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Decolorization of Iranian Date Syrup by Ultrafiltration

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ABSTRACT

Ultrafiltration (UF) was used for decolorization of an industrial Iranian date syrup. Experimental results were obtained by using two different concentrations of the date syrup (40 and 50 °Brix) and two different membrane pore sizes (15-20 and 30-50 kDa molecular weight cut-off values) under different trans-membrane pressures (TMP: 40, 70, 110 and 150 psi). The membrane with a pore size of 15-20 kDa resulted in average decolorization of 56% and turbidity reduction of 90%. Increasing TMP from 40 to 150 psi led to the decolorization and turbidity reduction of 48 and 82%, respectively. When the concentration of date syrup was increased from 40 to 50 °Brix, the levels of decolorization and turbidity reductions reached 47 and 78%, respectively. Reduction in the turbidity of date syrup was correlated with increases in the lightness (L*) and yellowness (b*), while the redness (a*) was decreased. Changes in the fructose and glucose levels due to the UF operation were much less than those of color and turbidity.

Keywords: Date juice, Color reduction, Turbidity, Trans membrane pressure.

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INTRODUCTION

A large quantity of the total dates produced in different provinces of Iran is unsuitable for consumption and are usually used as a feed. But, these dates contain high amounts of sugar that can be utilized as date syrup. Date syrup is probably the most common product of date. The large amount of suspended solids and coloring matters in date syrup is a limiting factor for its use in the food formulations.

Hence, in order for the date to be used as a source of sugar in such products as beverages, it is necessary for the date syrup to be clarified and decolorized (Mostafa and Ahmed, 1981). Several studies have focused on the production of liquid sugar from the dates. Mostafa and Ahmed (1981) reported that the color groups, degradation products of reducing sugars, melanoidines and iron-polyphenolic complexes contributed to the color of date syrup. They used phosphate precipitation as an efficient clarification method for date syrup. Ehrenberg *et al.* (1997) reported that treating the date extract with lime followed by purification with cation and anion exchangers resulted in syrup with a purity of 99%. Al-Farsi (2003) reported that high quality syrup could be produced by modifying the clarification process of the date juice using filtration, hot liming and filtration, cold liming and filtration, powder-activated carbon and filtration or granular-activated carbon and filtration. Farmani *et al.* (2008) reported a refining process for sugarcane juice using microfiltration. Nasehi *et al.* (2012) tried to optimize the adsorption process of dark colored compounds in date syrup using powdered activated carbon at different operating conditions including different concentrations as well as different temperatures, where 60°C was found the best temperature among the four

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temperatures they had applied within 30-60°C.

The current methods of clarification and decolorization such as liming, resins, activated carbon, and other chemical materials suffer from several major problems. Liming and use of activated carbon involve high costs of energy and also result in environmental pollution, which cannot be neglected (Gyura *et al*., 2005). In addition, resins involve high levels of water consumption and effluent disposal.

Due to the above mentioned issues, the possibility of using membrane processes as new separation techniques has been intensively investigated. Compared with the traditional processing methods, membrane processes have some advantages. They are more economical to operate in this method and, in comparison with thermal processes, they do not need extreme heat conditions, phase changes, and chemical agent (Cassano *et al*., 2007a). Lewandowski *et al*. (1999) used ultrafiltration (UF) and electrodialysis for the decolorization and demineralization of date syrup. Ultrafiltration was performed at ambient temperature through polyethersulfone membranes with 1 and 3 kDa molecular weight cut off (MWCO) values. According to their results, the UF with 1-3 kDa MWCO suffered from the problem of very low flow rates. However, UF has shown to have a great potential for the clarification and decolorization of sugar syrups and fruit juices (Hamachi *et al*., 2003). Use of UF for the clarification and decolorization processes presents several advantages. UF processes are easy to operate with moderate temperatures and need much less energy compared to the traditional methods. Also, continuous simplified processes are possible while using the UF (Cassano *et al*., 2007a). Fukumoto *et al*. (1998) applied microfiltration (0.2 μm) and UF (0.02 μm) for the clarification of apple juice and suggested that under optimal conditions (8 m s^{-1}, 61 psi and 50°C) UF resulted in higher steady state flux and less fouling than the microfiltration for both depectinized and ascorbic-acid-treated apple juices. UF can also offer the possibility of operating in a single step and reducing the working time and easy cleaning and maintenance of the equipment (Cassano *et al*., 2007a). Therefore, in order to identify the optimum process conditions that would ensure acceptable flux with adequate juice quality, the objective of the present study was to investigate the effects of different operating parameters such as transmembrane pressure (TMP), feed concentration, and pore size on the permeate flux, rate of decolorization, turbidity loss, sugar content, as well as the *L** (lightness), **a*** (redness), and *b** (yellowness) color indices.

MATERIALS AND METHODS

Membrane Parameters

The UF operation was conducted in a laboratory cross-flow mode filtration apparatus equipped with two spiral UF membrane modules provided by Permionics (Gujarat, India), a stainless steel feed tank, two manometers to determine the inlet and outlet pressures, a feed flow-meter, and a thermometer. The membranes were made of polyethersulfone with MWCO of 15-20 and 30-50 KDa. According to the manufacturer's data, these membranes had an effective area of 2.5 m^2, an operating pH range of 2-11, an operating temperature range of 0-30°C and pressure range of 10-150 psi.

Filtration Experiments

The UF process was operated in a batch concentration mode with recycling of the retentate stream to the feed tank. Each experiment was performed at constant temperature (ambient conditions), feed flow rate (according to the applied TMP), and initial feed concentration using 30 L of feed. Levels of TMP and feed concentrations for each experiment were selected according to the Taguchi's experimental approach (Table 1) (Roy, 1990). The permeates were

Table 1. Experimental conditions applied in this study for ultrafiltration based on Taguchi's experimental design.

MWCO[a] (kDa)	TMP[b] (psi)	°Brix
15-20	150	40
15-20	110	40
15-20	70	50
15-20	40	50
30-50	150	50
30-50	110	50
30-50	70	40
30-50	40	40

[a] Molecular weight cut-off, [b] Trans-membrane pressure.

collected separately at five different time intervals (4, 10, 16, 24 and 36 minutes) to determine the changes in the color and turbidity of the products. To perform the operation, the membrane was eluted using demineralized water and then water permeate flux was measured at different TMP levels. To perform the filtration experiments, the storage vessel was filled (30 L) with date syrup. To determine the flux levels at each stage (in sequence), permeate from that stage was weighed using a digital balance. Once each date syrup filtration experiment was finished, the membrane was rinsed with tap water for 30 minutes to eliminate the polarization and cake layer. Then, a cleaning process with NaOH solution at 0.05% (w/w) (pH= 12) was applied followed by a second cleaning step with acid solution (nitric) at 0.1% (w/w) (pH= 2.4) for 60 minutes at 40°C using a high water flow rate and low TMP (about 10 psi) in order to remove the reversible polarized layer (Cassano *et al*., 2007b). After each step, water permeate flux was measured in order to determine different resistances of the filtration process.

Date Syrup

Date syrup with a color index of 13697 ICUMSA Unit (IU) and turbidity level of 90 Formazin Attenuation Units (FAU), and total soluble solid (°Brix) of 72 and pH of 4.32 was supplied by Sibasan factory (Shahrekord, Iran). Before each experiment, date syrup was diluted to known Brix levels (Table 1) using the formula $C_1V_1= C_2V_2$, where C_1 was the Brix of the original date syrup and V_1 was the volume needed from that syrup to prepare a known volume (V_2) of given °Brix (C_2). pH of the initial date syrup was measured using a calibrated pH meter. The amounts of total soluble solids were measured using a refractometer (ATAGO-DTM-1, Japan). Samples of feed, permeate, and retentate were collected during the experiments and stored at 7°C for further analysis. The color estimation was accomplished using a method recommended by ICUMSA (International Commission for Uniform Methods of Sugar Analysis). Samples were diluted to adjust the Brix and then filtered (by using 0.45 µm membrane filters). Thereafter, samples were adjusted to pH=7 (using NaOH 0.1 N) in order to measure their absorption values by spectrophotometer at 420 nm. Finally, the results were expressed in IU (Lewandowski, 1999). Turbidity was measured using a spectrophotometer (Hach DR/4000U, Colorado, and USA) in 860 nm and expressed in NTU (Nephelometric Turbidity Units). The amounts of reducing sugars were measured by using HPLC method (high performance liquid chromatography). A Vertex column operated at 25°C, with sulfuric acid solution 0.01N as the mobile phase flowing at 0.5 ml/min, was used. CIE *L**, *a**, and *b** color values were measured using a spectrophotometer (Hach DR/4000U, Colorado, USA). *L** is a measure of lightness and varies from 0 (black) to 100 (white), the *a** value varies from -100 (green) to +100 (red), and the *b** value varies from -100 (blue) to +100 (yellow).

RESULTS AND DISCUSSION

Effect of MWCO on Color, Turbidity, and Reducing Sugar

Changes in the decolorization level of date syrup for the two membranes are shown in

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Figure 1-a. As is the case with membrane applications, the color reduction was more pronounced when using the membrane with the smaller pore size (MWCO of 15-20 kDa). Average decolorization level for the membrane with MWCO of 30-50 kDa was 26% while it increased to 56% when using a membrane with 15-20 kDa pore size. Such a difference in the color reduction was due to the removal of suspended solids and color components with molecular weights greater than the MWCO of the membranes. This phenomenon is due to the increasing resistance to the permeate flux exerted by the membrane with the lower MWCO. In agreement with this study, Kardoe *et al.* (2000) showed that the use of membrane with lower MWCO led to more decolorization of a sugar solution. They reported 80% color reduction when using a membrane with MWCO of 5 kDa. Also, Lewandowski *et al.* (1999) showed higher decolorization of date syrup and lower flow rate during the ultrafiltration while applying a membrane with lower MWCO. Borneman *et al.* (2001) reported a decolorization level of 48-58% for apple juice when using membrane with MWCO of 10 kDa while they showed that membrane with MWCO of 100 kDa was not effective for decolorization of apple juice.

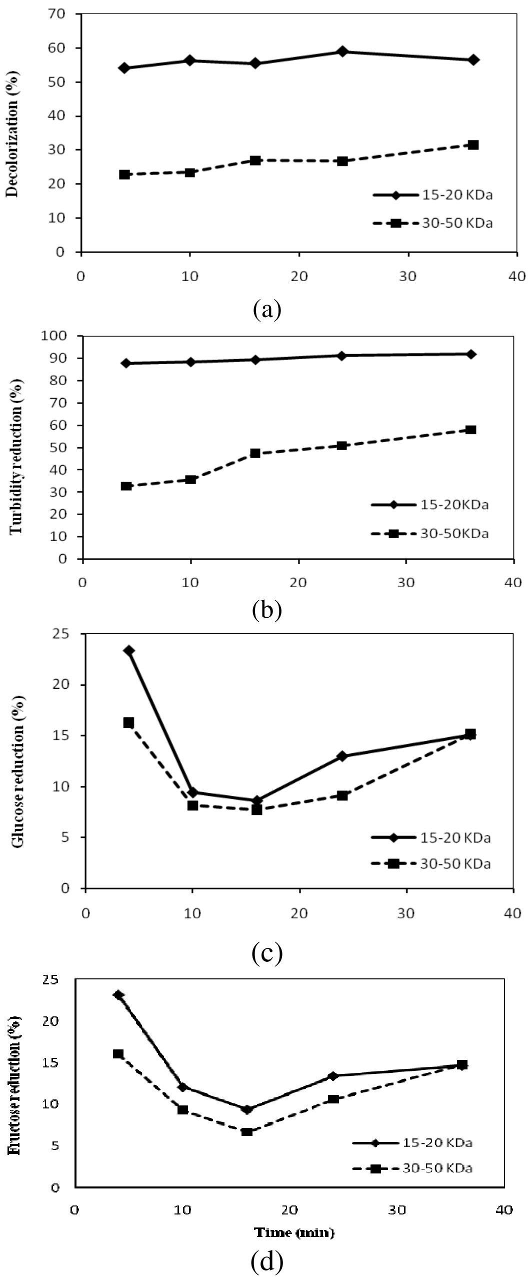

Figure 1. Effects of membrane pore size on the decolorization level of date syrup (a), turbidity reduction (b), glucose reduction (c), and fructose reduction (d) with time.

Changes in the turbidity reduction level of date syrup due to a change in the MWCO of membrane are shown in Figure 1-b. Turbidity losses of 30-60 and 89-91% were found for membranes with 30-50 and 15-20 kDa MWCO, respectively. Therefore, the best result was obtained from the membrane with lower MWCO (15-20 kDa) due to smaller pore size resulting in a higher removal of suspended solids. This is in agreement with the results of previous studies indicating higher turbidity loss when using membranes with lower MWCO (Vladisavljevic, *et al.*, 2003; Bruijn *et al.*, 2002; Cassano *et al.*, 2007a, b; Shahidi and Razavi, 2006; Balakrishnan *et al.*, 2000; Carvalho *et al.*, 1998). You *et al.* (2008) reported a 77% loss in the turbidity of apple juice when using a 50-kDa membrane. In the current study, the efficiencies of membranes in reducing the turbidity decreased during the process, due to the increase in the concentration polarization and membrane fouling.

Date syrup mainly contains sugars, 95% of which are reducing sugars (Lewandowski *et* *al*., 1999; Al-Farsi, 2003). Therefore, the effects of membrane parameters on the level of reducing sugars were investigated in this study. Reduction of glucose and fructose levels when using membranes with different MWCO are illustrated in Figure1c, d. In the case of membranes with 30-50 and 15-20 kDa MWCO, average reductions of glucose content were, respectively, 11.3 and 13.9%. These amounts for fructose content were 11.5 and 14.5%, respectively. These results are in good agreement with those reported by Carvalho *et al*. (1998) on the ultra- and microfiltration of pineapple juice. Their experiments with the 50 kDa MWCO membrane resulted in 10-11% glucose reduction and 12-16% sucrose reduction. Moreover, by applying the membrane with 0.22 μm pore size, the reduction of glucose and fructose contents were found at 9 and 10%, respectively (Carvalho *et al*., 1998). Balakrishnan *et al*. (2000) reported 2-4% reduction in sugar content when using a 50-kDa UF membrane for sugar cane juice. As reported above, there are no obvious differences in the behaviors of the two membranes in the removal of fructose and glucose, which is probably due to the smaller sizes of glucose and fructose molecules, compared to the MWCO values of both membrane modules used in the current study. On the other hand, a portion of these sugars did not pass through the membranes, indicating that they may have been absorbed in the membrane or bound with other ingredients not passing the membranes. Majority of the reduction in the sugar content occurred at the beginning of each process (Figures 1-c and -d.). According to the results of this study, glucose reduction through the UF membranes with 15-20 and 30-50 kDa MWCO were, respectively, 23.3 and 16.3%. These amounts were 23.2% and 16.0%, respectively, for fructose reduction. In addition, the lowest reduction happened in the middle (10-20 minutes) of each process. This phenomenon was probably due to the presence of free sites on the membrane surface at the beginning of the processes and led to the removal of sugar molecules from the date syrup. After a few minutes of the process, these sites were filled and, as a consequence, the efficiency of membrane filtration decreased. After 16 to 36 minutes, a gradual increase in the sugar reduction was observed, which was related to the increase in the concentration polarization and membrane fouling .

Effect of TMP

Effect of TMP on the decolorization level of the permeate samples during 36 minutes of UF process is shown in Figure 2-a. Increasing the TMP from 40 to 150 psi resulted in an increase in the decolorization level from 28% to 48%. A higher TMP results in the formation of a denser polarization layer; consequently, the membrane resistance against the crossing color components resulted in an increase in the level of color reduction as the process continued. Decloux *et al*. (2000) and Gyura *et al*. (2005) reported similar results on the decolorization level. But, according to Hamachi *et al*. (2003), such changes are only related to the difference in MWCO of the membrane used, where an increase in the pressure (at higher pressure levels) results in the formation of a cake layer on the membrane surface leading to an increase in the resistance of the membrane against the flux.

Also, increase of TMP from 40 to 150 psi led to an increase in the permeate flux (0.12 to 0.22 kg min^{-1} m^{-2}) as well as the decolorization levels of the date syrup (Figures 2-a and -b). Since the pressure is a driving force for the UF processes, with an increase in the pressure (from 40 to 110 psi), permeate flux also increased. However, after 110 psi TMP level, no clear changes were found in the permeate flux. At higher pressures, the formation of the cake layer on the membrane surface can lead to an increase in the resistance of membrane against the flux (Jacob and Jaffrin, 2007).

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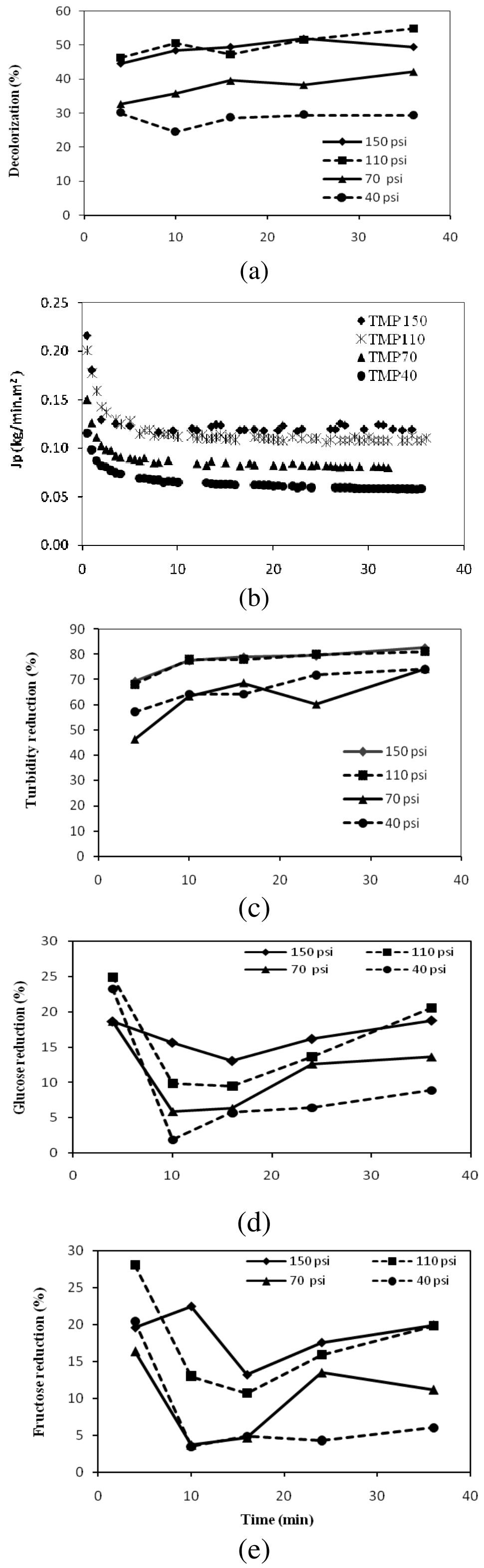


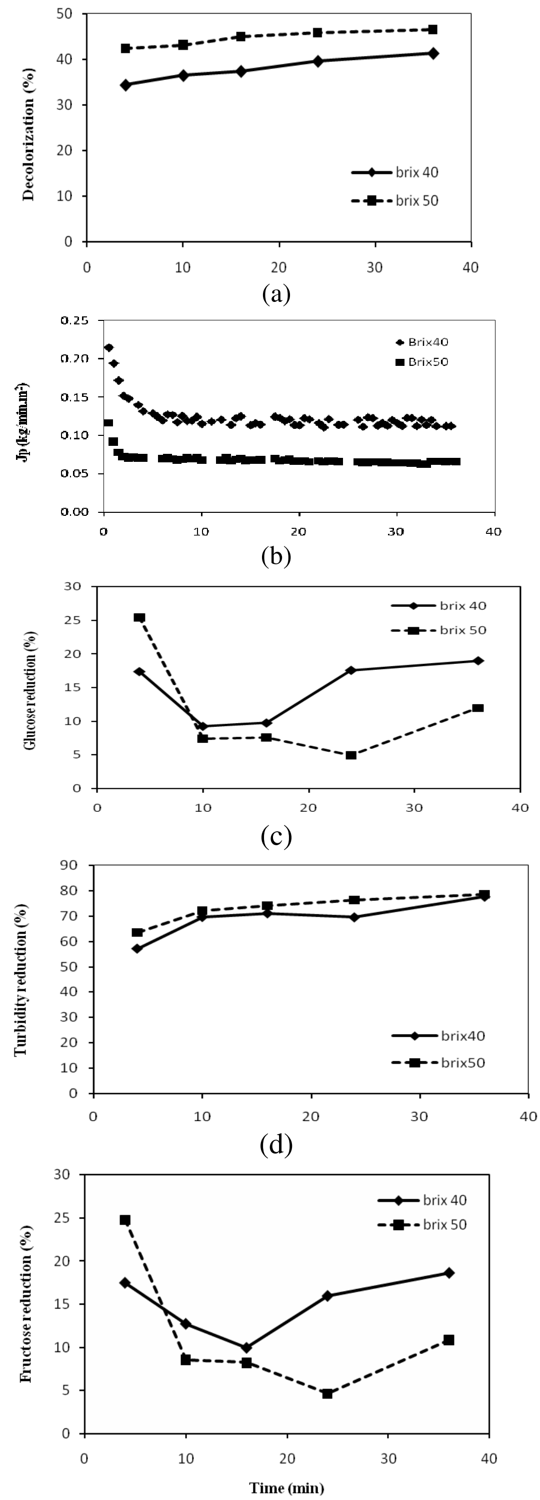
Figure 2. Effects of trans-membrane pressure (TMP) on the decolorization (a), permeate flux (b), turbidity reduction (c), glucose reduction (d) and fructose reduction (e) of date syrup with time.

Figure 2-c presents the effect of different pressure levels on the reduction of turbidity during the UF processes applied in the current study. The reduction in the turbidity level was increased as the TMP level increased from 40 to 110 psi (68 to 82%). However, no such reduction was found at TMP levels greater than 110 psi. At all pressure levels applied here, the turbidity reduction rate was low at the beginning and gradually increased as the processes advanced further. During each process, a cake layer gradually forms that can act as a secondary membrane and cause more resistance against crossing of the materials (Seres *et al*., 2004).

With an increase in the TMP level, reductions were found in both glucose and fructose levels (Figures 2d and -e). For instance, with raising the pressure from 40 to 150 psi, glucose and fructose reduction increased, respectively, from 9.2 to 16.5% and from 7.8 to 18.5%. As it is obvious in both figures, the declining trends of the sugars reduction in the initial stage were followed by rising trends in the final stages.

Effect of Brix

The changes of the color reduction efficiency at different syrup concentrations are shown in Figure 3-a. A decolorization level of 34 to 41% was found for the date syrup with a 40 °Brix during 0-36 minutes of the operation, while this range varied within 42-64% for the date syrup with Brix= 50. This means that higher level of color reduction is observed for the date syrup with greater °Brix due to the higher transfer rate of the components towards the membrane surface that causes the cake layer to thicken and results in a higher membrane resistance against the color components. During such changes in the color, the permeate flux decreased from 0.21 to 0.12 kg min^{-1} m^{-2} (Figure 3-b). That is, at lower concentration, permeate flux was higher. Decrease in the concentration of date syrup resulted in the lower transport rate of materials to the cake



(e)

Figure 3. Effects of feed concentration (°Brix) on the decolorization (a), permeate flux (b), turbidity reduction (c), glucose reduction (d) and fructose reduction (e) of date syrup with time.

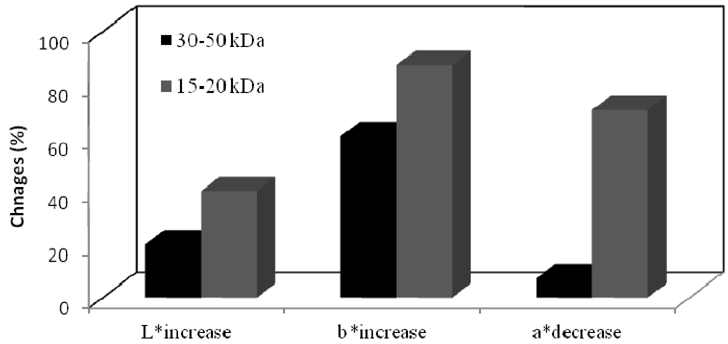
layer on the membrane surface. This phenomenon leads to a thinner thickness of the cake layer and, therefore, resistance of the membrane decreases.

The changes in the turbidity reduction efficiency at different syrup concentrations are shown in Figure 3-c. When °Brix increased from 40 to 50, turbidity decreased due to an increase in the crossing rate of materials through the membrane leading to formation of a denser cake layer and increase in the membrane resistance against the material transportation. The rate of turbidity reduction was increased from ~60% at the beginning of the 50 °Brix to 80% at the end of the process. For the other °Brix (40), the starting value was slightly lower.

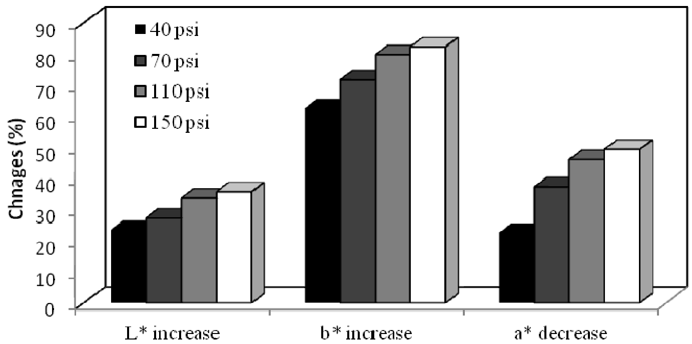
The changes in the sugar reduction for the two syrup concentrations studied here are shown in Figures 3-d and -e. Sugar reductions were found at higher levels when using the 40 °Brix as compared to 50 °Brix. This can be related to the total sugar contents of the original syrups, where a constant sugar removal from both syrups results in a higher contribution in the fraction of the sugars removed from the date syrup with the lower concentration i.e. 40 °Brix.

Changes in the CIE *L*, a** and *b** Color Indices during the UF Process

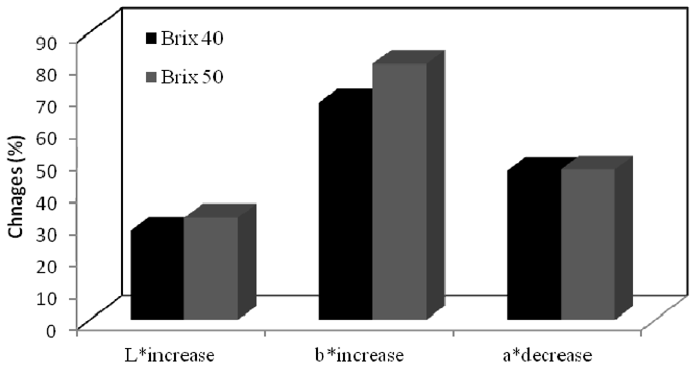
In addition to the ICUMSA method, to follow the changes in the color and turbidity of the date syrup (previous sections), a colorimetric procedure was applied in the current study to determine the changes in the color of date syrup in the UF process (Figure 4). Major changes were found in the three indices related to such colorimetric method (L*, a* and b*) including an increase in the *L** and *b** and a decrease in the *a** values of the date syrup. Main color components of date syrup include melanoidins, which contribute to the redness of date syrup (Mostafa and Ahmed., 1981). Since these compounds posses a polymeric structure with high molecular weight, UF processing is often capable of removing them resulting in the reductions in the red color of date



(a)



(b)



(c)

Figure 4. Effects of changes in molecular weight cut off value (a), trans-membrane pressure (b) and °Brix of date syrup (c) on *L** (lightness), *b** (yellowness) and *a** (redness) values of the permeate.

syrup due to the UF process (Belitz *et al.*, 2009). Following the reduction in the red color, the yellow color might be illuminated due to the presence of carotenoids and flavonoid pigments with lower molecular weights present in the permeate (MacDougall, 2002, Socaciu, 2008).

The effects of changes in the membrane pore size, TMP, and °Brix of the date syrup on *L*, a** and *b** indices are also shown in Figure 4. Although lightness levels of the permeate samples obtained from both membranes were higher than those from the feed samples, the membrane with 15-20 kDa MWCO was more effective in increasing the lightness resulting in an average increase of 40% in the *L** value when compared to the membrane with 30-50 kDa MWCO, which resulted in an increase of 20% in the *L** value (Figure 4-a). Part of the increase in the lightness could be related to the removal of haze-causing agents, which were reduced through the filtration process. Also, the membrane with 15-20 kDa MWCO resulted in greater decrease (70%) in the red color (a* value) of date syrup when compared to the other membrane, which resulted in 7% reduction in *a** value. A similar trend was found for the changes in the *b** value when using the two membranes of the current study. Increases in the *b** values were recorded at 60 and 87% for the 30-50 and 15-20 kDa membranes, respectively.

With an increase in the TMP level from 40 to 150 psi, *L** and *b** values also increased accordingly (Figure 4-b). Applying a higher pressure causes a denser cake layer on the membrane surface and subsequently results in blocking the passage of the components through the membrane. With increase in the applied pressure from 40 psi to 150 psi, the increase of *L** value improved from an average level of ~23 to 49%. *b** values corresponding to the permeates during such changes in the TMP level increased from 63% for the 40 psi to 82% for the 150 psi TMP levels. In addition, reduction in the *a** value changed from an average of 23% for the 40 psi to 49% for the 150 psi TMP levels.

The last part of the current study dealt with the changes in the *L*, b*,* and *a** values as the feed concentration was increased from 40 to 50 °Brix (Figure 4-c). The highest level of changes were found in the *b** values, which increased by a change in the °Brix from 40 to 50. This indicated that the yellowness of the product was greater (i.e., 80 *vs*. 68% for the increase in the b* values) when the feed concentration was at higher level.

CONCLUSIONS

This research aimed at investigating the effects of ultrafiltration process on the physical and chemical specifications of date syrup. The results show that applying the 15-20 kDa membrane instead of 30-50 kDa membrane as well as the increase in the °Brix of date syrup (from 40 to 50) improved its decolorization level. However, the use of syrup with higher Brix value with the current membrane resulted in a decrease in the flux rate. On the other hand, increasing the TMP not only compensated the drop in the flux, but also increased the level of decolorization. Moreover, in comparison with the TMP of 150 psi, 110 psi resulted in more optimized level of decolorization at similar flux and lower energy was required to operate at that pressure. A significant improvement was obtained in the syrup by applying a higher TMP and Brix levels. Also, the membrane with smaller MWCO was more effective in increasing the lightness of the permeate. Finally, the results showed that, during the process of ultrafiltration, the date syrup lost its sugar content, but the loss was not at a level to be concerned, considering the level of decolorization gained.

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رنگبری شهد خرمای ایرانی از طریق فراپالایش

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چکیده

به منظور رنگبری از شیره خرمای صنعتی از فن آوری نوین فرا پالایش استفاده گردید. نتایج تحقیقات حاصل از فرآیند شربت خرما با درجه بریکس ۴۰ و ۵۰ با دو غشاء با اندازه روزنه های ۲۰-۱۵ و ۵۰-۳۰ کیلو دالتون، در شرایط فرآیندی مختلف از جمله با اختلاف فشار غشایی ۴۰، ۷۰، ۱۱۰ و ۱۵۰ Psi نشان داد که غشاء با اندازه روزنه های ۲۰-۱۵ کیلو دالتون به طور متوسط تا سطح 56% قدرت رنگبری و ۹۰٪ کاهش کدورت شربت خرما را موجب شده است. افزایش اختلاف فشار غشایی از ۴۰ به ۱۵۰ Psi منجر به کاهش ۴۸٪ میزان رنگ و ۸۲ % کدورت از شربت گردید. با تغییر میزان درجه غلظت شربت از ۴۰ به ۵۰ درجه بریکس میزان کاهش رنگ تا ۴۷٪ و میزان کاهش کدورت تا سطح ۷۸٪ رسیده است . ارتباط کم شدن میزان کدورت با افزایش شاخص روشنایی (L*) و شاخص زردی (b*) بوضوح مشاهده گردید. در حالیکه میزان شاخص قرمزی (a*) کمتر گردید. تفاوت میزان فروکتوز و گلوکز در شربت فرآیند شده به طریق غشاء فراپالایش با نمونه شربت قبل از فرآیند در مقایسه با تغییرات رنگ و کدورت بسیار ناچیز بوده است.

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