

PURIFICATION OF DIFFUSION JUICE WITH ULTRAFILTRATION  
CERAMIC MEMBRANE\*

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**Abstract.** The aim of the present study was to determine the usefulness of a FAIRLEY 0.2  $\mu\text{m}$  ceramic membrane for diffusion juice purification. Filtration was carried out with: distilled water, sucrose solution of 15% concentration, diffusion juice from sugar factory and diffusion juice obtained under laboratory conditions. It was found that 15% sucrose solution had by ca. 35% smaller filtrate flux than water, under the same conditions. Purity of juices after ultrafiltration did not depend on process temperature. Juice elimination efficiency and filtration efficiency depend on diffusion juice quality. Filtration of diffusion juice with a 0.2  $\mu\text{m}$  ceramic membrane resulted in an elimination efficiency similar to that of classical method. It seems that, in the future, filtration may replace, partly or totally, the classical purification of juice, which however still requires much research.

**Key words:** diffusion juice, ultrafiltration, ceramic membrane, elimination efficiency

INTRODUCTION

Membrane processes are quite broadly used in the food technology. The examples are: removal of bacteria from milk and whey, production of lactose and protein preparations, condensation of juices and fruit pulps, and others [11, 20].

With membranes, substance separation can be achieved up to the molecular level. For that purpose different techniques are used, depending on the properties of the molecules separated and on the driving force of the process. Among the processes, the following can be named: heat, electric, chemical, diffusion, and the

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most common, pressure processes [10]. Pressure difference is the driving force for microfiltration, ultrafiltration, nanofiltration and reversed osmosis. These methods are used for the purification and concentration of water solutions, the separation achieved being according to particle sizes.

The modern membrane production, from materials durable and resistant in technological process and at lower costs, opens up new prospects for their use in other branches of the food industry.

In the sugar industry, for diffusion juice purification, the processes of liming and carbonation are used. They are very energy consuming, and the side products are polluting the environment. The utilization of membrane separation for diffusion juice purification may eliminate the negative side effects to the environment, provided the following conditions are satisfied:

- 1) the purified juice obtained should be characterized by purity not lesser than that after the conventional method,
- 2) the yield of the process should be the largest and the least dependent on the duration of the process,
- 3) sucrose losses should be limited and not to exceed those of conventional liming and carbonation.

In order for the thin juice obtained from membrane filtration to be of adequate purity, the membranes applied must have their cut-off point larger than the molecular mass of sucrose (342), i.e. micro- or ultrafiltration membranes.

First studies on the application of membrane techniques for purifying diffusion juice were conducted by Madsen [13]. As a result of ultrafiltration of diffusion juice (of 88.9% purity) by using cellulose nitrate membranes, a juice of 91.5% purity was obtained, while after a standard process – 92.0%. Vern and co-workers [19] of the Dow Chemical Company proposed a technological line for diffusion juice purification based on membranes made of polyethersulfone and polyvinyl. From the data it follows that the juice purified was of 90.30% purity, while the diffusion juice – of 88.62% purity (the elimination efficiency only 16.35%). Sarka and co-workers [16] studied juice ultrafiltration with various membranes. They found that juice purity increased from 90.72 to 92.16% (elimination efficiency 30.7%), while the coloring decreased by 60-70%. Application of cellulose nitrate membranes allowed obtaining an up-to 40% elimination efficiency, depending on diffusion juice quality [15], while that effect for classical methods was 21 to 43% [21]. Thus it is possible, using the membrane techniques, to obtain an elimination efficiency comparable with that of the traditional method – though it depends much on the kind of membranes used and on the quality of the juice.

The efficiency of membrane processes is limited by the following phenomena: concentration polarization (concentration gradient at membrane surface)

and the so-called fouling – a permanent and often irreversible change in membrane permeation [10].

To a considerable degree, the fouling of membranes is caused by pectins [7].

The initial processing of juice can have an effect on filtration efficiency and membrane durability. Schrevel [17] found that before ultrafiltration it is necessary to remove the large particles of pulp and sand which can damage the membrane surface fast. Studies on ultrafiltration of juice from sugar cane allowed the conclusion that application of Alfa-Laval centrifuge for initial juice purification resulted in 2-fold increase in filtration efficiency and a similar extension of the time between flushing cycles of membranes [18]. A good result was also achieved from the use of filters made of stainless steel [5]. On the other hand, Hanssens [6] and Vern [19] stated that no processing of juice before ultrafiltration is needed. The difference in opinion of the authors might be due to differences in the quality of the juices filtered. It seems, however, that at least for the sake of membrane durability it is necessary to apply devices for removing particles that can damage membrane surface. The longer the filtration process, the greater number of authors advised initial processing of juice.

In studies on diffusion juice ultrafiltration described in the literature, membranes made of various materials were used – plastics (polyethylene, polysulfone, vinyl polychloride and others) and ceramic membranes. The latter are remarkable for their chemical resistance, long life, possibility of back-flushing and high temperature resistance. All those features are very important for application of the membrane in the sugar industry, rendering the ceramic membrane potentially the best. The aim of the present study was to determine the usefulness of a FAIRLEY 0.2  $\mu\text{m}$  ceramic membrane for diffusion juice purification.

## MATERIALS AND METHODS

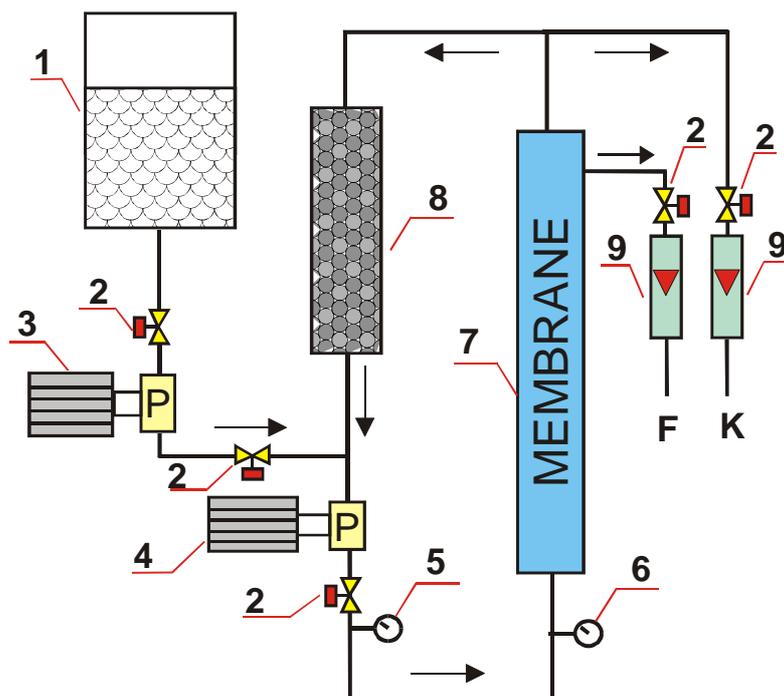
Filtration was carried out with:

- distilled water,
- sucrose solution of 15% concentration,
- diffusion juice from sugar factory (A),
- diffusion juice obtained under laboratory conditions (B).

Sucrose solution was made using distilled water and sucrose (refined sugar).

Diffusion juice (A) was taken from the “Wrocław” sugar plant in October of 2003. It was pre-filtered with a 0.07 mm filter. Diffusion juice (B) was obtained from dried beet pulp, sucrose (refined sugar), molasses and water. The pulp ( $150 \text{ g dm}^{-3}$  water) was heated with water for 30 minutes at  $80^\circ\text{C}$ . The supernatant was decanted and pre-filtered with a 0.07 mm filter, and then molasses was added ( $12 \text{ g l}^{-1}$  solution), and after thorough mixing sucrose was added at such an amount that the volume of the apparent dry substance in the juice was ca. 15%.

The investigation was conducted using the experimental setup shown schematically in Figure 1. A ceramic membrane module was applied, made by FAIRLEY, with 0.24 m<sup>2</sup> surface and 0.2 μm diameter.



**Fig. 1.** Diagram of filtration unit. 1 – feed, 2 – valves, 3 – inlet pump, 4 – circulation pump, 5 – temperature sensor, 6 – manometer, 7 – membrane, 8 – heater, 9 – rotameters

The filtration process of water and 15% sucrose solution was conducted using the pressures of 0.20, 0.25 and 0.30 MPa, and temperatures of 20-65°C. Filtration of diffusion juices was done at 0.25 MPa and 30-58°C. In each case the concentrate flux was at constant level – 130 l h<sup>-1</sup> m<sup>-2</sup>.

In the filtrates obtained the following were assayed [3]:

- dry real mass content by the thermogravimetric method
- sucrose content by the polarimetric method

The purity of the juices was calculated based on the sucrose and dry mass content, and the purification effect using the formula:  $E = 10000 \cdot (C_o - C_d) / (C_o(100 - C_d))$  where:  $E$  – elimination efficiency (%),  $C_o$  – purity of purified juice (%),  $C_d$  – purity of diffusion juice (%).

During filtration, the filtrate flux was also determined using a rotameter.

The results were statistically processed with Statistica 6.1 package, Tukey's test.

## RESULTS AND DISCUSSION

The ceramic membrane applied is commercially available. Its modular structure enables constructing installations of practically unlimited surface. Practical modules are of 0.06 to 6.48 m<sup>2</sup> area [8]. Determination of the properties of a single module is necessary for designing an installation of affective surface suitable for a given production scale.

Figure 2 shows the effect of temperature on the efficiency of water filtration. The applied pressures are 0.20, 0.25 and 0.30 Mpa, and temperatures in the range of 20-65°C. The relationships obtained are expressed by polynomials of the second order. The data presented in the figure show that filtration efficiency increases with temperature, though the increase is not proportional to temperature. Similar dependences were described by Sarka et al. [16]. The maximal possible temperatures and pressures are limited by pump cavitation. The results of measurements on water filtration are also a source of reference for determination of membrane fouling after filtration of diffusion juices. On that basis, the time of stopping the process of membrane flushing was also determined.

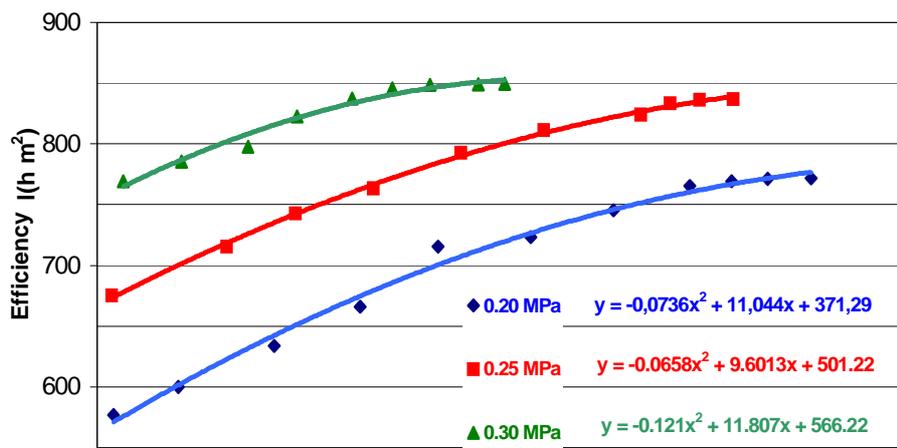
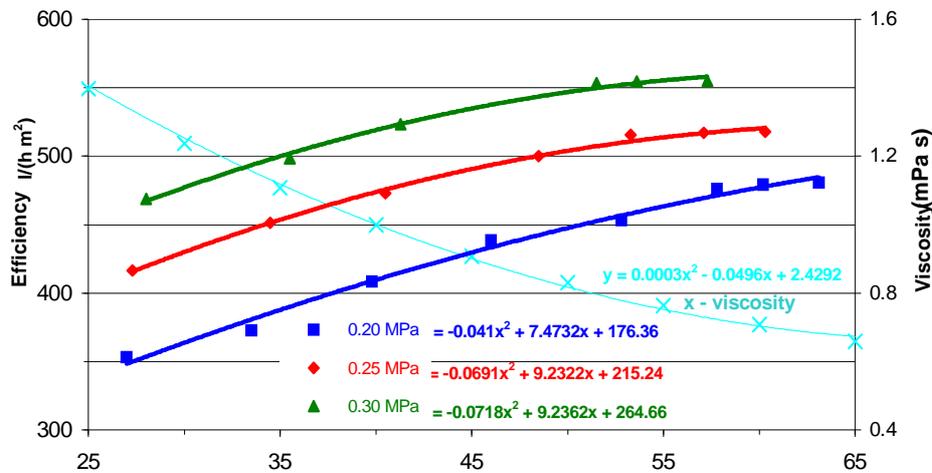


Fig. 2. The effect of temperature on efficiency of water filtration

Figure 3 shows the effect of temperature on the filtration efficiency of 15% sucrose solution. The pressures applied: 0.20, 0.25 and 0.30 MPa at 26-64°C. The relations obtained are described with second-order polynomials. The equations describing the dependence of efficiency on temperature, like in the case of water filtration, indicate that the relation is not linear. The efficiency of 15% sucrose

solution, for the same parameters of pressure and temperature, was ca. 62-65% of that for water. The molecular size of sucrose is markedly smaller than the membrane pore dimensions, and thus had no effect on efficiency. For comparison, in Figure 3 presents also the viscosity of 15% sucrose solution at same temperatures [9]. For the temperatures used, that dependence is fairly well represented by a second-order polynomial. The decrease in filtrate flux, as compared with the flux of pure water, is to a large degree due to changes in viscosity of the solution.



**Fig. 3.** The effect of temperature on filtration efficiency of 15% sucrose solution and viscosity of this solution [9], the applied pressures: 0.20; 0.25 and 0.30 MPa

The results on filtration of diffusion juice from sugar factory (A) and that produced in laboratory conditions (B) are presented in Table 1. Diffusion juice from factory (A) was characterized by 15.49% dry mass content, 13.6% sucrose and 87.80% purity, thus being similar to juices obtained under industrial conditions [21]. In order to get a more extensive characteristic of the membrane, juice (B) was obtained in laboratory conditions. Its quality was substantially worse. The dry mass content in that juice was 14.51%, sucrose 12.05% and purity only 83.05%. Such parameters are characteristic of diffusion juices obtained from roots of poor technological quality (e.g. damaged by frost). Based on the statistical analysis performed, it was found that the filtration temperature had no effect on dry mass and sucrose content in the juices. So, the mean value of juice purity after filtration and the mean elimination efficiency were calculated, the latter being 34.3% for juice A and 27.2% for juice B. Such a procedure is in accord with the recommendations by Dobrzycki [4]. The difference between the elimination

efficiency for juice from sugar plant (A) and juice (B) was ca. 7%. Similar relationships apply for diffusion juice purified by the classical method – the greater the diffusion juice purity the greater its elimination efficiency [4]. With classical juice purification, the elimination efficiency is usually 28-35%, rarely over 40% [21]. Thus it can be stated that the application of the 0.2  $\mu\text{m}$  ceramic membrane allows obtaining an elimination efficiency similar to that of the traditional method.

**Table 1.** The influence of ultrafiltration temperature on the content of dry mass and sucrose in diffusion juice before and after purification

Juice from sugar factory (A)				Laboratory juice (B)			
Temperature (°C)	Dry mass (%)	Sucrose (%)	Purity (%)	Temperature (°C)	Dry mass (%)	Sucrose (%)	Purity (%)
28.0	13.98	12.7	90.84	30.5	12.91	11.1	85.98
33.5	13.97	12.7	90.91	37.5	12.89	11.1	86.11
38.5	13.95	12.8	91.76	42.5	12.88	11.2	86.96
43.5	13.90	12.8	92.09	47.5	12.87	11.2	87.02
48.5	13.91	12.8	92.02	52.0	12.86	11.3	87.87
52.0	13.92	12.8	91.95	54.1	12.87	11.3	87.80
58.1	13.93	12.8	91.89	56.6	12.89	11.3	87.66
mean	13.94	12.8	91.64	mean	12.88	11.2	87.06
diffusion juice	15.49	13.6	87.8	diffusion juice	14.51	12.05	83.05

The dependence of juice filtration efficiency on temperature is shown in Figure 4. Like for water and sucrose solutions, it is described by second-order polynomials. The efficiencies obtained were 4-5 times smaller than for filtration of sucrose solutions. In spite of smaller dry mass content, the flux of juice (B) filtrate was always smaller than that of juice (A) by ca. 4-10%. That difference was due to a greater amount of non-sugars, which is testified by the lower purity of juice (B). The obtained fluxes of purified juice, at the level of  $130 \text{ l m}^{-2} \text{ h}^{-1}$ , are fairly high compared with literature data. Bubnik et al. [2], using a 20 nm ceramic membrane (pressure 0.1 MPa, temperature 22-24°C), obtained the filtrate flux at  $70 \text{ l m}^{-2} \text{ h}^{-1}$ .

The filtration efficiency can be hampered by the concentration polarization phenomenon or fouling. The results obtained so far, however, indicate that concentration polarization and fouling are not substantial [2, 19]. These harmful effects can be restricted by the application of the so-called back-flushing [12] or modification of the membrane surface [1,14]. It seems that, in the future, filtration

may replace, partly or totally, the classical purification of juice, which however still requires much research. The advantages of that process are: diminished environmental pollution, lower energy consumption and relatively low costs of membrane utilization.

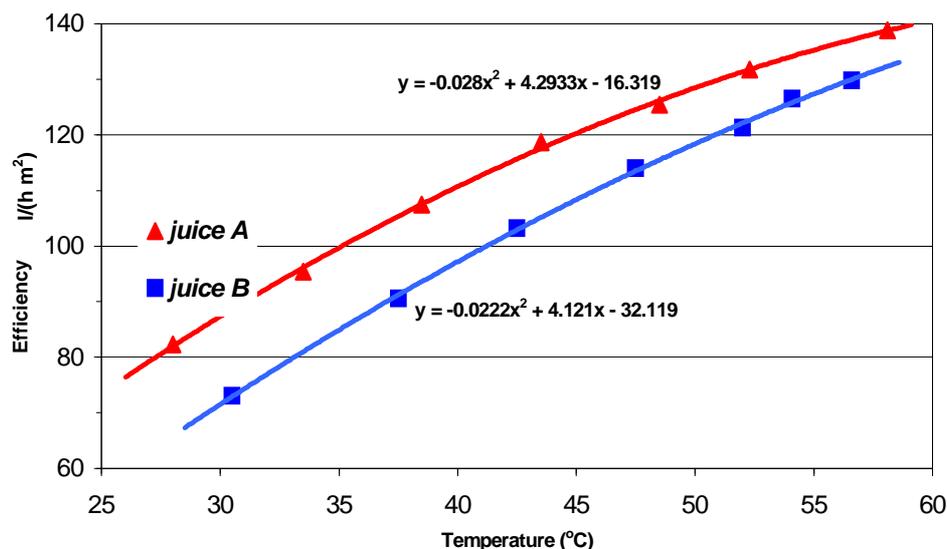


Fig. 4. The dependences of juice filtration efficiency on temperature

## CONCLUSIONS

1. 15% sucrose solution had by ca. 35% smaller filtrate flux than water, under the same conditions.
2. Purity of juices after ultrafiltration did not depend on process temperature.
3. Juice elimination efficiency and filtration efficiency depend on diffusion juice quality.
4. Filtration of diffusion juice with a 0.2  $\mu\text{m}$  ceramic membrane resulted in an elimination efficiency similar to that of the classical method.

## REFERENCES

1. **Akhtar S., Hawes C., Dudley L., Reed I., Stratford P.:** Coatings reduce the fouling of micro-filtration membranes. *Journal of Membrane Science*, 107, 209-218, 1995.
2. **Bubnik Z., Hinkova A., Kadlec P.:** Cross-flow micro- and ultrafiltration applied on ceramic membranes in impure sugar solutions. *Czech J. Food Sci.*, 1, 29-32, 1998.
3. **Butwiłowicz A.:** Analytical methods of production control in the sugar industry (in Polish). Warszawa, Instytut Przemysłu Cukrowniczego, 1997.

4. **Dobrzycki J.:** Juice elimination efficiency (in Polish). *Gazeta Cukrownicza*, 10, 153-156, 1982.
5. **Ghosh A., Balakrishnan M., Dua M., Bhagat J.:** Ultrafiltration of sugarcane juice with spiral wound modules: on-site pilot trials. *Journal of Membrane Science*, 174, 205-216, 2000.
6. **Hanssens T., van Nispen J., Koerts K., de Nie L.:** Ultrafiltration as an alternative for raw juice purification in the beet sugar industry. *Int. Sugar Journal*, 86, 228-243, 1984.
7. **Hatziantoniou D., Howell J.:** Influence of the properties and characteristics of sugar beet pulp extract on its fouling and rejection behaviour during membrane filtration. *Desalination*, 148, 62-72, 2002.
8. <http://www.faireyceramics.com/cpp/microfiltration/index.htm>
9. <http://www.univ-reims.fr/Externes/AVH/MementoSugar/001.htm#refs>
10. **Kołodziejczyk A.:** The efficiency of pressure membrane processes (in Polish). Wrocław, Oficyna Wydawnicza Politechniki Wrocławskiej, 1996.
11. **Kroll J.:** Application of membrane processing in apple juice concentration (in Polish). *Przemysł Spożywczy*, 8, 56-57, 2001.
12. **Ma H., Bowman Ch., Davis R.:** Membrane fouling reduction by backpulsing and surface modification. *Journal of Membrane Science*, 173, 191-200, 2000.
13. **Madsen R.:** Ultrafiltration as a method for juice purification. *Zuckerindustrie*, 12, 612-614, 1971.
14. **Moritz T., Benfer S., Arki P., Tomandl G.:** Influence of the surface charge on the permeate flux in the dead- end filtration with ceramic membranes. *Separation and Purification Technology*, 25, 501-508, 2001.
15. **Regiec P.:** Attempt to use cellulose nitrate membrane for the purification of diffusion juice. *Acta Agrophysica*, 2, 407-415, 2003.
16. **Sarka E., Smolik J., Gebler J., Bubnik Z.:** Ultrafiltration of raw juice. 21-st General Assembly CITS May 24-28, 1999 Antwerp.
17. **Schrevel G.:** Membrane filtration on beet raw juice and prelined juice. *Zuckerindustrie*, 5, 386, 2001
18. **Vercelotti J., Clarke M., Godshal M., Blanco R., Patout W., Florence R.:** Chemistry of membrane separation processes in sugar industry applications. *Zuckerindustrie*, 9, 736-745, 1998.
19. **Vern C.:** The beet sugar factory of the future. *International Sugar Journal*, 1159, 310-314, 1995.
20. **Witrowa-Rajchert D.:** Membrane processes in food technology (in Polish). *Przemysł Spożywczy*, 8, 52-55, 2001.
21. **Zero M.:** Information on production and technical data in the sugar industry 2002/2003 (in Polish). Warszawa, Instytut Przemysłu Cukrowniczego, 2003.

## OCZYSZCZANIE SOKU DYFUZYJNEGO ZA POMOCĄ ULTRAFILTRACYJNEJ MEMBRANY CERAMICZNEJ

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**Streszczenie.** Celem badań było określenie przydatności ceramicznej membrany FAIREY 0,2  $\mu\text{m}$  do oczyszczania soku dyfuzyjnego. Filtracji poddano: wodę destylowaną, roztwór sacharozы o stężeniu 15%, sok dyfuzyjny z cukrowni oraz sok dyfuzyjny otrzymany w warunkach laboratoryjnych. Stwierdzono, że 15% roztwór sacharozы odznaczał się o około 35% mniejszym strumieniem filtratu niż woda, w tych samych warunkach filtracji. Czystość soków po ultrafiltracji

nie zależała od temperatury procesu. Efekt oczyszczania soku i wydajność filtracji zależały od jakości soku dyfuzyjnego. Filtracja za pomocą membrany ceramicznej 0,2 mm, pozwoliła na uzyskanie podobnego efektu oczyszczania soku dyfuzyjnego, co metoda klasyczna. Wydaje się, że ultrafiltracja może w przyszłości zastąpić, częściowo lub całkowicie, klasyczne oczyszczanie soku, wymaga to jednak jeszcze wielu badań.

Słowa kluczowe: sok dyfuzyjny, ultrafiltracja, membrana ceramiczna, efekt oczyszczania