mezger, 'Model functions for flow curves including a yield point', in *The Rheology Handbook*, 2nd ed., Hannover, Germany: Vincentz Network, 2006, p. 57.
[9] I. Hornsey, 'Nitrogen Metabolism', in *Brewing*, 2nd ed., Cambridge, United Kingdom: RSC Publishing, p. 153.