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A comparative study on pomegranate juice concentration by osmotic distillation and thermal evaporation processes

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Abstract−A comparative study was performed on the concentration of pomegranate juice by the osmotic distillation (OD) and thermal evaporation processes. Nanofibrous polyether-block-amid (PEBA) membrane was prepared by the electrospinning technique, and the influence of operating parameters on the performance of the OD process was studied. The experimental results indicated that an increase in the temperature difference led to higher water flux, while a higher feed temperature resulted in undesirable color changes in the concentrate product. A comparison between the OD and evaporation processes revealed that the OD concentrate product had better quality than the evaporation product in terms of aroma and phenolic compounds retention. Furthermore, an economic analysis was performed by COMFAR III software to compare the OD and evaporation processes for the concentration of the pomegranate juice. It was also found that both OD and evaporation processes were feasible, although the evaporation process was more favorable in terms of the economic efficiency.

Keywords: Pomegranate Juice, Osmotic Distillation, Evaporation Concentration, Polyether-block-amid (PEBA), Electrospinning

INTRODUCTION

Pomegranate (*Punicagranatum L.*). which is cultivated extensively in Iran, Afghanistan, India, Spain, Turkey, Tunisia, Egypt and Morocco [1]. is a source of antioxidants. It has anti-inflammatory and anti-angiogenic properties that may protect against cancer, heart attack, high cholesterol and other chronic diseases [2,3]. The pomegranate fruit is consumed as fresh seeds, fresh juice, syrup and paste. The pomegranate syrup and paste, which are produced by the concentration of the fruit juice, can be used as a flavoring and coloring agent. The fruit juice concentration not only enhances the microbiological stability and fruit juice shelf life, but also reduces the cost of packaging, transportation and storage of the final products [4]. The concentration of fruit juices is generally achieved by techniques like evaporation [5,6] freeze concentration [7,8] and membrane processes [9-12]. Although the evaporation process as the conventional concentration technique is performed under vacuum, thermal evaporation generally leads to quality deterioration of the product due to damage to heat-sensitive components such as vitamins and antioxidants. In addition, the evaporation process may be responsible for color changes and the formation of unfavorable carcinogenic and mutagenic components during the heat treatment [13]. Therefore, appropriate alternative methods are desired for concentration of the pomegranate juice with high quality. Nowadays, membrane separation processes such as reverse osmosis (RO), membrane distillation (MD) and osmotic distillation (OD) are applied to concentrate the fruit juices under moderate operating temperatures. Among them, the OD process has successfully been employed to concentrate various liquid foods, including fruit juices, vegetable juices and milk [14].

Osmotic distillation, also known as osmotic evaporation, is a membrane process in which an aqueous solution or mixture containing one or more volatile components is contacted with a porous hydrophobic membrane in which an osmotic solution is passed on to the other side. The osmotic solution that is also known as stripping solution or brain solution is usually an aqueous solution of inorganic salts like NaCl, $CaCl_2$, $MgCl_2$, $MgSO_4$ and K_2HPO_4. The hydrophobic nature of the membrane prevents penetration of the aqueous feed solution into the membrane pores [15,16]. A number of polymeric membranes such as polypropylene (PP) [17-21] polytetrafluoroethylene (PTFE) [22-25] polyvinylidene fluoride (PVDF) [9,22,26] polyvinylpolypirrolidone (PVPP) [27] and polyethersulfone (PES) [28] have previously been used in the OD process for the concentration of various fruit juices.

Recently, Dincer [29] performed a comparative study on the concentration of black mulberry juice by thermal evaporation and OD processes and reported that the concentration by the OD process produces high-quality product and preserves its heat-sensitivity. A literature search revealed that the OD process has widely been used to produce various fruit concentrates. However, more research on the development of new membranes with high separation performance needs to be conducted in order for the OD technology to reach a foothold in juice processing. The main goal of this study was to develop a new nanofibrous polyether-block-amid (PEBA) membrane by the electrospinning method for the concentration of pomegranate juice using the OD process. Furthermore, the influ-

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ence of various operating parameters such as concentration of brine solution, temperature difference between feed and brine solutions and membrane thickness on the separation performance of the electrospun nanofibrous PEBA membrane was investigated. The potential of the OD process for the concentration of pomegranate juice was compared with the evaporation technique using a falling film evaporator. Finally, an economic analysis using COMFAR III software was performed to compare the OD process with the evaporation process. The main innovative aspect of this work is preparation of an electrospun nanofibrous PEBA membrane for the OD process.

MATERIALS AND METHODS

1. Materials

Fresh pomegranate fruits were purchased from a local market, washed in cold tap water and drained. By removing the outer leathery skin, the arils were separated. Then the juice was extracted by pressing the arils using an electric juice extruder. The obtained juice, having a deep-red color, was stored in a freezer at −17 °C and defrosted before use.

The commercial polyether-block-amide (Pebax®2533 SA 01) supplied from Arkema Inc. (Paris, France) was used as the membrane material. N-Butanol purchased from Merck Co. Ltd. (Darmstadt, Germany) was utilized as organic solvent. Calcium chloride ($CaCl_2$-$2H_2O$) that was used to prepare the osmotic salt solution and Triton X100 as a surfactant were received from Merck.

2. Preparation of Electrospun Nanofibrous Membrane

The nanofibrous PEBA membrane was prepared by the electrospinning of PEBA solutions on a non-woven polyester substrate. For this purpose, an electrospun polymer solution was prepared by dissolving PEBA (8%wt) and Triton X100 (1.2%wt) into n-butanol. The mixture was magnetically stirred for 6 h at 60 °C until a clear homogeneous solution was obtained and then sonicated for 30 min. The homogeneous electrospun solution was fed to a positively charged spinneret with a diameter of 1.16 mm attached to an electrospinning apparatus (Nanomeghias, Tehran, Iran). The flow rate was adjusted at 4 μl/min for an applied voltage of 25 kV and a fixed distance of 15 cm. At constant electrospinning condition, the membrane thickness was controlled by the electrospinning duration. The thickness of the prepared membrane was measured by a micrometer.

3. Membrane Characterization Tests

Scanning electron microscopy (SEM) analysis using an SEM device (AIS2100, Seron Technologies Inc., South Korea) was used to characterize the morphology of the prepared nanofibrous membranes. Before analysis, the samples were coated with a thin gold layer by the sputtering system under vacuum.

The mean pore size (D_m) of the membranes was determined via the filtration velocity method using the Guerout-Elford-Ferry relation [30]:

$$D_m = \sqrt{\frac{32(2.9-1.75\varepsilon)\eta_w l Q}{\varepsilon A \Delta P}} \quad (1)$$

where ε is the membrane porosity, η_w is the water viscosity (8.9×10^{-4} Pa s), l is the membrane thickness (m), Q is the volumetric flow rate (m^3/s) of the pure water permeation test, A is the membrane effective area (m^2) and ΔP is the operating pressure (MPa). A filtration experiment was carried out in a cross flow flat-frame stainless steel SEPA CF membrane module (Osmonics Inc., Minnetonka, MN, USA) with filtration area of 35 cm^2 at a temperature of 25 °C and transmembrane pressure (TMP) of 1.5 bar. The time duration of the experiment was 120 min.

A water content test was employed to determine the membrane porosity. In this test, the dry membrane was cut in a 5×7 cm^2 size and weighed. This weight was recorded as the membrane weight in dry state (W_d). Then the dried membrane was immersed in deionized water for 48 h. The water on the membrane surface was carefully cleaned with a clean tissue and the membrane was weighed (W_w). The membrane porosity was calculated using the following equation:

$$\varepsilon = \frac{W_w - W_d}{\rho_w A l} \quad (2)$$

where ρ_w is the water density at room temperature.

4. Concentration of Pomegranate Juice

The concentration of pomegranate juice was determined by

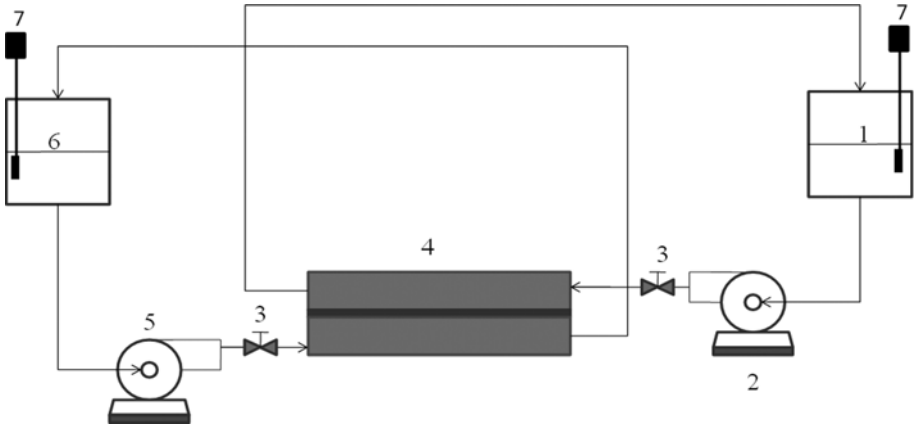

Fig. 1. The scheme of the OD pilot laboratory setup.

1. Feed tank
2. Feed pump
3. Manual valve
4. Membrane module
5. Osmotic solution pump
6. Osmotic solution tank
7 & 8. Temperature controller

two different techniques, the OD and evaporation processes. A laboratory OD experimental setup consisting of a cross flow flat-frame stainless steel SEPA CF membrane module (Osmonics Inc. Minnetonka, MN, USA) was used for the OD experiments. Aqueous solutions of calcium chloride with different concentrations were used as a stripping solution. Both feed solution (pomegranate juice) and osmotic agent solution were circulated on either side of the membrane in counter-current mode with the help of two independent pumps with variable velocity. The stripping solution reservoir was large enough to maintain a nearly constant salt concentration during the experiments. The effective area of the membrane used in the OD experiments was 137.5 cm^2 and a 5 lit tank used as the feed tank. The OD experiments were performed at atmospheric pressure. A schematic of the OD experimental apparatus is presented in Fig. 1. As shown, the feed and osmotic solution tanks were equipped with two temperature controllers. The temperature of the feed and osmotic solution tanks was measured by a thermocouple and adjusted by a temperature controller (TZN4S, Autonics, Seoul, Korea) with a precision of ±0.01 °C. The temperature of the feed tank was fixed at a constant temperature close to the ambient temperature, and the temperature of the osmotic solution tank was adjusted at a given value to reach a specific temperature difference using the temperature controller and a 2 kW heater for heating the osmotic solution.

The water mass flux (J_W) was calculated as follows:

$$J_W = \frac{W}{S\tau} \tag{3}$$

where W is the weight of water passed through the membrane (kg), S is the effective membrane area (m^2) and τ is the time duration of the experiments (h).

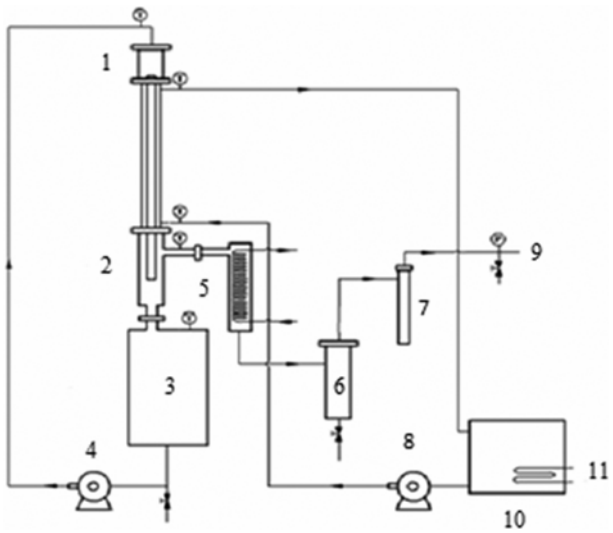

Fig. 2. The scheme of the falling film evaporator.

1. Feed distributer
2. Separator
3. Feed tank
4. Feed circulation pump
5. Condenser
6. Condensate tank
7. Trap
8. Water circulation pump
9. To vacuum pump
10. Water tank
11. AC power supply

Before the OD process, the membrane setup was sterilized by a cleaning in place method. In this procedure, the membrane setup was rinsed with tap water for 5 min. Then it was submitted to two washing steps using an aqueous NaOH solution (0.1 M) and an aqueous HCl solution (0.1 M). These two steps were carried out for 15 min at ambient temperature. At the end of each step, the membrane setup was rinsed with tap water for 5 min.

Furthermore, the concentration of pomegranate juice was determined via the evaporation process using a falling film evaporator. The operating pressure of the evaporator was 0.185 bar and the hot water with a temperature of 73 °C was used as a heating medium. The feed solution was heated to 60 °C using a preheater and then fed to the evaporator. A schematic of the falling film evaporator is presented in Fig. 2.

The concentration of the concentrate products was measured in terms of °Brix using an ABBE refractometer (2WAJ model, Fujian, China) at the ambient temperature. The aroma compounds in the pomegranate juice and concentrates were determined according to the procedure presented by Raisi et al. [31] using gas chromatography (GC) analysis. Finally, the total phenolic contents of the pomegranate juice and concentrate products were measured using the analysis method described by Mirsaeedghazi et al. [32] using a UV/Vis spectrophotometry.

RESULTS AND DISCUSSION

1. Characterization of Nanofibrous Membrane

The surface morphology and structure of the nanofibrous PEBA membranes prepared by the electrospinning technique were characterized by the SEM analysis. The SEM image from the top surface of the prepared membrane is presented in Fig. 3. It can be seen that the nanofibers were formed on the surface of the polyester substrate. The average fibers diameter is 50-150 nm.

Moreover, the porosity and mean pore diameter of the prepared nanofibrous PEBA membrane were determined by the water content and pure water permeability tests, respectively. The results revealed that the steady state pure water flux was 1476.8 kg/m^2h

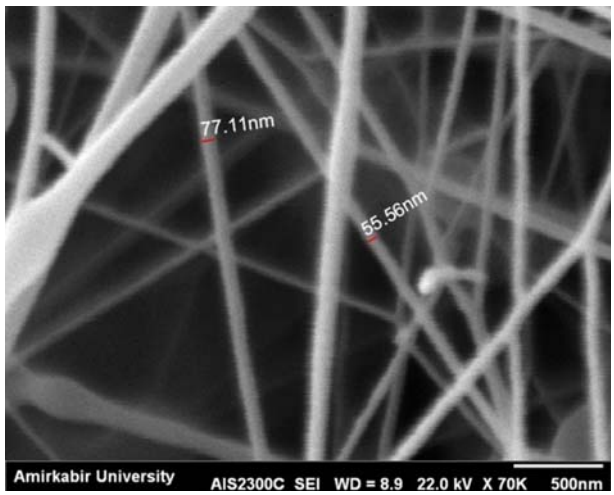

Fig. 3. The SEM image from the top surface of the prepared nanofibrous PEBA membrane.

and the mean pore diameter and porosity were 836 nm and 70.9%, respectively. These imply that the prepared electrospun membrane is a microfiltration membrane.

2. Pomegranate Juice Concentration by the OD Process

The pomegranate juice was concentrated by the laboratory OD experimental setup from an initial concentration of 12°Brix to a final concentration of about 25°Brix. In the following, a parametric study was performed to evaluate the effects of operating parameters like the concentration of the osmotic solution, temperature difference between the feed and osmotic solutions and membrane thickness on the separation performance of the prepared electrospun nanofibrous membrane.

3. Effect of Osmotic Solution Concentration

The salt concentration in the osmotic solution has a very important role in the OD process due its effect on the mass transfer driving force of the process. Three different salt concentrations of 3.62, 4.62 and 5.43 mol/lit (maximum solubility of $CaCl_2$ in water at 20 °C is 74.5 gram per 100 mlit) were chosen to investigate the influence of osmotic solution concentration on the separation performance of the OD process. The time dependency of the concentrate product concentration at various osmotic solution concentrations and effects of salt concentration on the transmembrane flux are presented in Fig. 4. The results indicated that the highest concentration and separation occurred at an osmotic solution concentration of 5.43 mol/lit. At this salt concentration, the water permeation flux was 1.18 kg/m^{2}h that was 31% higher than the flux at a salt concentration of 4.62 mol/lit. The transmembrane flux also increased with an increase in the concentration of osmotic agent solution. This trend can be attributed to changes in the mass transfer driving force with the salt concentration in the osmotic solution. In the OD process, the driving force for the component transport through the membrane is the difference in the component vapor pressure across the membrane. An enhancement in the osmotic solution concentration led to higher vapor pressure difference across the membrane and the driving force for the water transport through the membrane increased, so that the transmembrane flux enhanced. Similar results were reported by other researches for the fruit juice concentration by the OD process [17,19]. Based on these results, the salt concentration of 5.43 mol/lit was selected as the optimum value and the following OD experiments were conducted at this salt concentration.

4. Effect of Temperature Difference

The temperature difference between feed and osmotic solutions is another key operating parameter that affects the separation performance of the membrane in the OD process. The temperature not only affects the transport properties of the membrane, but also has significant influence on the mass transfer driving force of the OD process. To investigate the influence of operating temperature, experiments were performed for the concentration of the pomegranate juice by a varying temperature difference of 0, 15 and

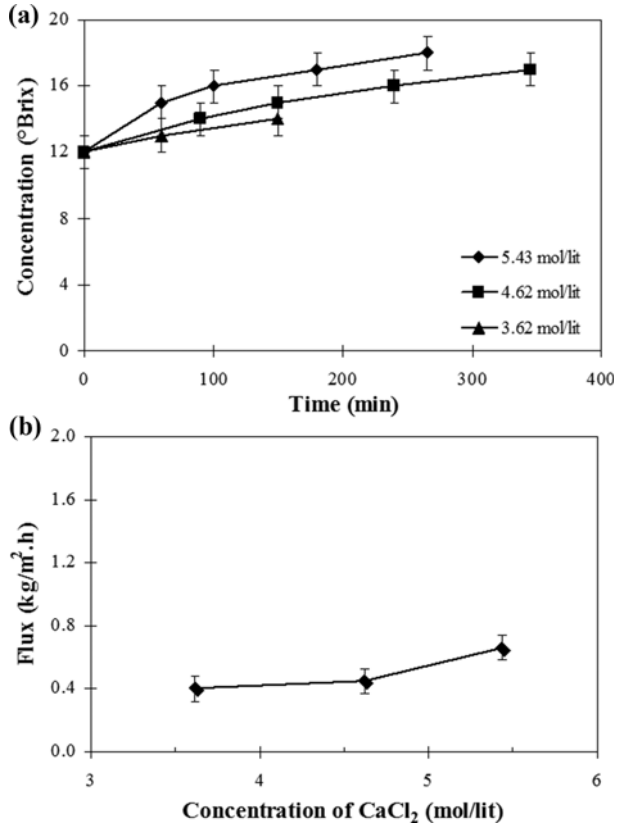

Fig. 4. The time dependency of the product concentration at various osmotic solution concentrations (a) and the effect of salt concentration on the transmembrane flux (b) for temperature difference of 0 °C and membrane thickness of 30 μm.

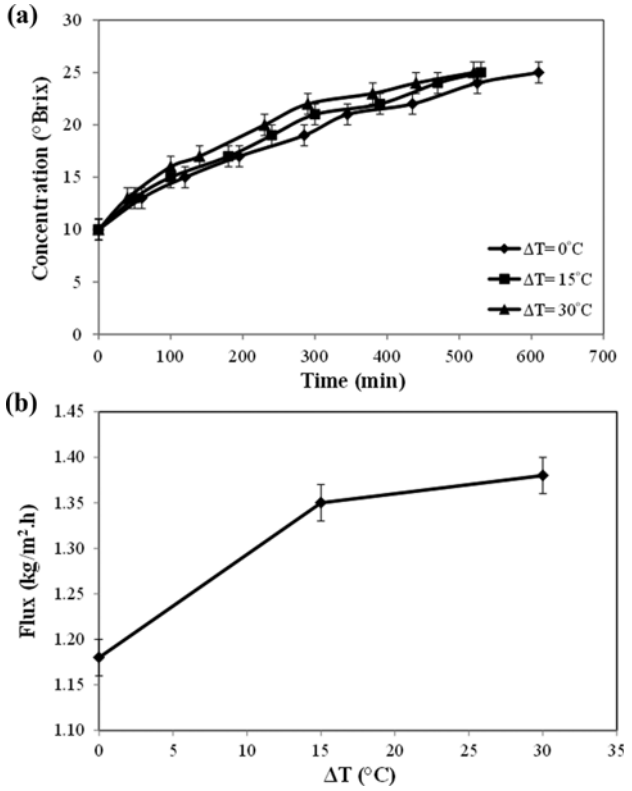

Fig. 5. The time dependency of the product concentration at various temperature differences (a) and the effect of temperature difference on the transmembrane flux (b) for osmotic salt concentration of 5.43 mol/lit and membrane thickness of 30 μm.

30 °C between the feed and osmotic solutions. For this purpose, the temperature of the osmotic solution was fixed at 20 °C and the temperature of the feed solution was adjusted by a temperature controller to obtain these temperature differences. The time dependency of the product concentration at various temperature differences and the effect of the temperature difference between feed and osmotic solutions on the transmembrane flux are indicated in Fig. 5. It can be seen that the transmembrane flux enhanced as the temperature difference between feed and osmotic solution increased. This behavior can be related to the effect of operating temperature on the water transport through the membrane. In the OD process, the mass transport across the membrane occurs through three seriate steps: i) water evaporation at the feed/membrane interface, ii) transport of water vapor through the membrane pores, and iii) vapor condensation at the membrane/osmotic solution interface. Generally, the vapor transport through the membrane pores takes place via one of three molecular diffusion, Knudsen diffusion and viscous diffusion mechanisms, depending on the pore size of the membrane and the characteristics of the diffusing component. In the present work, the mean pore size of the prepared nanofibrous PEBA membrane is 836 nm and the mean free path of the water vapour molecules is 0.3 nm [33]. Since the membrane pore size is larger than the water mean free path, the water vapor molecules transfer through the membrane pores by the molecular diffusion mechanism. The diffusion coefficient can be determined by the following relation [34]:

$$D = \frac{0.001858T^{1.5}}{P^{*}\sigma_{AB}^{2}\Omega_{D}}\left(\frac{1}{M_A}+\frac{1}{M_B}\right)^{0.5} \quad (4)$$

where D is the diffusion coefficient (m^2/s), T is the absolute temperature (K), P^* is saturated vapour pressure (kPa), σ_{AB} is the Lennard-Jones force constant, Ω_D is the collision integral (Å) and M is the molecular weight (g/gmol).

Based on Eq. (4), the water vapor diffusion coefficient through the membrane pores increases as the temperature goes higher. Consequently, the water transmembrane flux enhances with an increase in the temperature difference between feed and osmotic solutions. Although the higher feed temperature resulted in higher water flux through the membrane, these temperatures had adverse effects on the quality of the final concentrate product and led to undesirable color changes. Based on these results, the temperature difference of 0 °C was selected as the optimum value and the temperature difference between the feed and osmotic solutions was set at this value for the following OD experiments.

5. Effect of Membrane Thickness

The membrane thickness that determines the mechanical strength of the membrane can affect the mass and heat transfer through the membrane. Electrospun nanofibrous PEBA membranes with a thickness of 30 and 60 μm were used in the OD experiments to evaluate the influence of the membrane thickness. The time dependency of the product concentration and transmembrane flux at a membrane thickness of 30 and 60 μm are shown in Fig. 6. As observed, the thicker membrane had a higher transmembrane water flux than the thinner one. Based on Fick's law, the OD transmembrane flux is inversely proportional to the thickness of the membrane, because the intrinsic membrane resistance against the

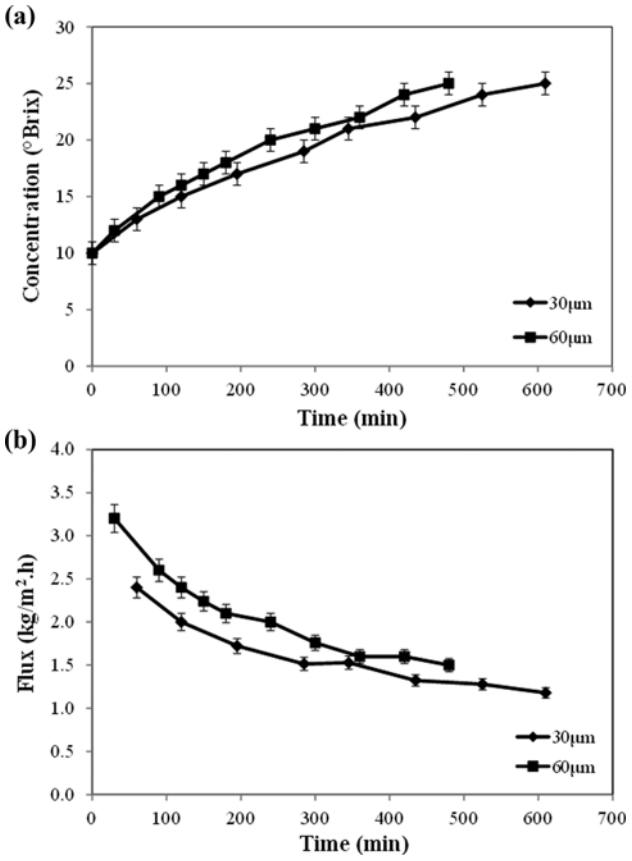

Fig. 6. The time dependency of the product concentration (a) and transmembrane flux, (b) at membrane thickness of 30 and 60 μm for osmotic salt concentration of 5.43 mol/lit and temperature difference of 0 °C.

component transport increases with the membrane thickness. Thus, the membrane thickness affects the thermal conductivity of the membrane in the OD process because the temperature polarization has a significant effect on the vapor pressure difference across the membrane and on the permeation flux. Therefore, a decrease in the membrane thickness enhances the heat loss from the membrane hot side to the membrane cold side, and consequently the vapor pressure difference as the mass transfer driving force reduces and results in a lower transmembrane water flux.

6. Pomegranate Juice Concentration by the Evaporation Process

The evaporation process as a conventional technology is well-known in the food industries, especially in the case of concentration of fruit juices and other liquid foods. Generally, fruit juices are heat-sensitive or contain heat-sensitive compounds. Due to the short time exposure, falling film evaporators which are usually operated at vacuum conditions are preferred. The pomegranate juice was also concentrated using a bench scale falling film evaporator. The time dependency of the concentrate product concentration is presented in Fig. 7.

To compare the quality of the pomegranate concentrate products obtained by the OD and evaporation processes, the retention of the volatile aroma compounds and phenolic compounds during the concentration process was evaluated. The aroma and phenolic

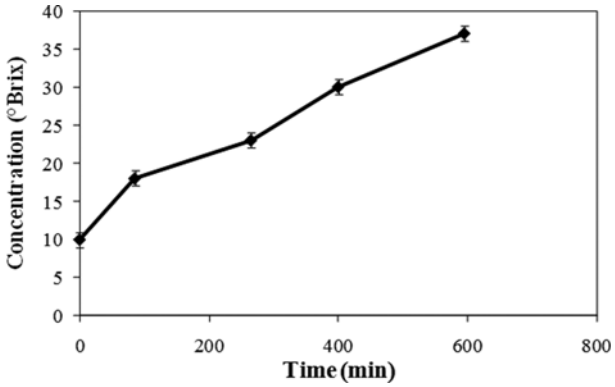

Fig. 7. The time dependency of the product concentration of the evaporation process.

compounds determine the sensory attributes of the pomegranate juice and concentrate. Also, these components are important for their several potential health benefits [35]. The aroma compounds which were identified in the pomegranate juice and concentrate are listed in Table 1. It can be seen that 61% of the total identified aroma compounds were retained in the concentrate product of the OD process, while 74% of the pomegranate aroma compounds were lost during the concentration by the evaporation process. In addition, the content of the phenolic compounds in the raw pomegranate juice and the concentrate products was determined. The concentration of the phenolic compounds in the pomegranate juice, OD concentrate product and evaporation concentrate product were 12.5, 12.4 and 10.5 ppm, respectively. These values indicate that the loss of phenolic compounds in the OD concentration process is lower than the evaporation process and the OD process is preferred to the evaporation process in terms of phenolic compound retention.

7. Economical Evaluation

Finally, an economic study of the pomegranate concentration process was performed to compare the OD process with the evaporation process. The COMFAR III software was employed for the economic analysis of a concentration plant with a capacity of 3,024 kg/h pomegranate juice. To perform the economic evaluation using the COMFAR III, the equipment in both OD and evaporation processes was sized and designed based on the obtained experimental data. For each process, the equipment costs were estimated based on the price in Iran (Table 2), then these data were imported into the COMFAR III software and the economical evaluation was performed using the economic analysis module of COMFAR III. The results of the economic analysis are presented in Table 3. The total investment costs, which include the fixed and working costs, were estimated using percentages of the delivered equipment costs. The methodology and applicable percentages are illustrated by Peters et al. [41] and Bejan et al. [42]. The applied percentages are based on the average percentages suggested by Bejan et al. [42]. Starting from the estimated equipment costs for each process, the total investment costs and working costs are estimated as shown in Table 3. The results indicated that both OD and evaporation processes are economically feasible for the concentration of the pomegranate juice. Due to higher equipment costs of the OD process

Table 1. List of identified aroma compounds in the pomegranate juice and concentrates

Component	Pomegranate juice	OD concentrate		Evaporation concentrate	
	Concentration (ppm)	Concentration (ppm)	Retention (%)	Concentration (ppm)	Retention (%)
3-Methyl butanal	9.7±1.1	6.4±0.9	66.1	1.3±0.4	13.3
Amyl butyrate	10.9±0.7	2.2±0.8	20.9	4.2±0.6	38.7
Ethyl pelargonate	9.9±0.5	2.5±0.7	26.0	0.9±0.7	9.1
α-Ionone	29.0±1.7	25.2±1.2	86.9	3.2±0.9	11.3
Total	59.6±1.9	36.5±1.3	61.2	9.7±1.3	16.3

Table 2. The specifications of the equipment used in both evaporation and OD processes

Process	Equipment	Count	Size	Reference for the equipment cost
Evaporation	Shell and tube heat exchanger	1	16.7 m^2	[36]
	Feed vessel	1	55 m^3	[36]
	Product vessel	1	15 m^3	[36]
	Feed pump	1	SHE-32-125/11A	[37]
	Product pump	1	FSE 4- 100-250/40	[37]
	Coils for water	2	16 m^2	[38]
OD	Membrane module	-	328 m^2	[39]
	Feed vessel	2	55 m^3	[36]
	Product vessel	1	15 m^3	[36]
	Feed pump	1	WHF-3 (900 rpm)	[40]
	Osmotic solution pump	1	WHF-3 (1,200 rpm)	[40]
	Osmotic solution vessel	1	61 m^3	[36]

Table 3. The economic indices of the concentration process by the OD and evaporation

	OD process	Evaporation process
Equipment costs ($)	74,446	34,499
Fixed costs ($)	481,057	221,378
Working capital ($)	10,019,053	9,920,708
Total investment costs ($)	10,572,276	10,175,205
Production costs ($)	12,172,070	11,901,000
Gross profit from operations ($)	61,499,774	61,615,771
Payback period (Year)	2	2
Internal rate of return on investment (IRR) (%)	66.47	68.09
Break-even point (%)	29.4	23.2

than the evaporation process, the profit of the OD process was lower than the evaporation. Moreover, the break-even point of the OD process was higher than that of the evaporation process. The break-even point is a point where the production costs and the revenue from sales are exactly equal to each other. This implies that the OD concentration process is more vulnerable to the variations in the production and sale.

CONCLUSIONS

Pomegranate juice was concentrated by the evaporation process with a falling film evaporator as well as by the OD process with a nanofibrous PEBA membrane prepared by the electrospinning technique. The influence of the OD process parameters, such as osmotic solution concentration, temperature difference between the feed and osmotic solutions and membrane thickness on the transmembrane flux and product concentration, was studied. The results showed that the water transmembrane flux increased by an enhancement in the osmotic solution concentration, temperature difference and membrane thickness. Based on the OD experiments, the salt concentration of 5.43 mol/lit, temperature difference of 0 °C and membrane thickness of 60 μm were chosen as optimum operating conditions for the OD concentration process. At these conditions, the final product concentration and permeation flux was 25°Brix and 1.5 kg/m^{2}h, respectively. The comparison of the pomegranate concentrate product of the OD process with that of the evaporation process indicated that most of the pomegranate aroma and phenolic compounds were retained in the OD product, while most of the volatile aroma compounds were lost during the evaporation process. Finally, the COMFAR III software was utilized to evaluate the economic aspects of both OD and evaporation concentration processes. It was observed that both processes were economically feasible and the profit of the evaporation process was higher than the OD process due to lower equipment costs of the evaporation concentration process.

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