Effects of Solvent Concentration on Refining (Degumming, Dewaxing, and Deacidification) of Canola Oil Using Membrane Filtration

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ABSTRACT: *Miscellas of canola were obtained by mixing its crude oil with hexane solvent at 80:20 and 70:30 ratios. Then, 16.1 M of phosphoric acid and 6.92 M of sodium hydroxide were mixed with the resulting micelles at 0.3% (w/w for degumming) and 13% (w/w for neutralizing), respectively, before two sequential Membrane Filtrations (MF). The MF unit had a crossflow mode equipped with three independent variables of transmembrane pressures or TMP (at 2, 3, and 4 bar), feed velocity (at 0.5 and 1 m/s), and temperatures (at 30, 40, and 50˚C) were used to determine the efficiencies of two MF processes and find out their optimum conditions. When the crude canola oil was mixed with 20-30% solvent and passed the two stages of MF (for degumming and refining) at TMP=2 bar, feed velocity=1 m/s, and temperature = 50˚C, the final polished canola oil had < 5% soap, < 5% phosphorus, < 5% fatty acids, and < 15% wax. Membrane refining, compared to chemical refining, significantly reduced the phosphorus content (50%), free fatty acids (29%), soap (99%), and wax (72%) of refined canola oil. While the permeate flux of canola miscella with 20% solvent increased with rising TMP, feed velocity, and temperature, the ones with 30% solvent did not increase with a similar trend. The highest permeate flux of refined canola oil reached 0.03 Kg/m^2, s for miscellany with 20% solvent when the feed velocity, TMP, and temperature of degumming or neutralization were 1m/s, 3-4bar, and 30-40ºC, respectively. The dominant fouling changed from standard to cake blocking when the crude oil was mixed with 20 or 30% solvent, and the TMP of the MF process in each stage of degumming and neutralization was > 2 bar.*

KEYWORDS: *Canola oil, Membrane, Miscella, Refining, Microfiltration, Polyvinylidene fluoride.*

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INTRODUCTION

Various methods, such as mechanical/full pressing (for palm and olive), direct solvent extraction (for soybean and rice bean), and pre-pressing followed by solvent extraction (for rapeseed, sunflower seed, palm kernel, cottonseed, and corn) have been applied to extract the vegetable oils. However, these procedures depend on the nature of the oil-bearing material. Direct solvent extraction is usually applied when the oil content is less than 20-25%. Canola is a bright yellow-flower plant of the *Brassicaceae* family [1], which is among the five most-grown vegetable oil crops worldwide [2,3].

Canola oil is one of the most common edible and healthy cooking oils due to its low content of saturated fatty acids (~7.0%), high content of monounsaturated fatty acids (~60.0%), adequate content of n3 fatty acids (8.0% - 12.0%) [4]. Also, canola has more phenolic compounds than other oilseeds [5].

Diverse unpleasant compounds, such as phospholipids (PLs), free fatty acids (FFAs), waxes, and pigments which can be the reason for reducing the quality of the oil, are found in instant crude canola oil as vegetable oil. Therefore, they must be refined in several stages, including degumming, neutralization, bleaching, and deodorization, which consumes a lot of energy [6,7]. Removal of phospholipids, i.e., degumming, is the first and main step of the crude canola oil refining process.

Water or dilute acid will be applied in the prevalent degumming process to precipitate phospholipids by hydration, followed by agitation and centrifugation [8]. Since a significant amount of oil (4-5%), along with a large amount of wastewater and relatively high-energy consumption, will occur in processes like these, some alternative methods should be considered [9].

In recent years, membrane technology, due to its high selectivity, environmental friendliness, and low cost, has been widely used and replaced the conventional methods and has been turned into an indispensable means in industrial production and processing [10-14].

In the past three decades, membrane technology has had a significant and practical role in the food industry. An array of food products, such as agricultural and agricultural by-products, dairy products, beverages, and edible oils, are connected to this technology [15,16].

However, this technology, especially in the refining of vegetable oils, is theoretical. The main goal of the previous studies was the removal of the solvent (desolventizing) using ultrafiltration [17].

Rangaswmy et al. used membrane technology for vegetable oil processing. They concluded that this process was successfully conducted on individual steps of the prevalent refining process. Also, desolventizing revealed that approximately 65% of energy could be saved for solvent evaporation in an industrial environment.

An integrated membrane process that is focused on pretreatment and desolventizing along with physical refining would be a practical approach to fortify the benefits [18].

Recently, in some studies, the successful use of membranes in the degumming of crude vegetable oil/hexane miscella has been observed [19, 20]. However, the use of membrane technology in vegetable oil refining processes has been limited due to membrane instability in organic solvents, reduced permeate flux as a result of fouling, and the large scale of operation associated with the vegetable oil industry. Improvements in membrane technology have been observed in a large number of studies in the last decade [21,22].

Niazmand et al. investigated the effect of process conditions on the colloid-enhanced ultrafiltration of canola oil and stated that the permeate flux would be increased by raising the pressure and temperature from 25 to 55 ^{o}C [23]. Based on the studies of *Niazmand et al*. on the quality and stability of refined canola oil by adding chemical agents such as $CaCl_2$, EDTA, and SDS aqueous solutions, and membrane processing, SDS solution almost totally decreased phospholipids content and phenolic compounds reduced in SDS- and /EDTA-pretreated oil. Eventually, they noticed the vital role of SDS and EDTA in oil oxidation. Adding SDS and EDTA led to the high stability of membrane processing of canola oil [24].

Also, the result of another study conducted on canola oil miscella was the same, and the reduction of phenolic compounds in SDS and EDTA-pretreated filtered oil was more noticeable than in the processed miscella. The filtering without any chemical agents showed more reduction of FFAs [25].

Ochoa et al. [26] investigated the vegetable oils degumming using ultrafiltration by polyvinyldenfluoride (PVDF), polyethersulfone (PES), and polysulfone (PSF) membranes and stated that PVDF is more resistant to hexane than others. *Kim et al*. [19] investigated the separation of phospholipids from crude vegetable oil by

ultrafiltration with a polyimide membrane and found that phospholipids could be reduced by more than 90%. *De Souza et al*. [27] used ceramic membranes to degumming corn oil/hexane miscella at different transmembrane pressures (TMP) (0.5 and 1.5 bar) and tangential velocities (1.4 and 2.4 ms^{-1}) and concluded that an increase in TMP had a positive effect on phosphorus retention, while the tangential velocity had a negative effect.

Abdellah et al. studied on efficient degumming of crude canola oil by using ultrafiltration membranes and bio-derived solvents. Polysulfone (PSF), polyethersulfone (PES), and ceramic membranes were used to refine canola oil. It was believed that ceramic membranes had better performance than other membranes due to good cleaning. This membrane showed high phospholipid retention (95±2 %), although some oil was also retained (16±3%). The information was provided on the application of terpenes as hexane substituted. Since polymeric membranes are not readily cleaned for reuse, it seems unlikely to be applicable at an industrial scale. The best results were obtained with cymene, suggesting it is a reliable target for industrial usage [28].

To eliminate phospholipid from crude rapeseed و along with investigating their fouling mechanism, control, cleaning, and influence on oil quality, enzyme-membrane binding was used by *Hou et al*. According to the results, by using the hydrophobic ceramic, the highest efficiency for degumming was for enzyme-membrane binding. The initial fouling mechanism consisted of intermediate blockage and cake. To reduce the available area, pressure and pore size was the most significant. Also, increasing the resistance, temperature, and cross-flow velocity significantly reduced the resistance of cleanable pollution. Moreover, it was noticed that the membranes eliminated the phospholipids and water and were beneficial for decreasing free fatty acids and peroxide values. Therefore, the enzyme-membrane binding can be considered for gradual use in small-scale oil production [29].

The second process of chemical vegetable oil refining is neutralization which begins when degumming is done [30]. It can be used during or after the degumming process [31].

The neutralization is mainly conducted to eliminate free fatty acids (FFAs) contained in crude vegetable oils. In chemical refining, an alkali is used to neutralize FFAs and remove oil acidity [30].

It has been reported in some studies that membrane technology has been applied for the neutralization of vegetable oils. The molecular weight of free fatty acids is about one-third that of triglycerides, so they can be removed from crude vegetable oil using a suitable membrane. Also, methanol was used to extract FFAs from crude oil in some cases, and then FFAs were recovered from methanol solution using nanofiltration [32]. In the membrane technology method, more than 90% of FFAs can be recovered from crude vegetable oil [33]. *Ailcieo et al*. [34] treated crude soybean oil using ultrafiltration with two different membranes and reported that more than 50% of FFA was retained in oil with a commercial ceramic membrane. In contrast, 35% was retained using a polysulfone hollow fiber.

Based on the above-mentioned information, it was our objective to apply membrane filtration (MF) for canola oil mixed with 20 or 30% solvent and refine it by using phosphoric acid and NaOH solutions in the two stages of degumming and neutralization. We planned to test different levels of independent variables (feed velocity, pressure, and temperature) and find out the optimum conditions for achieving the highest efficiency as well as the lowest membrane fouling. We hypothesized that using two sequential MF processes for the two stages of refining would substantially minimize the phosphorus, soaps, acidity, and wax of crude canola oil and prevent soap-making in the final product.

EXPERIMENTAL SECTION

Canola crude oil was obtained from Mahidasht Vegetable Oil Co. (Kermanshah, Iran). All of the used chemicals were purchased from Merck (Germany).

Chemical analytical methods

The oil samples in the feed and the permeate were analyzed based on the methodologies of the AOCS (2017). The phosphorous content of oil was measured by the standard molybdenum blue method (Ca 12–55). Phospholipid level was calculated by multiplying the phosphorous content by a factor of 30; wax content (Ch 8-02); soap content (Cc 17-95); The acid value, which indicates free fatty acids (FFAs) in the sample, can be considered as a measure of the progress of the hydrolysis reaction in the oil (Cd 3d-63) [35].

Preparation of canola oil miscella

Canola oil miscella was prepared by mixing 80% and 70% (w/w) crude canola oil with 20% and 30% (w/w,

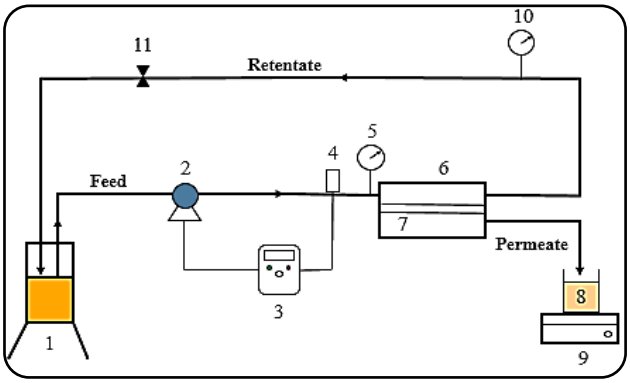

Fig. 1: A schematic view of the used experimental setup consisting of a feed tank, pump, inverter, transmitter, inlet pressure meter, membrane module, membrane, permeate tank, balance, outlet pressure meter, and floe valve, respectively)

respectively) industrial-grade hexane (90%). They were named canola 20% and canola 30% in the results.

Membrane unit

The used hydrophobic polyvinylidene fluoride (PVDF) (0.22 µm pore size and 109 cm^2 active area) (Millipore, USA) in a crossflow mode (Fig. 1). A rotary van pump transferred the feed to the module in the batch mode (PROCON, Series 2, Milano, Italy).

The feed pressure was maintained at the desired levels by a transmitter coupled with an inverter (SV004ic6-1, KOREA). The weight of permeate, as a function of time, was recorded. Meanwhile, the retentate was recycled into the feed tank. Using two separate pressure meters, pressures were recorded on both sides of the feed and retentate. Processes were performed at transmembrane pressures (TMPs) of 2, 3, and 4 bar, flow velocities of 0.5 and 1 m/s, and temperatures of 30, 40, and 50 ℃.

Oil refining was performed in two stages, including degumming and neutralization processes. In the degumming process, canola oil miscella was mixed with 85% phosphoric acid (0.3% w/w ratio) and shaken for 30 min. The mixture was processed with a membrane, and the permeate was collected. In the neutralization process, the permeate was mixed with NaOH, shaken for 30 minutes, and then treated by the membrane in which the permeate was the final product.

The percentage of NaOH solution used was calculated according to Eq. 1.

$$B = \frac{FFA \times 0.142}{\frac{A}{100}} \quad (1)$$

B is the percentage of used NaOH solution, FFA is the percentage of free fatty acids in canola oil, and A is the percentage of used NaOH solution concentration.

Theory

The permeate flux can be calculated by equation 2.

$$J_p = \frac{\Delta m}{A \times t} \quad (2)$$

The permeate flux is represented by J_p (kg/m^2.s), the permeate weight is represented by Δm (kg) collected in *t* (s), and the effective membrane surface is represented by *A* (m^2) [36].

The blocking mechanism in membrane processing was determined according to Hermia's theory. Thus, when the curve of *t/v* vs. *v* is linear, the formation of cake is the dominant mechanism.

Also, the dominant mechanism will be the standard blocking if the curve of *t/v* vs. *t* is linear. Moreover, the intermediate blocking mechanism will be dominant if Ln (*t*) vs. *v* is linear [37]. In addition, the proposed formula by Hermia (Eq. 3) can assign the duration of any onset fouling mechanism during the process.

$$\frac{d^2t}{dv^2} = k\left(\frac{dt}{dv}\right)^i \quad (3)$$

In this equation *k* is the resistance coefficient and *i* is the blocking index. If blocking index was 0, 1, 1.5 and 2, the fouling mechanism was cake formation, intermediate blocking, standard blocking and complete blocking, respectively.

Statistical analysis

All of the experiments were conducted in triplicate orders. The Minitab 16 software analyzed all the data. One-way analysis of variance (ANOVA) was used to determine the effect of membrane refining, while Turkey's test was used to determine the differences between the means ($(p < 0.05)$).

RESULTS AND DISCUSSIONS

Physicochemical characteristics

Crude and miscella canola oils (20% and 30%) were subjected to chemical and membrane refining in two significant steps of degumming and neutralization, respectively. The phosphorous content in both refining methods compared to crude oil was reduced significantly

Table 1: Physicochemical characteristics of the crude, chemical and membrane refined oils.

	Crude oil	Chemical refining	Canola 20%*	Canola 30%*
Phosphorous content (mg/kg)	294.6 ± 2.51[a]	0.066± 0.00[b]	0.032± 0.00[b]	0.031± 0.00[b]
Free fatty acid (%)	1.40± 0.011[a]	0.07± 0.00[b]	0.05± 0.00[c]	0.05± 0.00 [c]
Soap (%)	0.00+0.00 [c]	99.30± 0.57[a]	5.60± 1.15[b]	0.00+0.00 [c]
Wax (mg/kg)	201.40± 0.86[a]	91.5± 0.93[b]	25.50± 0.68[c]	25.80± 0.55[c]

Results were reported as mean ± SD. Different superscript letters in the same row represent significant differences ($p < 0.05$).

($p < 0.05$). The differences between the two methods in the degumming step were insignificant. However, microfiltration better decreased phosphorous content.

The Free fatty acid content in crude oil (1.40%) significantly decreased with both refining methods (Table 1). However, microfiltration (0.05%) was more successful than chemical refining (0.07%) ($p < 0.05$). Numerous studies prove that FFA, specifically at a high concentration, is a pro-oxidant [38]. The decomposition of hydroperoxides by the carboxyl group of the fatty acid might be its reason [39-41]. Avoid enzyme-catalyzed hydrolysis, which mostly depends on the overall quality of oilseeds, the initial content of moisture, and the condition of storage [42,43]. The bacterial, yeast, and mold contamination during growth, storage, and spices processing can enhance enzymatic hydrolysis. Due to the number of carbon atoms in the fatty acid, unpleasant flavor and odor can be generated because of non-esterified fatty acids, even at low levels [44]. Even though the hydrolytic rancidity initial products (FFA) are not toxic, they can accelerate the oxidation of oil, which can lead to a similar harmful effect of oxidized oils. These products might adversely and diversely affect human health, such as rapid weight loss, high death rate, digestive disturbances, dermatitis, reproductive failure, and anemia [45].

Free fatty acids are removed in the soap form from edible oils in neutralization when oils are exposed to an alkali solution such as Sodium hydroxide (NaOH). According to Table 1, microfiltration in the case of canola was 30% completely removed soaps in neutralized oil, while the highest amount of soaps was observed in the chemically refined oil ($p < 0.05$) (Table 1).

Only certain oils, such as corn, sunflower, canola, and rice bran, require dewaxing. Chilling, settling, and separation are the stages of wax removal in an integrated commercial refinery. However, wax content is significantly reduced through both methods, compared with crude oil (201.40 mg/kg) ($p < 0.05$). However, microfiltration in both miscella canola oils (20% and 30%) was more efficient than chemical refining (91.5 mg/kg) (Table 1).

A novel method for simultaneous degumming and deacidification of corn oil by miscella refining in one step was studied by *Wang et al*. Results showed that phospholipids and free fatty acids could be simultaneously successfully removed using this one-step method. Under the optimized conditions (50 g/100g miscella oil concentration, 30 g/100g excess alkali, 10 ◦Be′ alkali concentration, and 150 rpm at 50 ◦C for 60 min), the phosphorus content and acid value of corn oil can be decreased from 587.57 mg/kg and 5.13 mg KOH/g to 5.79 mg/kg and 0.10 mg KOH/g, respectively. High oil yield (95.3 g/100g) was achieved at low temperatures (50 ◦C). The washing process was saved, and no wastewater was formed in this method [46].

Wang et al. studied a novel method for corn oil degumming and deacidification by refining miscella. According to the results, phospholipids and free fatty acids could be successfully eliminated at one time using this one-step method. The phosphorus content and acid value of corn oil can be decreased from 587.57 mg/kg and 5.13 mg KOH/g to 5.79 mg/kg and 0.10 mg KOH/g, respectively, in the desired optimized conditions of 50 g/100g miscella oil concentration, 30 g/100g excess alkali, 10◦ Be′ alkali concentration and 150 rpm at 50 ◦C for 60 min. A high oil yield was achieved at a low temperature of 50 ◦C (95.3 g/100g). No wastewater was observed since the washing process was saved in this method [46].

Doshi et al. conducted a study on crude peanut oil miscella degumming using the membrane of PVDF. The results indicated that by optimally operating the PVDF membrane at 10 bar at room temperature, an excellent phospholipid (gum) rejection (95%) with 70 and 46 liters per square meter hour ($L/m^2.h$) of hexane and miscella permeate could be occurred [47].

In another study, the investigation was conducted on crude palm oil degumming and deacidification by using a

Fig. 2: View of the used experimental setup with mentioned components

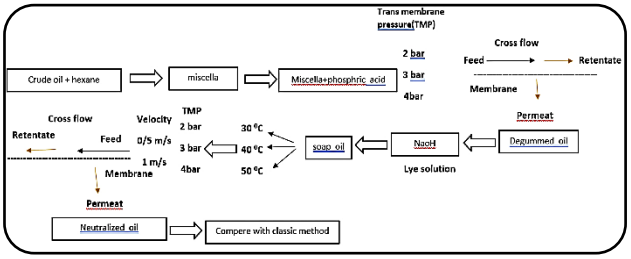

Fig. 3: Schematic view of the Flow diagram of oil treatment and membrane filtration on two stage

mixed matrix PVDF membrane. The results recorded an increase in the concentration of magnesium silicate from 3 to 8 wt% in the polymer matrix as the highest rate of FFA elimination at 16.51%, phospholipid at 93.31 %, and color at 18.8 %, respectively. These mentioned results are consistent with the present study, as well [48].

Membrane refining process

The TMP effect, along with the impact of velocity and temperature on the flux of permeate, was evaluated. In addition, the dominant fouling mechanism throughout the crude canola oil/hexane miscella degumming and neutralization was investigated, as well.

The TMP impact on the permeate flux throughout the process of membrane refining

All membrane refining processes were performed at three transmembrane pressures (2, 3, and 4 bar) to investigate their effect on the permeate flux in both degumming and neutralization processes. According to the results, increasing TMP in all experiments in the degumming process leads to an increase in permeate flux (Fig. 2)

The reason for these findings was that microfiltration is a pressure-driven process; as a result, increasing the driving force increased the permeate flux. The difference in the amount of permeate flux was significant at the beginning of the experiment and decreased over time. The reason for this was to drive more large particles towards the membrane surface at higher pressures, reducing pressure's positive effect in increasing the flux [10]. On the other hand, the flux difference at different transmembrane pressures was lower in the canola by 30%. The reason for this observation was the evaporation of the solvent at high pressures during the process, which led to an increase in the oil concentration in this miscella. In the next section, this fact will be discussed more.

The effect of the pressure of the transmembrane on permeate flux during the neutralization process was investigated in a similar study. Similar outcomes were observed in the canola 20% process processing, while the result in the processing of canola 30% was different (Fig. 3).

Contrary to expectations, the permeate flux decreased with increasing transmembrane pressure in this experiment. This is the greater evaporation of hexane solvent at high pressures, which caused the ratio of pure oil in miscella to increase at high pressures, resulting in a decrease in the permeate flux due to increased viscosity. These observations contradicted similar studies in the processing of fluid foods with membrane processing because, in all of them, unlike the present study, there was no volatile component in the feed.

The flow velocity effect on the permeate flux in the membrane refining process

All membrane refining processes were performed at two flow velocities of 0.5 and 1 m/s to examine the effects on the permeate flux in both degumming and neutralization processes. As the results stated, increasing the flow velocity in both the degumming and neutralization processes increased the permeate flux (Figs. 4 and 5).

These observations are the shear stress forces applied to the cake surface formed on the membrane, which will cause this layer to peel off and increase the permeate flux.

In all tests (especially in the neutralization process), the increase in flux due to increased velocity was reduced at

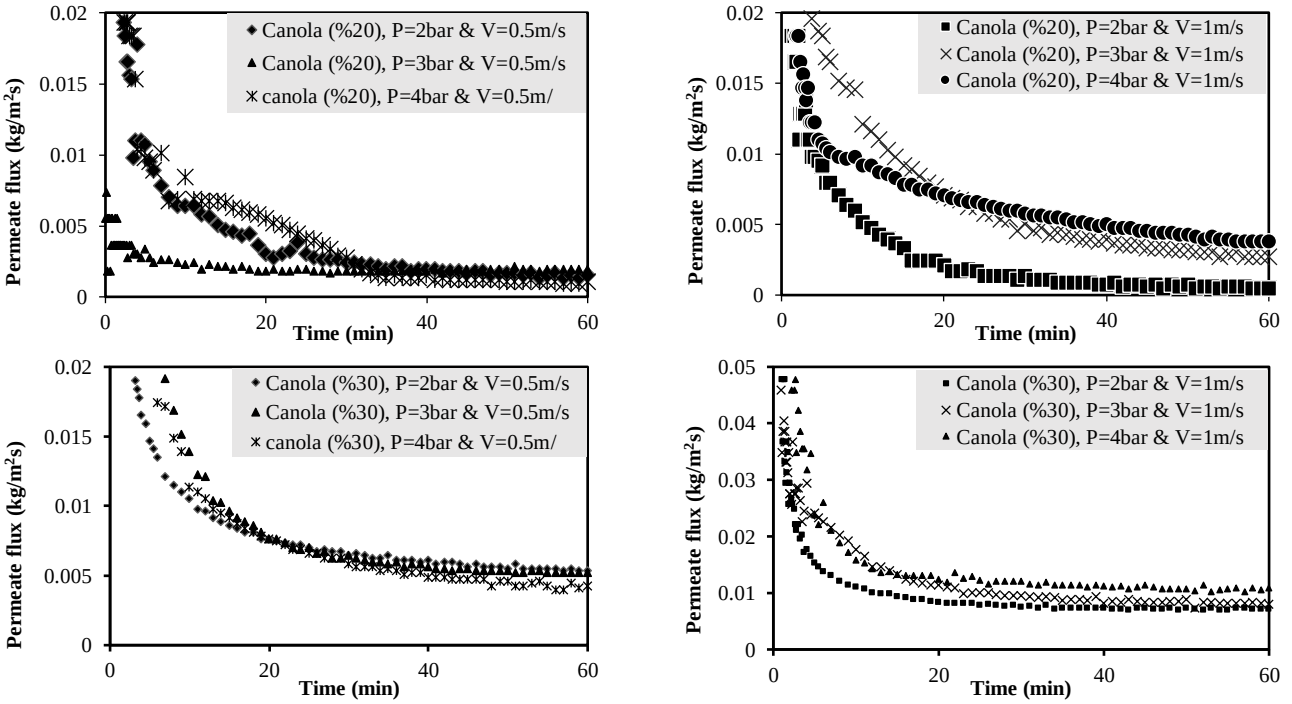

Fig. 4: The TMP impact on the permeate flux during first stage of membrane refining process of canola oil (degumming process)

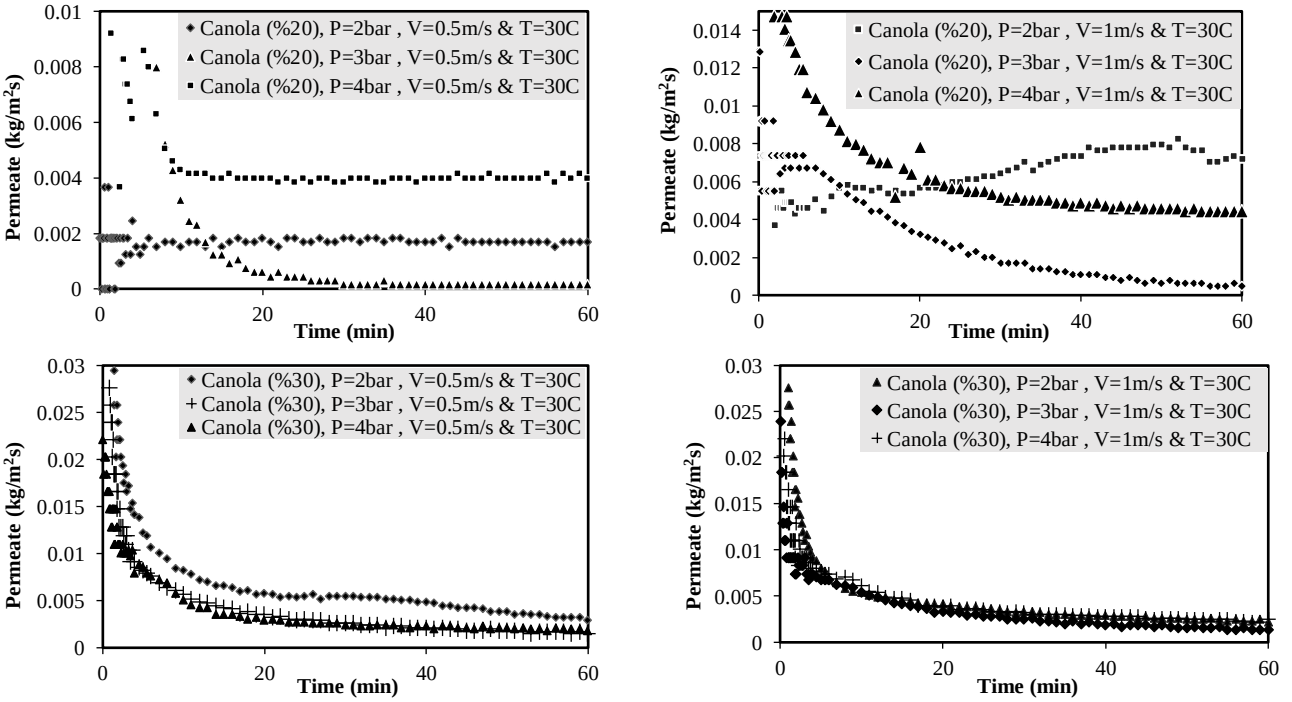

Fig. 5: The TMP effect on the permeate flux in the second stage of the process of membrane refining of canola oil (neutralization process)

the canola 30%. The reason could be the greater evaporation of the hexane solvent at high velocities and pressures, which would neutralize the high-velocity effect due to the increase in crude oil concentration in the miscella composition.

The feed temperature impacts on the permeate flux in the process of membrane refining

All membrane refining processes were performed at three feed temperatures (30, 40, and 50˚C) to investigate

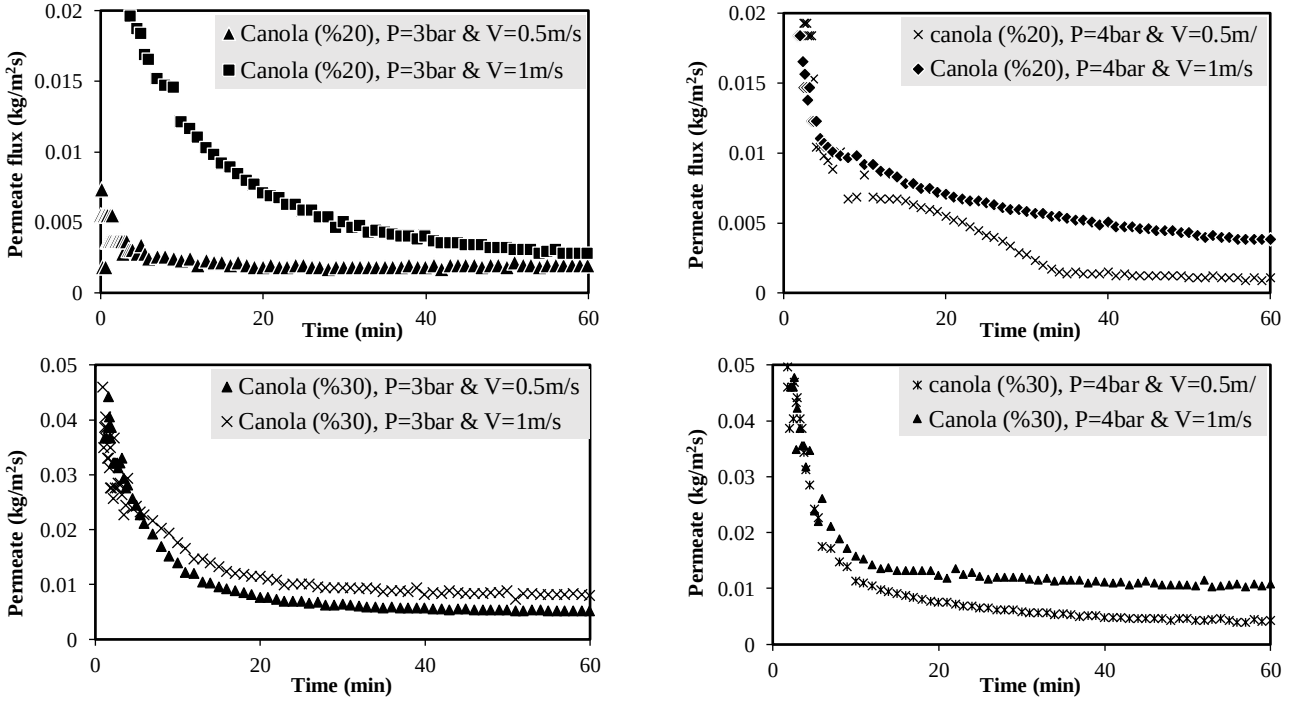

Fig. 6: The feed velocity effect on the permeate flux in the first stage of the membrane refining process of canola oil (degumming process)

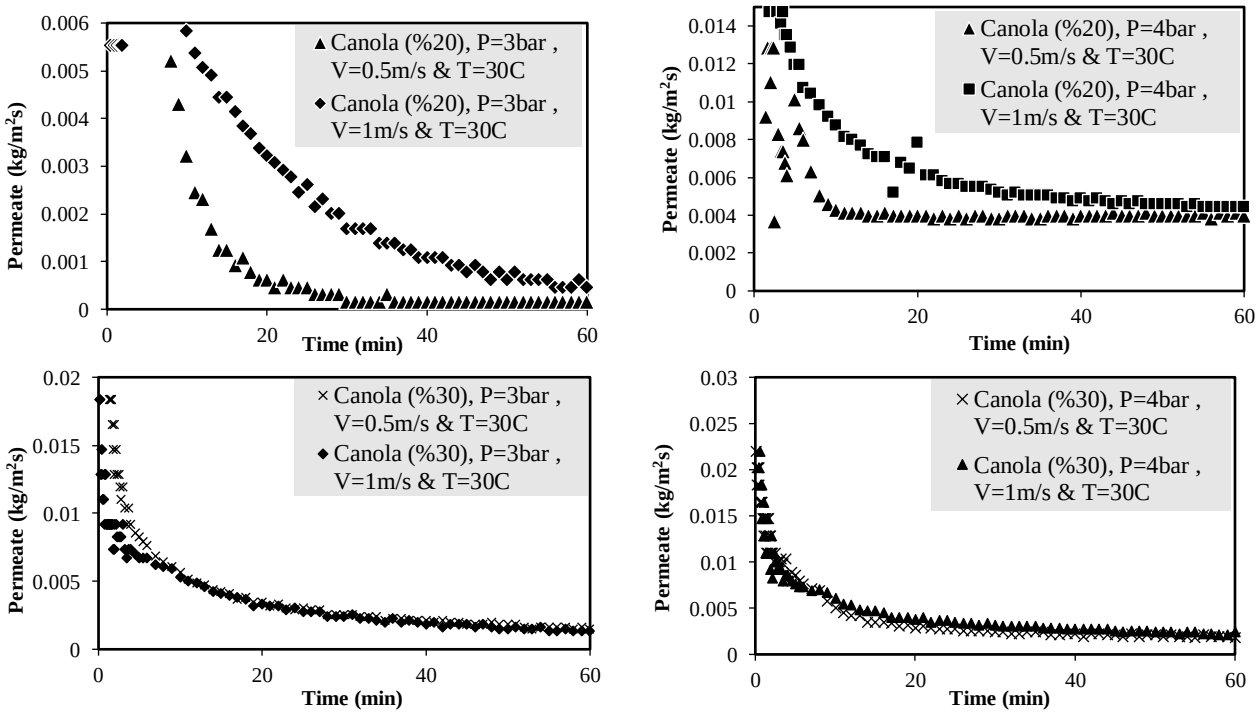

Fig. 7: The feed velocity effect on the permeate flux in the second stage of membrane refining process of canola oil (neutralization process)

their effect on the permeate flux in both degumming and neutralization processes. The results showed that in most tests and both degumming and neutralization processes, by increasing the temperature from 30 to 40 ˚C, the permeate flux increases, as well (Figs. 6 and 7).

The reason for this is the decrease in flow viscosity, which will increase the hydraulic permeability and consequently increase the permeate flux.

Table 2: Dominate blocking mechanism in degumming and neutralization processes

Dominate blocking mechanism								
Number	Type of test	P (bar)	V (m/s)	T (℃)	R			Type of blocking
					t/v vs. v	t/v vs. t	Lnt vs. v	
1	Degumming	2	0.5	–	0.9056	0.9853	0.9709	Standard
2			1		0.7559	0.997	0.9892	
3		3	0.5		0.7933	0.7459	0.7538	Cake
4			1		0.9187	0.9911	0.9354	Standard
5		4	0.5		0.7848	0.9958	0.992	
6			1		0.9957	0.948	0.8905	Cake
7	Neutralization	2	0.5	30	0.3494	0.3377	0.7111	Intermediate
8				40	0.9175	0.8914	0.8081	Cake
9				50	0.9269	0.9837	0.9443	Standard
10			1	30	0.0049	0.6812	0.0738	
11				40	0.9391	0.9129	0.7466	Cake
12				50	0.4505	0.4425	0.7271	Intermediate
13		3	0.5	30	0.4465	0.9997	0.9141	Standard
14				40	0.9947	0.9392	0.8758	Cake
15				50	0.8575	0.9779	0.973	Standard
16			1	30	0.685	0.9924	0.9377	
17				40	0.9273	0.8715	0.7805	Cake
18				50	0.9095	0.854	0.8025	
19		4	0.5	30	0.8962	0.8389	0.8008	
20				40	0.8359	0.7838	0.7675	
21				50	0.9937	0.9701	0.9152	
22			1	30	0.9738	0.8933	0.8404	
23				40	0.9906	0.9617	0.901	
24				50	0.9958	0.9562	0.8769	

It was expected that the permeate flux would increase again as the temperature increased to 50 °C, but contrary to expectations, the permeate flux decreased so that the highest permeate flux was at 40 °C. The reason for this finding is that the increase in temperature in solvent-containing foods such as miscella has two positive and negative effects on permeate flux. Its positive effect is to reduce the feed viscosity by increasing the temperature, thus increasing the hydraulic permeability and permeate flux. Its negative effect is more evaporation of hexane solvent with increasing temperature and consequently increasing the concentration of crude oil in the miscella, leading to increased membrane fouling.

Evaluation of dominate blocking mechanism in degumming and neutralization processes

The permeate volume (v) and the processing time (t) correlation were studied in all tests. The determination of the dominant fouling mechanism was the result that showed that in most degumming processes, the dominant mechanism in the whole process is the standard blocking (Table 2).

However, in most neutralization processes, especially at high pressures, the cake mechanism has been the dominant fouling mechanism. These findings indicate that in the degumming stage, smaller particles are separated from the oil that can enter the membrane's pores and deposit on the wall of these pores. However, the separating particles are more significant in the neutralization stage and cause the cake mechanism, the dominant mechanism.

To specify the occurrence time of each fouling mechanism during the process, the blocking index was calculated. The reported results showed that in the degumming process of canola 20%, the intermediate, standard, and complete blocking mechanisms are the dominant mechanism in the initial minutes of the process, even though the formation of cake will be the dominant mechanism over time in all tests (Fig. 8).

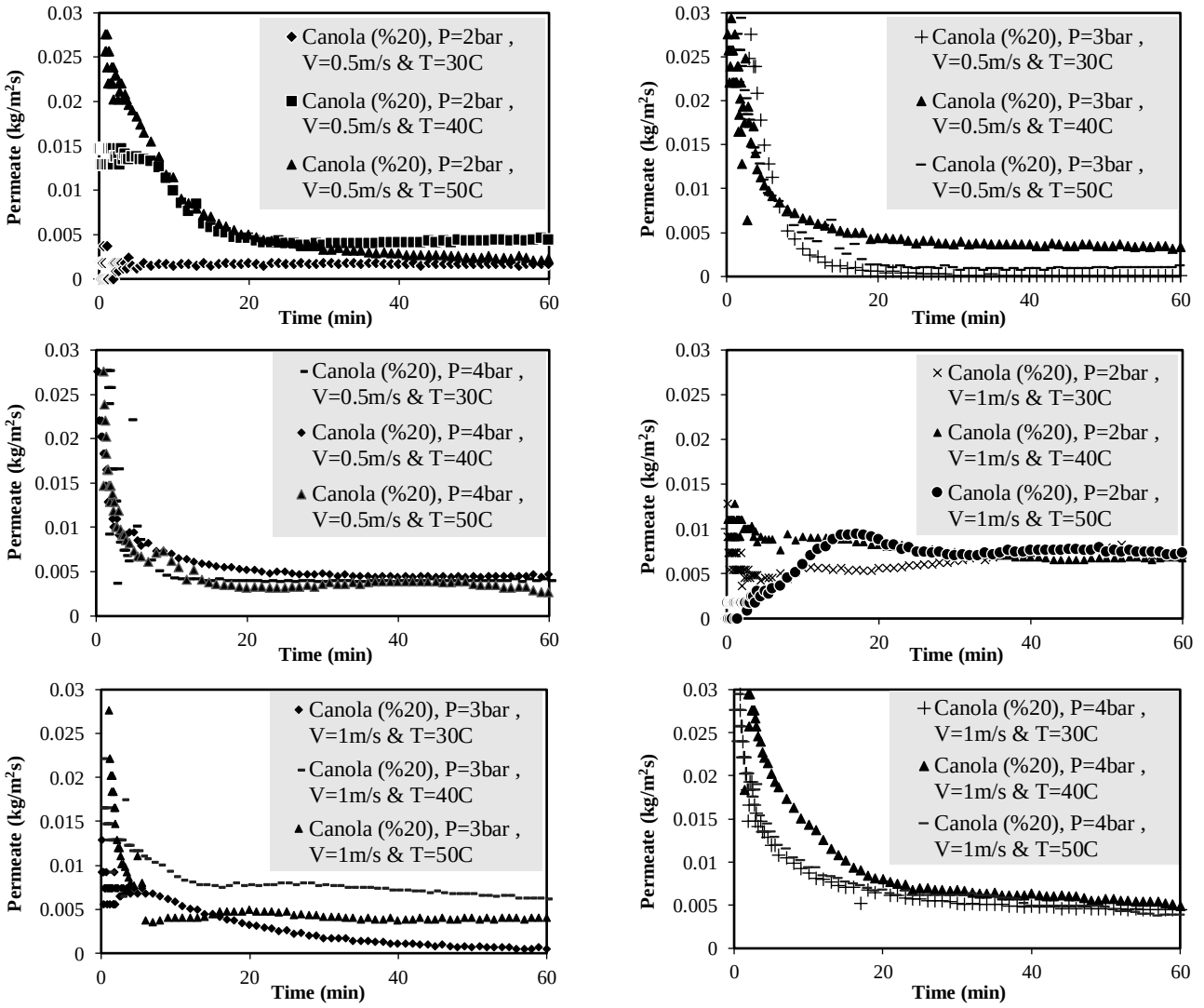

Fig. 8: The feed temperature effect on the permeate flux during second stage of membrane refining process of canola 20% (neutralization process)

However, in the degumming process of canola 30%, the cake formation mechanism was dominant at the beginning of the process, and over time other mechanisms emerged. The point about this oil was that as the pressure decreased and the flow rate increased, the intermediate and standard blocking mechanisms had more opportunities to emerge.

These findings were consistent with what was reported about permeate flux. The formation of the cake was the dominant fouling mechanism at the low pressures of the neutralization stage. However, at high pressures, canola oils with different percentages of hexane showed different behaviors (Fig. 9).

At low percentages of hexane, other fouling mechanisms also appeared at different times at high pressures. However, at high percentages of hexane, at high pressures, the cake formation mechanism was still the predominant fouling mechanism, probably due to more evaporation of hexane and oil condensation.

de Souza et al. conducted a study that applied a ceramic membrane (0.05 µm of pore diameter, on average) to degum corn oil/hexane miscella and to investigate the influx of transmembrane pressure (TMP) (0.5 and 1.5 bar), tangential velocity (1.4 and 2.4 m s^{-1}), and percentage of corn oil on the miscella (25% and 35% w/w), in terms of the permeate flux and elimination of phospholipids, and elimination of 65% to 93.5% of phospholipids was done. The result of this was achieving a minimum phosphorus content in the permeate of 23 mg/kg and color and waxes

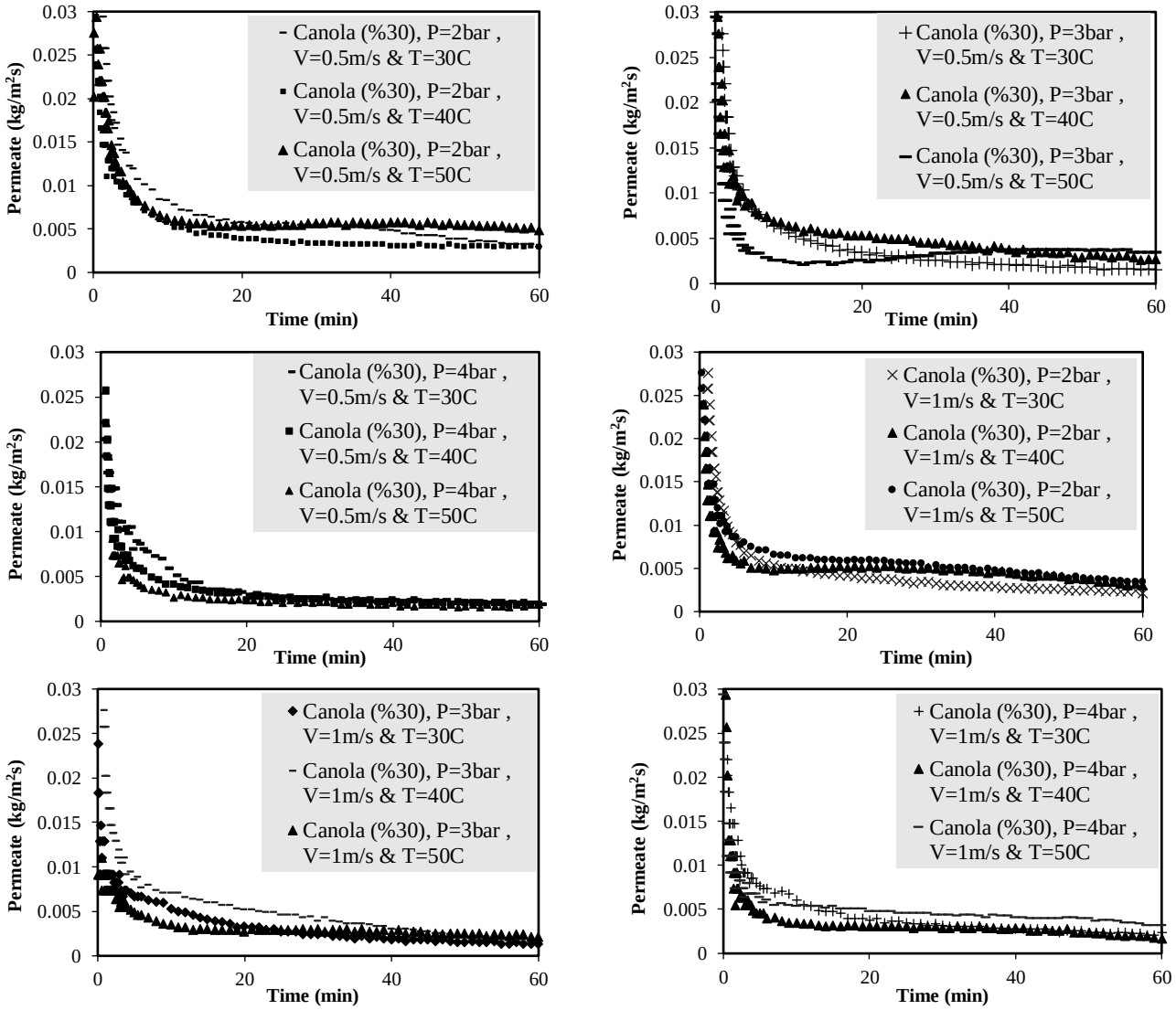

***Fig. 9: The effect of feed temperature on the permeate flux during the second stage of the membrane refining process of canola 30% (neutralization process*)**

decrement, along with the tocopherols and tocotrienols conservation in the crude oil. A raised TMP and a more significant percentage of oil in the miscella positively affected the retention of phosphorus, while the tangential velocity had a negative influence. Under the best operational conditions, the permeate flux reached 120 kg/h.m^2 at 40 ^{o}C [27].

Ribeiro et al. studied on optimization of degumming of soybean oil on a pilot plant scale, using a multi-channel ceramic membrane with a permeation area and a pore diameter of 0.2 m^2 and 0.01 µm, respectively. The concentration of phosphorous represented dependent variables in degummed oil or the permeate and the permeate flux. The tangential velocity and the transmembrane pressure (TMP) are varied from 2.9 to 3.9 m/s and 1 to 2 bar, respectively. The phospholipids were retained up to 99.7%, resulting in a 2.2 mg/kg of phosphorous concentration. The permeate flux, with a mass reduction factor (MRF) of 3.2, is varied from 21.5 to 40.5 L/m^2.h. The only variable shown to impact the process was the TMP. The lowest phosphorous content (2.2 mg/kg) in the degummed oil and the highest permeate flux (40.5 kg/m^2.h) were observed at 2 bar [49].

Basso et al. investigated the degumming and production of soy lecithin from crude soybean oil using ultrafiltration. The main goal of this research was to examine

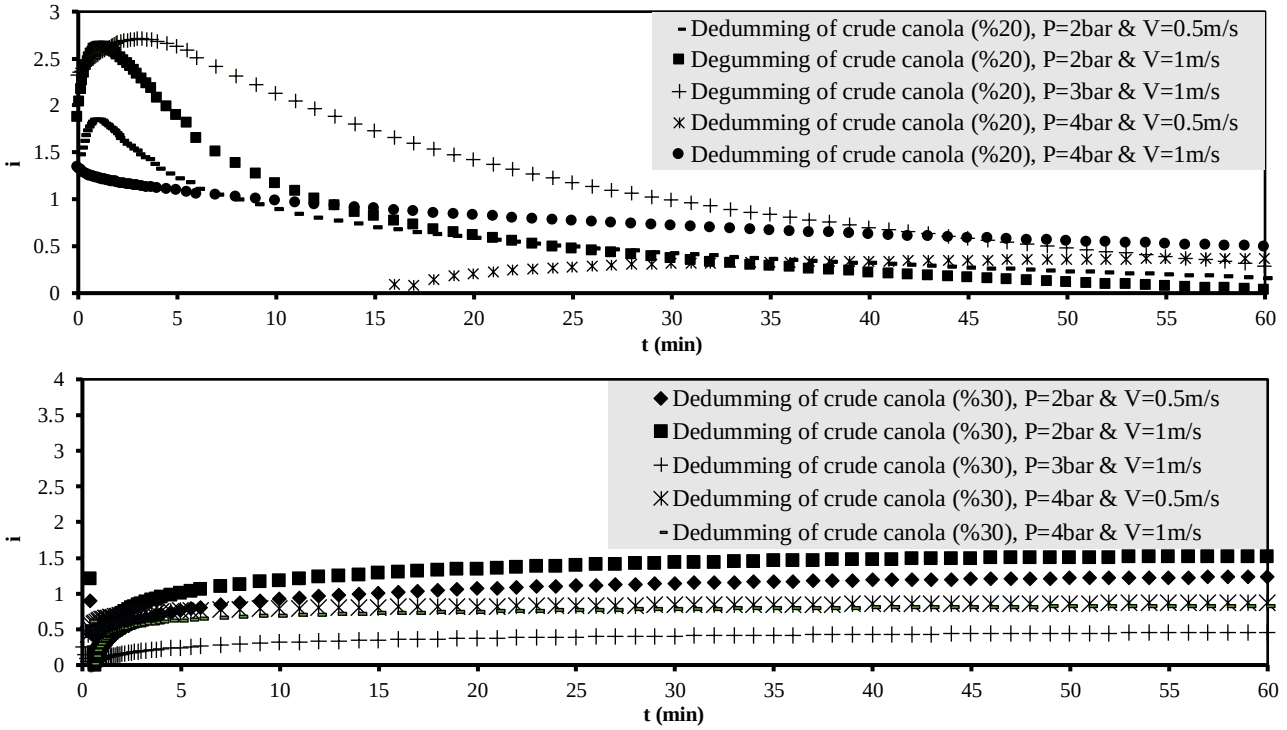

Fig. 10: Changes in blocking index (i) during the first stage of the membrane refining process of canola oil (degumming)

the impact of the pressure of the transmembrane, the velocity of cross-flow, and the opening of the permeate valve in the cleaning process, which means the circulation of hexane on a ceramic membrane with 0.2 m^2 permeation area and 0.01 mm pore diameter in a pilot unit, which have 40 L processing capacity.

In this research, four different operational conditions for cleaning, combinations of pressure (0.5–2.0 bar) and velocity (1.0–5.0 m/s), as well as the effect of opening the permeate valve were investigated.

In addition, soybean lecithin production and purification were conducted using diafiltration of the retentates derived from the UF of the miscella. Its result is a product that has 90% acetone insoluble matter. The best cleaning condition was in low pressure (0.5 bar) and elevated velocity (5.0 m/s), which made the recovery of the permeate flux possible in about 85 min [50].

In another study, *Rafe et al*. used a polysulfone amide (PSA) ultrafiltration membrane to investigate refining crude canola oil. The results showed a considerable reduction in the permeate flux with increasing the time of the process, even though it was increased by increasing the temperature from 30 to 50 °C and transmembrane pressure from 1.5 to 2 bar, as well.

Increasing the temperature or transmembrane pressure leads to a reduction of the irreversible fouling resistance (R_{if}) and percentage of fouling. According to the results, the concentration polarization resistance (R_{rf}) was much higher than other resistances. Therefore, an essential role in total hydraulic resistance was played by reversible resistance.

Regarding improving the oil refining process efficiency, the phospholipids retention, FFAs, and color were so intriguing in this study. The temperature increased the percentage of retention of phospholipids and FFAs, while transmembrane pressure and time decreased it. However, no significant difference in removing color under different operating conditions was observed [51].

CONCLUSIONS

Degumming, dewaxing, and deacidification processes of canola oil were performed in the present study's successive and successful steps. As feedstock, canola oil hexane miscella was used. Moreover, for phospholipids and FFAs, 85% phosphoric acid and alkali aqueous solution were used, respectively.

Based on the results, the permeate flux decreased sharply in the first moments and reached a steady state throughout the experiments. As with all membrane

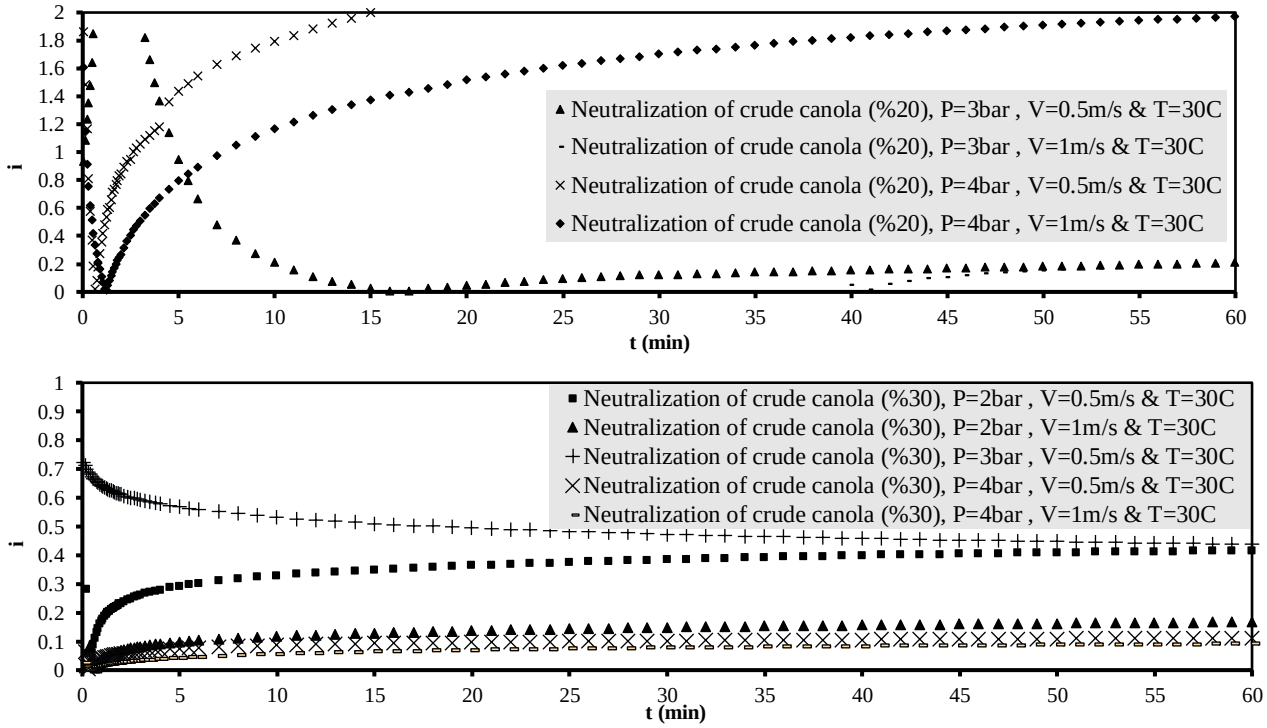

Fig. 11: Changes in blocking index (i) during the second stage of the membrane refining process of canola oil (neutralization)

processes, increasing transmembrane pressure, flow rate, and temperature increase the permeate flux. The present study showed that when the share of hexane solvent increases, the results will differ from what was expected.

So in miscella with 30% hexane, with increasing pressure from a certain amount onwards, due to more evaporation of hexane and increasing oil concentration in miscella, the permeate flux will not change, and sometimes it will decrease. For a similar reason, increasing the flow rate and feed temperature increased the permeate flux to a certain extent, but no increase was observed afterward. Studies of the predominant membrane fouling mechanisms have also shown a trend similar to that observed by permeate fluxes.

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