

# Effects of concentration method and storage time on some bioactive compounds and color of jujube (*Ziziphus jujuba* var *vulgaris*) concentrate

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**Abstract** Jujube extract was concentrated by various heating methods including vacuum, microwave and conventional heating. The effect of concentration methods and storage time (for 90 days at 4 °C) on total phenolic and total monomeric anthocyanin contents, individual anthocyanins, individual organic acids, and color values of jujube concentrate was investigated separately. The desired level of concentration (65.0 °Brix) was achieved in 45, 96 and 117 min by the microwave, vacuum and conventional heating methods, respectively. The concentrate obtained with microwave method had the highest total phenolic content (159.32 mg GAE/g DW) and total monomeric anthocyanin content (48.84 mg cyn-3-glu/100 g DW) in comparison to the other methods at the beginning of storage. Hunter color parameters ( $L^*$ ,  $a^*$  and  $b^*$ ) decreased significantly with increasing the time of storage in all cases; however, this effect was more obvious in the vacuum heating. Cyanidin-3,5-diglucoside was determined as the major anthocyanin in all concentrates, while its degradation was more pronounced in the conventional heating (25.59%) comparing to the microwave (11.14%) and vacuum methods (17.59%) during the 90-day storage. The jujube concentrate prepared with the microwave method had the highest organic acid contents (e.g. malic, citric, succinic and ascorbic acids) as compared to the other methods. Thus, according to the results, the heating method and storage time had significant effects on the bioactive compounds and color values of jujube concentrate. In general, microwave energy could be successfully used in

production of jujube concentrate followed by 90-day storage.

**Keywords** Jujube · Microwave · Vacuum heating · Storage · Bioactive compounds · Color

## Introduction

Nowadays, consumers' demand of new functional foods, as a result of their increasing consciousness of food-health interactions, is clearly rising. In addition, interest in protection of bioactive compounds and a safe alternative method for preservation of processed fruits and fruit juices has recently increased significantly throughout the world (Ekici 2014). In industrial processing, juices are usually concentrated for ensuring longer storage life, easier transportation, and higher resistance to microbial and chemical deterioration than the original juices (Onsekizoglu 2013). The traditional concentration of fruit juices is done by thermal evaporation technique, resulting in degradation of anthocyanins, loss of color, and declination of volatile compounds of the final product (Maskan 2006). In addition to concentration process, storage time may change the quality of the final product (Ekici 2014; Alighourchi and Barzegar 2009).

The jujube (*Ziziphus* spp.) from Rhamnaceae family is extensively grown throughout the tropical and subtropical areas of the world, especially in Asia and America as well as in the Mediterranean region (Kou et al. 2015). Iran is one of the main producers of jujube with an annual production of 3000 ton, and the extent of its cultivation is 1350 ha (Golmohammadi 2013). Jujube fruit is a considerable source of phenolic compounds, anthocyanins, flavonoids, organic acids, etc. (Wu et al. 2012; Gao et al.

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2012). The consumption of jujube fruit and its products due to its health promoting effects has considerably increased. Jujube fruit can be eaten fresh or used to make beverages, jam, jelly, pickle and other traditional products (Sun et al. 2011). Hence, in the present research, jujube concentrate is considered as a functional food product. Moreover, since jujube fruit is obtained at special months of the year, some techniques should be applied for preparing its concentrate.

To the best of our knowledge, nobody has studied the effect of different concentration methods on bioactive compounds and color values of jujube fruit concentrate and their changes during storage. Therefore, the objective of the present study was to investigate the effects of various concentration methods (microwave, vacuum and conventional heating) and selected storage times at 4 °C on the total phenolic and total monomeric anthocyanin contents, individual anthocyanins, individual organic acids, and color values of jujube concentrate.

## Materials and methods

### Materials

Sweet-mature jujube fruits (*Ziziphus jujuba* var *vulgaris*) were originally obtained from South Khorasan Agricultural and Natural Resources Research and Training Center, Iran. The fruits were washed in cold tap water and their stones were removed manually. Then they were dried in hot air oven (Memmert, Germany) at 40 °C for 8 h. The initial moisture content of jujube fruit was found to be 47.51% that was reduced to 3.46% on drying. Then a fine powder was obtained using a mill (Moulinex, Type DPA1, CMMF 800 W, France), and was passed through a 30-mesh sieve. The jujube powder was freeze-dried at −18 °C in dark conditions until use.

### Preparation of jujube extract

500 ml distilled water was added on 50 g jujube powder; then the mixture was slightly shaken at  $20 \pm 2$  °C in rotary shaker (IKA®, KS 4000i Control, India) at a speed of 180 rpm for 24 h. The extract was filtered with filter cloth and then double layered filter paper. Finally, the clear extract was stored in colored vials at 4 °C (Ekici 2014).

### Concentration of jujube extract

The extract was concentrated to a final °Brix of 65.0 from the initial °Brix of 25.3 by the following evaporation processes:

1. *Microwave heating* A programmable domestic microwave oven (Arçelik ARMD 580, Turkey) with the

maximum output of 900 W at 2450 MHz was used. The oven has adjustable power (wattage) and time controllers and is fitted with a turntable. Heating above or below 350 W power level results in some problems such as foaming, charring of juice, or lengthy of concentration time. Therefore, the study was carried out at 350 W power level. 500 ml of the extract sample was put in a beaker and placed in the microwave oven.

2. *Rotary vacuum evaporator* 500 ml of the extract sample was concentrated under vacuum at 40 °C using a rotary evaporator (Heidolph, Schwabach, Germany).

3. *Evaporating at atmospheric pressure (conventional heating)* The jujube extract was concentrated using an electromagnetic heater (IKA, Staufen, Germany) by stirring continuously. For this purpose, 500 ml of the extract sample was continuously heated and stirred during this process.

The temperature for the microwave, vacuum and conventional heating was 96, 40 and 98 °C, respectively. Samples were taken for the measurement of °Brix periodically in all processes. Then they were allowed to cool down at room temperature. Each experiment was performed in triplicate.

### Determination of total soluble solids (TSSs)

During each concentration process, the total soluble solid content of the samples was measured with a digital refractometer (DR-AIATAGO, Japan) at 20 °C and expressed in °Brix (AOAC 2016).

### Color measurement

The color of concentrates was measured using a Hunterlab Color Flex (Hunterlab, Reston, Virginia, USA). The instrument was standardized each time with black and white tiles ( $L = 92.23$ ,  $a = -1.29$ ,  $b = 1.19$ ). The Hunter color values ( $L^*$  lightness,  $a^*$  redness and  $b^*$  yellowness) were evaluated (Timmermans et al. 2011).

### Determination of total phenolic content (TPC) and total monomeric anthocyanin content (TMAC)

Total phenolic content (TPC) was determined using spectrophotometer (Cary 60, Agilent, US) by Folin–Ciocalteu colorimetric method (Yang et al. 2010; Girones-Vilaplana et al. 2012). Briefly, 20 µl of the sample was mixed first with 1.58 ml of distilled water and then with 100 µl of Folin–Ciocalteu reagent. Then 300 µl of saturated Na<sub>2</sub>CO<sub>3</sub> (20%) was added. After the mixture was allowed to stand for 30 min at 40 °C. Then the absorbance was measured at 765 nm. The standard curve of the absorbance of gallic acid was used, and the results were reported as mg gallic-

acid equivalents per g dry weight of the extract (mg GAE/g DW).

Total monomeric anthocyanin content (TMAC) was estimated by pH-differential method (Cam et al. 2009). Two buffer systems were used: 0.025 M potassium chloride buffer (pH 1.0), and 0.4 M sodium acetate buffer (pH 4.5). 200  $\mu$ l of the sample was mixed with 1.8 ml of either potassium chloride or sodium acetate buffer, and the absorbance of the sample was recorded at 510 and 700 nm according to the following equation:

$$A = (A_{510 \text{ nm}} - A_{700 \text{ nm}})pH_{1.0} - (A_{510 \text{ nm}} - A_{700 \text{ nm}})pH_{4.5} \quad (1)$$

The results were reported as mg of cyaniding-3-glucoside equivalents per 100 g dry weight of extract (mg cy-3-glu/100 g DW) according to the following equation:

$$\text{Monomeric anthocyanin pigment (mg/l)} \\ = A \times MW \times DF \times 1000 / (\epsilon \times L) \quad (2)$$

where A is absorption value, MW is molecular weight (449.2 g/mol), DF is dilution factor,  $\epsilon$  is molar absorptive coefficient (26,900 l/mol/cm), and L is the cell path length (1 cm).

### Analysis of individual organic acids

Jujube concentrate was diluted to 5° Brix with 50 mM  $H_3PO_4$  solution prior to organic acid analysis. The diluted sample was directly loaded onto a Sep-Pak C18 cartridge (Millipore, Milford, USA), previously conditioned with 2 ml of methanol. Then it was equilibrated by passing 2 ml of distilled water at a flow rate of approximately two drops per second using a 2 ml plastic syringe. The separation of organic acids in the clarified sample was carried out using a Waters HPLC system, isocratically, with Empower software, equipped with a pump (Waters 600), a Rheodyne 7125i six-way injector with 20  $\mu$ l sample loop and a UV-Vis detector (Waters model 2487). A column (Prontosil 120-5 C18 AQ 250  $\times$  4.6 mm, dp 3  $\mu$ m with precolumn; Knauer, Berlin, Germany) was used for separation. The injected samples volume was 20  $\mu$ l at room temperature, and the mobile phase was  $H_3PO_4$  (50 mM) at a flow rate of 0.7 ml/min and wavelength of 205 nm. The individual organic acids were identified by comparison of their retention times with those of pure standards and spiking some samples. Calculation of the concentrations was based on the external standard method (multiple point calibration curves). Organic acid standards including oxalic, tartaric, malic, shikimic, ascorbic, citric, maleic, succinic, quinic and malonic acids were purchased from Supelco (USA). The obtained results were expressed as mg per 100 ml (Berenji Ardestani et al. 2015).

### Analysis of individual anthocyanins

Identification of anthocyanins was performed using Waters HPLC unit with a Nucleodur C18 Gravity column (4.6  $\times$  250 mm, dp 5  $\mu$ m) from Macherey–Nagel (Düren, Germany). Assessment of anthocyanins was according to a modified procedure of Del Carpio Jiménez et al. (2011). 20  $\mu$ l of the sample clarified by a Sep-Pak C18 cartridge (Millipore, Milford, USA) and a 0.45  $\mu$ m acetate cellulose filter was injected into the HPLC system. The elution was carried out at room temperature at a flow rate of 0.8 ml/min by UV-Vis detector at the wavelength of 520 nm using aqueous solvent A (10% formic acid) and acetonitrile (B) in a linear gradient from 95% A and 5% B for 0–1.67 min, 90% A and 10% B for 3.34 min, 80% A and 20% B for 20 min to 95% A and 5% B for 25 min. The individual anthocyanins were identified and quantified by comparing to the retention times of pure external standards such as cyanidin-3-glucoside (Sigma-Aldrich, >95% purity), cyanidin-3,5-diglucoside (Sigma-Aldrich, >90% purity), delphinidin-3-glucoside (Santa Cruz Biotechnology, >95% purity), delphinidin-3-5 diglucoside (Santa Cruz Biotechnology, >90% purity), pelargonidin-3-glucoside (Sigma-Aldrich, >97% purity), pelargonidin-3,5-diglucoside (Sigma-Aldrich, >95% purity) and peonidin-3-glucoside (Sigma-Aldrich, >95% purity); and their content was expressed as mg/100 ml.

### Statistical analysis

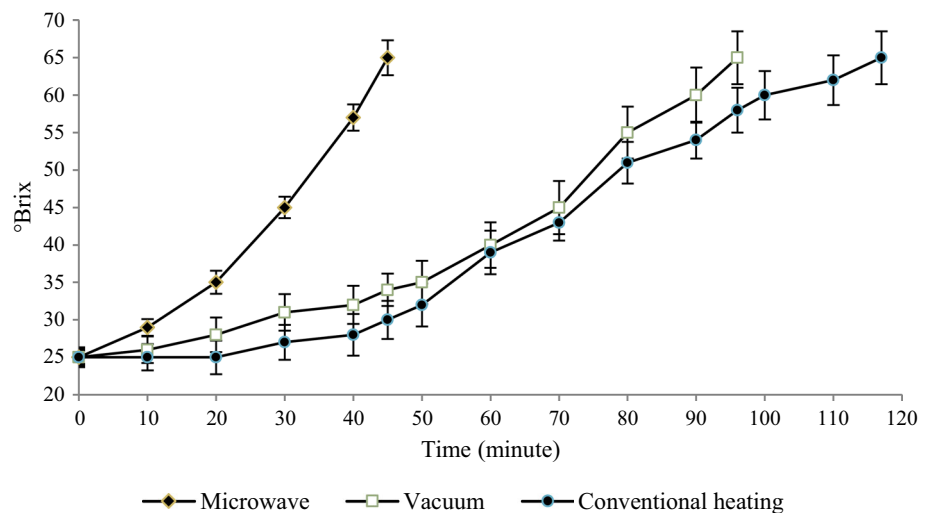
All treatments were performed in triplicate. Data analyses were carried out using the SPSS software (ver. 20; SPSS Inst., Cary, NC, USA). All results were shown as the mean  $\pm$  standard deviation (SD) of three replicates. Means comparisons were used by the one-way analysis of variance (ANOVA) followed by Duncan's test ( $P < 0.05$ ).

## Results and discussion

### Soluble solid

The soluble solid concentration (°Brix) of jujube extract for three concentration methods is shown in Fig. 1. The time required to obtain the desired final concentration (65.0 °Brix) was 45, 96 and 117 min for the microwave, vacuum and conventional heating processes, respectively. The results represented a significant difference among the mean evaporation times of the three techniques ( $P < 0.05$ ).

**Fig. 1** Change in °Brix of jujube concentrate produced by various concentration processes



### Color parameter

Hunter color parameters ( $L^*$ ,  $a^*$  and  $b^*$ ) of the jujube concentrate produced by different methods and stored at 4 °C for 90 days can be seen in Fig. 2. As shown, all concentration methods and prolongation of storage time decreased the color parameters ( $L^*$ ,  $a^*$  and  $b^*$ ) of jujube concentrate significantly, and all the products turned red-dish brown.

The  $L^*$  (lightness) value decreased with increasing the time of storage, and the color of samples got darker. The loss of  $L^*$  value of the samples concentrated by the microwave, conventional and vacuum heating methods after a 90 day-storage at 4 °C was 32.94, 37.16 and 44.17%, respectively. The highest loss of  $L^*$  value was determined in the samples treated with the vacuum process. There was significant difference ( $P < 0.05$ ) between the mean  $L^*$  values of different concentration methods.

The  $a^*$  (redness) value of the jujube concentrate decreased during storage by all methods of concentration. The reduction in this parameter was not very severe as compared to the  $L^*$  value. Loss of  $a^*$  value was 10.34, 15.38 and 20.0% for the conventional, microwave and vacuum heating methods during the storage period, respectively. Concentration of the jujube extract by various heating methods had effect on the mean  $a^*$  values statistically ( $P < 0.05$ ). A similar behavior of this parameter was described by Alighourchi and Barzegar (2009) for reconstituted pomegranate juice stored at 4, 20, and 37 °C for 210 days, and by Kammerer et al. (2007) for canned strawberry fruits with added black carrot concentrate during storage over 24 weeks.

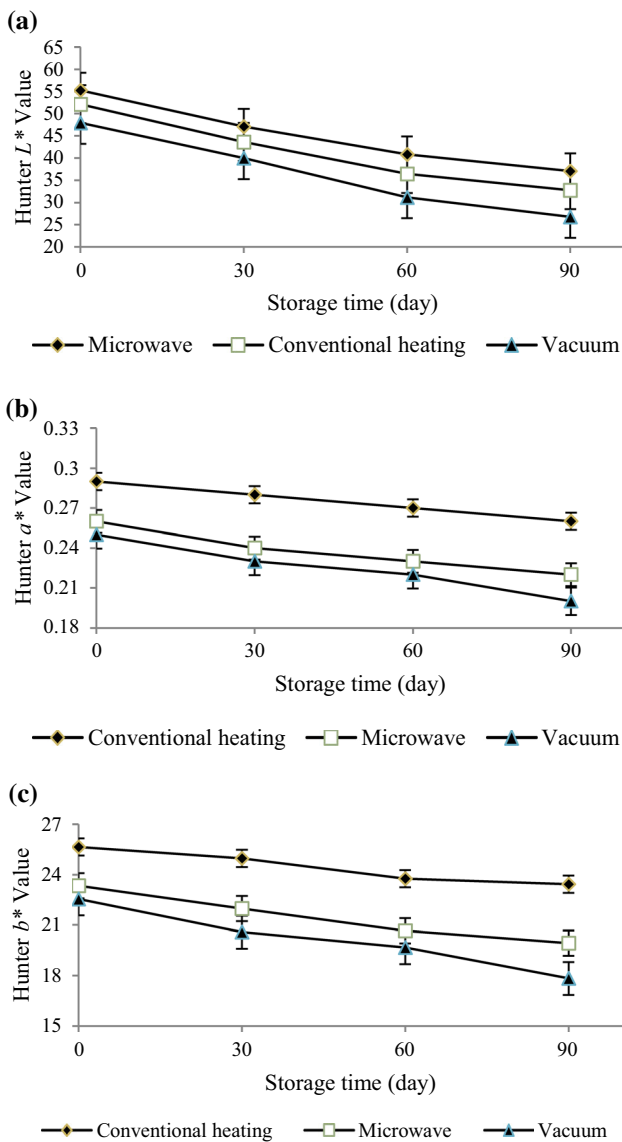
A decreasing trend of  $b^*$  (yellowness) value was observed in all samples during storage at 4 °C. The loss of  $b^*$  value by the conventional, microwave and vacuum processes was 8.61, 14.61 and 20.93%, respectively, at

4 °C after a 90 day-storage. Statistically, the change in  $b^*$  value for the above processes was significantly different ( $P < 0.05$ ). A similar trend was reported by Ahmed et al. (2004) in plum puree during heating and by Hojjatpanah et al. (2011) in black mulberry (*Morus nigra*) juice concentrate during storage.

Several researchers have reported a significant correlation between decrease in the  $L^*$  and  $a^*$  values and increase in the browning of foods during thermal processing and color destruction (Ekici 2014; Maskan et al. 2002). Losses of  $L^*$ ,  $a^*$ , and  $b^*$  values were accompanied by increased polymeric color values, which can be attributed to the degradation or polymerization of anthocyanins with different phenolics. Consequently, the compounds formed by the co-pigmentation phenomenon lead to increase in polymeric color values, as well as formation of brown pigments of fruit concentrate (Patras et al. 2010). Chromatic parameters of the samples in the present work showed that the amount of loss was higher in the vacuum heating method ( $L^*$ : 44.17%,  $a^*$ : 20.0% and  $b^*$ : 20.93%) than in the other methods. This may be attributed to low temperature (40 °C), used which could not inactivate the naturally present oxidative enzymes such as polyphenol oxidase, resulting in color deterioration during the processing and specially during the storage (Maskan 2006; Patras et al. 2010).

### Total phenolic content (TPC)

TPC of jujube concentrates is given in Fig. 3. As shown, the different concentration techniques significantly affected on the TPC of jujube concentrates ( $P < 0.05$ ). The sample obtained with the microwave method (159.32 mg GAE/g DW) had the highest phenolic content in comparison with the vacuum (130.43 mg GAE/g DW) and conventional methods (100.23 mg GAE/g DW) at the beginning of the storage.



**Fig. 2** Variation of Hunter color  $L^*$  (a),  $a^*$  (b) and  $b^*$  (c) values of jujube concentrate produced by various concentration processes at storage time 0, 30, 60 and 90 days

Moreover, the highest total phenolic stability was observed in the jujube concentrate treated with the microwave method during storage. The loss of TPC of the samples concentrated by the microwave, vacuum and conventional methods after a 90 day-storage at 4 °C was 11.36, 25.94 and 29.71%, respectively. The results showed that the storage time had significant ( $P < 0.05$ ) effect on the TPC of jujube concentrates. Similar to the present results, Begić-Akagić et al. (2011) reported that the phenolic content decreased by 20.19–24.49% in apple cultivar juice during a 60 day-storage at 1 °C, and Varela-Santos et al. (2012) observed a significant decrease of 39.6% in the phenolic content of pomegranate juice during a 35 day-storage at 4 °C ( $P < 0.05$ ).

In the present research, the degradation rate of phenolic compounds in the microwave heating method was lower comparing to the other two methods. This could be explained by the fact that in microwave heating, the heat as controlled is generated throughout the material (volumetric heating), leading to faster heating rates, compared to the other methods where the heat is usually transferred from the surface to the interior parts (Yousefi et al. 2012; Fazaeli et al. 2013). The slight degradation of phenolic compounds with microwave energy indicates that the esterified and glycoside bound phenolic acids could be cleaved by microwave treatment; this might be attributed to the heating effect caused by the electromagnetic radiations during the microwave heating process (Hayat et al. 2010).

**Total monomeric anthocyanin content (TMAC)**

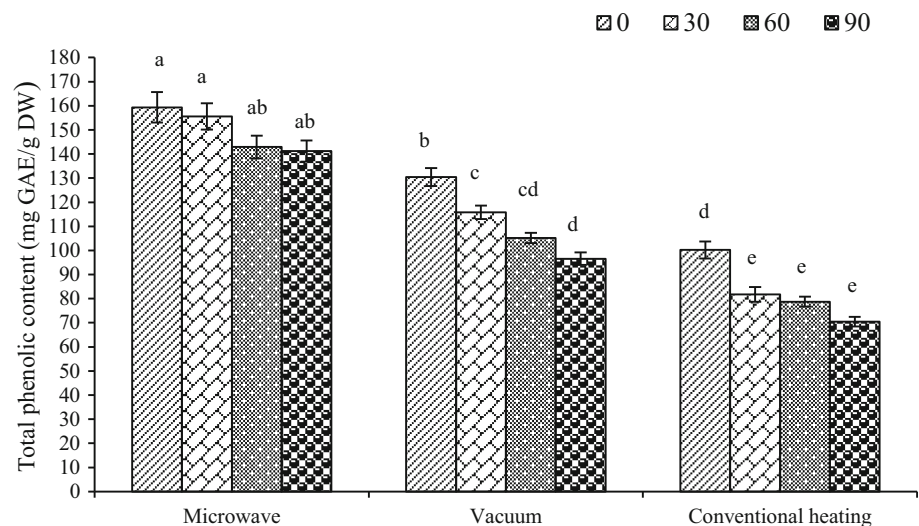
Figure 4 illustrates the effects of different concentration methods and storage time on the TMAC (cyn-3-glu/100 g DW) of jujube concentrates during a 90 day-storage at 4 °C. The various concentration methods indicated significant ( $P < 0.05$ ) differences in the TMAC values of jujube concentrates. The initial TMAC (storage time 0 day) of the jujube concentrate obtained by the microwave method (48.84 cyn-3-glu/100 g DW) was considerably higher compared to those of the concentrates obtained by the vacuum (30.07 cyn-3-glu/100 g DW) and conventional methods (27.71 cyn-3-glu/100 g DW). Degradation percentage of the TMAC of jujube concentrates achieved by the conventional, vacuum and microwave methods was 15.77, 10.94, and 6.81% after a 90 day-storage at 4 °C, respectively. The jujube concentrate treated with the conventional method had the highest anthocyanin loss in comparison to the other methods. It was observed that storage time had significant effect ( $P < 0.05$ ) on the TMAC of jujube concentrates (Fig. 4). Similarly, Ekici (2014) reported that the total anthocyanin content of poppy sorbet concentrated by the conventional, microwave and vacuum methods was lost at the rate of 14.43, 7.05 and 4.30%, respectively after 3 months at 4 °C. Moreover, a research by Alighourchi and Barzegar (2009) on reconstituted pomegranate juice showed that the degradation percentage of total anthocyanin content was 71.8, 91.3, and 96.9%, respectively at 4, 20 and 37 °C after a 210 day-storage.

**Effect of various concentration methods and storage time on individual anthocyanins**

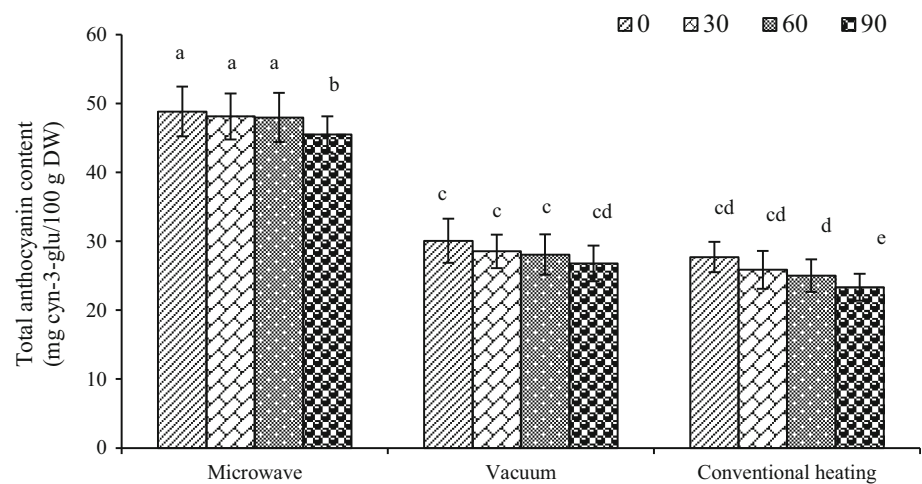
The profile of the individual anthocyanins of jujube concentrates characterized seven major peaks in the 4–13 min region of chromatogram (chromatogram not shown). Delphinidin-3,5-diglucoside, cyanidin-3,5-



**Fig. 3** The change of total phenolic content of jujube concentrated by microwave, vacuum and conventional heating methods stored at 4 °C for 90 days



**Fig. 4** The change of total anthocyanin content of jujube concentrate treated by microwave, vacuum and conventional heating methods stored at 4 °C for 90 days



diglucoside, delphinidin-3-glucoside, pelargonidin-3,5-diglucoside, peonidin-3-glucoside, cyanidin-3-glucoside, and pelargonidin-3-glucoside were well separated in all concentrates by comparing their retention times with those of pure standards (and also by spiking).

The changes in the individual anthocyanins (mg/100 ml concentrate) of various jujube concentrates after treatment with different concentration processes and during the storage time of 90 days at 4 °C are presented in Table 1. Concentrating by different methods did not affect the profile of the individual anthocyanins of concentrates. However, it can be seen that the different methods had significant ( $P < 0.05$ ) influence on the individual anthocyanins of all samples after concentration. The jujube concentrate treated with the microwave method had the highest individual anthocyanin content as compared to the other methods. Moreover, Table 1 shows that increasing of storage time caused a slight but significant decrease ( $P < 0.05$ ) on the individual anthocyanin content in

comparison with the initial anthocyanin concentration (storage time 0 day) due to degrading of anthocyanins ( $P < 0.05$ ). Cyanidin-3,5-diglucoside was determined as the major anthocyanin in all concentrates. The degradation percentage of cyanidin-3,5-diglucoside of the samples concentrated by the microwave, vacuum and conventional methods after a 90 day-storage at 4 °C was 11.14, 17.59 and 25.59%, respectively. These results demonstrate that the stability of diglycosides anthocyanins (e.g. cyanidin-3,5-diglucoside and delphinidin-3,5-diglucoside) to degradation during cold storage was higher than that of monoglycosides (e.g. cyanidin-3-glucoside, delphinidin-3-glucoside, peonidin-3-glucoside, and pelargonidin-3-glucoside). Similarly, higher stability of diglycosides anthocyanins at cold storage conditions has been documented by Alighourchi and Barzegar (2009). It is possible that sugar substitution enhances the stability of anthocyanin molecule. Increase in hydroxylation increases the instability of anthocyanins, whereas increase in glucosylation confers

**Table 1** Effect of various concentration methods and storage at 4 °C on anthocyanins profile

Heating method	Storage time (day)	Anthocyanin (mg/100 ml concentrate)						
		dp-3,5-dg	cy-3,5-dg	dp-3-g	pg-3,5-dg	p-3-g	cy-3-g	pg-3-g
Microwave	0	3.82 ± 0.75 <sup>a</sup>	27.99 ± 1.52 <sup>a</sup>	5.15 ± 0.44 <sup>a</sup>	0.45 ± 0.09 <sup>a</sup>	0.89 ± 0.12 <sup>a</sup>	5.67 ± 0.21 <sup>a</sup>	0.61 ± 0.06 <sup>ab</sup>
	30	3.65 ± 0.61 <sup>ab</sup>	25.34 ± 2.11 <sup>b</sup>	4.23 ± 0.76 <sup>bc</sup>	0.41 ± 0.07 <sup>ab</sup>	0.75 ± 0.13 <sup>b</sup>	5.18 ± 0.11 <sup>a</sup>	0.60 ± 0.01 <sup>ab</sup>
	60	3.45 ± 0.22 <sup>b</sup>	24.92 ± 2.55 <sup>c</sup>	4.11 ± 0.22 <sup>b</sup>	0.41 ± 0.08 <sup>ab</sup>	0.62 ± 0.06 <sup>cd</sup>	3.69 ± 0.09 <sup>b</sup>	0.58 ± 0.02 <sup>b</sup>
	90	2.90 ± 0.11 <sup>c</sup>	24.87 ± 0.98 <sup>c</sup>	3.37 ± 0.11 <sup>cd</sup>	0.40 ± 0.11 <sup>b</sup>	0.60 ± 0.09 <sup>c</sup>	3.42 ± 0.07 <sup>b</sup>	0.40 ± 0.01 <sup>a</sup>
Vacuum	0	2.75 ± 0.98 <sup>c</sup>	24.55 ± 2.01 <sup>cd</sup>	4.58 ± 0.90 <sup>bc</sup>	0.37 ± 0.01 <sup>b</sup>	0.62 ± 0.11 <sup>cd</sup>	4.43 ± 0.08 <sup>c</sup>	0.52 ± 0.02 <sup>b</sup>
	30	2.14 ± 0.11 <sup>d</sup>	24.28 ± 2.65 <sup>cd</sup>	3.14 ± 0.31 <sup>d</sup>	0.34 ± 0.01 <sup>c</sup>	0.55 ± 0.03 <sup>d</sup>	3.58 ± 0.02 <sup>d</sup>	0.40 ± 0.05 <sup>c</sup>
	60	2.11 ± 0.32 <sup>d</sup>	24.05 ± 1.32 <sup>d</sup>	2.34 ± 0.56 <sup>e</sup>	0.33 ± 0.07 <sup>c</sup>	0.37 ± 0.01 <sup>e</sup>	3.35 ± 0.01 <sup>d</sup>	0.34 ± 0.02 <sup>d</sup>
	90	1.95 ± 0.13 <sup>e</sup>	20.23 ± 1.43 <sup>e</sup>	2.17 ± 0.21 <sup>e</sup>	0.30 ± 0.02 <sup>cd</sup>	0.35 ± 0.02 <sup>e</sup>	2.25 ± 0.09 <sup>e</sup>	0.25 ± 0.00 <sup>e</sup>
Conventional	0	2.29 ± 0.06 <sup>d</sup>	20.59 ± 1.22 <sup>e</sup>	3.46 ± 0.61 <sup>cd</sup>	0.29 ± 0.02 <sup>d</sup>	0.54 ± 0.07 <sup>d</sup>	3.11 ± 0.02 <sup>df</sup>	0.41 ± 0.01 <sup>c</sup>
	30	1.65 ± 0.34 <sup>ef</sup>	18.77 ± 0.87 <sup>f</sup>	2.15 ± 0.51 <sup>e</sup>	0.22 ± 0.00 <sup>e</sup>	0.52 ± 0.04 <sup>d</sup>	3.05 ± 0.01 <sup>f</sup>	0.40 ± 0.02 <sup>c</sup>
	60	1.55 ± 0.19 <sup>f</sup>	16.09 ± 0.69 <sup>g</sup>	1.67 ± 0.15 <sup>f</sup>	0.22 ± 0.01 <sup>e</sup>	0.39 ± 0.01 <sup>f</sup>	1.55 ± 0.01 <sup>g</sup>	0.22 ± 0.02 <sup>d</sup>
	90	1.49 ± 0.02 <sup>f</sup>	15.32 ± 0.61 <sup>g</sup>	0.94 ± 0.09 <sup>g</sup>	0.19 ± 0.02 <sup>f</sup>	0.25 ± 0.02 <sup>g</sup>	1.14 ± 0.00 <sup>g</sup>	0.18 ± 0.00 <sup>d</sup>

Data are mean ± SD (n = 3). Similar letters in each column in the level  $P < 0.05$  were not significantly different. delphinidin-3, 5-diglucoside (dp-3,5-dg); cyaniding-3, 5-diglucoside (cy-3,5-dg); delphinidin-3-glucoside (dp-3-g); pelargonidin-3, 5-diglucoside (pg-3,5-dg); peonidin-3-glucoside (p-3-g); cyaniding-3-glucoside (cy-3-g); pelargonidin-3-glucoside (pg-3-g)

greater stability at different conditions (Reque et al. 2014; Castañeda-Ovando et al. 2009).

Overall, some factors influencing anthocyanins stability during the processing and storage are chemical structure of anthocyanins, pH, temperature, light, oxygen, presence of enzymes, etc. (Patras et al. 2010). Loss of anthocyanins in fruit concentrate during storage may be due to the activity of residual enzymes (e.g. polyphenol oxidase and peroxidase) or condensation reactions with other phenolic compounds (Reque et al. 2014). In addition, thermal processing, especially at above 50 °C, could be effective on degradation of the bioactive compounds such as anthocyanin and other polyphenol compounds (Onsekizoglu 2013; Reque et al. 2014). The present study indicated that heating temperature and time processing were higher in the conventional method than in the other methods. Also loss of TMAC (cyn-3-glu/100 g DW) and individual anthocyanins (mg/100 ml concentrate) in the jujube concentrates was more evident under the vacuum heating method as compared to the microwave method. Thus, according to our findings, it is possible that, firstly, speedy destruction of anthocyanins at higher temperatures (conventional heating method) is due to the hydrolyzation of 3-glycoside structure, which may increase the instability of anthocyanins (Fazaeli et al. 2013). Secondly, heating by the microwave method is evidently rapid and uniform, thus inactivating enzymes and minimizing degradation of anthocyanins more quickly (Maskan 2006). However, more research is needed to define the mechanism(s) responsible for the deterioration of anthocyanins and polyphenolic compounds during the storage of concentrates.

#### Effect of various concentration methods and storage time on organic acids profile

In this study, the organic acids profile of various jujube concentrates was determined by HPLC. Ten organic acids (oxalic, citric, tartaric, quinic, malic, shikimic, ascorbic, succinic, maleic, and malonic acids) were identified in all the samples (chromatogram not shown). The changes of the individual organic acids of concentrates obtained from the various concentration processes and the storage time of 90 days at 4 °C are presented in Table 2. In general, malic, citric, succinic and ascorbic acids were found to be orderly the most predominant organic acids in the jujube concentrates studied. Concentrating by different methods did not affect the profile of the organic acids of the concentrates; however, their contents were significantly ( $P < 0.05$ ) different. The jujube concentrate treated with the microwave method had the highest organic acid content as compared to the other methods. Based on the present research findings, storage time had significant ( $P < 0.05$ ) effect on declining the organic acid content of all the samples. The loss of malic, citric, succinic and ascorbic acids of the concentrate prepared by the microwave method after a 90 day-storage at 4 °C was 13.36, 10.98, 15.13, and 31.33%, respectively.

Our findings revealed that the degradation of organic acids was more pronounced in the conventional heating compared to the other methods during the storage time. It is known that heat-induced reactions between nitrogen-free carboxylic acids and sugars are the most affecting parameters in organic acids, causing important changes in their

**Table 2** Effect of various concentration methods and storage at 4 °C on organic acids profile

Heating method	Storage time (day)	Organic acid (mg/100 ml concentrate)									
		Oxalic	Citric	Tartaric	Quinic	Malic	Shikimic	Ascorbic	Succinic	Maleic	Malonic
Microwave	0	18.01 ± 2.34 <sup>a</sup>	158.31 ± 3.54 <sup>a</sup>	28.98 ± 2.21 <sup>f</sup>	19.31 ± 3.19 <sup>d</sup>	193.54 ± 4.11 <sup>a</sup>	12.22 ± 1.89 <sup>e</sup>	137.87 ± 3.29 <sup>f</sup>	145.85 ± 3.13 <sup>g</sup>	10.21 ± 1.75 <sup>a</sup>	17.04 ± 2.65 <sup>e</sup>
	30	17.87 ± 1.11 <sup>ab</sup>	151.76 ± 3.26 <sup>b</sup>	27.32 ± 2.74 <sup>f</sup>	19.01 ± 3.41 <sup>d</sup>	180.75 ± 3.54 <sup>c</sup>	11.99 ± 1.34 <sup>b</sup>	114.19 ± 3.54 <sup>e</sup>	139.55 ± 3.43 <sup>a</sup>	9.55 ± 1.11 <sup>a</sup>	15.76 ± 2.54 <sup>d</sup>
	60	17.34 ± 1.76 <sup>b</sup>	141.21 ± 2.97 <sup>c</sup>	25.87 ± 1.13 <sup>e</sup>	17.32 ± 2.14 <sup>ef</sup>	174.32 ± 2.14 <sup>b</sup>	11.32 ± 1.06 <sup>ab</sup>	105.19 ± 3.08 <sup>de</sup>	135.41 ± 2.14 <sup>ab</sup>	9.12 ± 0.54 <sup>ab</sup>	9.12 ± 0.54 <sup>ab</sup>
Vacuum	0	16.13 ± 0.89 <sup>c</sup>	140.92 ± 2.61 <sup>cd</sup>	25.21 ± 1.09 <sup>e</sup>	16.98 ± 1.14 <sup>e</sup>	167.68 ± 3.04 <sup>ab</sup>	10.34 ± 0.94 <sup>a</sup>	94.67 ± 2.67 <sup>c</sup>	123.78 ± 2.58 <sup>b</sup>	8.01 ± 0.28 <sup>b</sup>	13.87 ± 1.27 <sup>f</sup>
	30	17.43 ± 1.16 <sup>b</sup>	150.12 ± 2.76 <sup>b</sup>	24.99 ± 3.43 <sup>e</sup>	16.25 ± 1.65 <sup>e</sup>	181.62 ± 2.64 <sup>c</sup>	10.55 ± 1.32 <sup>a</sup>	125.55 ± 2.76 <sup>g</sup>	130.65 ± 3.64 <sup>ab</sup>	9.05 ± 1.98 <sup>ab</sup>	15.32 ± 1.93 <sup>d</sup>
	60	16.21 ± 2.08 <sup>cb</sup>	138.97 ± 3.32 <sup>cd</sup>	21.18 ± 2.85 <sup>d</sup>	15.37 ± 1.76 <sup>a</sup>	164.71 ± 1.98 <sup>ab</sup>	9.31 ± 1.14 <sup>d</sup>	106.90 ± 1.98 <sup>de</sup>	124.34 ± 3.72 <sup>b</sup>	8.15 ± 1.18 <sup>bc</sup>	13.97 ± 1.76 <sup>f</sup>
Conventional	0	16.14 ± 1.24 <sup>cb</sup>	129.97 ± 1.32 <sup>e</sup>	20.95 ± 2.14 <sup>d</sup>	13.74 ± 2.14 <sup>bc</sup>	155.42 ± 2.14 <sup>d</sup>	7.32 ± 0.96 <sup>ef</sup>	92.32 ± 2.14 <sup>c</sup>	110.32 ± 2.14 <sup>c</sup>	6.33 ± 0.84 <sup>d</sup>	11.72 ± 1.14 <sup>b</sup>
	30	13.96 ± 1.27 <sup>d</sup>	123.38 ± 2.51 <sup>f</sup>	18.38 ± 2.43 <sup>a</sup>	13.58 ± 1.52 <sup>b</sup>	146.32 ± 1.34 <sup>e</sup>	8.10 ± 0.85 <sup>g</sup>	80.42 ± 1.59 <sup>b</sup>	104.76 ± 1.24 <sup>de</sup>	6.12 ± 0.73 <sup>d</sup>	10.43 ± 0.93 <sup>cb</sup>
	60	15.33 ± 2.09 <sup>e</sup>	143.24 ± 1.29 <sup>d</sup>	19.46 ± 2.76 <sup>cd</sup>	15.55 ± 1.56 <sup>a</sup>	172.70 ± 3.54 <sup>ab</sup>	9.45 ± 0.74 <sup>d</sup>	115.45 ± 1.76 <sup>d</sup>	120.11 ± 2.53 <sup>b</sup>	8.19 ± 1.32 <sup>bc</sup>	14.75 ± 1.64 <sup>ab</sup>
	0	14.12 ± 1.32 <sup>de</sup>	141.91 ± 1.94 <sup>e</sup>	15.29 ± 1.15 <sup>b</sup>	13.98 ± 1.61 <sup>bc</sup>	150.98 ± 3.87 <sup>d</sup>	7.52 ± 0.21 <sup>e</sup>	101.57 ± 1.34 <sup>de</sup>	109.64 ± 1.27 <sup>c</sup>	7.57 ± 0.48 <sup>c</sup>	11.58 ± 1.05 <sup>b</sup>
	30	13.92 ± 1.43 <sup>de</sup>	122.97 ± 3.32 <sup>f</sup>	13.32 ± 2.14 <sup>e</sup>	13.02 ± 2.14 <sup>e</sup>	133.67 ± 2.14 <sup>f</sup>	7.08 ± 0.14 <sup>ef</sup>	84.62 ± 2.14 <sup>b</sup>	100.87 ± 2.14 <sup>d</sup>	7.22 ± 0.14 <sup>c</sup>	9.97 ± 1.14 <sup>c</sup>
	60	10.65 ± 0.84 <sup>f</sup>	112.58 ± 1.74 <sup>g</sup>	12.68 ± 1.08 <sup>e</sup>	10.54 ± 1.86 <sup>g</sup>	125.45 ± 2.43 <sup>g</sup>	6.76 ± 0.91 <sup>f</sup>	70.18 ± 2.43 <sup>a</sup>	91.45 ± 1.87 <sup>e</sup>	6.34 ± 0.87 <sup>d</sup>	9.32 ± 0.91 <sup>c</sup>

Data are mean ± SD (n = 3). Similar letters in each column in the level  $P < 0.05$  were not significantly different

contents (Piva et al. 2008; Ribeiro et al. 2007). The microwave pasteurisation of fruit juices has been previously reported by several researchers, as it preserves the natural organoleptic characteristics of the juice and reduces the time of exposure to energy, with the subsequently lower risk of losing essential thermolabile nutrients such as organic acids (Cañumir et al. 2002; Igual et al. 2010).

## Conclusion

Based on the achieved results, jujube concentrate is an excellent source of phenolic, anthocyanin and organic acids. The results showed that all the concentration methods as well as storage time affected the bioactive compounds and color values of jujube concentrate. In comparison to the conventional and vacuum heating methods, microwave processing had less destructive effects on the bioactive compounds (e.g. total phenolic content, total and individual anthocyanins and organic acid content) of the jujube concentrates during the 90-day storage. In addition, after concentration processing and during storage of the samples, the color values of  $L^*$ ,  $a^*$  and  $b^*$  decreased significantly. However, the microwave method led to the lowest color degradation; therefore, to obtain a high quality jujube concentrate, microwave concentration method seems to be the most appropriate process.

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