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Pomegranate Juice Clarification Using Ultrafiltration: Influence of the Type of Variety and Degree of Ripeness

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Abstract: Fruit consumption guarantees the supply of most of the necessary nutrients for a complete and balanced diet, as it is a relevant source of vitamins, minerals, and antioxidants. In particular, pomegranate has very interesting medicinal properties, such as an anti-inflammatory effect and the protection of the cardiovascular system, among others. During pomegranate juice production, it appears cloudy and must be clarified to remove suspended solids such as colloids and high-molecular weight tannins. The membrane clarification process is a cost-effective alternative to the conventional methods, resulting in a high-quality product. In this work, the clarification of pomegranate juice using the Triple System Model F1 membrane module was carried out for the *Mollar* and *Wonderful* varieties with early and late maturity. Three ultrafiltration membranes with different molecular weight cut-off and different chemical compositions were used. The rejection coefficient and permeate flux (which represent the selectivity of the membranes and the process efficiency, respectively) were measured. GR-40PP showed the best results in terms of membrane selectivity and process efficiency, achieving adequate physicochemical juice parameters. Regarding the comparison of the maturity degree, in general terms, the *Mollar* variety showed better results. Ripe pomegranates showed greater selectivity, while the process efficiency was higher for the early samples.

Keywords: pomegranate juice; ultrafiltration; membranes; clarification; *Punica granatum*

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1. Introduction

Almost 4 million deaths were associated with the inadequate consumption of fruits and vegetables in 2017. In fact, last year, the World Health Organization (WHO) recommended a healthy diet low in fat, sugar and sodium, and rich in fruits and vegetables. Specifically, WHO suggests that an intake of more than 400 g per day of fruits and vegetables improves overall health and may reduce the risk of non-communicable diseases [1].

Recently, there has been a worldwide increase in interest in pomegranate juice due to numerous health benefits of its consumption [2]. Because of its high antioxidant capacity, pomegranate and its components such as juice, seeds and peel, have favorable health effects, including antibacterial and anticarcinogenic properties [2].

Bioactive compounds of pomegranate include phenolic components such as hydrolysable tannins (ellagitannins and gallotannins), phenolic acids (gallic acid) and flavonoids (anthocyanins) [3]. The major anthocyanins in pomegranate juice are as follows: delphinidin-3,5-diglucoside; cyanidin-3,5-diglucoside; cyanidin-3-O-glucoside and delphinidin-3-O-glucoside, pelargonidin-3-0-glucoside and the 3,5-diglucoside [4]. Its high polyphenol content implies remarkable anti-inflammatory, antioxidant, antitumor, antimicrobial, anti-obesity, antidiabetic, diuretic and depurative qualities, among other, so it can be used as a preventive measure against cardiovascular diseases, cancer, and neurodegenerative diseases. Its ability to slow down ageing has also been demonstrated [3,5–7].

Pomegranate juice has a low caloric value due to its light fat and protein levels. The sugar is represented by glucose and fructose, with the content of the last one being normally higher than that of glucose. It also contains potassium, as well as phosphorus, magnesium, calcium and iron [4].

The natural appearance of pomegranate juice is turbid, which makes it difficult to preserve. Therefore, it must undergo a clarification process before being placed on the market. In this process, suspended solids are removed to produce a clear juice with a better taste and to avoid the appearance of turbidity after bottling. One method to carry out this process is through membrane ultrafiltration (UF) [8–11].

Different techniques have been used to conditionate and clarify pomegranate juice, such as pasteurization, thermal concentration, and the use of fining agents [12]. However, these techniques modify the bioactive compounds and the antioxidant activity of the juice.

An increased consumer demand for high-quality pomegranate juice, as well as a growing industrial interest in the production of different products (functional food, nutraceuticals, etc.), have promoted interest in minimal-processing technologies [13,14]. In this context, membrane processes represent an innovative approach to improve the quality of pomegranate juice.

Microfiltration and UF membrane processes have proven to be comparable to pasteurization in guaranteeing the microbiological stability of juice, and avoiding the deterioration of the final product. In addition, UF and nanofiltration membranes offer new perspectives in juice fractionation with the aim of recovering and purifying bioactive compounds of interest to produce functional ingredients [12].

For all the above reasons, the introduction of the UF technology represents a turning point in the production of high-quality, naturally flavored juice [2]. Through UF, it is possible to obtain an additive-free juice, avoid temperature-induced degradation, and maintain a constant pH for the final product [8].

Different authors use UF membranes to clarify pomegranate juice. For example, Conidi et al. [15] tested UF and nanofiltration flat-sheet membranes with nominal molecular weight cut-off (MWCO) ranging from 1000 to 4000 Da to biologically purify active compounds from clarified pomegranate juice. Other authors showed that clarified juice obtained by membrane processing has a more attractive color than fresh juice, which can improve the marketability of the product [10].

However, the main drawback of these processes is the fouling of membranes [15]. Therefore, the expansion of membrane technology in the juice clarification industry has been limited by membrane fouling [16–18]. Consequently, there is still a need for an in-depth study of the factors affecting the process from the perspective of membrane selection and process conditions.

The objective of this study is, therefore, to screen different membranes in the clarification process of pomegranate juice. The selectivity of the membranes and the efficiency of the process have been compared by means of the rejection coefficient and the permeate flux. Relevant physicochemical parameters such as transmittance, degrees Brix and turbidity, among others, have also been measured. Finally, analysis of the influence of the different fruit varieties and the degree of maturity on the pomegranate juice clarification process by membranes has been carried out.

2. Materials and Methods

2.1. Reagents and Pomegranate Juice

Pomegranate of the *Mollar* and *Wonderful* varieties, in the early and late ripening stages, was purchased from a local supermarket (Murcia, Spain). Juice from every pomegranate variety and maturity stage was produced in a pilot plant. In addition, the following reagents were used in the different tests carried out: water of HPLC purity distributed by J.T. Baker (Madrid, Spain), formaldehyde 37–38% w/w stabilized with methanol, NaOH 0.1 mol/L and phenolphthalein 1% solution distributed by Panreac (Barcelona, Spain), pH calibration standards (4.01; 7.00; 9.21) supplied by Hach (Düsseldorf, Germany), standard K^+ and Na^+ solution of 1 g/L distributed by Scharlab S.L. (Barcelona, Spain) and Merck KGaA (Darmstadt, Germany), respectively.

2.2. Membranes

Three different UF membranes (GR-40PP, GR-60PP and FS-40PP) were supplied by Alfa Laval (Madrid, Spain) and the specifications are shown in Table 1.

Table 1. Characteristics of UF membranes.

Type of Membrane	GR-40PP	GR-60PP	FS-40PP
Manufacturer	Alfa Laval	Alfa Laval	Alfa Laval
Supporting material	Polypropylene	Polypropylene	Polypropylene
Composition	Polysulphone	Polysulphone	Fluoropolymer
Surface area (cm^2)	84.82	84.82	84.82
Typical operating pressure (bar)	1–10	1–10	1–10
Tolerated pH range (at 25 °C)	1–13	1–13	1–11
Temperature range (°C)	5–75	5–75	5–60
MWCO * (kDa)	100	25	100

* Molecular weight cut-off.

2.3. Experimental Equipment

The equipment used in this work was supplied by MMS AG Membrane Systems (Swiss Biotech). It consisted of a Triple System Model F1 membrane module, which enables the use of any type of flat membrane to carry out laboratory-scale tests in a relatively short time and under transverse flow, obtaining data for the rejection coefficient of each membrane for the different pomegranate juice samples. Figure 1 shows the flow diagram of the membrane module used in the present study [19].

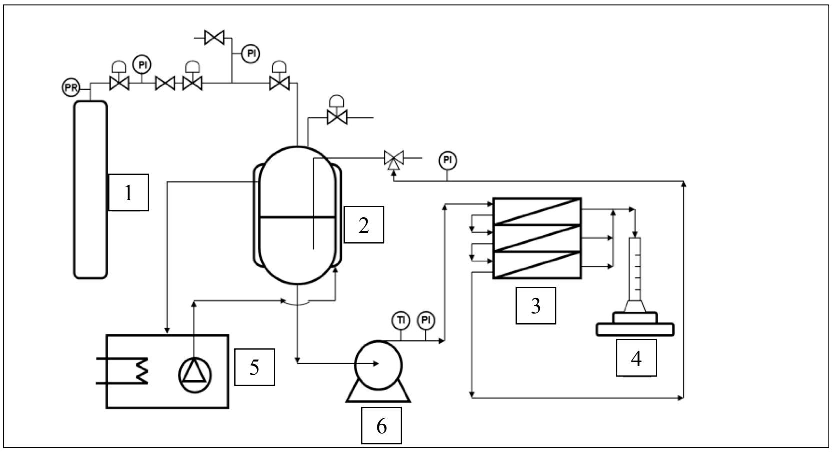

Figure 1. Flow diagram of the Triple System Model F1 membrane module designed by Hidalgo et al. [19]: (1) nitrogen bottle (outer cage); (2) feed tank; (3) membrane Triple System Model F1; (4) digital scale; (5) thermostat; (6) pressure pump.

2.4. Experimental Procedure

The methods used in this study are further divided into experimental and analytical methods.

2.4.1. Experimental Methods

The membrane module was started up by introducing 800 mL of the pomegranate juice into the feed tank and opening the nitrogen valve to operate at the desired pressure.

The membrane module consisted of three UF membranes, through which the feed stream was separated into two: retentate and permeate. In this way, we were able to know the membrane permeate flux and the permeate concentration.

Data on the input and output temperatures and pressures of the system were obtained from a computer connected to the membrane module. A first set of experiments was performed according to the batch concentration configuration in which the permeate stream was collected separately while the retentate was recycled back to the batch with a feed reservoir up to a VRF (volume reduction factor) of 1.5.

All the experiments were performed at different applied pressures (ranging from 4 to 8 bar) and at an operating temperature of 25 ± 2 °C. The UF membrane performance was evaluated in terms of productivity (permeate flux), solute rejection and fouling index. The permeate flux (J_p) was determined by measuring the collected permeate weight in a specific time through the membrane surface area as follows:

$$J_p = \frac{Q_p}{t \cdot A}$$

where J_p is the permeate flux (kg/sm^2), Q_p is the permeate weight (kg) at time (s) and A is the membrane surface area (m^2).

The rejection coefficient (R) of the selected membrane towards a specific compound was determined by the following:

$$R = \frac{\left(C_f - C_p\right)}{C_f} \cdot 100$$

where C_p and C_f are the concentration of the specific compound in the permeate and feed, respectively.

The fouling index was calculated by comparing the pure water permeability before and after the juice filtration according to following equation:

$$\% F = \frac{\left(J_{wi} - J_{wf}\right)}{J_{wi}} \cdot 100 \quad (1)$$

where J_{wi} is the pure water permeability of the native membrane and J_{wf} is the pure water permeability after pomegranate juice ultrafiltration.

2.4.2. Analytical Methods

The absorbance of samples was measured at 420 nm by using a Thermo Electron Evolution 300 UV–Vis Spectrophotometer (Thermo Electron, Waltham, MA, USA). Transmittance (650 nm) was determined by GENESYS 10S Series UV–Visible Spectrophotometer (Thermo Scientific, Madrid, Spain). Degrees Brix (°Bx) of the juice samples were analyzed by using an automatic refractometer (ATAGO™ RX-5000α-Bev, ATAGO, Tokyo, Japan). A Hach 2100AN Turbidimeter (Hach, Germany) and Jenway PFP7/C clinical Flame Photometer (Fisher Scientific, Sweden) were used for determining turbidity and Na^+ and K^+ ions (ppm). Finally, an automatic Titrator (Metrohm 916 Ti-Touch, Metrohm, Madrid, Spain) and pH-meter (Crison GLP-21, Crison Instrument Hach LANGE, Barcelona, Spain) were used to measure acidity and the formaldehyde index (FI) and pH, respectively.

3. Results and Discussion

The ultrafiltration membranes were pre-treated prior to the filtration of the pomegranate juice. This treatment consisted of the immersion of the membranes in distilled water for 10 min and their passing through the membranes on the membrane module system. Similarly, after the filtration of the different varieties of juice, the equipment was cleaned with distilled water again.

3.1. Screening of the Optimal Membrane for Pomegranate Juice Clarification

This study was carried out using two pomegranate varieties, *Mollar* and *Wonderful*, in the initial stage of the season, which involved the low-ripening stage of the fruit. Three types of membranes, with two different chemical compositions, were used, two of them having the same MWCO. The parameters studied in order to choose the optimal membrane were the rejection coefficient and the permeate flux, which represented the selectivity of the membranes and the efficiency of the process, respectively.

Figure 2 shows the rejection coefficient and permeate flux obtained from the different UF membranes for the different varieties of the pomegranate juice used.

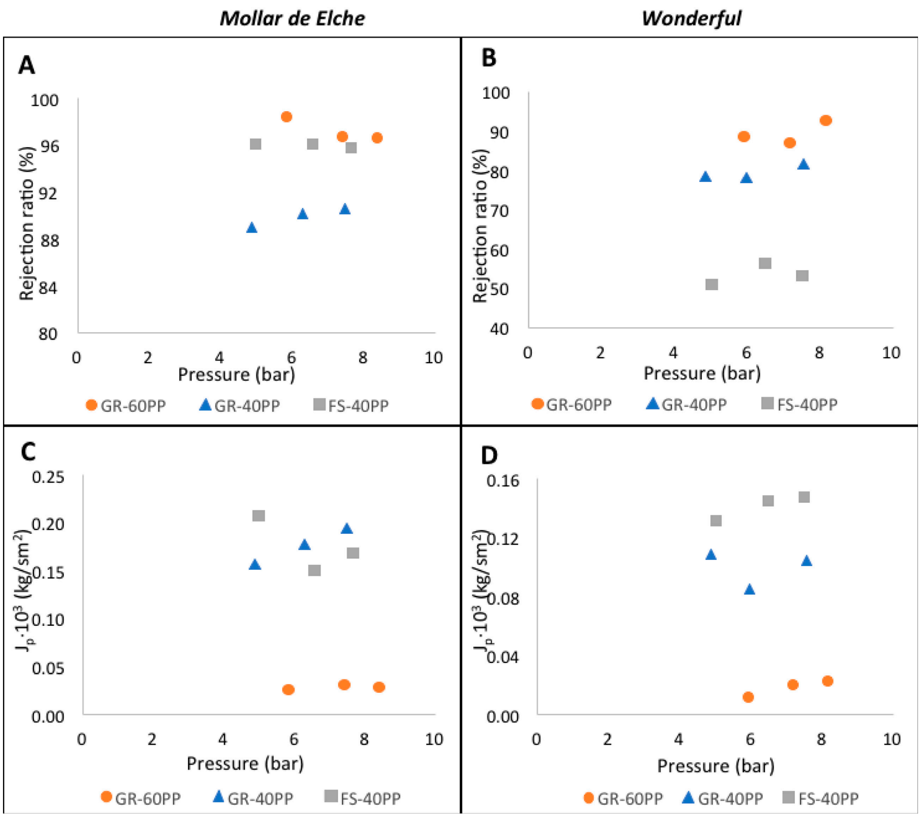

Figure 2. Comparison of selectivity and efficiency, using the rejection coefficient and permeate flux versus pressure, respectively, for *Mollar* (**A**,**C**) and *Wonderful* (**B**,**D**) varieties.

The rejection coefficients obtained from the GR-60PP membrane using different pomegranate varieties were around 90%. However, the permeate fluxes were extremely low. The FS-40 membrane obtained a high rejection coefficient from the *Mollar* variety, but it was low from *Wonderful*. Although a high permeate flux was obtained from the two varieties, this membrane was discarded because of the rejection coefficient values.

The GR-40PP membrane was selected because of its excellent results; the values of the rejection coefficient were 90% and 80% from the *Mollar* and *Wonderful* varieties, respectively, whereas the permeate flux values were similar to those obtained from the FS-40 membrane. These results were similar to those obtained by other authors using a PVDF membrane of a similar cut-off size [20].

Different samples of feed, permeate and concentrate were obtained from the UF process, and parameters such as °Bx, pH, Turbidity, Na^+ and K^+ ions were analyzed. Tables 2 and 3 show the values of different physicochemical parameters for the varieties *Mollar* and *Wonderful*, respectively.

Table 2. Physicochemical parameters for the different membranes of the *Mollar* pomegranate variety.

Parameters	Pomegranate *Mollar* Juice								
	GR-60PP			GR-40PP			FS-40PP		
	S_{feed}	S_{perm}	S_{ret}	S_{feed}	S_{perm}	S_{ret}	S_{feed}	S_{perm}	S_{ret}
ABS (420 nm)	7.45	0.23	11.32	5.63	0.63	8.53	19.91	0.90	33.55
%T (650 nm)	0.33	100.80	1.23	5.88	91.20	1.53	0.95	26.10	0.61
°Bx	15.88	6.28	8.69	16.01	14.78	16.17	12.3	10.89	12.77
Na^+ (ppm)	20	6	10	22	19	20	18	15	22
K^+ (ppm)	1427	504	734	1386	1365	1407	1224	1345	1448
Ph	3.95	4.06	3.93	3.97	3.96	3.97	3.95	3.86	4.12
%ACA	0.27	0.07	0.16	0.27	0.21	0.28	0.22	0.20	0.24
FI	11.50	4.66	6.04	10.62	10.06	10.88	11.50	8.64	9.28
Turbidity (NTU)	2957	2.25	1940	578	1.02	1145	2882	83	4086

S: sample; FI: Formaldehyde Index.

Table 3. Physicochemical parameters for the different membranes of the *Wonderful* pomegranate variety.

Parameters	Pomegranate *Wonderful* Juice								
	GR-60PP			GR-40PP			FS-40PP		
	S_{feed}	S_{perm}	S_{ret}	S_{feed}	S_{perm}	S_{ret}	S_{feed}	S_{perm}	S_{ret}
ABS (420 nm)	3.10	0.34	3.05	9.71	0.72	4.97	6.94	0.60	26.78
%T (650 nm)	1.30	100.60	31.60	0.40	57.00	0.20	1.27	3.71	0.01
°Bx	16.01	6.09	8.43	16.25	15.31	16.23	15.88	14.94	15.20
Na^+(ppm)	15	8	6	26	20	26	27	22	26
K^+(ppm)	2945	766	1244	2488	2250	1812	2772	2408	2448
pH	3.09	3.12	3.09	3.07	3.08	3.07	3.22	3.16	3.14
%ACA	1.45	0.51	0.74	1.45	1.36	1.48	1.56	1.44	1.60
FI	-	6.24	7.12	15.26	13.40	13.14	12.10	10.88	11.96
Turbidity (NTU)	1691	2.70	1641	2587	6.57	2339	814	371	4176

S: sample; FI: Formaldehyde Index.

The absorbance values in the feed, permeate and concentrate samples evolved in the same way for the pomegranate juice of the *Mollar* variety as for the *Wonderful* variety when using the FS-40PP membrane. An increase in the absorbance values in the retentate samples with respect to the feed samples, and a decrease in the permeate samples, could be observed. The transmittance of the samples in the concentrate stream was higher than that of the feed for all the membranes tested, with particularly high values for both varieties of pomegranate juice in the GR60-PP membrane, and in the GR-40PP membrane for the *Mollar* variety.

Regarding sugars measured as °Bx, a decrease in content was observed in the permeate samples, obtaining higher values in the concentrate samples for the GR-40PP and FS-40PP membranes. A similar behavior was observed for both varieties of pomegranate juice with respect to the degree of acidity. The salt content (sodium and potassium) decreased more considerably in the case of the GR-60PP membrane, which could be explained due to the smaller molecular cut-off size of this membrane. The pH value of the pomegranate juice was not affected by the UF process. In the case of the *Mollar* variety, pH values oscillated around 4, while for the *Wonderful* variety, these values were slightly higher than 3, similar to the results obtained by other authors [10].

Turbidity showed a significant decrease when comparing the feed and permeate samples, being more pronounced for the GR-60PP and GR-40PP membranes. This may be because both membranes have the same chemical composition (polysulphone). The physicochemical parameters shown in Tables 2 and 3 confirm that the GR-40PP membrane performs adequately for both varieties, confirming the proposed choice.

The above results were compared with the study carried out by Mirsaeedghazi et al. [20] in which turbidity, pH, °Bx and acidity were measured after the clarification of pomegranate juice with different membranes. It was found that the results for these parameters were similar to those obtained in this study [10].

In addition, in Table 4, the absolute quality requirements of the Reference Guide for pomegranate juice of the European Fruit Juice Association (AIJN) have been reviewed and compared with the values obtained in our study [4].

Table 4. Comparison between the values established by the European Fruit Juice Association and the values obtained for the different membranes and pomegranate juice varieties.

Membrane			GR-60PP		GR-40PP		FS-40PP	
			Permeate		Permeate		Permeate	
Specifications	**Min.**	**Max.**	**M**	**W**	**M**	**W**	**M**	**W**
°Bx	14.00	-	6.28	6.09	14.78	15.31	10.89	14.94
Na^+ (ppm)	-	30	6	8	19	20	15	22
K^+ (ppm)	1300	3000	504	766	1365	2250	1345	2408
FI	5.00	20.00	4.66	6.24	10.06	13.4	8.64	10.88

M: *Mollar*; W: *Wonderful*; FI: Formaldehyde Index.

The GR-60PP and FS-40PP membranes were able to retain a higher amount of sugars for the *Mollar* variety, and a value below minimum for the °Bx in the permeate. The values for sodium and the formaldehyde index (FI) were within the limits in all samples, while potassium was outside the accepted range for both varieties in the GR-60PP membrane.

3.2. Influence of the Chemical Composition of Membranes on Clarification

The GR-60PP and GR-40PP membranes have the same chemical composition but different MWCO, 25 and 100 kDa, respectively. To compare how the chemical composition affects the clarification of pomegranate juice, FS-40PP and GR-40PP membranes were chosen as they have the same molecular cut-off size (100 kDa), so the only difference between them is the chemical composition. While GR-40PP is composed of polysulphone, the FS-40PP membrane is composed of fluoropolymer. Both materials confer hydrophobic characteristics to membranes, but polysuphone shows oxygen and sulphur dioxide subunits, providing some hydrophobicity to the GR-40PP membrane. Contrarily, fluoropolymer shows CH_2 and $CF_{2,}$ providing high hydrophobicity to this membrane. Thus, hydrophilic subunits of polysulphone could provide higher capacity to create hydrogen bonds and Van der Waals interaction with phenolic compounds in the pomegranate juice [12], which confers better characteristics for pomegranate juice clarification.

In Figure 2A,C, it is observed that, for the *Mollar* variety, high rejection coefficients (88 and 96% for GR-40PP and FS-40PP membranes, respectively) and high permeate fluxes (around 0.17 and 0.195 10^{-3} kg/sm^2 for FS-40PP and GR-40PP membranes, respectively) were obtained. However, when studying the pomegranate juice of the *Wonderful* variety (Figure 2B,D), the rejection coefficient was higher for the GR-40PP membrane and the permeate flux was higher for the FS-40PP, but both parameters were lower in this variety.

3.3. Influence of the Degree of Ripeness on Pomegranate Juice Clarification

The ripeness stage of pomegranate affects the initial point for clarification. Fernandes et al. [21] described that the highest flavonoid, phenolic compounds and antioxidant activity were found in juices from the ripeness stage; meanwhile, they were reduced from skin and pellicles within the ripeness process. The amount of sugars is proportional to the ripeness stage, so higher amounts are found in ripe fruits. Something similar was found by Ydjedd et al. [22] in carob, where the flavonoids and phenolic compound were reduced during ripening.

Figure 3 shows the selectivity and efficiency values for the two types of samples and two maturity stages. In both cases, the membrane selected as optimal in the previous section (GR-40PP) was used.

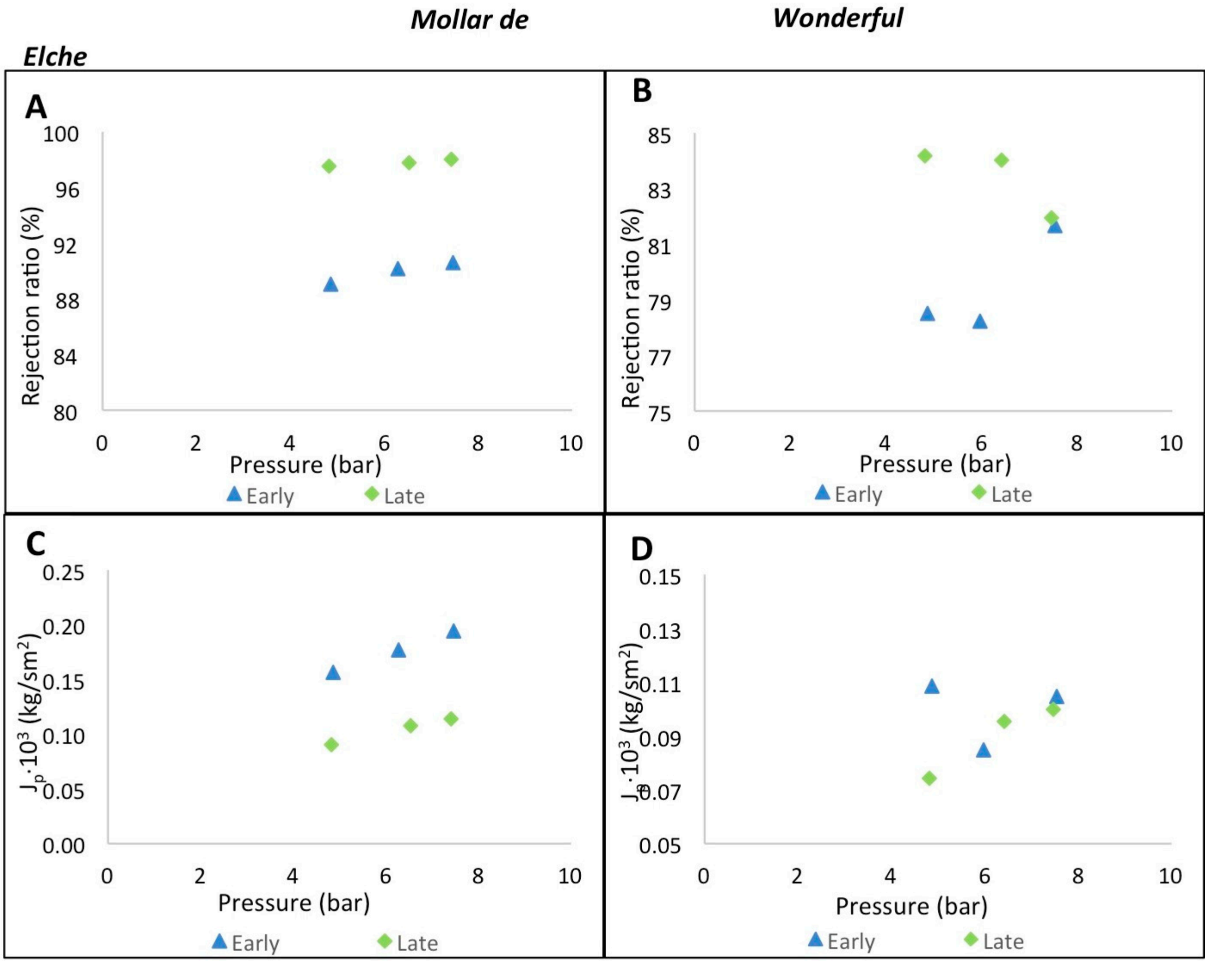

Figure 3. Selectivity and efficiency comparison using the rejection coefficient and permeate flux versus pressure, respectively, for *Mollar* (**A**,**C**) and *Wonderful* (**B**,**D**) varieties at the early and late maturity stage.

As it can be observed, the rejection coefficient for both the *Mollar* and *Wonderful* varieties was higher for the late maturity samples than for the early maturity ones. However, higher permeate fluxes were obtained for the early maturity samples. In the case of the *Wonderful* variety, these fluxes were found to be very similar. These expected results were similar to those found by other researchers [23]. The GR-40PP membrane rejected sugars and phenolics in juice, whose content was higher in the late maturity stage.

Although phenolic compounds were rejected, the clarification processes showed beneficial effects since sugars were also rejected, a finding similar to that of other authors [24]. Furthermore, the reduction of these substances, and other solids, could decrease turbidity and sensory properties, as described by several authors [12,25]. According to Baklouti [16], the application of UF technology to clarified pomegranate juice decreased the amount of phenolic compounds that cause astringency and bitterness, thus improving clarity but reducing the natural red color.

Table 5 shows the values of the physicochemical parameters for *Mollar* and *Wonderful* varieties.

Table 5. Physicochemical parameters of pomegranate juice from the *Mollar* and *Wonderful* varieties for both degrees of maturity.

Parameters	Pomegranate *Mollar* Juice						Pomegranate *Wonderful* Juice					
	Early			Late			Early			Late		
	S_{feed}	S_{perm}	S_{ret}	S_{feed}	S_{perm}	S_{ret}	S_{feed}	S_{perm}	S_{ret}	S_{feed}	S_{perm}	S_{ret}
ABS (420 nm)	5.63	0.63	8.53	19.11	0.46	57.89	9.71	0.72	4.97	9.91	0.90	30.16
%T (650 nm)	5.88	91.2	1.53	3.00	36.5	0.04	0.40	57.00	0.20	1.50	54.50	−0.02
°Brix	16.01	14.78	16.17	15.56	11.66	12.82	16.25	15.31	16.23	16.36	14.79	17.39
Na^+ (ppm)	22	19	20	13	12	15	26	20	26	15	13	26
K^+ (ppm)	1386	1365	1407	1417	1224	1365	2488	2250	1812	1724	2210	989
pH	3.97	3.96	3.97	4.45	4.49	4.43	3.07	3.08	3.07	3.16	3.53	3.50
%ACA	0.27	0.21	0.28	0.21	0.17	0.33	1.45	1.36	1.48	0.93	0.90	1.10
FI	10.62	10.06	10.88	14.22	17.06	11.96	15.26	13.40	13.14	18.72	23.94	20.20
Turbidity (NTU)	578	1.02	1145	901	31.2	5440	2587	6.57	2339	500	12.80	4433

S: sample; FI: Formaldehyde Index.

The behavior observed in these assays was similar to that obtained from the analysis of the different membranes. The pH value did not undergo significant modifications after passing through the membrane, while the percentage of acidity and the FI decreased in the permeate samples and increased in the samples extracted from the retentate stream. Absorbance values decreased in the permeate samples and increased in the retentate samples, while the opposite behavior was found when studying the transmittance. Sugars decreased after juice clarification, especially for the late samples. When studying the sugar content, it can be found that early samples of the *Mollar* variety had higher levels of sugars than the late ones, while in the *Wonderful* variety, the late samples had a greater sugar content. The salt content (sodium and potassium) decreased in the permeate samples, being higher in the early maturity samples than in the late maturity ones. In the retentate streams, the salt content behaved differently, depending on the variety and the degree of maturity.

When comparing the results obtained in the present study on the influence of the ripening degree in the clarification process, it was found that other authors showed similar results (pH values and °Bx) [26]. The study carried out by Onsekizoglu [27] highlighted that the organic acid content was maintained during the clarification process due to the low molecular weights of these compounds.

Table 6 shows the values of the Reference Guide for pomegranate juice of the European Fruit Juice Association (AIJN) and the values obtained for both degrees of maturity [4].

Table 6. Comparison between the values established by the European Fruit Juice Association and the values obtained for both degrees of maturity of the *Mollar* and *Wonderful* varieties.

			MOLLAR		*WONDERFUL*	
Specifications	**Min.**	**Max.**	**Early**	**Late**	**Early**	**Late**
°Bx	14	-	14.78	11.66	15.31	14.79
Na^+ (ppm)	-	30	19	12	20	13
K^+ (ppm)	1300	3000	1365	1224	2250	2210
FI	5.00	20.00	10.06	17.06	13.4	23.94

The late *Mollar* variety failed to obtain a below-minimum value for °Bx and potassium, while the late *Wonderful* variety obtained an above-maximum value for the FI.

3.4. Fouling Behavior of the Membranes

In order to study the fouling in each membrane, the filtration of water was carried out both before and after the filtration of the pomegranate juice. In this way, the permeate fluxes obtained in both situations could be compared. As the water fluxes are represented as J_w, they are shown in Figure 4.

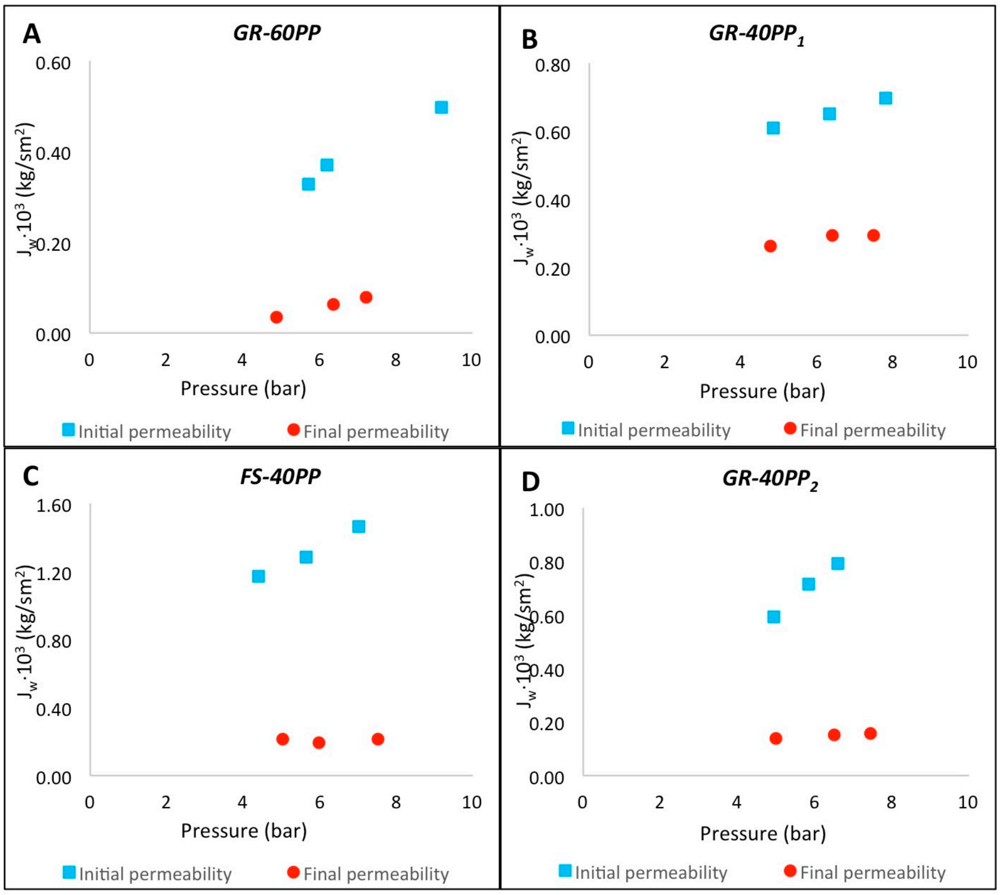

Figure 4. Comparison of water mass fluxes before and after clarification of pomegranate juice for GR-60PP (**A**), GR-40PP$_1$ (**B**), FS-40PP (**C**) and GR-40PP$_2$ (**D**) membranes.

It is evident that the permeability of the membranes decreased after the clarification process for all the membranes used. The rapid decrease in the permeate flux was a clear indicator that membrane fouling may have occurred, which also meant a decrease in membrane efficiency.

In Table 7, fouling index values divided by pressure ranges for the different membranes are shown.

Table 7. Fouling index of the different membranes.

	Pressure Range (bar)		
Membranes	**[4.4–5.75]**	**[5.75–6.5]**	**[6.5–9.2]**
GR-60PP	89.50	83.11	99.14
GR-40PP$_1$	57.12	54.78	57.80
FS-40PP	81.57	85.12	85.34
GR-40PP$_2$	76.54	78.88	80.29

It is observed that the GR-40PP membranes obtained a lower percentage of fouling than GR-60PP and FS-40PP. In general, as the applied pressure increased, the fouling was higher. The GR-40PP1 membrane was used for early varieties and the GR-40PP2 membrane for late varieties. In the tests carried out with the GR-40PP membrane, a higher soiling was obtained for the late samples.

When comparing the results with other research, which used a different ultrafiltration membrane to study the fouling and quality of pomegranate juice, the results showed that the main limiting factor in the use of this type of membrane in the clarification of pomegranate juice was fouling. This phenomenon causes a decrease in permeate flows and, in turn, a decrease in the efficiency of the process [15]. This occurs because particles larger than the molecular cut-off size of the membranes accumulate on the membrane surface, forming a layer that prevents the filtration of smaller particles.

4. Conclusions

The following conclusions can be drawn from the results obtained in this research.

Comparing the results from the study carried out for the *Mollar* and *Wonderful* varieties with the early degree of maturity, it was determined that the optimal membrane was GR-40PP, since it was the one that offered the best results in terms of membrane selectivity and process efficiency. In the same way, the physicochemical parameters studied indicated that this membrane showed excellent results.

In relation to the study of the maturity degree, it was found that the selectivity of the membrane was higher for samples with a late maturity degree, but the process efficiency was higher in early samples. Concerning the study of the chemical composition of the membranes, it could be seen that similar and higher results were obtained for the *Mollar* variety, whereas for the *Wonderful* variety, they were lower, with the membrane selectivity being higher for the GR-40PP membrane and the process efficiency being higher for the FS-40 PP membrane.

Finally, after the pomegranate juice clarification process, all the membranes suffered a high decrease in permeability due to the fouling that occurred during the process, which led to a reduction in the process efficiency. The membrane with the lowest percentage of fouling was the GR-40PP, which has been called the optimal membrane and, in particular, the one used with the early samples.

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