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Ceramic membrane cross-flow microfiltration for beer recovery from tank bottoms

Cross-flow microfiltration with ceramic membranes was applied for beer recovery from tank bottoms. The cross-flow microfiltration was optimised using model yeast suspension and the effects of pore size, flow speed and transmembrane pressure were characterised. Using 200 µm pore-size membranes and back-flush in 5 min intervals during filtration of tank bottoms, filtration rate 20 kg/m^2 h^{-1} compared to 3 kg/m^2 h^{-1} without back-flush was achieved. The quality of beer recovered from tank bottoms was extensively evaluated. Increased values of some beer components were found in recovered beer when 200 µm membranes were used: bitterness increased from 20 to 36 ºEBC, total nitrogen from 78.4 to 172.1 mg/100 ml, polyphenols from 20.4 to 56.4 mg/l and diacetyl from 0.11 to 0.41 mg/l. Pore-size in range 100–500 µm has effect on filtration rate but no influence on beer quality. Some parameters of recovered beer, such as extract, alcohol, pH are not changed. Analyse of 7 fractions collected during beer recovery showed that values are generally increased in last fractions.

BC 24 Bottling

(Descriptors: Beer, microfiltration, tank bottoms.

Deskriptoren: Bier, Mikrofiltration, Tankböden).

1 Introduction

The filtration techniques currently employed in beer filtration at industrial scale are candle filters and horizontal leaf filters using diatomaceous earth. During recent years cross-flow microfiltration has also been successfully applied. Ceramic microfiltration membranes may be applied mainly in beer filtration as substitute for classical filtration techniques (1). Using small pore sizes 0.1 – 0.5 µm near-sterility is achieved while increased pore-size up to 1.3 µm leads to improved filtration rates (1).

Inorganic microfiltration membranes provide a good performance and durability but their practical applications in beverage industry are considerably hampered by membrane fouling. The membrane fouling is a serious problem mainly in the microfiltration of real food products, such are beer, wine and milk, where fouling mechanisms are not completely clarified (2). The filtration rate decrease during beer clarification was reported to be caused mainly by reversible cake formation (3) and can be efficiently reduced by using oscillatory flow (4). A more serious problem is the membrane fouling. The membrane fouling by macromolecular fractions of beer and wine (5) and cleaning procedure for membranes fouled by beer (6) were reported. Several membrane foulants in beer filtration were identified: malt derived beer components, such are β-glucan, arabinoxylan, protein and polyphenol (7), carbohydrates and minerals (8), β-glucane (9).

A new pioneering application of ceramic membranes is beer recovery from tank bottoms. However, there are only a few literature reports on microfiltration application in this field (10, 11, 12). Spent yeast is mostly considered to be a useless waste product of fermentation. Former process technologies did not allow any economic recovery of beer from fermentation yeast and tank bottoms. Another product from tank bottoms is brewer's yeasts containing high-quality proteins, carbohydrates, vitamins that may be obtained after yeast complex fractionation and used as quality nutrients (13).

The aim of this study was to evaluate application of ceramic membrane cross-flow microfiltration for beer recovery from tank bottoms. The particular aim was to evaluate quality of recovered beer because these data are missing in literature.

2 Materials and methods

2.1 Membranes

Ceramic membranes Membralox (SCT, France), pore-sizes 100, 200 and 500 µm, length 0.25 m, inner diameter 7 mm, outer diameter 10 mm, active surface area 55 cm^2 were used in this study.

Membranes were cleaned according to the recommendation of the membrane supplier. The cleaning procedure was performed after each experiment and usually 80 – 90% of first-use water flow was achieved.

2.2 Filtration

Measurements were performed using a cross-flow microfiltration unit. The suspension to be filtered was circulated by a membrane pump equipped with a pulse absorber. Filtrate was circulated through a heat exchanger temperated at 20 °C. Transmembrane pressure difference was adjusted by a regulation valve and measured by a pressure gauge. Retenate was returned to a feed reservoir and permeate was collected in a vessel placed on balances. The balances, the pressure gauge and the regulation

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valve were connected to a computer. Computer program was used to regulate constant pressure and calculate actual permeate flow.

Tank bottoms for filtration experiments, beer and dried yeasts were supplied from a local brewery.

2.3 Analytical methods

Basic brewery characteristics of the beer were determined by a SCABA 5600 (Automatic Beer Analyser, Tecator AB, Sweden). Color, bitterness, nitrogen and polyphenols were determined according to the European Brewery Convention approved methods (14). Ethanol and diacetyl were determined by a gas chromatograph with a FID detector (Laboratorni Pristroje Praha, Czech Republic) using Porapak SQ (Sigma, USA) column.

3 Results and discussion

3.1 Effect of yeasts on microfiltration

Tank bottoms contain high concentration of yeasts and therefore main contribution to membrane resistance is expected from yeast layer formation over the membrane surface. Model yeasts solutions prepared from dried brewery yeasts resuspended in water were used to study the effect of yeasts on microfiltration. The effect of yeast concentration was evaluated using concentrations 1 and 5% (w/w), this range corresponds to yeasts concentrations in tank bottoms. A typical course of microfiltration is shown in Figure 1. Transmembrane pressure was gradually increased and as it can be seen from Figure 1 the pressure had no effect on the permeate flow. Analogical experiments were performed at different yeast concentrations and flow speeds and results are summarised in Figure 2. As it was expected, increased yeast concentration led to increased membrane resistancy and increased flow speed efficiently increased permeate flow.

3.2 Microfiltration of tank bottoms

A typical beer recovery from tank bottom using microfiltration with back-flush is presented in Figure 3 A. The achieved permeate flow (20 kg m^{-2} h^{-1}) was considerably lower compared to flows obtained with model yeast solutions. This can be attributed to membrane fouling by substances, mainly polymeric, present in beer. During microfiltration without back-flush (Fig. 3 B) only 3 kg m^{-2} h^{-1} was achieved. For comparison also pore-sizes 100 and 500 µm were tested (Fig. 4). Similar permeate flows 17 and 19 kg m^{-2} h^{-1} were obtained with 100 and 200 µm membranes while increased flow 30 kg m^{-2} h^{-1} was obtained with 500 µm membrane.

The tank bottoms used for microfiltration experiments were collected from cylindro-conical tank after fermentation and immediately used for beer recovery. Yeast concentration in tank bottoms varied usually in the range of 3.6 – 4.2%. In the presented microfiltrations usually 50 – 60% of beer was recovered from tank bottoms. After 60% beer beeing recovered filtration was finished because retenate usually became too viscous for any further microfiltration.

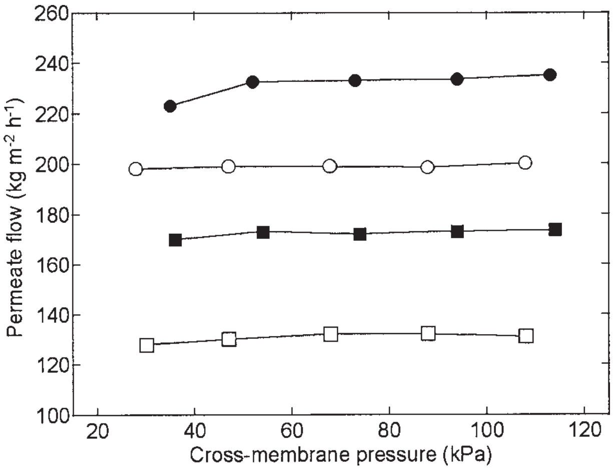

Fig. 2 Effect of transmembrane pressure and flow velocity on cross-flow microfiltration rate. Legend: yeasts concentration 1% (w/w) (○, ●); 5% (w/w) (□, ■); flow velocity 1.5 m s^{-1} (empty symbols), 2.5 m s^{-1} (full symbols)

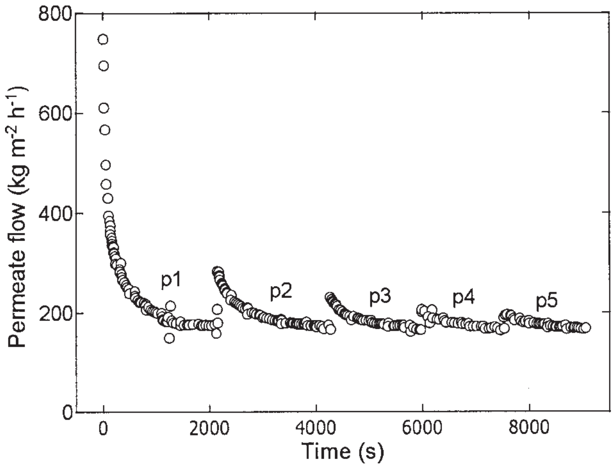

Fig. 1 Cross-flow microfiltration of yeasts at various pressures. Parameters of filtration: membrane pores 200 µm; yeasts concentration 1% (w/w); flow velocity 2.5 m s^{-1}; temperature 20 °C; back-flush in 5 min intervals; transmembrane pressure (kPa): 34.4 (p1), 54.2 (p2), 74.0 (p3), 93.4 (p4), 113.9 (p5)

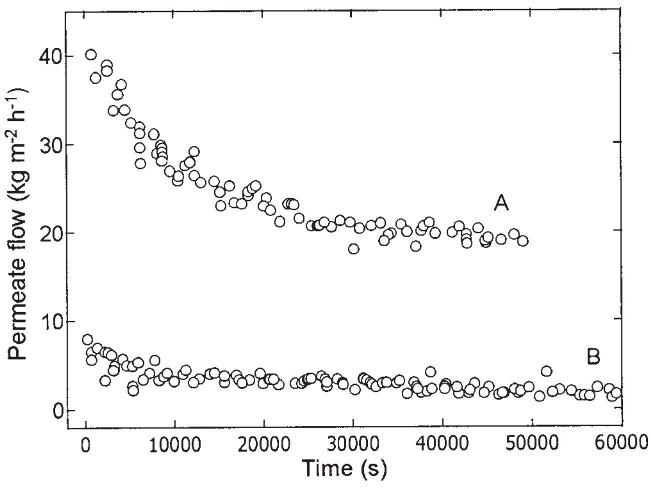

Fig. 3 Cross-flow microfiltration of tank bottom. Parameters of filtration: membrane pores 200 µm; transmembrane pressure 150 kPa; yeasts concentration 4.2% (w/w). The filtrations were performed with (A) and without (B) back-flush.

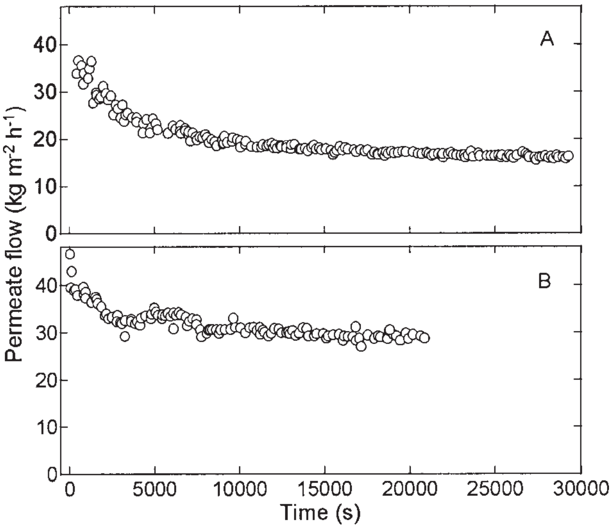

Fig. 4 Effect of pore-size of ceramic membrane on microfiltration of tank bottoms; pore-sizes: 100 µm (A), 500 µm (B). Parameter of filtration were similar to Fig. 3 B

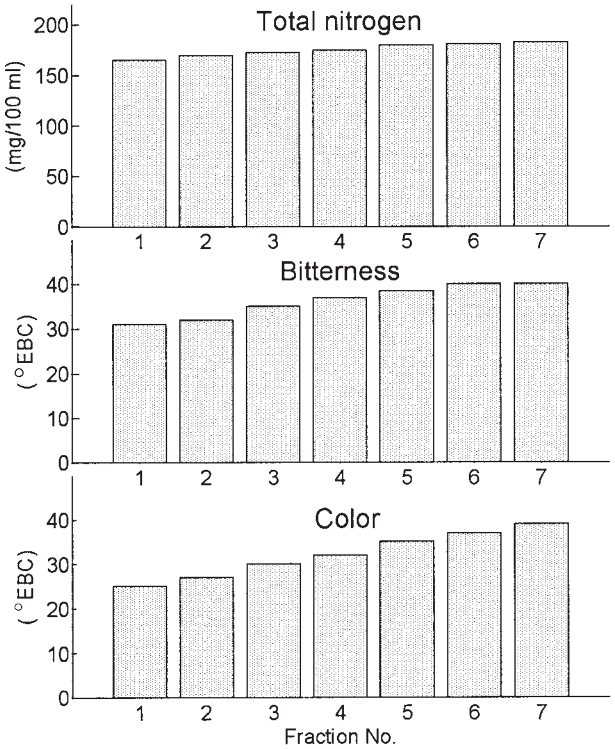

Fig. 5 Parameters of beer collected during tank bottom microfiltration. Beer was collected in 7 equal-volume fractions. Parameters of filtration: membrane pores 200 µm; transmembrane pressure 150 kPa; yeasts concentration 4.2% (w/w)

3.3 Recovered beer quality

The crucial point for any practical applicability of cross-flow microfiltration is the quality of recovered beer. The analyses of beer recovered by cross-flow microfiltration are presented in Table 1 together with the analyses of beer obtained from the tame tank. Some parameters remain substantially unchanged: extracts, ethanol, pH. But, as it can be seen from Table 1, beer obtained by microfiltration with back-flush contained considerably increased color, concentrations of bitter compounds, total and free nitrogen, polyphenols and diacetyl. The most increased value was observed for free nitrogen, for which a 2.5-times increased value compared to beer was obtained. To reveal possible effects of microfiltration itself on beer parameters the recovered beer is compared to beer recovered from tank bottoms by centrifugation. This comparison shows almost equal values of bitter compounds and diacetyl. Nevertheless, color, free and total nitrogen and polyphenols have higher values in microfiltrate than centrifugate which indicates that this may be an effect of microfiltration. We assume that the increased polyphenols may be caused by releasing adsorbed polyphenols from cells and increased nitrogen may be the consequence of cell disruption, both caused by shear stress during microfiltration. It can be concluded from comparison of experiments with back-flush and without back-flush (Tab. 1) that back-flush has no significant effect on beer parameters, only slightly increased total nitrogen which perhaps may be attributed to back-flush was observed.

Table 1 Comparison of beers recovered using ceramic membrane of 200 µm pore-size with and without back-flush.

Parameter	No backflush			With backflush		
	Beer	Centrif.	Filter	Beer	Centrif.	Filter
Original extract (% w/w)	11.8	11.9	11.3	11.3	11.2	11.2
Apparent extract (% w/w)	4.98	4.89	3.86	2.54	2.32	3.00
Real extract (% w/w)	5.13	5.04	4.1	3.12	3.09	3.87
Ethanol (% w/w)	4.11	4.08	3.9	4.13	4.08	3.54
Yield (%)	56.5	57.6	63.7	72.4	72.41	65.57
Color (ºEBC)	18	23	31	17	24	32.1
pH	4.31	4.01	4.32	4.41	3.98	3.99
Bitterness (ºEBC)	18	38	34	20	37	36
Total nitrogen (mg/100 ml)	88.6	61.2	154.0	78.4	60.4	172.1
Free nitrogen (mg/100 ml)	23.5	31.2	75.6	33.2	34.6	83.0
Polyphenols (mg/l)	18.6	18.9	54.6	20.4	19.8	56.4
Diacetyl (mg/l)	0.12	0.30	0.51	0.11	0.31	0.41

Table 2 Effect of pore-size of microfiltration membrane on recovered beer parameters.

Parameter	Pore size 100 µm			Pore size 200 µm			Pore size 500 µm		
	Beer	**Centrif.**	**Filter**	**Beer**	**Centrif.**	**Filter**	**Beer**	**Centrif.**	**Filter**
Original extract (% w/w)	11.1	11.4	10.9	11.3	11.2	11.2	9.7	10.2	11.5
Apparent extract (% w/w)	2.54	2.56	3.53	2.54	2.32	3.00	2.03	2.20	0.73
Real extract (% w/w)	3.11	3.14	4.04	3.12	3.09	3.87	3.50	3.40	2.78
Ethanol (% w/w)	4.3	4.2	2.79	4.13	4.08	3.54	4.05	4.10	4.58
Yield (%)	71.9	72.5	62.8	72.4	72.41	65.57	65.2	68.3	75.8
Color (ºEBC)	19	23	29	17	24	32.1	22	26	31
pH	4.23	4.08	4.08	4.41	3.98	3.99	4.75	4.60	4.75
Bitterness (ºEBC)	19	36	36	20	37	36	18	22	29
Total nitrogen (mg/100 ml)	86.3	60.9	165.0	78.4	60.4	172.1	98.7	71.5	167.8
Free nitrogen (mg/100 ml)	33.4	36.2	87.7	33.2	34.6	83.0	32.3	35.6	101.8
Polyphenols (mg/l)	21.2	21.4	51.1	20.4	19.8	56.4	22.3	19.6	55.8
Diacetyl (mg/l)	0.01	0.30	0.61	0.11	0.31	0.41	0.16	0.26	0.60

How parameters of recovered beer change during microfiltration was investigated by collecting fractions. Seven equal-volume fractions were collected and analysed. Almost all parameters were found constant only total nitrogen and bitter compounds were increasing over the course of beer recovery (Fig. 5). Relatively intensive increase of color in beers recovered by microfiltration, compared to centrifuged beers, was observed. This was probably caused by oxidation, as well as all increased color values (presented in Tab. 1 and Tab. 2) as microfiltration allows beer aeration over long period of time. Thus beer recovery under carbon dioxide atmosphere is desirable.

Table 2 summarises effects of used pore-sizes on recovered beer parameters. Pore-size was expected to have effect on high-molecular weight compounds but only a slight increase of free nitrogen with 500 µm membrane was observed.

4 Conclusions

It can be concluded that beer recovered from tank bottoms is significantly changed in some parameters. Substantial increase of concentrations of bitter compounds, total and free nitrogen, polyphenols and diacetyl was observed in recovered beer. We assume that increased polyphenols may be caused by releasing of adsorbed substances from cells and increased nitrogen may be a consequence of cell disruption, both caused by shear stress during microfiltration. Some parameters remain substantially unchanged: extracts, ethanol, pH. Pore-size does not influence beer quality, for practical reasons 500 µm membranes which provide a higher filtration rate can be recommended. Another practical precaution should be filtering under carbon dioxide atmosphere to avoid color increase due to oxidation.

5 Zusammenfassung

Bugan, S. G., Dömény, Z., Šmogrovičová, D., Švitel, J., Schlosser, Š., und Stopka, J.: Crossflow-Mikrofiltration mit Keramikmembranen zur Rückgewinnung von Bier von Tankgelägen — Monatsschrift für Brauwissenschaft 53, Nr. 11/12, 229 – 233, 2000

BC 24 Abfüllung

Die Crossflow-Mikrofiltration mit Keramikmembranen wurde zur Rückgewinnung von Bier von Tankgelägen eingesetzt. Die Crossflow-Mikrofiltration wurde mit Hilfe einer Modell-Hefesuspension optimiert, und die Auswirkungen der Porengröße, der Fließgeschwindigkeit und des Transmembrandrucks wurden charakterisiert. Bei Verwendung von Membranen mit einer Porengröße von 200 µm und Rückspülung in Intervallen von 5 Minuten während der Filtration von Tankgelägen wurde eine Filtrationsrate von 20 kg/m^2 h^{-1} gegenüber 3 kg/m^2 h^{-1} ohne Rückspülung erreicht. Die Qualität des von den Tankgelägen zurückgewonnenen Bieres wurde ausführlich bewertet. Erhöhte Werte einiger Bierbestandteile wurden bei zurückgewonnenem Bier festgestellt, wenn Membranen mit 200 µm verwendet wurden: Die Bittere stieg von 20 auf 36 ºEBC, der Gesamtstickstoff von 78,4 auf 172,1 mg/100 ml, die Polyphenole von 20,4 auf 56,4 mg/l und Diacetyl von 0,11 auf 0,41 mg/l. Porengrößen im Bereich von 100 – 500 µm wirken sich auf die Filtrationsrate aus, haben aber keinen Einfluß auf die Qualität des Bieres. Einige Parameter des zurückgewonnenen Bieres, wie etwa Extrakt, Alkohol und pH-Wert, bleiben unverändert. Die Analyse von 7 während der Rückgewinnung des Bieres aufgefangenen Fraktionen zeigte, daß die Werte im allgemeinen in den letzten Fraktionen erhöht sind.

Bugan, S. G., Dömény, Z., Šmogrovičová, D., Švitel, J., Schlosser, Š., und Stopka, J.: Cross-flow microfiltration sur membrane de céramique pour la récupération de la bière des fonds de tanks — Monatsschrift für Brauwissenschaft 53, Nr. 11/12, 229 – 233, 2000

BC 24 Soutirage

La cross-flow microfiltration sur membrane de céramique a été appliquée pour la récupération de la bière des fonds de tanks. La cross-flow microfiltration a été optimisée en utilisant le modèle de levures en suspension et les effets de la taille des pores, la vitesse de circulation et la vitesse transmembranaire ont été caracterisés. En utilisant des membranes avec une taille des pores de 200 µm et un rétro-lavage toutes les 5 minutes pendant la filtration des fonds de tanks, un débit de filtration de 20 kg/m^{2}h^{-1} a été obtenu comparé a 3 kg/m^{2}h^{-1} sans rétro-lavage. La qualité de la bière récupérée des fonds de tanks a été largement évaluée. On a observé une augmentation de certaines valeurs des composés de la bière récupérée en utilisant des membranes de 200 µm: l'amertume passe de 20 à 36 °EBC, l'azote total de 78,4 à 172,1 mg/100ml, les polyphénols de 20,4 à 56,4 mg/l et le diacétyle de 0,11 à 0,41 mg/l. La taille des pores des membranes entre 100 et 500 µm a un effet sur le débit de filtration mais n'a pas d'influence sur la qualité de la bière. Quelques paramètres de la bière récupérée, tels que l'extrait, l'alcool, le pH n'ont pas changé. L'analyse de 7 fractions collectées pendant la récupération de la bière montrait que les valeurs augmentaient généralement pour les dernières fractions.

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