

Clarification of Apple Juice with a Scraped-Surface Membrane Unit: The Effect of System Parameters on Process Efficiency

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ABSTRACT: *Apple juice can be clarified by ultrafiltration; however, fouling is a limiting factor against the industrialization of the process. A scraped-surface membrane unit was designed and constructed to reduce fouling during membrane clarification of apple juice using a polyether sulfone membrane with a pore size of 4 kDa. The effects of blade rotation speed (0, 600, 1400, and 2200 rpm), transmembrane pressure (0.5, 1, and 1.5 bar), feed flow rate (10, 15, and 20 mL/s), and distance between the blade and the membrane surface (2 and 5 mm) were evaluated on the permeate flux. The results revealed that increasing the blade rotation speed up to 600 rpm would increase the permeate flux, but further elevation of rotation did not affect the permeate flux. The optimized operating condition was obtained at low transmembrane pressure, feed flow rate, and short distance between the blade edge and the membrane surface. The study of the physicochemical properties of apple juice during clarification indicated that the designed system could reduce the turbidity of the juice by more than 99%. Other properties also changed to almost the same extent as in non-rotating systems.*

KEYWORDS: *Apple juice; Clarification; Membrane; Fouling; Rotation; Scraped-surface.*

INTRODUCTION

Apple (*Malus domestica*) is a nutritious fruit that is cultivated in many parts of the world [1]. Apple juice has

medicinal properties and contains nutrients, minerals, and micronutrients [2]. Large particles in the juice cause

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it to become cloudy; these particles also stick to the walls of the evaporator in the process of concentration and burn, causing an unpleasant taste of concentration [3]. Thus, the juice must be clarified before concentration. In traditional methods, large particles are bound together by enzymes and then separated from the juice by a filter press [4]. The use of enzymes reduces the viscosity of juice and its color [5]. On the other hand, fining agents, such as gelatin, bentonite, silica-sol, and diatomaceous earth can cause environmental problems while also changing the properties of fruit juice and reducing some active ingredients [6].

Membrane processes are a viable alternative to traditional methods of clarifying fruit juices. Due to their non-thermal nature, these processes can have a positive effect on reducing energy consumption and costs [7]. Ultrafiltration (UF) is one of the pressure-driven membrane techniques that has satisfactorily replaced the traditional methods of clarifying apple juice [8, 9]. The most important problem of this method is membrane fouling, which causes problems at the industrial level [10, 11]. Many studies have been conducted on membrane clarification of apple juice and on methods for reducing fouling. In recent years, chemical, hydraulic, electrical, and mechanical methods have been used to remove membrane fouling [12, 13]. Some of these methods, such as chemical methods, are expensive and pollute the environment [14].

Fukumoto, Delaquis, and Girard used microfiltration (MF) and UF ceramic membranes to clarify apple juice. They reported that UF membranes resulted in higher steady-state flux and less fouling than MF membranes [8]. *Cai et al.* studied the changes in the physicochemical properties of apple juice after UF clarification. They concluded that with increasing pressure and flow rate, the permeate flux increased and some polyphenols decreased [7]. *Bahceci* explored the effect of temperature, transmembrane pressure (TMP), and application of gelatin and bentonite as two filtrating aids agents on the membrane performance during clarification of apple juice. It was found that the increase in temperature and TMP enhanced the permeate flux. Further, changes in these two parameters had opposing effects on fouling resistance. While elevated temperatures reduced the fouling resistance, higher TMPs increased it. Also, pretreatment with gelatin (150 mg/L) and bentonite (750 mg/L) successfully eliminated the haze formed by polyphenols to a desirable extent [15]. *Gulec et al.*

evaluated the performance of a modified polysulfone membrane in clarification of apple juice, using low-pressure oxygen plasma treatment. They reported that the plasma-treated US100 membrane had an increased initial permeate flux of 75 kg/m.h which was approximately 1.5 times higher compared to using a plain membrane. Antifouling investigations revealed improved fouling resistance as well as prevention from initial permeate flux reduction. These results indicated that improved hydrophilicity and reduced surface roughness arose from plasma treatment which would prevent the unfavorable foulant attachment to the membrane surface. Overall, the effect of plasma treatment on the UF performance dispelled the need for an additional pre-flocculation step [16]. Elsewhere, *Sarkar* reported the potentiality of an electric field-assisted cross-flow UF system for different operating conditions using a modeled film theory, as a promising alternative to conventional approaches in the clarification of raw apple juice. Some estimated parameters involved in the mass transfer phenomenon such as diffusion rate of apple juice, Concentration Polarization (CP), gel layer features, and viscosity were examined for evaluating the permeate flux and flux decline. It was indicated that application of direct current (DC) effectively dragged the charged species, and owing to reduced CP effect, the permeate flux was enhanced. Furthermore, the gel layer was found to have a compressible nature as its resistance was dependent on TMP drop [17].

In addition to the membrane clarification studies of apple juice mentioned above, studies have also been conducted on the methods of fouling reduction in the membrane clarification of other fruit juices. For example, *Hemmati, Mirsaeedghazi, and Aboonajmi* applied ultrasound in the membrane clarification of carrot juice and concluded that it had a positive effect on the permeate flux; with elevation of the transmembrane pressure, its positive effect was enhanced [10]. *Aliasghari Aghdam et al.* obtained similar results during membrane clarification of pomegranate juice in the presence of ultrasound [3]. *Salehinia, Mirsaeedghazi, and Khashehchi* explored the effect of laser on the efficiency of the membrane process of pomegranate juice and concluded that laser assistance boosted the permeate flux due to the reduction of total resistance [18]. *Zhao et al.* utilized a shearing vibration membrane system to reduce fouling due to uniform shear action. They found that higher vibration frequency could enhance the

reversible membrane anti-fouling property. It was observed that elevation of the frequency from 1 to 5 Hz led to diminished TransMembrane Pressure (TMP). Interestingly, it was shown that even a low frequency of 5 Hz positively affected the membrane fouling behavior [19]. Nurra *et al.* investigated the effect of shear-enhanced filtration using a vibratory system in the dewatering of microalgae. It was reported that the use of innovative vibrated membrane filtration technology was far more effective in concentrating microalgae than conventional tangential cross-flow filtration. Also, while the filtration permeability values of the conventional method were approximately 10 L/h/m²/bar in the steady state phase, they found that in the new method, the dynamic filtration permeability increased by three times. They concluded that this result was mainly attributed to the elimination of the fouling in dynamic filtration [20]. Zarouk *et al.* used a magnetic field to reduce fouling during membrane clarification of pomegranate juice. They concluded that application of a magnetic field at the high intensity, low level of the feed flow rate and the direction of the magnetic field from the feed to the permeate side has the best effect on the efficiency of the process [21].

As mentioned above, mechanical methods are one of the techniques to reduce fouling of membrane processes. Thus, in this study, a scraped-surface membrane unit was designed and constructed to investigate the effect of physical stress resulting from the rotation of the blades on the membrane clarification performance of apple juice. Then, the effectiveness of the unit was investigated under various system parameters.

EXPERIMENTAL SECTION

Preparation of apple juice

Fresh apples (a variety of Granny smith) were prepared from the local market (Tehran, Iran) and washed. After peeling, the juice was extracted by an electric juicer (Parskhazar, Iran).

Design and construction of the membrane module

The scraped-surface membrane module was designed and fabricated (Fig. 1). The material of the module was polyethylene. The membrane was enclosed between the lower and upper parts of the module. These two parts were tightened with three bolts and formed a cylindrical part.

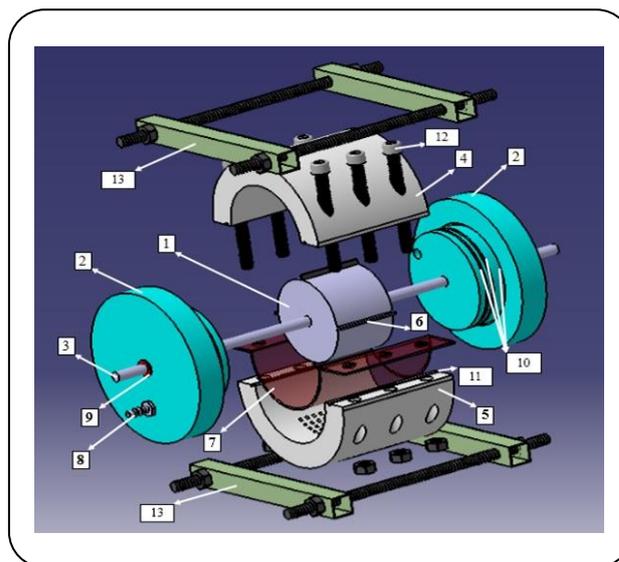


Fig. 1: The exploded parts of module 1) Rotor, 2) Cap, 3) Shaft, 4) Upper part of module (meshed body), 5) Lower part of module, 6) Blade, 7) Membrane, 8) Port, 9) Packing, 10) O ring, 11) Longitudinal sealing, 12) Bolt, 13) Frame.

Also, the two caps were compressed axially to this cylindrical part by a metal frame to eliminate any unwanted leakage. The shaft of the rotor was coupled with an electromotor (Motogen, Tabriz, Iran) and the rotation of the output shaft was adjusted to a desired value by an inverter (SV004ic5-1, KOREA). A pump transferred fresh juice to the module through the inlet port (No. 8 in Fig. 1), after which it is divided into two outflows, retentate and permeate. Permeate was collected from the bottom of the module for further investigation and retentate was returned to the juice tank for reuse.

Membrane unit

Polyethersulfone (PES) membrane with a pore size of 4 kDa (Sepanta polymer, Iran) with an active area of 1.12×10^{-2} m² was used in a lab-scale scraped-surface module (Fig. 2). In the system, the flow was adjusted with a manual valve and measured using a flowmeter, while the pressure was adjusted using an inverter coupled with a transmitter. A rotary vane pump (PROCON, Series 2, Milano, Italy) was employed to pump the feed to the membrane module and the retentate was recycled to the feed tank. A digital balance (A&D CO., LTD, EK-2000, Japan) was utilized to measure the permeate weight. Feed and retentate pressures were measured using two pressure meters. A coupled

Table 1: The specifications of the constructed module.

Module specification	Value
Flowing working volume	$604 \times 10^{-6} \text{ (m}^3\text{)}$
Total volume of module	$3089 \times 10^{-6} \text{ (m}^3\text{)}$
Clearance	2 and $5.25 \times 10^{-3} \text{ (m)}$
Length of rotor	$57.8 \times 10^{-3} \text{ (m)}$
Diameter of rotor	95 and $89 \times 10^{-3} \text{ (m)}$
Inside diameter of module	$99 \times 10^{-3} \text{ (m)}$
Outside diameter of module	$153 \times 10^{-3} \text{ (m)}$
Shaft diameter	$12 \times 10^{-3} \text{ (m)}$

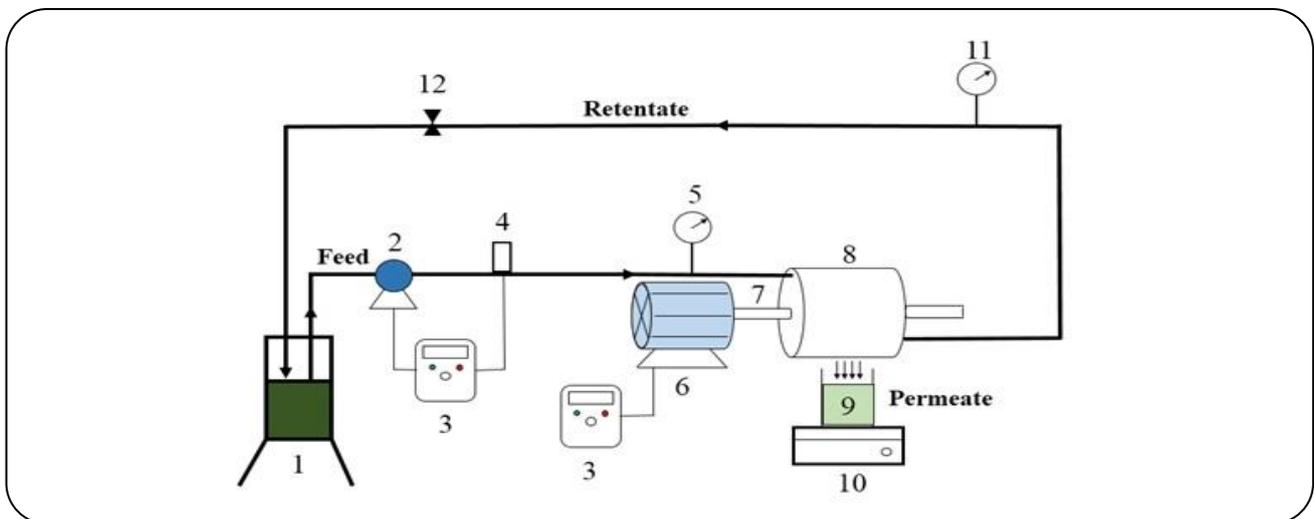


Fig. 2: Schematic of membrane unit 1) Feed tank, 2) Pump, 3) Inverter, 4) Transmitter, 5) Input pressure meter, 6) Electromotor, 7) Shaft, 8) Membrane module, 9) Permeate tank, 10) Digital balance, 11) Output pressure meter, 12) Flow control valve.

electromotor (Motogen, Tabriz, Iran)-inverter (SV004ic 5-1, KOREA) was used to rotate the rotor at the desired rotation speed.

Table 1 reports the specifications of the constructed module in detail.

Design of experiments

The values of the selected variable were:

- Transmembrane pressure at three levels (0.5, 1, and 1.5 bar)
- Flow rate at three levels (10, 15, and 20 mL/s)
- The speed of the rotor at four levels (0, 600, 1400, and 2200 rpm). An optical-mechanical tachometer (Lutron, DT-2236, Taiwan) was used to determine the rotor speed.

- The distance of the blade edge from the membrane surface (clearance distance) at two levels (2 and 5×10^{-3} m)
- The permeate flux (kg/m.s) was calculated using Eq. (1).

$$\text{Permeate flux} = \frac{\Delta m}{A \cdot t} \quad (1)$$

Where Δm is the permeate weight (kg), t denotes the time (s), and A represents the membrane active surface (m^2).

Evaluation of physicochemical properties of juice

The physicochemical properties of fresh and clarified apple juices in the membrane system with and without blade rotation were investigated.

The pH was measured using a digital pH meter (Metrohm-780 Switzerland). The total Soluble Solid Content (TSS) was determined by a portable refractometer ATAGO, HSR500, Japan) and expressed as Brix. Turbidity was measured using a digital turbid meter (Tu-2016, Taiwan) at 25 ° C. The titratable acidity was measured by titrating 2.5 mL of juice (diluted to 25 mL with water) with 0.1N NaOH in the presence of phenolphthalein as the indicator and the result was reported as g citric acid/ 100 mL juice [22, 23].

Total polyphenolic compounds were measured using the Folin-Ciocalteu colorimetric method [7]. In this method, 0.2 mL of the sample was mixed with 1 mL of 10 times diluted Folin-Ciocalteu reagent and 0.8 mL of 7.5% sodium carbonate. The prepared solutions were kept at room temperature for 30 minutes and then measured by a UV-visible spectrophotometer at 760 nm (UV-Vis Perkin Lambda 25, USA). Gallic acid (10 to 100 mg/L) was used as the standard solution with the results expressed in mg/L Gallic Acid Equivalent (GAE).

The color of apple juice was measured using CIE L* a* b* method. In this method, L* a* b* represents lightness, red/green coordinate, and yellow/blue coordinate, respectively.

All tests were performed three times and average values were reported. The rejection factor (R, %) was calculated according to Eq. (2).

$$R\% = \frac{P_f - P_m}{P_f} \quad (2)$$

Where P_f and P_m are parameter values in feed and permeate, respectively.

RESULTS AND DISCUSSION

Apple juice was clarified in the scraped-surface membrane unit. The effects of various parameters such as blade rotation speed, pressure, feed flow rate, and clearance distance of the blade to the membrane surface were evaluated on the process efficiency.

Effect of blade rotation speed on the permeate flux

The difference between permeate flux with and without the rotation of the blades was significant in all experiments.

The rotation of the blades enhanced the permeate flux due to the turbulence caused by the rotation of the blade. Various researchers emphasize that concentration polarization (CP) in membrane processes will eventually lead to fouling in the process [24, 25]. The rotation of the blades reduced the

concentration polarization, thus reducing the fouling. Membrane clarification of apple juice was performed at three blade rotation speeds (0, 600, 1400, and 2200 rpm) to study its effect on the permeate flux. The results revealed blade rotation increased the permeate flux, but the blade rotation speed did not affect the permeate flux (Fig. 3).

The Reynolds number (Re) in the constructed system was measured using Eq. (3).

$$Re = \frac{\rho D \omega^2}{\mu} \quad (3)$$

Where ρ , ω , D , and μ are juice density, angular velocity, the diameter of the rotor, and dynamic viscosity, respectively [26]. The particle inside the fruit juice is subjected to two counteracting forces, centrifugal and drag forces, which affect the laminar sublayer and gel layer on the surface of the membrane. The increase in the angular velocity led to the elevation of Reynolds number to 2.3×10^9 . At higher revolutions, the centrifugal force dominates the drag force, consequently a constant permeate is obtained sooner. The results showed that the revolution of the impeller-induced permeate until a specific value of ω . On the other hand the effect of Re on the concentration polarization region seems to be limited and similar to the friction factor of flow in a pipeline or coefficient of drag in external flow, where the friction factor or coefficient of drag became independent of Re at higher values of Reynolds number [27]. Since rotation at a higher speed did not have a considerable effect on reducing the concentration polarization, thus the rotation of 600 rpm was selected due to low energy consumption and the most efficient performance for subsequent experiments.

Effect of pressure, flow rate, and rotation speed on permeate flux

Apple juice was clarified by the scraped-surface membrane system at a given rotation of 600 rpm, 10 mL/s, and three transmembrane pressures of 0.5, 1, and 1.5 bar. At each pressure, it was compared with the efficiency of the system without the rotation of the blade at the same pressure. The results indicated that the difference between the permeate flux in the rotating and non-rotating states increased with the reduction of the transmembrane pressure. In other words, the rotation of the blade in the process at a pressure of 0.5 bar had the highest positive effect

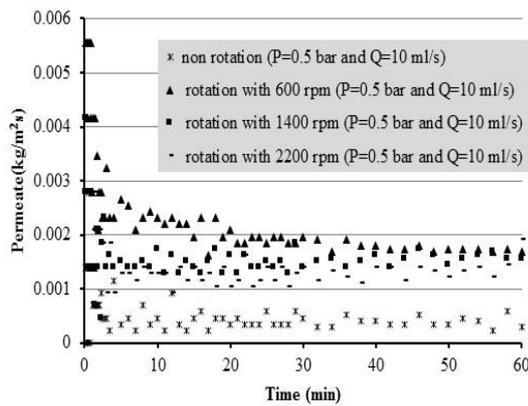


Fig. 3: Effect of blade rotation speed on the permeate flux.

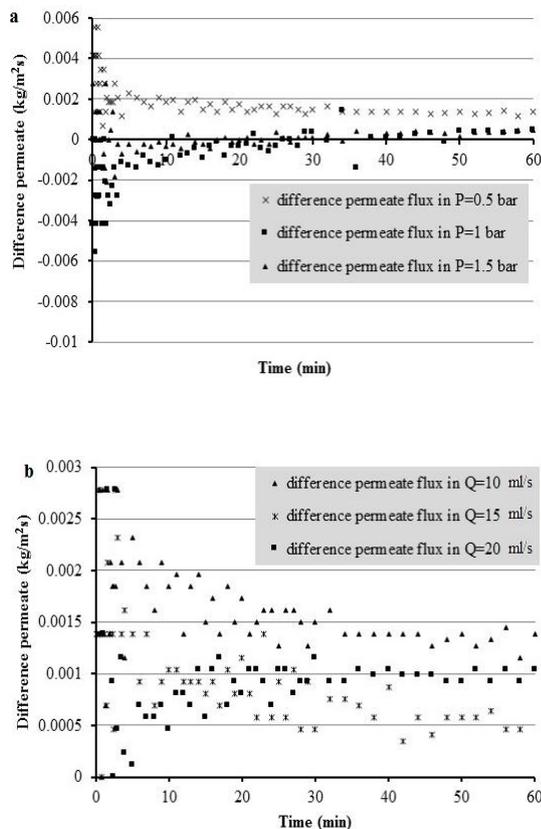


Fig. 4: Effect of transmembrane pressure (at $Q=10$ mL/s) and feed flow rate (at $P=0.5$ bar) on the blade rotation efficiency.

on the efficiency of the membrane process (Fig. 4a). This result may be explained by the fact that at high pressures, more large particles are pushed to the membrane surface causing higher fouling. As a result, the turbulence caused by the rotation of the blade cannot reduce such fouling;

however, at low pressures, fouling will be less and turbulent flow caused by the blade rotation can prevent particles from settling on the membrane surface.

Effect of feed flow rate on blade rotation efficiency

The performance of membrane clarification of apple juice was investigated at three different flow rates (10, 15 and 20 mL/s, all at 0.5 bar) with two modes of speed operation, zero and 600 rpm. The results indicated that the efficiency of blade rotation in enhancing the permeate flux increased with reduction of feed flow rate; so, the best performance was achieved at the feed flow rate of 10 mL/s (Fig. 4b). The reason for this result was that at high velocities, the flow was more turbulent, which reduced the concentration polarization; the results were the same when the speed of rotation increased.

The effect of the clearance distance of the blade from the membrane surface on the permeate flux

The membrane clarification of apple juice was performed in a scraped-surface membrane unit with two different clearance distances of the blade from the membrane surface (2 and 5×10^{-3} m) to examine its effect on the membrane efficiency. The results indicated that the permeate flux grew with decreasing the clearance distance (Fig. 5). The rotation of the blade creates a velocity gradient between the rotor and the membrane surface. This velocity gradient grows with a reduction of the distance between the blade tip and the membrane surface (clearance distance), resulting in a higher shear rate on the membrane surface [27]. The higher shear rate delays as well as reduces the amount of cake formation, and accelerates the passage of material through the membrane, explaining why the lower clearance distance caused a higher permeate flux.

The effect of process on the physicochemical properties of apple juice

The effect of rotation (with and without) on different physicochemical parameters (pH, turbidity (NTU), Total Soluble Solid (TSS, Brix), acidity (g citric acid/ 100 mL juice), total polyphenol (mg/L Gallic acid), and color parameters L^* , a^* , and b^*) is presented in Table 2. The effective process (0.5 bar and 10 mL/s) was selected to investigate the physicochemical properties in the feed and permeate.

Table 2: Changes in physicochemical properties of apple juice during membrane clarification (0.5 bar, 10 mL/s, and clearance distance of 2×10^{-3} m).

properties		Without rotation			With rotation (600 rpm)		
		Feed	Permeate	Rejection (%)	Feed	Permeate	Rejection (%)
pH		3.48 ^{c,*}	3.51 ^b	0.9	3.51 ^b	3.54 ^a	0.9
Turbidity (NTU)		964 ^a	22.48 ^c	98	864.3 ^b	2.94 ^d	99.7
Total soluble solid (TSS, °Brix)		13.8 ^b	11.8 ^c	14.5	14.3 ^a	11.8 ^c	17.5
Acidity (g citric acid/ 100 mL juice)		0.61 ^a	0.48 ^c	21.3	0.53 ^b	0.43 ^d	18.9
Total polyphenol (mg/L Gallic acid)		129.6 ^a	86 ^b	33.6	72.9 ^c	44.6 ^d	38.8
Color	L*	5.46 ^c	96.48 ^a	1667	5.46 ^c	87.25 ^b	1498
	a*	9.88 ^a	-5.66 ^c	157.3	9.88 ^a	-4.22 ^b	142.7
	b*	23.31 ^b	22.62 ^c	3	23.31 ^b	34.12 ^a	46.4

* In each row, same letter means no significant difference ($P > 0.05$)

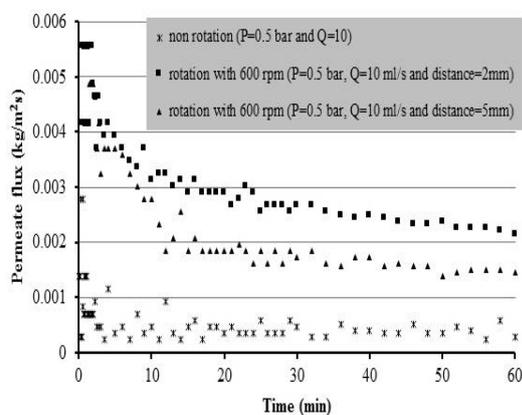


Fig. 5: The effect of clearance distance on the permeate flux at pressure of 0.5 bar and flow rate of 10 mL/s.

The pH of apple juice increased after membrane clarification, but the rejection factor was the same in ‘with rotation’ and ‘without rotation’ units (Table 2). However, the findings of the current study do not support past research. *Salehinia et al.* as well as *Hemmati et al.* found that pH did not change during the membrane clarification of pomegranate and carrot juice in the presence of laser and ultrasonic, respectively [18, 10]. Apple juice turbidity decreased significantly after membrane clarification and the extent of this reduction was almost the same in both rotating and non-rotating mode of operation. Total soluble solid and acidity of apple juice diminished slightly after membrane clarification in both states of operation. The variations of these two parameters in the two systems were almost the same. Also, the results showed that the total

polyphenol of apple juice after the clarification process was significantly reduced in both cases with and without rotation of the blades. Experiments indicated that the reduction of polyphenols in the rotating state was slightly greater than in the non-rotating state. The reduction of polyphenols can be attributed to their interaction with the membrane material and/or cake layer [7]. The results of color parameters revealed that the luminosity (L^*) was enhanced after the clarification process, while the reddish parameter (a^*) diminished in both rotation and non-rotation states. The value of the yellowish (b^*) changed in a different way. This property decreased in non-rotation mode, while an increase was observed during clarification when rotating mode was selected. The increase in b^* is due to the higher permeation of some pigments, including enzymatic browning compounds in the rotating mode where a high permeate flux was observed [28].

The trend of changes in the tested parameters was following the findings of *Cai et al.* as well as *Gulec et al.* reported for the clarification of apple juice via ultrafiltration [7, 28].

CONCLUSIONS

The use of a scraped-surface membrane unit in membrane clarification of apple juice boosted the process efficiency. The results revealed that blade rotation up to 600 rpm was sufficient to enhance the system efficiency, where faster rotation speed caused no further improvement of performance, yet this rotation demanded more energy consumption. The performance of the designed unit

increased with reduction of the transmembrane pressure, feed flow rate, and the distance between the blade edge and the membrane surface.

Examination of the physicochemical properties of apple juice during membrane clarification indicated that the use of the designed scraped-surface unit had no different effects on the physicochemical properties of apple juice, and the manner of change of these parameters was similar to other apple juice membrane clarification methods.

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