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Dead-end Microfiltration of Rough Nonalcoholic Beer by Different Polymeric Membranes

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ABSTRACT

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Clarification of rough nonalcoholic beer using microfiltration as an alternative to conventional filtration with filter aids presents scientific and technical challenges for the brewing industry. An experimental pilot plant was used to evaluate the permeability and selectivity of polymeric membranes in the clarification process. Cellulose acetate (CA) with pore sizes of 0.2, 0.45, 0.8, and 1.2 μm , together with cellulose nitrate (CN), nylon (NY), and polytetrafluoroethylene (PTFE) with a pore size of 0.45 μm , were used at transmembrane pressures (TMP) of 1.0 and 2.0 bar. The data corroborated that the flux values of the CA, CN, and NY membranes were almost the same and reduced drastically, whereas PTFE was not permeable at 1.0 bar and its flux at 2.0 bar increased at the beginning and decreased after reaching a maximum. At both TMP, the CN membrane displayed the highest rejection of suspended particles and haze-active proteins, while retention of polyphenols and color were highest for the NY membrane. The lower selectivity of the PTFE membrane can be attributed to its hydrophobicity and less interaction with the hydrophilic haze-active proteins. The results confirmed that by increasing the pore size of CA membranes the separation factor of the sensitive proteins and suspended particles decreased and permeability increased.

Keywords: Clarification, Haze-active proteins, Malt beverage, Nonalcoholic beer, Polyphenols, Pore size

RESUMEN

Aclaración de la cerveza sin alcohol en bruto por microfiltración como una alternativa a la filtración convencional con ayudas del filtro presenta desafíos científicos y técnicos para la industria cervecera. Una planta piloto experimental fue utilizada para evaluar la permeabilidad y selectividad de las membranas poliméricas en el proceso de clarificación. El acetato de celulosa (CA), con tamaños de poro de 0.2, 0.45, 0.8, y 1.2 μm , junto con el nitrato de celulosa (CN), el nylon (NY), y el politetrafluoroetileno (PTFE) con un tamaño de poro de 0.45 μm , se utilizaron en las presiones transmembrana (PGT) de 1.0 y 2.0 barra. Los datos corroboran que los valores de flujo de la CA, CN, y las membranas NY fueron casi los mismos y se redujeron drásticamente, mientras que el PTFE no era permeable a 1.0 barra y su flujo a 2.0 barra mayor al principio y han disminuido tras alcanzar un máximo. Por los dos PGT, la membrana CN muestra el mayor rechazo de partículas en suspensión y turbidez proteínas activas, mientras que la retención de los polifenoles y el color eran los más altos de la membrana NY. La selectividad inferior de la membrana PTFE se puede atribuir a su hidrofobicidad y menor interacción con las proteínas activa turbidez hidrofílicas. Los resultados confirmaron que al aumentar el tamaño de los poros de las membranas CA el factor de separación de las proteínas y partículas en suspensión sensible disminución y el aumento de la permeabilidad.

Palabras claves: Aclaración, Bebidas de malta, Cerveza sin alcohol, Polifenoles, Proteínas activa turbidez, Tamaño de los poros

Beer is the fifth most widely consumed beverage in the world and is a popular beverage, with an average consumption of 23 L per person per year. The consumption of nonalcoholic beer is increasing significantly due to its lower calorie content; health concerns associated with alcoholic beverages; and social, legal, and religious issues associated with alcohol in some parts of the world. This has led to the development of a market for health-promoting beverages, and most major brewers are now including alcohol-free beers in their product portfolios (2,4,6). Clarification is a critical stage that involves the removal of yeast and particles to achieve a bright beer with colloidal stability. In addition, this stage reduces the microbiological count of the filtrate. Standard filtration consists of retention of solid particles during dead-end filtration with filter aids (6,8,17,19). Recently, membrane technology has been successfully used in a clarification process, wherein kieselguhr filtration is replaced by cross-flow microfiltration. It has been found that this technology could provide further improvements to quality, environmental, and health issues, as well as process safety, simplicity, flexibility, and operational costs (7,11,13,17,18,23).

Numerous studies have been done on the use of polymeric membranes in microfiltration of beer. The first polymeric membrane used for this application was typically polypropylene. Despite its good resistance to many cleaning agents, this polymer has one main disadvantage—its hydrophobic nature. In recent years, a wide range of hydrophilic polymeric membranes have been tested for use in microfiltration (15). Czekaj et al (3) studied dead-end microfiltration of beer using hydrophilic cellulose acetate (CA) and polycarbonate (PC) membranes with a nominal pore size of 0.2 μm . They found that the CA membrane, which has higher surface porosity, led to a more gradual decrease in flux than did PC (3). Yokhanis et al (24) used a stirred cell with 0.45- μm hydrophobic polytetrafluoroethylene (PTFE) and hydrophilic surface-modified polyethersulphone membranes to investigate the effects of membrane hydrophilicity on flux and resistance. They concluded that fouling on hydrophobic membranes was severe and involved adsorption of beer components onto the membrane surface, while surface-modified hydrophilic membranes provided higher flux (24).

Reed (15) reported on a breakthrough in the application of membranes in beer filtration in a study of the effects of membrane pore size on final product quality. Fillaudeau and Carrère (9) demonstrated that membranes with mean pore diameters <0.2 and 0.45 μm provided high retention of essential compounds and a clear, bright, almost sterile, but insipid product (less color, dry matter, bitterness, and head-retention value). Below 0.80 μm , retention remained too high and induced low-value permeates. Membranes with a mean pore diameter of ≥ 0.80 μm satisfied a retention rate of essential compounds of <10% (8,9). The advantage of pore sizes >0.4 to 0.5 μm was clear when studying the transmission of beer components, but according to prior research, application of larger pore sizes (<1 μm) did not result in considerably higher flux even though flux declined at a substantially lower rate (8,15). Gan et al (11) showed that chill haze in beer clarified with a ceramic membrane with a 1.3- μm pore

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diameter was a potential problem of colloidal instability. On the other hand, the same membrane with a 0.2- μm pore diameter was too small to allow passage of sufficient proteinaceous components for a good head-retention value. Therefore, an optimum pore diameter should be chosen according to the feed and process conditions (11). Fillaudeau et al (8) evaluated analytical profiles of the permeate and retention rate for membranes with different mean pore diameters in a rotating vibrating filtration plant. According to their research, for pore diameters $>1\ \mu\text{m}$ and $<3\ \mu\text{m}$, the clarified beer met European Brewery Convention (EBC) qualitative standards (11).

Nonalcoholic beer (NAB) contains a maximum of 0.5% (vol/vol) alcohol, which is produced by either removal of alcohol during post-fermentation or restriction of alcohol production during the brewing process. Fermentation bypass is the most common method used in areas where use of alcohol is prohibited according to religious, social, and legal restrictions or concerns about nonfermented and halal products (2). The fermentation bypass method involves a different feed to the filtration stage compared with lager beer production.

In addition to the alcohol content, the yeast content of rough non-alcoholic beer (RNAB) is limited. RNAB does not contain carbon dioxide, and in the absence of biological modification, the molecular and macromolecular composition of the filtration feed (such as proteins, amino acids, and acetals) is different. These make the physicochemical properties of this type of beer entirely distinct from lager beer. These distinctions require specific consideration in process design and operation.

Despite the long history of applied research and development in membrane clarification of lager beer and substantial differences in properties between NAB and lager beer, very few studies have been performed on the clarification process for NAB. The ever-growing market demands for NAB necessitate further work on process design of NAB brewing.

The objective of this work was to evaluate the effects of pore size and polymeric material on the permeability and selectivity of membranes in dead-end microfiltration of RNAB at two transmembrane pressures (TMP): 1.0 and 2.0 bar. For this purpose, CA membranes with pore sizes of 0.2, 0.45, 0.8, and 1.2 μm and cellulose nitrate (CN), nylon (NY), and PTFE membranes with a pore size of 0.45 μm were used as polymeric membranes.

EXPERIMENTAL

The apparatus used in this study is shown schematically in Figure 1. It was constructed entirely of stainless steel, and the membrane module was capable of holding a flat-sheet polymeric membrane. The membrane was supported on a sintered stainless-steel plate, providing an active filtration area of 3.46 cm^2 . The membrane types and pore sizes used are summarized in Table I.

Before each experiment, the membranes were cut into 25-mm-diameter circles and gently placed in the module above the stainless-steel support. The module was then closed, and permeation was started by opening the feed valve above the module (V2 in Figure 1). The membranes did not go through special wetting processes, even though during the short time period after placement on the support and before opening the feed valve the membranes were only wet by the water already present on the support.

At the beginning of the experiment, with valves V2 and V3 (Fig. 1) closed, a certain amount of RNAB was filled into the feed vessel, and after adjusting the pressure by regulator, the V3 and V2 valves were opened consecutively to start the permeation. Feed temperature and TMP were measured with an accuracy of $\pm 0.1^\circ\text{C}$ and $\pm 0.05\ \text{bar}$, respectively. The permeate was collected in a reservoir placed on an electronic microbalance (accuracy $\pm 0.1\ \text{g}$; model GP 5202, Sartorius) interfaced to a PC to collect and record mass versus time data (every 0.4 sec). The permeate flux was obtained by numerical differentiation of the filtrate volume versus time data.

The NAB for this study was produced, using the fermentation bypass process, from pure malt (provided by Iran Behnouth Co., Tehran, Iran). The RNAB was prepared by taking samples from the

Fig. 1. Experimental dead-end microfiltration apparatus. TMP = transmembrane pressure; RNAB = rough nonalcoholic beer.

TABLE I
Polymeric Membranes Used in This Study^a

Membrane Material	Nominal Pore Size (μm)	Thickness (μm)	Water Permeability at TMP = 1.0 bar ($\text{L}/\text{m}^2\text{-hr}$)	Bubble Point (bar)	Manufacturer
Cellulose acetate	0.2	120	14,400	>2.9	ALBET
Cellulose acetate	0.45	120	41,400	1.9	ALBET
Cellulose acetate	0.8	120	120,000	0.8	ALBET
Cellulose acetate	1.2	140	228,000	NA	M&N
Cellulose nitrate	0.45	130	41,400	2.4	M&N
Polytetrafluoroethylene	0.45	65	180	0.7	ALBET
Nylon	0.45	115	15,600	2.2	ALBET

^a TMP = transmembrane pressure; NA = not available.

upper part of the conditioning vessel and adjusting them to the desired concentration. Physical and chemical measurements were performed on collected permeate samples during experiments according to EBC protocols (5). This included measurement of haze-turbidity (EBC method 9.29), sensitive proteins (EBC method 9.40), total polyphenols (EBC method 9.11), and color (EBC method 9.6). The soluble solids concentration of feeds was adjusted to $3.8 \pm 0.1^\circ\text{Bx}$, which was determined by digital refractometer (model PR-32 α , Atago Co., Ltd.). The haze was determined in nephelometric turbidity unit (NTU) scales using a portable turbidimeter (model 2100P, Hach). Each permeation experiment was 90 min long, and the rejection values were calculated from total permeate over feed parameters.

RESULTS AND DISCUSSIONS

Overview of Filtration Runs

The comparative effects of the type of membrane materials used were evaluated for membranes of the same pore size (0.45 μm) at TMP of 1.0 and 2.0 bar. For evaluation of the effects of pore size on qualitative and quantitative parameters in RNAB clarification, CA membranes with pore diameters of 0.2, 0.45, 0.8, and 1.2 μm were tested at a TMP of 1.0 bar. A summary of the results is presented in Table II in the form of maximum flux (J_{max}), its average in the first 20 min of operation ($J_{20\text{min}}$), and qualitative parameters for feed and permeate. The repetition of the experiments and standard deviation of the results are presented in Table II (25).

TABLE II
Summary of Feed Parameters Measured and Quantitative and Qualitative Results for Polymeric Membrane Materials Tested^a

Parameter ^b	No. of Tests	J_{max} (L/m ² ·hr)		$J_{20\text{min}}$ (L/m ² ·hr)		Haze (NTU)		Color (EBC)		Sensitive Proteins (NTU)		Polyphenols (mg/L)	
		Avg.	SD	Avg.	SD	Avg.	SD	Avg.	SD	Avg.	SD	Avg.	SD
Feed	—	—	—	—	—	74.69	25.0	3.00	0.00	78.1	18.5	63.45	15.4
TMP = 1.0 bar													
0.2- μm CA	1	5,980	—	2,016	—	2.25	—	2.50	—	23.1	—	—	—
0.45- μm CA	5	8,256	1,165	2,930	889	2.61	1.45	2.60	0.14	58	29.7	63	8.5
0.8- μm CA	3	38,961	1,202	12,191	1,411	7.08	0.92	2.75	0.21	82	3.2	56	—
1.2- μm CA	2	51,668	1,278	12,647	1,084	7.06	0.62	2.80	0.00	78	1.4	51	—
0.45- μm CN	2	7,511	1,669	2,844	899	1.68	0.11	2.50	—	58	—	66	—
0.45- μm NY	1	3,795	—	1,740	—	3.21	—	2.50	—	60	—	32	—
0.45- μm PTFE	2	—	—	—	—	—	—	—	—	—	—	—	—
TMP = 2.0 bar													
0.45- μm CA	3	18,327	998	6,857	1,530	2.2	0.76	2.65	0.21	75	3.5	76	—
0.45- μm CN	1	12,326	—	5,631	—	1.33	—	2.50	—	56	—	48	—
0.45- μm NY	1	15,323	—	6,712	—	1.45	—	2.20	—	60	—	31	—
0.45- μm PTFE	3	120	35	21	35	2.27	0.64	2.75	0.07	77	15.9	56	—

^a J_{max} = maximum flux; $J_{20\text{min}}$ = average flux in first 20 min of operation; NTU = nephelometric turbidity unit.

^b TMP = transmembrane pressure; CA = cellulose acetate; CN = cellulose nitrate; NY = nylon; and PTFE = polytetrafluoroethylene membranes.

Fig. 2. Scanning electron microscope images of new polymeric membranes: A, cellulose acetate (0.45 μm); B, cellulose nitrate (0.45 μm); C, polytetrafluoroethylene (0.45 μm); and D, nylon (0.65 μm).

Fig. 3. Flux behavior of different polymeric membranes (CA = cellulose acetate, CN = cellulose nitrate, NY = nylon, and PTFE = polytetrafluoroethylene) with a pore diameter of 0.45 μm under transmembrane pressure (TMP) of 1 or 2 bar.

Fig. 4. Turbidity rejection and color permeation obtained with different polymeric membranes (CA = cellulose acetate, CN = cellulose nitrate, NY = nylon, and PTFE = polytetrafluoroethylene) with a pore diameter of 0.45 μm under transmembrane pressure (TMP) of 1 or 2 bar.

According to Table II at a TMP of 1.0 bar, 1.2- μm CA had the highest flux and was one order of magnitude higher compared with that of 0.45- μm CA. However, the turbidity of the permeates from membranes with larger pore sizes (0.8 and 1.2 μm) were higher and approached the upper limit of 8.0 NTU (equivalent to 2 EBC) (12). Moreover, the more highly sensitive (haze active) proteins in the permeates would cause colloidal instability in the product (1,21). Among different types of membranes with the same pore size, 0.45- μm CA had the highest permeation with both TMP; 0.45- μm PTFE did not have any permeate at a TMP of 1.0 bar, and its flux at a TMP of 2.0 bar was far less than was observed for the other membrane materials. Turbidity and sensitive protein content were lowest for 0.45- μm CN, CA and PTFE showed better color permeation, and NY had the lowest content of polyphenols in the permeate. Scanning electron microscopy illustrated the structural differences among the membranes (Fig. 2).

Effects of Polymeric Membrane Material

Permeation flux behavior through different polymeric membranes with a pore diameter of 0.45 μm is shown in Figure 3. According to this results, at a TMP of 1.0 bar, CN and NY membrane flux behaviors were the same, CA had higher permeation flux, and PTFE did not show any permeation. At a TMP of 2.0 bar, there was no significant difference between the flux behavior of CA, CN, or NY, but PTFE behaved quite differently and unexpectedly. The flux behavior of PTFE membrane at a TMP of 2 bar increased initially

and, after reaching a maximum of approx. 80 $\text{L}/\text{m}^2\text{-hr}$, decreased. This phenomenon can be attributed to a shift in hydrophobicity in the PTFE matrix during filtration. The data corroborate the theory that under high pressure hydrophilic sites or zones began to develop within the matrix, which initially increased the flux and through filtration fouling eventually caused a drop in flux.

The objective of the clarification stage is to maintain the color while removing the maximum amount of haze-active proteins and particles (8,11). The average turbidity rejection and color permeation at two TMP (1.0 and 2.0 bar) for all four types of membranes with a pore diameter of 0.45 μm is shown in Figure 4. According to the results, the 0.45- μm CN membrane had the highest turbidity rejection at both TMP. Turbidity rejection increased at higher pressure for all four membranes (Fig. 4). This can be attributed to the compression of the filtration cake formed on the membrane, which acts as a secondary membrane and increases selectivity. CA and PTFE had better color permeation at both TMP compared with NY.

The colloidal stability of beer depends primarily on its haze-active (sensitive) proteins and polyphenol content (1,14,20,21). The effects of four membrane materials with a pore size diameter of 0.45 μm on the retention of these components at two TMP (1.0 and 2.0 bar) are shown in Figure 5. CN had the highest rejection of sensitive proteins at both pressures (Fig. 5). Despite low selectivity of sensitive proteins by NY, it had the highest rejection of polyphenols, due to the adsorptive property of NY to the polyphenols, which has been reported previously (10,11,16,19,22). The retention

Fig. 5. Rejection of haze-active materials from rough nonalcoholic beer with different polymeric membranes (CA = cellulose acetate, CN = cellulose nitrate, NY = nylon, and PTFE = polytetrafluoroethylene) with a pore diameter of 0.45 μm under transmembrane pressure (TMP) of 1 or 2 bar.

Fig. 6. Flux behavior of cellulose acetate membrane with different pore sizes at a transmembrane pressure of 1.0 bar.

Fig. 7. Relative flux of cellulose acetate membrane with different pore sizes at a transmembrane pressure of 1.0 bar.

of polyphenols was low for the other membranes and reduced at the higher pressure, indicating that the formation of the cake layer (acting as a secondary membrane) and its compactness at higher pressure did not influence the rejection of polyphenols. This may be due to the mechanism of polyphenol retention, which seems to be mainly adsorption on the membrane. The lowest rejection of sensitive proteins at a TMP of 2.0 bar was found for PTFE, which could be due to the hydrophobic nature of the PTFE and less interaction with hydrophilic haze-active proteins. This explanation agrees with previous work done on the subject (10,11).

Effects of Membrane Pore Size

The performance of CA membranes with pore sizes of 0.2, 0.45, 0.8, and 1.2 μm with regard to flux behavior and product quality was observed. Flux values for 0.2- and 0.45- μm CA were almost the same but much lower than those of 0.8- and 1.2- μm CA (Fig. 6). In spite of this significant difference, the changes in relative flux (the ratio of flux to maximum flux, which normally occurs at the beginning of microfiltration) were quite similar (Fig. 7). Relative flux was higher for smaller pore sizes, whereas larger pore size led to a higher rate of flux reduction (Fig. 7), which indicates that fouling was higher for larger pore diameters.

The effects of CA pore diameter on selectivity for turbidity, color, sensitive proteins, and polyphenols are shown in Figure 8. Accord-

ing to the results, rejection for all components (other than polyphenols) increased with smaller pore sizes. Turbidity retention for membranes with pore sizes of 0.2 and 0.45 μm was almost the same and higher than those membranes with pore sizes of 0.8 and 1.2 μm , but color permeation increased almost linearly with increasing pore size. Larger pore sizes (0.8 and 1.2 μm) did not have a large effect on sensitive (haze-active) proteins, but with smaller pore sizes of 0.45 and 0.2 μm , rejection of these proteins increased.

CONCLUSIONS

In this study the effects of the membrane material and pore size of polymeric membranes on permeability and selectivity in dead-end microfiltration of RNAB were observed and analyzed. An experimental pilot plant was used to evaluate CA membranes with pore sizes of 0.2, 0.45, 0.8, and 1.2 μm and CN, PTFE, and NY membranes with a pore size of 0.45 μm at two TMP (1.0 and 2.0 bar).

Flux values for CA, CN, and NY membranes with a pore size of 0.45 μm were almost the same at the two TMP, with CA showing slightly higher flux behavior. As an unexpected result, the 0.45- μm PTFE membrane did not allow any permeation at a TMP of 1.0 bar, whereas at a TMP of 2.0 bar, flux increased initially and, after reaching a maximum of approx. 80 L/m²·hr, decreased to a final flux value similar to the other membranes. This behavior can be attributed to

Fig. 8. Selectivity of cellulose acetate membrane with different pore sizes at a transmembrane pressure of 1.0 bar.

the hydrophobicity of the PTFE membrane and formation of hydrophilic sites during permeation. Despite the higher flux value for CA with 1.2- and 0.8-μm pore sizes than for those with 0.45- and 0.2-μm pore sizes, the flux ratio to maximum flux was higher for the CA membranes with smaller pore sizes.

CN had the highest rejection for turbidity and haze-active proteins, NY retained the most polyphenols, and CA had the highest color permeation. Higher pressure led to better turbidity removal, which could be due to the compression of the cake layer and the resultant increase in selectivity for suspended particles. Pore size had a diminishing effect on selectivity for turbidity and sensitive proteins, even though it had no obvious effect on polyphenol retention. Color permeation increased with the pore diameter of the CA membrane.

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