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CONCENTRATION OF APRICOT JUICE USING COMPLEX MEMBRANE TECHNOLOGY

E. Fogarassy[a], E. Bekassy-Molnar[a]*, Cs. Balla[b] and Gy. Vatai[a]

[a]Department of Food Engineering, Faculty of Food Science, Corvinus University of Budapest, H-1118 Budapest, Ménesi út 44. Hungary
[b]Department of Refrigeration and Livestocks' Products Technology, Faculty of Food Science, Corvinus University of Budapest, H-1118 Budapest, Ménesi út 43-45. Hungary

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In this study, pressed apricot (*Prunus armeniaca* L.) juice was concentrated using complex membrane technology with different module combinations: UF-RO-OD, UF-RO-MD, UF-NF-OD and UF-NF-MD. In case of the best combination a cross-flow polyethylene ultrafiltration membrane (UF) was applied for clarification, after which pre-concentration was done using reverse osmosis (RO) with a polyamide membrane, and the final concentration was completed by osmotic distillation (OD) using a polypropylene module. The UF-RO-OD procedure resulted in a final concentrate with a 65–70 °Brix dry solid content and an excellent quality juice with high polyphenol content and high antioxidant capacity.

Nanofiltration (NF) and membrane distillation (MD) were not proper economic solutions.

The influence of certain operation parameters was examined experimentally. Temperatures of UF and RO were: 25, 30, and 35 °C, and of OD 25 °C. Recycle flow rates were: UF: 1, 1.5, and 2 m^3 h^{-1}; RO: 200, 400, and 600 l h^{-1}; OD: 20, 30 and 40 l h^{-1}. The flow rates in the module were expressed by the Reynolds number, as well. Based on preliminary experiments, the transmembrane pressures of UF and RO filtration were 4 bar and 50 bar, respectively. Each experimental run was performed three times. The following optimal operation parameters provided the lowest total cost: UF: 35 °C, 2 m^3 h^{-1}, 4 bar; RO: 35 °C, 600 l h^{-1}, 50 bar; OD: 20, 30 and 40 l h^{-1}; temperature 25 °C.

In addition, experiments were performed for apricot juice concentration by evaporation, which technique is widely applied in the industry using vacuum and low temperature.

For description the UF filtration, a dynamic model and regression by SPSS 14.0 statistics software were applied.

Keywords: apricot juice, ultrafiltration, reverse osmosis, osmotic distillation, evaporation, economic evaluation

Apricot (*Prunus armeniaca* L.) is the third most commonly produced stone fruit. The main areas of production are the Mediterranean countries, which contribute 40% of the world's supply (Versari et al., 2008). Hungary also occupies a respectable place among producer countries. The apricot is used for consumption as fresh fruit, jams and preserves, juice, conserve, and for making alcoholic spirits. The nature of apricot means that it is only available for fresh consumption during the ripening period of 4–5 weeks. It is a delicate fruit that tends to go bad quickly. The harvested fruit endures only a short storage time of 2–3 d at room temperature. The food industry often produces a concentrated apricot juice that contains the fibres of the fruit. With membrane technology the apricot liquor is concentrated without fibres, but after the concentration the sterilized squeezed fibre can be added to the juice.

* To whom correspondence should be addressed.
Phone: 36-1-482-6233; fax: 36-1-482-6223; e-mail: erika.bekassy@uni-corvinus.hu

The greatest percentage of the edible portion of an apricot is water, followed by carbohydrates, organic acids, amino acids, and phenolic compounds. The most common acids found in apricot are malic acid and citric acid (Bureau et al., 2009). The rest of the ingredients are cellulose, fibre, mineral salts, trace elements, pectin, tannic acid, and colouring and volatile components. In addition to trace elements (iron, copper, manganese, zinc, chromium, and selenium), in recent times the influence of heavy metals, as pollutants, has been realised in the makeup of the fruits (Saracoglu et al., 2009).

For traditional concentration of fruit juices, pressed juice is subjected to heat treatment. Modern processes generally use simple flow-through evaporators for concentration; while heat may damage the valuable components and flavours of fruit juice (Ashurst, 2005).

Cryoconcentration (Aider & De Halleux, 2008) is another method in which heat is removed from the fruit until it is frozen, then formed ice crystals are physically separated. The advantage of this treatment is that it occurs at low temperature (below 0 °C), but the disadvantages are the loss of juice remaining in the ice crystals and the considerably high cost of the deep-freezing.

Membrane technology has an advantage over cryoconcentration and evaporation: it is performed at room temperature and requires low energy, meaning that vitamins, antioxidants, and other valuable elements are not damaged during processing. In our research group the concentration of different fruit juices (grape, black currant, red currant, cherry, apple, raspberry, etc.) was performed by membrane techniques (Vincze et al., 2006, 2007; Kozák et al., 2007; Rektor et al., 2007; Banvolgyi et al., 2009; Molnar et al., 2012). The behaviour of the fruits is different during the processing, therefore it is very important to determine experimentally what sort of membrane operations, how many membrane steps, which operational parameters give the best result. There are numerous membrane operations, each of them differing in the porosity of the membrane and the applied driving force. Microfiltration (MF) can be used for clarification and sterilization of juices (Gomes et al., 2013). On the other hand, numerous studies have involved ultrafiltration (UF) for sterilization of fruit juices (He et al., 2007; Tasselli et al., 2007; Jaeger De Calvalho et al., 2008), because UF is a more reliable way to clarify the juice microbiologically. Nanofiltration (NF) and reverse osmosis (RO) are applied in fruit juice pre-concentration (Vincze et al., 2006, 2007; Kozák et al., 2007; Rektor et al., 2007), whereas osmotic distillation (OD) and membrane distillation (MD) are applied in the final concentration of juice (Rektor et al., 2006; Kozák et al., 2007; Kujawski et al., 2013).

1. Materials and methods

The Magyar Gönczi apricot (*Prunus armeniaca* L.) species, purchased from an original Hungarian producer, was used in the experiments. After sorting, washing, and removal of solid particles, the fruit was chopped and enzyme-treated. Enzyme treatment is necessary to break down pectin. The influence of three types of enzyme was examined, using the data of Novozymes Switzerland AG (Table 1.), and at room temperature. These enzymes are used in the pectin decomposition of grapes (Biozym XP), apple and pear (Pectinex YieldMASH), and blackcurrant and strawberry (Pektopol PT-400). Room temperature (25 °C) was chosen to protect the valuable components. Following each of the three enzyme treatments, the treated juice was pressed with pneumatic fruit press and the effects were examined. From the standpoint of juice yield (fresh apricot juice pressed from seedless fruit) Pectinex YieldMASH enzyme proved best.

Table 1. Relative effects of enzymes used for breakdown of pectin

Enzyme	Amount ($g\ l^{-1}$)	Time required for breakdown (min)	Temperature (°C)	Yield (%)
Biozym XP	0.002	120	16	30
Pektopol PT-400	0.09	30	25	55
Pectinex YieldMASH	0.06	30	25	70

Combined membrane technology was applied for concentration of apricot juice in batch mode (Fig. 1.): UF sterilization, RO pre-concentration, and OD final concentration processes were carried out. All together 300 kg apricot was processed. First UF was used for sterilization at 3 different temperatures and 3 different flow rates. Then the 9 sterilized UF-permeates were pre-concentrated with RO at 3 different temperatures and 3 different flow rates. The final concentrations of the 9 retentates were reached by OD using the optimal temperature and pressure. In all 9 cases 65–70 °Brix was achieved, therefore the high final concentrations are trusty and accurate.

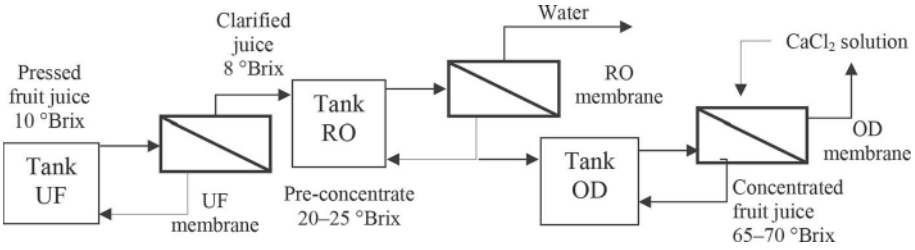

Fig. 1. Combined membrane technology for concentration of apricot juice

On the basis of a series of preliminary experiments, UF-RO-OD, UF-RO-MD, UF-NF-OD, and UF-NF-MD systems were experimentally tested. In another experiment instead of UF, MF membrane was tested, but the UF provided the proper sterilization and clarification, therefore the MF was omitted. RO achieved higher retentate concentration and the apricot loss was negligible, consequently the NF was not used in the complex system. The OD produced higher final concentrate without damaging the valuable components, while the MD, which needed a higher temperature to be a feasible process, was left out. Finally, the UF-RO-OD system was found to be optimal for apricot juice concentration. For membrane filtrations (UF, RO), the transmembrane pressure difference is the driving force of the process, and the recirculation of the retentate by pump completes it. In OD the driving force is the difference of concentration between the two sides of the membrane (Mulder, 1997; Cheryan, 1998). In the experiments membranes of different material, manufacturer, and surface were used, and Table 2 shows the main data of membrane modules. The producer was the Hidrofilt Co, Hungary.

Table 2. Types of laboratory membranes (the producer was the Hidrofilt Co, Hungary)

Membrane type	Manufacturer	Membrane shape and material	Membrane area (m^2)
UF	Berghof	Tubular/polyethylene (PE)	0.41
RO	Trisep	Flat/polyamide (PA)	0.18
OD	Microdyn	Capillary tube/polypropylene (PP), hydrophobic	0.10

In the first step, the pressed, fresh apricot juice with about 10 °Brix concentration was filtered by ultrafiltration (UF), at which point the suspended particles and microorganisms were removed from the juice. The sediment-free UF permeate was the feed of the RO. The retentate of RO, the pre-concentrated juice with about 20–25 °Brix, was pumped into the tubes of the capillary OD membrane module, resulting in an approximately 65–70 °Brix final concentrate of apricot juice.

On the shell side of the OD module 70 °Brix $CaCl_2$ solution was circulated. The water from the tube side through the hydrophobic membrane permeates to the shell side in the form of vapour. A scale connected to a computer was used to weigh the permeate accumulated at the $CaCl_2$ side.

Batch mode was applied and the influences of various process parameters were examined (Table 3). The temperature during the experiments was kept constant by a heating-cooling system. In each case, temperature was below 35 °C, in order to save the valuable materials in the concentrate.

Table 3. Process parameters

Process	Temperature (°C)	Volumetric recycle rate	Reynolds number in the module	Transmembrane pressure (bar)
UF	25;30;35	1; 1.5; 2 $m^3\ h^{-1}$	4278–9355	4
RO	25;30;35	200;400;600 $l\ h^{-1}$	1010–3140	50
OD	25	40 $l\ h^{-1}$	283	–

Each experimental run was performed three times. The standard deviations were calculated by the SPSS 14.0 program.

Traditional juice concentration was also done to compare the methods from the economic point of view. Two evaporation experiments with fresh apricot juice were carried out at laboratory scale (we had no possibility to evaporate at lower pressure, as it is applied in the industry):

a) 1 bar pressure (105 °C), Elettronica Veneta Film Evaporator, Italy

b) 0.45 bar pressure (79 °C), MTA Kutesz LD601 Rotadest, Hungary

As quality parameters, polyphenol content (Folin-Ciocalteu reagent, according to Singleton and Rossi, (1965)) and antioxidant capacity (the method of Benzie and Strain, (1999)) were measured before and after complex membrane operations, and before and after evaporations. Total solid content and two valuable components were analysed: the total solid content was measured by digital refractometer (Atago Pal-α), the polyphenol concentration by spectrophotometer (λ=760 nm), and the antioxidant activity by spectrophotometer (λ=593 nm).

For the description of the UF process, the permeate flux in function of time (J ~ t) was calculated using the following dynamic model:

$$\frac{J - J_0}{J_{SS} - J_0} = 1 - e^{-\frac{t}{T_i}} \quad (1)$$

where J_0 is the initial permeate flux (l m^{-2} h^{-1}), J_s is the nearly steady state permeate flux (l m^{-2} h^{-1}), t is the time (h), T_i is the time constant (h) that expresses the ease of completion of the process. The faster the process, the lower the T_i value is.

2. Results and discussion

2.1. Sterilization and clarification with ultrafiltration (UF)

The pressed apricot juice was filtered with a UF membrane, the temperature was set at 25, 30, and 35 °C, while the transmembrane pressure was maintained at 4 bar, and under these conditions the change of permeate flux was noted.

Figure 2 shows the ultrafiltration permeate fluxes as a function of time, at constant temperature and at various pressures using different recycle flow rates (1, 1.5, 2 m^3 h^{-1}). The strictly controlled temperature (e.g. at UF 30±0.5 °C) provided a narrow range of deviation in flux curves. It can be observed that an increase of approximately 0.5 m^3 h^{-1} produces an average increase of 25% in permeate flux. Figure 3 shows results obtained at various temperatures with recirculation flow rate as a constant and time as a variable. The temperature has a significant effect: 5 °C increase generates approximately 30% increase in the initial permeate flux. It can be stated that in the measured range the influence of temperature and flow is significant. The greater the recycled retentate, the faster the filtration. A decrease of total suspended solid concentration (2 °Brix, Fig. 1) was observed during UF filtration, along with a slight decrease of polyphenol content and antioxidant capacity (5–10%). The UF retentate of the concentrated juice is not a waste, but valuable material, which can be applied in the food industry after proper sterilization.

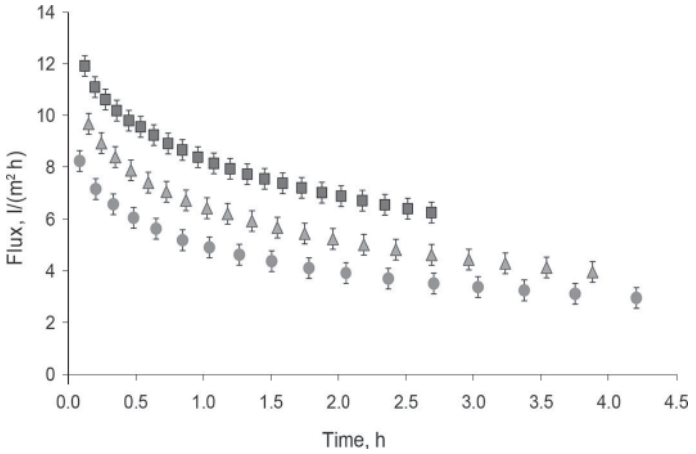

Fig. 2. Filtration of raw juice at constant temperature with ultrafiltration membrane (ultrafiltration 4 bar, 30 °C). □: 2 m^3 h^{-1}; △: 1.5 m^3 h^{-1}; ●: 1 m^3 h^{-1}

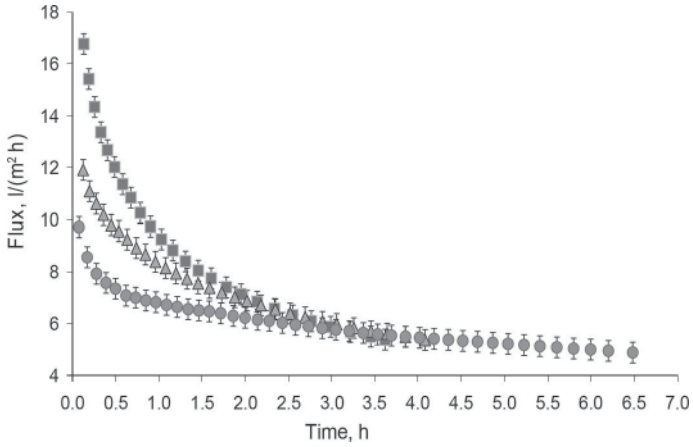

Fig. 3. Raw juice filtration with ultrafiltration membrane at constant recycle flow rate (Ultrafiltration 4 bar, 2 $m^3 h^{-1}$). □: 35 °C; △: 30 °C; ●: 20 °C

2.2. Pre-concentration with reverse osmosis (RO)

Juice permeates from the ultrafiltration membranes were concentrated to 20–25 °Brix dry content, using reverse osmosis (RO). In order to ensure the high osmotic pressure, a transmembrane pressure difference of 50 bar was applied. Clear water (0–0.3 °Brix dry solid content) came out on the permeate side. In order to determine optimal operation parameters, permeate flux was measured. Starting at 15 bar, pressure was increased in 5 bar increments up to 50 bar. It was not possible to take measurements under 15 bar, since the osmotic pressure of the apricot juice prevented making the amount of permeate necessary for measurement.

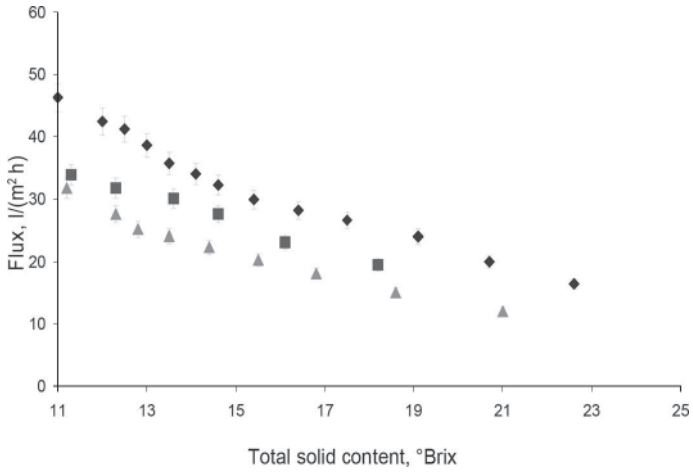

Fig. 4. Clarified juice pre-concentration with reverse osmosis at various temperatures and constant recycle flow rate (pre-concentration with reverse osmosis 50 bar, 600 l h^{-1}). ▲: 25 °C; ■: 30 °C; ◆: 35 °C

At 35 °C, 50 bar, the initial permeate flux value was 48 l m^{-2} h^{-1}, and because the steady state was low, we assumed 50 bar as a standard value for every pre-concentration test. The influence of the temperature and recycle flow rate has been studied in juice pre-concentration experiments using RO. Compared to ultrafiltration, in RO there was no great increase of permeate flux at higher temperature. Here, 5 °C increase in temperature (between 30 °C and 35 °C) resulted in a ~20% rise, whereas between 25 °C and 30 °C only a ~10% larger permeate flux appeared (Fig. 4).

Tests were performed to determine the effect of temperature and recycle rate on RO concentration. Figure 5 shows flux values based on solid content, while in Fig. 6 the increase of polyphenol content and antioxidant capacity can be seen. Concentration was examined using 25 °C and 50 bar transmembrane pressure difference at various recycle flow rates. No large changes in permeate flux were noted at either high or low temperatures. There was no loss of valuable components during RO (Fig. 6). The RO is a relatively cheap concentration method, therefore producers could aim for the highest possible concentration with it.

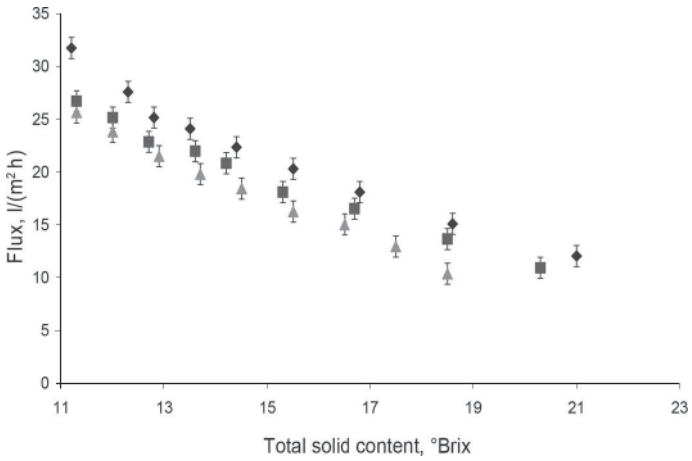

Fig. 5. Clarified juice pre-concentration with reverse osmosis at various recycle flow rates (pre-concentration with reverse osmosis 50 bar, 25 °C). ▲: 200 l h^{-1}; ■: 400 l h^{-1}; ◆: 600 l h^{-1}

2.3. Final concentration with osmotic distillation (OD)

The retentate, which was previously concentrated by RO, was further concentrated using OD. Like reverse osmosis, this procedure also reduces water content. Its advantage is that there is no need for a large pressure difference between the two sides of the membrane, and its disadvantage is the application of salt solution and the cost of salt regeneration. With OD a 65-70 °Brix solid content final concentrate could be produced, while the permeate solid content was ~ 0 °Brix and 10–30% loss of valuable components was observed (Fig. 6), which was similar to the result of reverse osmosis.

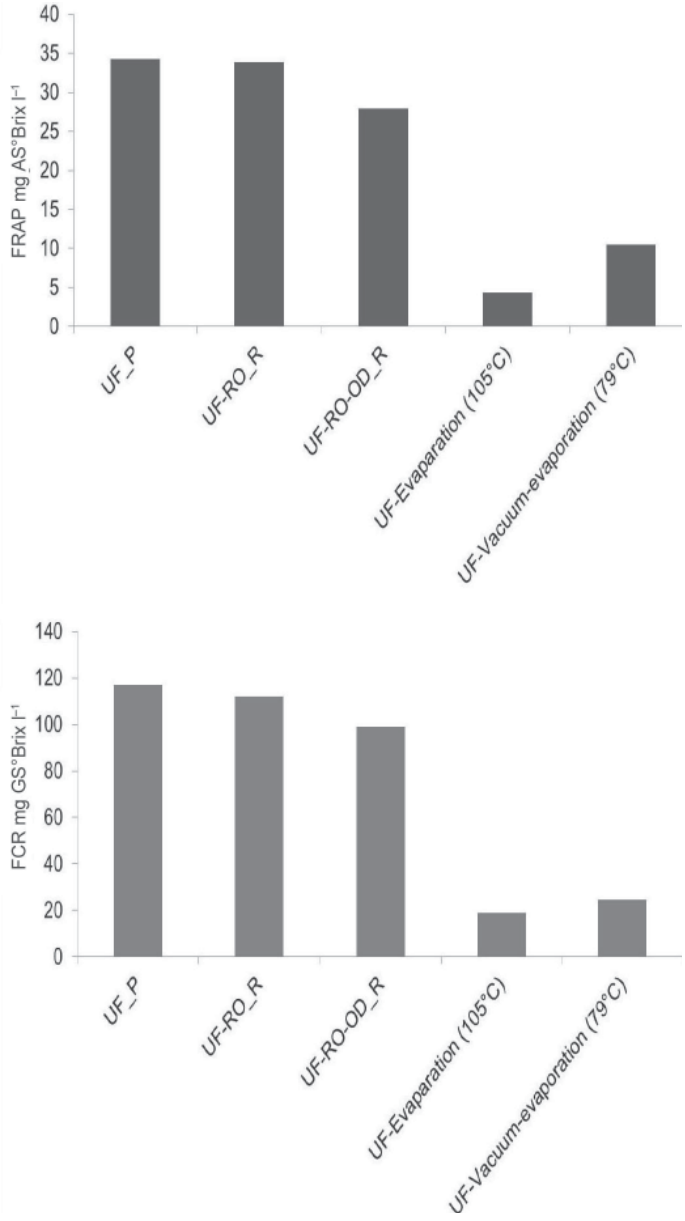

Fig. 6. Antioxidant capacity (FRAP) and total polyphenol content (FCR) in the products – related to dry matter – after membrane operation steps and evaporations

Figue 7 displays OD permeate flux and concentrate content results in relation to different recycle flow rates. Flux values ranged from 2.2–1.5 kg m^{-2} h^{-1} to ~ 1.1 kg m^{-2} h^{-1}. The experiments with OD were performed in closed loop to decrease the loss of volatile components (Cissé et al., 2011; Hasanoglu et al., 2012).

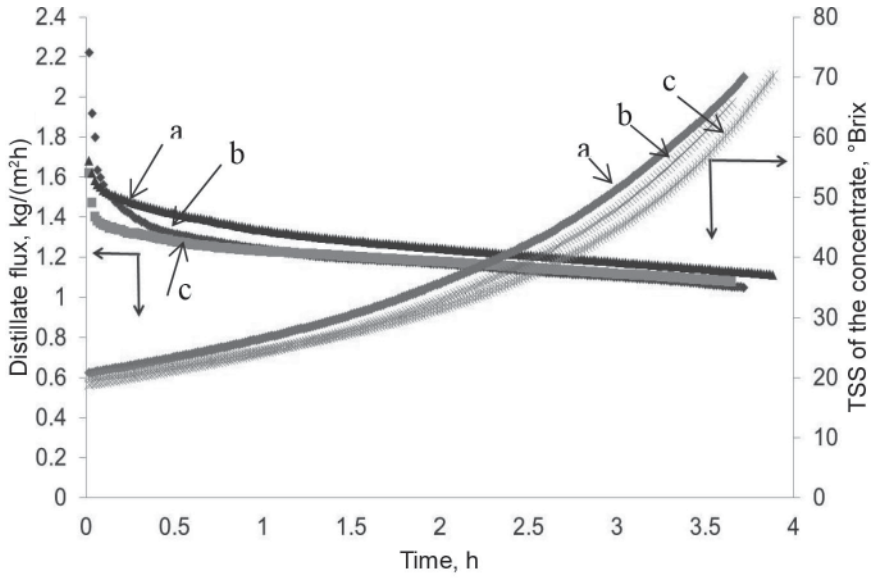

Fig. 7. Distillate flux and final TSS concentration of pre-concentrated juice with osmotic distillation (a: 25 °C, 20 l h^{-1}; b: 25 °C, 30 l h^{-1} ; c: 25 °C, 40 l h^{-1})

2.4. Evaporation

During evaporation, 65–70% solid content was produced for comparison of parameters with the complex membrane system. Figure 6 shows the total polyphenol content (FCR) and the antioxidant capacity (FRAP) of the different products. The evaporation values serve only for comparison of the different methods. By evaporation, 62–84% loss of valuable components was observed, therefore the complex membrane system is recommended if one wishes to produce a healthy apricot juice

2.5. Regression of a dynamic model for ultrafiltration (UF)

Laboratory experiments provided the model parameters and the time constant was determined from the logarithmic form of Eq. (1) (Fig. 8).

The time constant is related to the operation parameters (temperature and recirculate flow rate). Based on our experiments, this relationship was estimated with the following equation:

$$T_i = B \cdot Re^c \cdot \left(\frac{T}{T_0}\right)^d \tag{2}$$

where T_i is the time constant (h), Re is the Reynolds number, T is the temperature (°C), T_0 is the reference temperature (25 °C), and B, c, and d are constants.

Using the logarithmic form of Eq. 1, the time constants were calculated and provided the following (3):

$$T_i = 86.4 \cdot Re^{-0.52} \cdot \left(\frac{T}{T_0}\right)^{2.31} \tag{3}$$

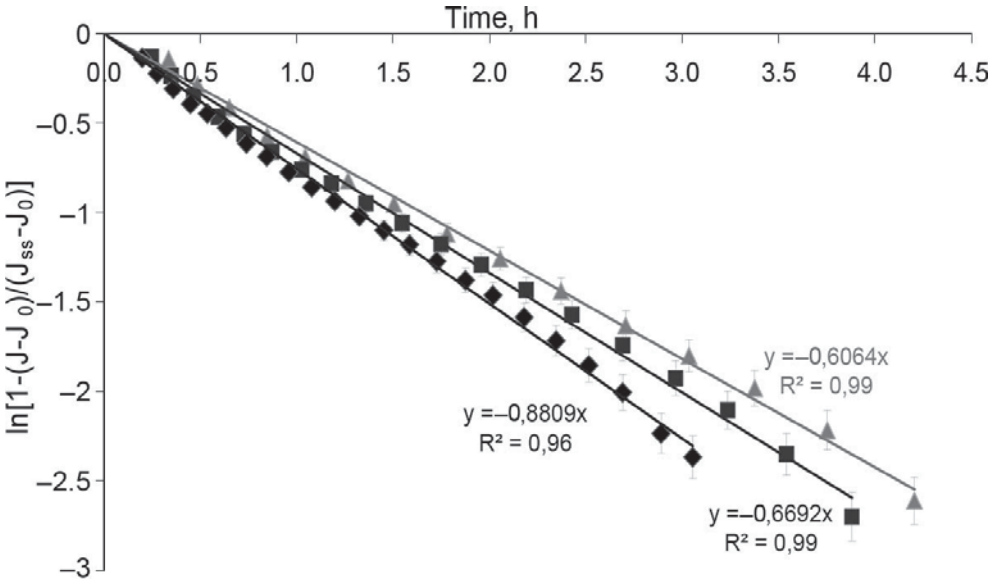

Fig. 8. Calculation of time constants of UF permeate flux at constant temperature and different recycle flow rates (▲: UF 30 °C, 1 $m^3 h^{-1}$; ■: UF 30 °C, 1.5 $m^3 h^{-1}$; ◆: UF 30 °C, 2 $m^3 h^{-1}$)

As an example, Table 4 contains the results obtained at 30 °C. Based on data in Fig. 8, the correlation of the residuum was calculated with the SPSS 14.0 program, using linear regression. The Durbin-Watson probe showed a linear correlation of the data, therefore the model of Eq. 3. is proper for describing the time constant of UF with a 95% confidence.

Table 4. Measured and calculated time constants ($T_{i\ meas.}$ and $T_{i\ calc.}$) for UF model

T (°C)	Q_{rec} ($m^3 h^{-1}$)	Re number	T/T_0 (°C °C^{-1})	$T_{i\ meas.}$ (h)	DW	$T_{i\ calc.}$ (h)	R^2
30	1	4278	1.2	1.70	1.838	1.65	0.99
30	1.5	6367	1.2	1.38	1.507	1.49	0.99
30	2	8456	1.2	1.19	1.452	1.14	0.96

Q_{rec}: recycle flow rate; T_0 = 25 °C; DW: Durbin-Watson constant; CI: 95%

3. Conclusions

On the basis of a series of preliminary experiments with different combinations of MF, UF, NF, RO, OD, and MD, a complex UF-RO-OD system was found feasible for apricot juice concentration. The optimal operation parameters of the complex membrane system were measured, calculated, and compared with the economic data of traditional evaporation.

Ultrafiltration (UF) can be used for clarification and sterilization of fresh, enzyme treated (Pectinex YieldMASH enzyme) apricot juice. Solid content of initial base juice was

around 10 °Brix, depending on degree of fruit ripeness. After UF clarification and sterilization about 8 °Brix juice was gained. It became apparent that raising the temperature causes a significant increase in the initial permeate flux, and increasing recycle flow results in a remarkable change, too, in the measured ranges.

A dynamic model was successfully applied for the description of time constant of UF filtration, the measured and calculated values overlapped well.

Using reverse osmosis (RO) for pre-concentration of clarified apricot juice, the solid content of the concentrate reached about 25 °Brix. The effect of temperature was considerable. It was a great advantage that only water passed to the permeate side.

When pre-concentrated apricot juice was further concentrated with OD, by the removal of water only, the final product had a 65–70 °Brix value. With such a large solid content the fruit concentrate can be stored at room temperature, because harmful microorganisms are not viable under these conditions (Rektor et al., 2006).

Two very important quality parameters: total polyphenol content (FCR) and the antioxidant capacity (FRAP) were analysed in the different products. The membrane operations using a 25–35 °C temperature range did not cause any damage to the valuable parts (FRAP and FCR) of the apricot juice. Using the FRAP method, the valuable components related to the dry matter were as follows: UF 34 mg AS °Brix l^{-1}, RO 34 mg AS °Brix l^{-1}, OD 28 mg AS °Brix l^{-1}, so the loss of the valuable components are about 20%. Using FCR method the valuable components were: UF 117 mg GS °Brix l^{-1}, RO 112 mg GS °Brix l^{-1}, OD 99 mg GS °Brix l^{-1}, so the loss of the valuable components are about 10–30%. We observed 62–84% loss of valuable components in the final product due to evaporation at 79–105 °C.

The complex UF-RO-OD membrane system is proposed as an economic method to create a healthy apricot juice. The following optimal operation parameters provided the lowest total cost: UF: 4 bar, 2 m^3 h^{-1}, 35 °C; RO: 50 bar, 600 l h^{-1}, 35 °C; OD: 20, 30 and 40 l h^{-1}; temperature 25 °C. A decrease in the optimal temperature saves the good quality of the juice, but it requires more membrane surface.

Based on the results of the experimental tests and data obtained from the SuperPro Designer program for comparison of traditional vacuum evaporation with multiple step (UF-RO-OD) membrane technique, it is apparent that the new process is 20–30% less expensive. The energy consumption of membrane filtrations is much less (35–45%), because in case of filtrations no phase conversion is needed, like in the case of evaporation. To produce apricot juice with combined membrane technique, a high final concentration can be achieved by membrane filtrations (until 25%) and osmotic distillation (from 25 until 65–70%).

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