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Research Paper

Effect of Brine Type on the Performance of Red Grapefruit Juice Concentration by Osmotic Distillation

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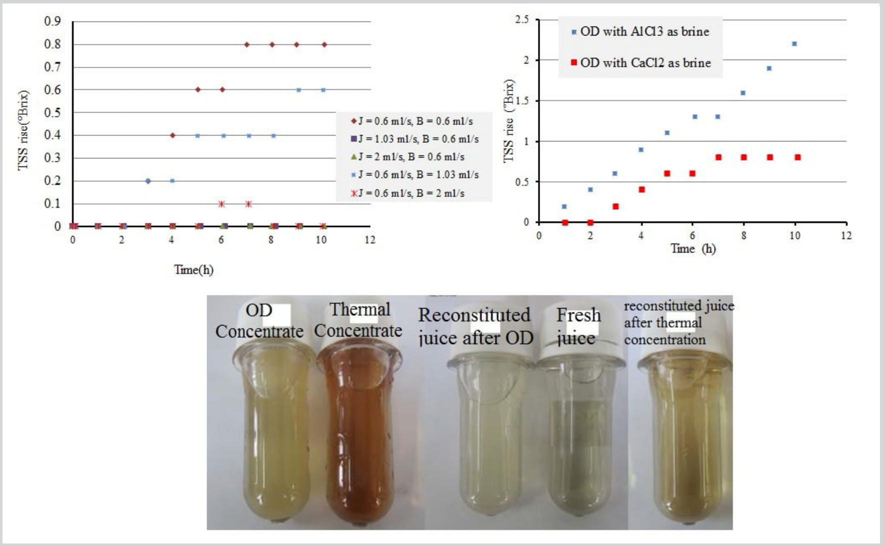
Highlights

- $AlCl_3$ and LiCl were the best salts during concentration of grapefruit juice with OD.
- Low flow rates of brine and juice were much more effective than other processes.
- OD makes a desirable concentrated grapefruit juice.

Graphical abstract



Abstract

Grapefruit juice contains a large number of nutrients that should be preserved during processes such as concentration. In the present study, osmotic distillation (OD) was used to concentrate this juice compared to the thermal process. The extracted juice was centrifuged (2000 rpm, 5 min) and osmotic distillation was performed in a laboratory scale flat sheet module by PVDF membrane (0.22 µm). After evaluation of osmotic pressure of 40 salts, $AlCl_3$ and LiCl were selected because of their high potential to produce osmotic pressure, and the volumetric rates of the brine and the juice were 0.6, 1.3 and 2 $mL.s^{-1}$. Thermal concentration was done in a rotary evaporator at 70˚C to 30 °Brix to compare the changes in physicochemical properties. The results showed that the low flow-rate of brine and juice was more effective during OD. It was also concluded that concentration of grapefruit juice with $AlCl_3$ and LiCl was more efficient than concentration with $CaCl_2$. The vitamin C content did not significantly change comparing to 40% decrease in thermal concentration. Also, polyphenol content increased after OD but did not change significantly after thermal concentration. The antioxidant activity and pH were constant in both concentration methods. The total acidity of fresh juice increased by about 23% after OD, but it remained constant with thermal concentration. Juice turbidity increased after both concentration methods.

1. Introduction

Red grapefruit, which is one of the most nutritious members of the citrus family, has bioactive components with antioxidant activity, such as vitamin C, flavonoids, and carotenoids. These components reduce the risk of cardiovascular disease and cancer and have antimicrobial, antiviral, anti-ulcer, and anti-allergy effects [1-3].

Due to the limited growing season of grapefruit, year-round consumption requires that it is preserved. Thermal concentration is the most common preservation method; however, it reduces vitamins, aroma, color, and other valuable qualities of the juice; Thus the concentrated product has significantly reduced the overall quality [4-6]. Osmotic distillation (OD) is a membrane technology that can concentrate sensitive foodstuffs such as fruit juices at

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room temperature and normal pressure to minimize the destruction of their chemical component [7-10]. In OD, a hydrophobic membrane separates juice and brine, which have different vapor pressures. The water vapor passes through the membrane from the juice to the brine side, causing both diluted brine and concentrated juice [11-14].

Several parameters, such as brine type and concentration, would affect OD performance. Hence, studies have focused on changing such parameters to increase process efficiency and nutrient concentration. Cassano et al. [15] produced pomegranate-juice concentrate using OD after juice clarification with ultrafiltration and concluded that the beneficial physicochemical characteristics in the concentrated juice, such as antioxidant activity, were preserved. Belafi-Bako and Boor [16] concentrated cranberry juice using both osmotic evaporation and osmotic membrane distillation and confirmed that nutrient compounds were retained. Kujawski et al. [17] concentrated red grape juice with OD and concluded that antioxidant activity and total phenolic content were preserved in the concentrated juice. Destani et al. [18] successfully used a combination of ultrafiltration and osmotic distillation in the laboratory scale to concentrate blood-orange juice. Achili et al. [19] used 14 osmotic solutions at an osmotic pressure of more than 1 MPa to select the optimal solution for forward osmosis. Results showed that $NaHCO_3$ and $KHCO_3$ caused the highest flux. Kujawski et al. [20] studied the concentration process of red grape juice using OD with polytetrafluoroethylene membrane and calcium chloride (50 wt.%) as brine solution. They concluded that the final TSS changes with time and the initial TSS. Also, the nutritive compounds of juice were preserved after the OD process. Cassano et al. [21] introduced OD and membrane distillation (MD) processes as a potential process for fruit and vegetable juice concentration. They believed that product quality improvement and energy saving are two important parameters affecting the attractiveness of these two processes. Boór et al. [22] concentrated the juice of cornelian cherry, blackthorn, white beam, and elderberry using OD. They resulted that 60 °Brix with a flux of 0.3-2.4 $Lm^{-2}h^{-1}$ could be achieved. Also, they concluded that the valuable compounds of all juices are preserved during the concentration process. Roozitalab et al. [23] compared the concentration process of pomegranate juice by OD with the thermal evaporation method. They concluded that the preservation of phenol content and aroma is higher in OD than in the thermal process; however, they present the thermal process as a more economical process.

One of the parameters affecting the efficiency of the osmotic distillation process is the type of salt used in the brine. Choosing the right type of salt in making salt water can have a favorable effect on the osmotic pressure created and finally on the efficiency of the concentration process by OD. Despite the importance of the topic, no comprehensive research has been done on it so far. In this study, the osmotic pressures of 40 salts were measured. Three salts that theoretically had the highest osmotic pressure were used to concentrate the red grapefruit juice through OD. The effect of selected salts on the performance of the concentration process was studied. Also, its effect on the physicochemical properties of red grapefruit juice was evaluated.

2. Materials and method

2.1. Extraction of fruit juice

The grapefruit was prepared from a local market (Tehran, Iran), and the fresh fruits were washed and manually peeled. The juice was produced using a domestic juicer (Tefal, Prepline 600 mL, France). The extracted juice was clarified using a centrifuge at 2000 rpm for 5 min (Ortoalresa S11, spin) and the clarified juice was stored at -25 °C in PET bottles until concentration time.

2.2. Osmotic distillation (OD)

Osmotic distillation was performed at 25˚C using a laboratory-scale OD unit (Fig.1). A 0.22 μm hydrophobic Polyvinylidene fluoride (PVFD) membrane (Millipore, USA) was used to separate the brine solution and the grapefruit juice. The brine on one side of the membrane and the fruit juice on the other side of the membrane were both passed and recycled by a peristaltic pump (Etatron DS-Rome, POP TR ACQUE, Italy). The juice tank was placed on a digital balance coupled with a computer to determine the water-vapor flux. Several volumetric rates of the brine and the juice were selected and examined (0.6, 1.3, and 2 mLs^{-1}) to evaluate their effect on the concentration performance.

Grapefruit juice was also concentrated using thermal processing in a rotary evaporator (Heidolph, Germany) at 70˚C to study the effect of the process on the juice's physicochemical characteristics compared to the effect of membrane concentration. The final total soluble solids in the concentrated juice were the same for both thermal and membrane methods (up to 30 °Brix).

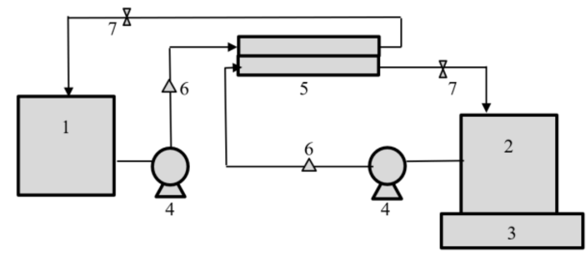

Fig. 1. Scheme of the osmotic-distillation unit (1: brine tank; 2: juice tank; 3: digital balance; 4: peristaltic pump; 5: membrane module; 6: pressure meter; 7: flow meter)

2.3. Selection of appropriate brine

The literature suggests that the difference between osmotic pressures in brine and feed solutions is the driving force in OD; for this study, 40 salts were selected and their osmotic pressures were calculated using Eq. 1.

$$\pi = i\varphi CRT \tag{1}$$

Where, i is the number of ions produced with salt degradation; and φ, C, R, and T are the osmotic coefficient, concentration of solute components ($mol.L^{-1}$), gas constant ($L.bar.mol^{-1}.K^{-1}$), and temperature (K), respectively [24].

All experiments were conducted at a similar temperature to remove its effect on the solubility of different salts.

2.4. Measurement of mass transfer coefficient

The water transfer coefficient was measured using Eq. 2.

$$J_W = K(a_2 - a_1) \tag{2}$$

Where K, J_W, a_1 and a_2 are the mass transfer coefficient ($kg.m^{-2}s^{-1}$), the water flux ($kg.m^{-2}s^{-1}$), and the water activities of the feed and brine, respectively.

A water-activity meter (Lab Master-aw, Novasina, Switzerland) was used to measure the water activity at 25˚C.

2.5. Assessment of physicochemical characteristics of juice

Changes in the physicochemical characteristics of the samples were determined in both the membrane and the thermal concentration. The total soluble solid content (TSS) of the concentrated juice was adjusted to the initial TSS using fresh juice to study only the effect of the concentration method.

TSS of the grapefruit juice was measured at 25˚C by a portable refractometer (ATAGO, HSR-500, Japan), and the data was reported in degrees Brix. The dry-matter content was determined by the difference between the initial and the final weights after removing its moisture and achieving constant weight at 105 °C. Turbidity was measured by a digital turbidity meter (TU-2016, Germany) and expressed in NTU.

The antioxidant activity was evaluated by measuring the radical-scavenging property of the juice in presence of 2,2-diphenyl-1 picrylhydrazyl (DPPH). First, 0.1 mL juice was mixed with 3.9 mL of methanolic solution of DPPH (25 $mg.L^{-1}$), and the absorbance of the mixture was measured using a UV-Vis spectrophotometer at 515 nm (Perkin Elmer LAMBDA 25 US/VIS, America) which was reported as $[DPPH]_t$ using a standard curve. The test was repeated with methanol instead of the juice, and the measured DPPH content was reported as $[DPPH]_{t=0}$. The remaining DPPH was obtained from Eq. 3 [25].

$$DPPH_{rem} = \frac{[DPPH]_t}{[DPPH]_{t=0}} \tag{3}$$

To determine the amount of phenolic compound, the fruit juice (1 mL), HCL (1 mL, 6M), and the solution of methanol in water (75% v/v) were mixed together. The final solution was stirred in a water bath (Memmret, Germany) at 90 ℃ for 2 hours. It was cooled to 25℃ and diluted to 10 mL, and 1 mL of the resulting solution was mixed with 5 mL folin that was 10 times diluted. This mixture was added to 15 mL of Na_2Co_3 (0.07 gmL^{-1}) and its volume was increased to 100 mL. The absorbance of the resulting solution was measured using the UV-Vis spectrophotometer (Perkin Elmer LAMBDA 25 US/VIS, America) at 760 nm against a control sample of distilled water. A standard curve was plotted using gallic acid, and the value of phenolic components was expressed as mg gallic acid per 100 mL.

To study total acidity, 2.5 g of grapefruit juice was mixed with 22.5 mL of distilled water, and titration with NaOH (0.1 N) was done in the presence of phenolphthalein until pH = 8.1 was achieved. Acidity was measured using Eq. 4.

$$A=100\frac{0.0046\ M}{W} \quad (4)$$

Where A, M, and W are total acidity (g citric acid per 100 g fruit juice), the volume of NaOH, and the weight of the juice sample, respectively. pH was measured using a pH meter (Metrohm-691, Switzerland).

To determine the value of vitamin C, 15 g of meta-phosphoric acid was dissolved in 40 mL of pure acetic acid and 200 mL of distilled water. Its volume was increased to 500 mL, and it was immediately filtered with a filter paper (expressed as an extraction solution). Next, 10 g of the sample (m_0) was mixed with one to three times its weight of extraction solution and was filtered. Three different volumes of the filtrate were immediately titrated by color reagent until a stable, bright pink color was achieved. The amount of color reagent consumed was expressed as A. A control test was also performed with the extraction solution in which the value of the consumed color reagent was expressed as B. To prepare the color reagent, 50 mg sodium salt of 2,6 dichlorophenolindophenol was dissolved in 150cc warm distilled water (50-60°C) that contained 42 mg sodium bicarbonate, and its volume was increased to 200 mL. The test was repeated with 5 mL of the standard solution of ascorbic acid instead of the sample solution to standardization; ascorbic-acid weight (mg) equal to 1 mL color reagent was expressed as m_1 (Eq. 5).

$$\text{Ascorbic acid content}\,(\text{mg})\,\text{in 100 g sample} = \frac{(A-B)m_1}{m_0}.100 \quad (5)$$

2.6. Statistical analysis

The mean value of data was reported after three times repetitions. The one-way ANOVA method was chosen for statistical analysis of the data, and mean values were compared by applying Duncan's multiple range tests using Minitab 15 software.

3. Results and discussion

3.1. Effect of brine and juice flow rate on the concentration performance

Three different flow rates for both the brine and the grapefruit juice (0.6, 1.03, and 2 mLs^{-1}) were selected to evaluate their effect on the membrane concentration. Results showed that the minimum value for both juice and the brine flow rates caused the maximum increase in brine weight (Fig. 2). The best concentration performance was achieved with these parameters, as shown by the increased juice TSS (Fig. 3). The minimum increase in brine weight was achieved when one of the flow rates was at the highest value. Changes in the juice TSS confirmed the data. Studies have specified three steps for mass transfer inside the membrane in an OD unit (Fig. 4):

Vaporization of water in the membrane interface – liquid on the juice side

Transfer of water vapor inside the membrane pores

Condensation of water vapor in the membrane interface – liquid on the brine side [17, 26, 27].

Low flow rates for both the juice and the brine give enough time to all three steps for concentration performance will be at its maximum.

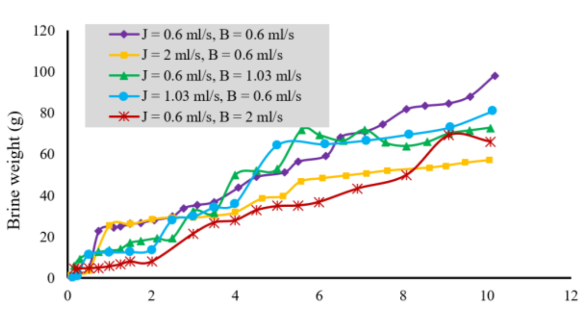

Fig. 2. Effect of juice and brine flow rates on the brine weight during membrane concentration of grapefruit juice (J = juice flow rate, B = brine flow rate)

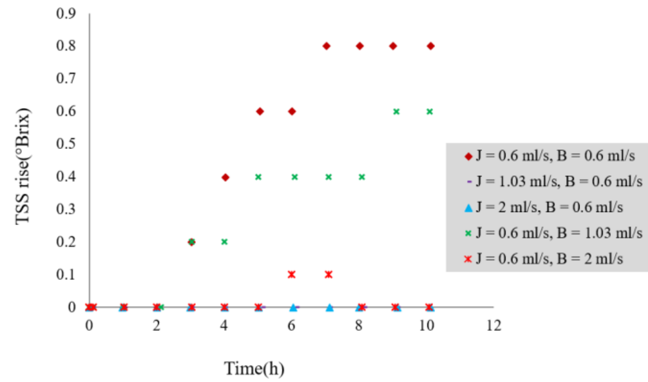

Fig. 3. Effect of juice and brine flow rates on the total soluble solid content during membrane concentration of grapefruit juice (J = juice flow rate, B = brine flow rate)

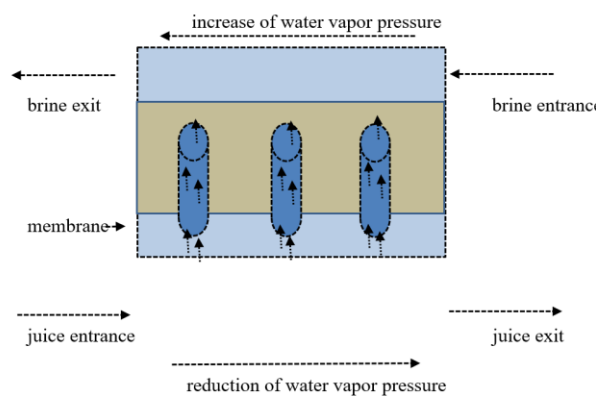

Fig. 4. Mechanism of mass transfer in the osmotic-distillation unit (Hogan et al., 1998)

3.2. Theoretical selection of the best salts

To select the most efficient brine, 40 salts were selected and their osmotic pressures were theoretically calculated (Table 1). Two aspects were considered in this evaluation:

To remove the effect of salt quantity on the process performance while evaluating osmotic pressures of different salts in similar concentrations, salt saturation concentration was ignored; $AlCl_3$ was selected from this point of view, as it had the highest value for ion number multiplied by the osmotic coefficient.

In the second step, the saturation concentration of salts was included in the evaluation of the osmotic pressure to select the salt that would produce the most osmotic pressure; LiCl was selected as the most efficient salt type from this point of view.

The concentration performance of these salts was compared with $CaCl_2$, which is commonly used in OD.

Results showed that the saturated solution of lithium chloride and aluminum chloride caused more osmotic pressure, and consequently more vapor flux, than calcium chloride during membrane concentration of grapefruit juice using OD (Figs. 5, 6). Overall, the water vapor flux in this study was much lower than that obtained by Kujawski et al. [20] in the membrane concentration of red grape juice. The reason for this is the lower temperature of the process compared to that research and probably the different hydrophobic nature of the membrane used in the two processes and the difference in the type of fruit juice used. $AlCl_3$ increased the TSS of grapefruit juice two to three times more than $CaCl_2$, which confirms the theoretical measurements (0.8 °Brix TSS rises for $CaCl_2$ and 2.2 °Brix TSS rises for $AlCl_3$ after 10h processing, Fig. 7). On the other hand, compression between LiCl and $CaCl_2$ showed that LiCl was more efficient in concentrating the grapefruit juice than $CaCl_2$. The concentration of grapefruit juice was followed for about 21 h, and it was observed that LiCl can increase the TSS of juice from 10 to about 30 °Brix (Figs. 8, 9). In contrast, the water transfer coefficient during OD of the grapefruit juice at 10 h was 0.764 $kg.m^{-2}h^{-1}$ when a saturated solution of $CaCl_2$ was chosen as the brine, and 1.003 $kg.m^{-2}h^{-1}$ when a saturated solution of LiCl was selected.

Table 1. Osmotic pressures of several salts that were theoretically measured

Salt name	M_s	φ_s	i	$i \times \varphi_s \times M_s$	$i \times \varphi_s$
$BaCl_2$	1.624	0.964	3	4.697	2.89
$Mg(NO_3)_2$	3.67	2.289	3	25.202	6.87
$Ca(NO_3)_2$	5.57	1.238	3	20.687	3.71
K_2CO_3	5.41	1.66	3	26.942	4.98
$ZnSO_4$	3.104	1.254	2	7.785	2.51
LiCl	14.47	2.936	2	84.968	5.87
NaBr	7.09	1.656	2	23.485	3.31
NH_4Cl	5.887	0.934	2	10.997	1.87
NH_4NO_3	11.89	0.495	2	11.771	0.99
$NaNO_3$	7.697	0.783	2	12.053	1.57
KBr	4.39	1.087	2	9.544	2.17
$(NH_4)_2SO_4$	4.04	0.663	3	8.036	1.99
$CuSO_4$	1.295	1.12	2	2.901	2.24
$FeSO_4$	1.796	0.61	2	2.191	1.22
$MnCl_2$	4.88	1.354	3	19.823	4.06
NaCl	5.28	1.276	2	13.475	2.55
$NaNO_2$	10.106	1.167	2	23.587	2.33
KiO_3	0.42	4.21	2	3.536	8.42
RbCl	5.79	1.156	2	13.386	2.31
$AlCl_3$	2.86	3.36	4	38.438	13.44
$Al(NO_3)_3$	2.31	2.24	4	20.698	8.96
$Al_2(SO_4)_3$	0.984	1.927	5	9.481	9.63
NaF	0.968	0.871	2	1.686	1.74
$NaClO_4$	4.65	1.062	2	9.877	2.12
$NaBrO_3$	2.34	0.792	2	3.707	1.58
KCl	4.077	0.99	2	8.072	1.98
$AgNO_3$	8.953	0.351	2	6.285	0.70
NH_4NO_3	11.759	0.507	2	11.924	1.01
$LiNO_3$	10.35	1.8	2	37.260	3.6
NaI	7.091	2.164	2	30.690	4.33
$NaClO_3$	8.174	1.855	2	30.325	3.71
NaCNS	9.967	1.639	2	32.672	3.28
KF	12.459	1.953	2	48.665	3.91
CsCl	11.36	1.019	2	23.152	2.04
CsF	16.87	1.189	2	40.117	2.38
$ZnCl_2$	11.113	2.252	3	75.089	6.76
KNO_2	13.55	0.560	2	15.189	1.12
$ZnBr_2$	10.02	2.326	3	69.932	6.98
$CaCl_2$	5.154	3.083	3	47.674	9.25
$Cu(NO_3)_2$	5.254	2.396	3	37.763	7.19

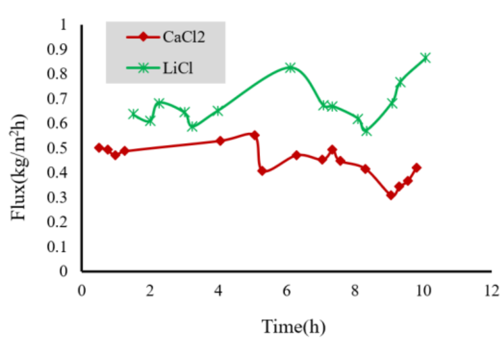

Fig. 5. Water-vapor flux during the membrane concentration of grapefruit juice using LiCl and $CaCl_2$

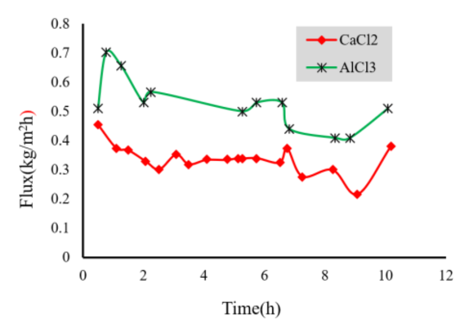

Fig. 6. Water-vapor flux during the membrane concentration of grapefruit juice using $AlCl_3$ and $CaCl_2$

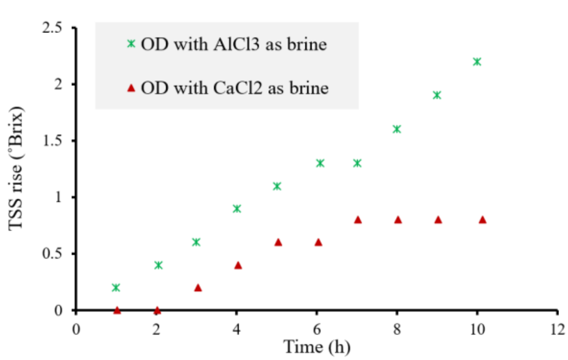

Fig. 7. Difference between application of $AlCl_3$ and $CaCl_2$ in concentration

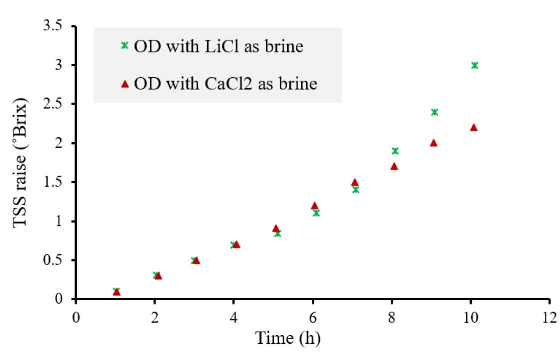

Fig. 8. Difference between application of LiCl and $CaCl_2$ in concentration

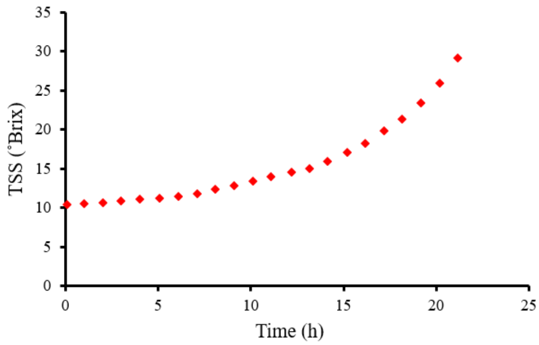

Fig. 9. Performance of LiCl in increasing the total soluble solid contents of grapefruit juice

3.3. Changes in physicochemical properties of grapefruit juice during membrane concentration

Grapefruit juice was concentrated using both OD and thermal processes, and the TSS of concentrated juice was adjusted to its initial value in fresh juice to study the net effect of the concentration method on the physicochemical characteristics of the grapefruit juice.

Results indicated that the content of vitamin C in the reconstituted juice made from the membrane-concentrated sample was not significantly different from the fresh juice; however, the thermal concentration method decreased ascorbic acid by about 40% (Table 2). Similar results were obtained by Bahçeci et al. [28]. They concluded that OD preserved more vitamin C compared to the thermal process. Evaluation of the polyphenol content of grapefruit juice before and after concentration showed that its value increased after membrane concentration; however, it did not significantly change after thermal concentration. The polyphenol content of apple and beet juices did not change after concentration with OD [29].

The antioxidant activity of grapefruit juice did not significantly change after concentration in either method. This result was in accordance with other studies in which apple, grape, and some vegetable juices were concentrated by thermal and OD methods and the antioxidant activity of juices did not change with either concentration method [30, 31]. The pH of samples behaved in a similar way to antioxidant activity; however, the total acidity of fresh juice increased by about 23% after membrane concentration, while the acidity level remained constant with thermal concentration; this difference was due to the long processing time of OD. Juice turbidity increased after the concentration process in both methods.

Visual assessment of different grapefruit-juice samples after both the membrane and the thermal concentration showed that the browning reaction in thermally concentrated juice was much more than in juice produced with OD (Fig. 10). In contrast, reconstituted grapefruit juice after OD was more similar to fresh juice than the reconstituted juice after thermal processing.

Table 2. Changes in physicochemical characteristics of grapefruit juice.

Characteristic	Concentrated using osmotic distillation		Concentrated using thermal processing	
	Fresh juice	Reconstituted concentrate	Fresh juice	Reconstituted concentrate
Turbidity (NTU)	23.99±0.11^{a,*}	36.66±0.57^{b}	27.55±0.3^{a}	32.04±0.1^{b}
Acidity (g citric acid/100g sample)	1.22±0.22^{a}	1.5±0.25^{b}	1.48±0.05^{a}	1.43±0.07^{a}
pH	3.37±0.06^{a}	3.32±0.04^{a}	3.31±0.02^{a}	3.27±0.06^{a}
Ascorbic acid (mg/100g)	13.23±1.80^{a}	11.73±3.16^{a}	15.05±0.04^{a}	9.03±1.04^{b}
Total phenolic component (mg Gallic acid/100 mL)	0.913±0.002^{a}	1.034±0.001^{b}	0.973±0.001^{a}	0.972±0.001^{a}
Antioxidant activity (mL juice/ g DPPH)	19.64±6.02^{a}	19.4±5.57^{a}	19.85±6.02^{a}	20.13±6.01^{a}

* The same letter in each row and for each concentration method shows no significant difference between the data.

Fig.10. Grapefruit-juice samples (a: concentrate produced with OD; b: concentrate produced with the thermal process; c: reconstituted juice after OD process; d: fresh juice; e: reconstituted juice after thermal concentration).

4. Conclusion

Osmotic distillation is a potential method to concentrate grapefruit juice. $AlCl_3$ and LiCl are introduced as two desirable salts that can efficiently concentrate grapefruit juice. The high efficiency of LiCl is due to its high saturation concentration; however, the high number of ions is responsible for the performance of $AlCl_3$. Both reasons led to the high osmotic pressure difference between the juice and the brine which is the driving force of the process. So, the rate of mass transfer increases due to the high level of juice concentration. Reduction of brine and juice flow rates can increase the concentration of juice during the process.

Nutrient components of grapefruit juice such as vitamin C and phenolic compounds are sensitive to high temperature; so, each process at high temperature destroys nutrients. As mentioned, OD is a concentration process operating at low temperatures. Since the concentration of grapefruit juice with OD retains a high level of nutrient components compared to the thermal method.

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