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ORIGINAL PAPER

Determination of phenolics, organic acids, minerals and volatile compounds of jujube (*Ziziphus jujuba* miller) jam produced by under vacuum evaporation compared with open pan method

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Abstract
The jujube fruit (*Ziziphus jujuba* Miller) is used in alternative medicine due to its important bioactive components and is well-suited for jam processing due to its high sugar content. In this study, changes in the chemical and aroma compounds of jujube jams produced using the open-pan (OPJ) and vacuum evaporation (VEJ) methods were determined immediately after production and after a storage period of 4 months. Concentrations of 5-hydroxymethylfurfural (HMF) in the OPJ and VEJ were 13.94 and 1.53 mg/kg initially and 22.63 and 1.66 mg/kg after storage, respectively. Epicatechin was identified as the major phenolic component in jujube fruit and jams. Concentrations of phenolic compounds were higher in the VEJ both before and after storage. It was determined that jujube jam is a good source of the minerals Fe, Ca, Zn, Se, and Cu. Ascorbic acid loss was 86.47% in the OPJ versus 73.32% in the VEJ after production and continued during storage in both jams. Numerous novel aroma compounds that are not found in fresh jujube fruit were produced as a result of the different jam production methods. Significant advantages of the vacuum evaporation process have been determined in the production of jujube jam compared to the open pan process.

Keywords Jujube jam · Vacuum evaporation · HMF · Organic acids · Phenolic compounds · Aroma compounds

Introduction

The jujube (*Ziziphus jujuba* Miller) is a unique fruit of the Rhamnaceae family that has been used by people for over 4000 years. Its natural distribution extends from Asia (Afghanistan, China, India, Iran, Pakistan, Turkey, South Korea, etc.) to Europe (Greece, Italy, Russia, Spain, etc.) and the Australian continent [1, 2].

The jujube fruit is a good source of minerals, proteins, sugars, organic acids, vitamin C, phenolics, and polysaccharides. It also contains various bioactive compounds that imbue the fruit with high nutritional value, functional properties, and health benefits [3, 4]. In traditional Chinese medicine, the pharmaceutical effects of jujube fruit are utilized in the treatment of diabetes, jaundice, diarrhea, urinary problems, ulcers, constipation, chronic bronchitis, liver disease, wounds, depression, and insomnia. Previous studies have shown that the bioactive components in jujube have antiproliferative, anti-inflammatory, antioxidant, antiobesity and antitumor effects and were protective against cardiovascular diseases and type 2 diabetes [1, 5, 6]. Jujube fruit is processed via numerous methods for consumption in various forms, as a dried snack, sweetened tea syrup or teabags, paste, soup, compote, pickle, jam, and wine.

Fruits and vegetables are preserved through various methods. One of these methods is processing fruits and vegetables into jam by concentrating and adding sugar. Jams are popular food products due to their low cost, year-round accessibility, and organoleptic properties. The consumption of commercial jams has become increasingly popular due to consumers' modern lifestyles. Although modern technologies are used today in commercial jam production, they are still produced using home-cooking methods [7]. In homemade jam production, fruit and sugar are mixed at a certain ratio and the mixture is concentrated at high temperatures to reach the final total soluble solid

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content [8]. High thermal processing and storage conditions directly affect the sensorial properties and nutritional constituents in jams, and the processes applied can significantly reduce their nutritional value. Combination of these processes causes undesirable losses in color, aroma, and nutritional components, often without attaining the desired consistency. It also leads to the production of unwanted compounds such as 5-hydroxymethylfurfural (HMF), furfural, and melanoidins. These problems are known to be caused by the application of extreme heat, prolonged thermal processing times and storage [9, 10]. Therefore, food processing methods must be carefully selected to minimize nutritional losses. Good manufacturing processes are expected to prevent loss of nutritional and sensorial characteristics of food products substantially throughout their extended storage.

Vacuum processing techniques provide a low-temperature, low-oxygen cooking environment with shorter processing time, which better preserves the nutritional value and physical structure of foods. In the food industry, the use of industrial-scale vacuum cooking equipment is usually preferred for the production of fruit juice concentrates, paste, and jam. Vacuum cooking better preserves the natural color and flavor of the food, and because there is reduced production of carcinogenic substances resulting from oxidation and high temperatures, the nutritional value of the food is also better preserved [11]. Moreover, vacuum cooking reduces the enzymatic and nonenzymatic degradation reactions which occur during the traditional cooking process and impact the quality of the final product [12].

The previous literature on jujube jam includes reports from Xiang [13], Guo and Cheng-rui [14], Hai-ou [15] on the production of low-sugar jujube jam. Uddin and Hussain [16] produced jujube jam using the open-pan method and reported an estimated 98.19% loss of ascorbic acid. These studies indicate that jujube jams have not been produced using vacuum methods, instead of produced traditionally in open pans, and that the chemical properties of the jams produced have not been adequately investigated. Rababah et al. [17] also reported substantial phenolic and antioxidant losses in jams produced from five different fruits using the open-pan method. A review of the literature on jam processing shows that most studies have examined open-pan methods, while there are few studies of jams produced using vacuum evaporation. In light of this information, the aim of this study was to address gaps in the literature regarding jujube jam production and highlight the benefits of vacuum jam production.

In this study, we prepared jujube jam using two different methods, open-pan (OPJ) as a control and vacuum evaporation (VEJ), and compared quality criteria (total dry matter, °Brix, pH, total and reducing sugar concentrations, phenolic and mineral content, HMF formation, organic acids, and aroma compounds) to determine the superior method of producing jujube jam.

Materials and methods

Raw material and preliminary trials

The jujube fruit used as the raw material in this study was obtained from the village of Harmanören, located in the Isparta province of Turkey. All fruits used to produce the jams was the same Jujube variety (*Ziziphus jujuba* Miller) and obtained from the same tree. Preliminary trials were conducted to develop the recipe and cooking technique for jujube jam. An optimum formulation was obtained based on factors such as fruit-to-sugar ratios, cooking times, applied temperature, and final soluble solid content (°Brix). This formulation was modified independently for each of the two production methods used (traditional open-pan and vacuum evaporation). In preliminary trials, three different fruit: sucrose: glucose syrup: water (g: g: ml: ml) ratio was tested (60: 40: 10: 25, 50: 50: 10: 25 and 40: 60: 10: 25). The pH of the medium was adjusted to 2.8, 3.0 and 3.2 with addition of citric acid (50%). An amount of pectin (1.1, 2.1 and 3.1 g) were added according to the recipe. The formulation with the highest score in sensory evaluation (consistency, color, taste, general overview) was preferred in the main jam production. In here, the value of the highest score was determined as 500 g fruit, 500 g sucrose, 100 ml glucose syrup, 250 ml water, medium of the pH 3.0 and 2.1 g of pectin. This recipe was used in the each of the jam production. The produced jams were filled into a total of 30 (250-cc) jars, 15 of open-pan jam (OPJ) and 15 vacuum-evaporation jam (VEJ). Jujube jams after production and after 4 months of storage at 20 °C were analyzed.

Jam production

Traditional open-pan method

In the laboratory, the quantities of jujube fruit, glucose syrup, and water specified in the recipe were added to a pan on a home-style cooking range. Half of the indicated quantity of sugar (sucrose) was added to the pan and the mixture was heated while stirring. The mixture was stirred until it came to a boil. After 3–4 min of boiling, the remaining sugar (sucrose) was added to the pan. The mean boiling temperature during open-pan production was measured as 104.5 °C. During the cooking process, °Brix was monitored; the pectin and acid were added when 68 °Brix was reached. Boiling was continued until the mixture reached its final °Brix value then the product was hot-filled at 85 °C into

sterile jars. The filled jars were closed, inverted for 5 min for pasteurization of the lids and the upper portion of the glass jars. Some of the jam product was reserved for analysis of initial concentrations, while the rest was stored at 20 °C for 4 months for analysis of jams after storage.

Vacuum evaporation method

A vacuum evaporator (Heidolph Laborota 4000, Germany), vacuum pump (ZX2, China), and vacuum gauge (Wika, China) were combined to create a vacuum cooking apparatus for jam production. The specified quantities of fruit, sugar, glucose syrup, citric acid, pectin, and water indicated in the recipe were placed in a rotary evaporator flask and heated to 65 °C to completely dissolve the sugar. The vacuum pump was adjusted to produce a vacuum of 560–600 mmHg. When the amount of condensed water in the condenser balloon equaled the amount of water determined in the preliminary trial, the vacuum pump was turned off and °Brix was measured. After removing the vacuum, the jam was pasteurized at 85 °C for 10 min, adjusted to a pH of about 3.0 and filled into sterile jars. The tops and lids of the jars were pasteurized as described above. Some of the jam product was reserved for analysis of initial concentrations, while the rest was stored at 20 °C for 4 months for analysis of jams after storage.

Analyses

Total dry matter, soluble solids, pH and sugar analyses

Total dry matter (TDM), soluble solids (SS), pH, total and invert sugar content were determined as described by Cemeroğlu [18].

5-Hydroxymethylfurfural (HMF) analysis

HMF analysis was performed with a high-performance liquid chromatography (HPLC) device (Shimadzu, Kyoto, Japan) using a method modified from Gökmen and Acar [19].

A 5-g sample of jujube jam was dissolved in 20 ml of distilled water. Five ml of this solution was mixed vigorously with 10 ml ethyl acetate (Merck) in a separatory funnel for 5 min. The ethyl acetate (upper) phase was collected and the sample was mixed with another 10 ml ethyl acetate for 5 min. The collected ethyl acetate phases were combined and mixed again with 2 ml of 1.5% Na_2CO_3 (Merck). The ethyl acetate phase was collected and the remaining Na_2CO_3 phase was re-extracted with 10 ml of ethyl acetate. The ethyl acetate phases were combined and filtered through ashless quantitative filter paper (Whatman Grade 589/3 blue ribbon). The obtained filtrate was acidified with 5 drops of acetic acid and dried at 40 °C in an evaporator. The residue was dissolved in 1 ml of acetic acid (pH 4.0) and 20 µL of this solution was injected into the HPLC device.

Amount of HMF in the final solution was calculated using the area under the peak corresponding to the concentration in the calibration plot and expressed as 1 g/ml. HMF concentration of the jam was calculated using the equation

$$C_{HMF}(mg/l) = \frac{C^*_{HMF} V}{m}$$

Where C^*_{HMF} represents HMF concentration (µg/ml) in the final solution, V represents the volume (ml) of the final solution, and m represents the volume of jam taken from the extract and diluted.

A Shimadzu 20 AD series HPLC system (Shimadzu, Tokyo, Japan) was used for analysis and comprised a SPD-M10Avp DAD detector, LC-10ADvp pump, SCL-10Avp system controller, CTO 10Avp thermostatted column compartment, and 2 Luna C18 (250 × 4.60 mm) 5-µm particle size columns (Phenomenex, USA). Chromatographic separations were performed using a mobile phase of ACN/H_2O (5:95 *v*/v) at a flow rate of 1 ml/min. Injection volume used for the analysis was 20 µl and column temperature was 30 °C.

Analysis of phenolic compounds

Analysis of phenolic compounds was conducted using HPLC (Shimadzu, Kyoto, Japan) with protocol modified from Caponio et al. [20]. The sample preparation procedure was modified from Wang and Mazza [21]. For analysis, 10-g samples of jam were weighed. To these samples, 0.1 g of BHT (2,6-di-tert-butyl-4-methylphenol) (Merck) and 15 ml extraction solution (80% methanol (JT Baker) containing 1% HCl [*v*/v]) was added and mixed for 45 min in an ultrasonic bath. The upper phase was collected into another volumetric flask. Another 15 ml extraction solution was added to the remaining lower phase and mixed in the ultrasonic bath for 45 min. The upper phase was collected and combined with the previously collected upper phase, and the mixture was filtered through Whatman No: 4 filter paper. The filtrate was passed through a 0.45 µm filter and 20 µl of this final filtrate was injected into the HPLC device.

Phenolic component peaks obtained in the chromatograms were identified by comparing with the arrival times and UV spectra of the reference standards. The concentration of phenolic compounds in the sample was calculated from standard curves of phenolic compound standards (catechin, chlorogenic acid, epicatechin, ferulic acid, rutin, and p-hydroxy benzoic acid).

The analysis was conducted using Eclipse XDB-C18 (250 × 4.60 mm) 5-µm particle column (Agilent, USA), chromatographic separations were done with mobile phases A: 3% acetic acid and B: methanol at a flow rate of 0.8 ml/min. Injection volume was 20 µl and column temperature was 30 °C. The elution profile was a binary gradient that started at 7% B and reached 20% B at 20 min, 25% B at 28 min, 27% B at 35 min, 30% B at 50 min, 33% B at 60 min, 42% B at 62 min, 50% B at 70 min, 70% B at 73 min, 80% B at 75 min, 100% B at 80 min, and 7% B at 81 min.

Determination of organic acids

Organic acid analysis was performed using a protocol modified from Kordis-Krapez et al. [22]. Merck chromatographic analytic chemicals were used in the analysis.

In organic acid sample preparation, a Supelco C18 solid phase cartridge (Waters Associates, Ireland) was first conditioned in 3 ml of methanol and washed with 10 ml of distilled water. A 5-ml sample was homogenized with 5 ml of 2% H_3PO_4 and filtered with coarse filter paper. One ml of the filtrate was diluted with 3 ml extraction solution (0.01 M KH_2PO_4, pH 8.0). One ml of this solution was passed through the cartridge and the eluate was collected into a tube. The cartridge was washed with 2 ml extraction solution. The eluates were combined and a volume of 20 µl was injected into the HPLC device.

The HPLC system used in the study comprised a SPD-10Avp UV-VIS detector (210 nm), LC-20AT prominence pump, SIL–20 AC prominence auto sampler, DGU-20A5 prominence degasser, and LC- 20AT prominence system controller. A Prodigy ODS-2 (250 × 4.6 mm) 5-µm particle column (Agilent, USA) was used. Separation of organic acids was achieved by isocratic elution using a mobile phase of H_3PO_4 (pH 2.3) at a flow rate of 0.8 ml/min. Injection volume used in the analysis was 20 µl and column temperature was 30 °C.

Determination of mineral content

Mineral analysis in the fresh jujube and jujube jams were performed using Perkin Elmer OPTIMA 5300 DV ICP OES device according to EPA method 6010 [23]. Merck chromatographic analytic chemicals were used in the analysis. The samples were prepared according to EPA method 3015A using a Milestone ETHOS ONE microwave sample preparation unit by adding 5 ml HNO_3 and 1 ml H_2O_2 to 0.6 g of jam and using wet digestion method. The final volume was adjusted to 15 ml.

Operating conditions of Perkin Elmer OPTIMA 5300 DV ICP OES device used in the analysis were as follows: RF power was 1450 W; plasma coolant gas flow was 15 l/min; auxiliary gas flow was 0.2 l/min; nebulizer gas flow was 0.6 l/min; heater was set to 30 °C, plasma view was axial, sample flow rate was 1.5 ml/min; integration time was 10 s; delay time was 60 s; and resolution was normal. The sensitive wavelength for mineral identification was determined according to the tables in the device manual. Signal changes over time were corrected using an external drift monitor. Averages from the sample replicates were recorded.

Extraction of aroma volatiles and GC–MS analysis

Aroma compound analysis was modified from Zhang et al. [24]. A 2-g sample of jujube jam (incubated at 45 °C for 30 min) was injected into a solid-phase microextraction (SPME) apparatus (Supelco Inc., Bellefonte, PA, USA) and analyzed using a Shimadzu (Japan) GC 2010 Plus device. Fresh jujube puree was prepared by manually removing the seeds and homogenizing the fruit with a kitchen-type hand blender (Braun MR 404, Germany). A 2-g sample of fresh puree and samples of jam were placed in 15 ml headspace vials and sealed with a septum and aluminum cap. Aroma compounds were extracted using carboxen-polydimethylsiloxane (CAR/PDMS, 75 lm) fiber in a manual holder (Supelco, Inc., Bellefonte, PA, USA). Before use, the fiber was preconditioned in the GC injector according to the manufacturer's instructions.

Analyses were performed using a GC 2010 SE GC/MS (Shimadzu, Japan) equipped with an injection block at 250 °C, operated in electron ionization positive mode, energy level 70 eV. The aroma compounds were separated using a Restek Rxi-5 Rxi-5Sil MS column (30 m × 25 mm, 0.25 µm) (Restek, Bellefonte, USA) held at 40 °C for 2 min, raised to 250 °C at 4 min, then held at 230 °C for 5 min. Helium was used as the carrier gas at a flow rate of 1.61 ml/min and desorption time was 5 min.

The adsorption and desorption of SPME fiber procedure with GC–MS technique was tested for repeatability, sensitivity, and stability in the determination of volatiles. Repeatability expressed as relative standard deviation (RSD) of volatiles was 2.2–5.8%. The solution concentration was used to determine the limit of detection (LOD) if the average signal-to-noise ratio was 3. The limit of quantitation (LOQ) value was also calculated from the calibration curve.

Linear retention indexes (LRI) of the aroma compounds were calculated for GC-MS data according to C7–C30 straight-chain alkanes (Sigma-Aldrich, Germany). The mass spectra of the individual compounds were compared with those in the National Institute of Standards and Technology (NIST), Wiley, Tutor, and Flavor and Fragrance Natural and Synthetic Compounds (FFNSC) libraries.

Statistical analysis

A student t-test was performed to determine the statistical significance of the difference in both the processing methods (OPJ and VEJ) and it's at the end of storage period (4 month) seperately using analysis of variance (ANOVA) in a 95% confidence interval.

Results and discussion

The TDM, SS, pH, total and reducing sugar, and HMF concentration results for fresh jujube fruit and jams are shown in Table 1.

Initial TDM values determined on wet weight basis were 34.10, 72.12 and 72.01% for fresh jujube fruit OPJ and VEJ respectively. After 4 months of storage, TDM values were 71.33% and 71.29% for OPJ and VEJ, respectively, with no significant difference between the two jams ($p > 0.05$). There was decrease in the amount of dry matter after storage. Initial SS values were 71.34 and 71.17 °brix for OPJ and VEJ, respectively ($p < 0.05$). The SS values after 4 months were 70.56 and 70.35 °brix in OPJ and VEJ, respectively, indicating a slight decrease in SS with storage. This was attributed to the fact that inversion continues during storage and reducing sugars were used in browning reactions. The pH values of the OPJ and VEJ were 3.05 and 3.01, respectively, while fresh jujube fruit had a pH of 4.80. When making jam, pH must be 2.8–3.2 for effective gelling. In our study, we added citric acid to facilitate gel formation.

In our study, total sugar content was 24.75% in fresh jujube fruit, consistent with values reported by Gao et al. [25]. Initial and post-storage invert sugar content in dry weight (DW) was 40.98 and 37.79% in OPJ and 34.41 and 33.89% in VEJ, respectively indicating that OPJ had higher invert sugar content than VEJ. Initial and post-storage total sugar content in DW was 71.11 and 70.23% in OPJ and 73.05 and 72.41% in VEJ, respectively. This suggested that the vacuum method better preserved sugar content due to the lower temperatures used. Our findings were similar to those in a study by Tomruk et al. [26] on sugar content of strawberry jams. Sugar content decreased in both types of jams in our study, which may be a result of reducing sugars entering Maillard reactions and increasing HMF.

Total sugar content was higher in VEJ than OPJ because the sugar degraded less in the low temperatures used in vacuum jam processing. However, invert sugar content was higher in OPJ because the longer heating time and high temperature under atmospheric conditions caused more inversion. This suggests that sugar crystallization may occur less in OPJ compared to VEJ, while crystallization may be a problem in VEJ. Tosun and Ustun [27] emphasized that 30–35% of the final total sugar content should be invert sugar, which is lower than the ratios detected in our study. After 4 months of storage, crystallization was not detected in either of the jams.

HMF content

The Maillard reaction can occur during production or storage of foods containing amino acids and sugar, altering the color and flavor and reducing the nutritional value of the food, and posing a threat to human health. HMF is an important intermediate product of the Maillard reaction. It's used as an indicator of the intensity of thermal processing in production and unsuitable storage conditions of various food products such as jam, fruit juice and concentrate, dried fruits, honey, and baby formulas [28]. HMF is an end-product of ascorbic acid or carbohydrate degradation. Processing at high temperatures is known to cause Maillard reaction and caramelization, depending on high carbohydrate content and low pH of the product (under acidic conditions) [29].

In our study, initial HMF concentration in jams produced using two different methods were 1.53 and 13.94 mg/kg (in DW) in VEJ and OPJ, respectively ($p < 0.05$). The higher temperatures used in OPJ production correspond with its

Table 1 Total dry matter, soluble solids, pH, invert sugar, total sugar and hydroxymethylfurfural values in fresh jujube fruit and jujube jams

Time	Raw material and jam sample	TDM (%)	SS (°brix)	pH	Invert sugar (%, in DW)	Total sugar (%, in DW)	HMF (mg/kg in DW)
	Jujube	34.10 ± 0.05	33.65 ± 0.12	4.80 ± 0.21	8.95 ± 0.57	24.75 ± 0.94	0
0 months	OPJ	72.12 ± 0.12^{a}	71.34 ± 0.14^{b}	3.05 ± 0.03^{b}	40.98 ± 1.28^{b}	71.11 ± 2.46^{a}	13.94 ± 0.12^{b}
	VEJ	72.01 ± 0.47^{a}	71.17 ± 0.2^{a}	3.01 ± 0.03^{a}	34.41 ± 1.86^{a}	73.05 ± 2.21^{a}	1.53 ± 0.07^{a}
4 months	OPJ	71.33 ± 0.34^{A}	70.56 ± 0.11^{B}	3.02 ± 0.02^{B}	37.79 ± 2.12^{A}	70.23 ± 2.67^{A}	22.63 ± 0.35^{B}
	VEJ	71.29 ± 0.39^{A}	70.35 ± 0.18^{A}	2.97 ± 0.04^{A}	33.89 ± 2.26^{A}	72.41 ± 2.02^{B}	1.66 ± 0.04^{A}

OPJ Open pan evaporation jam, *VEJ* Vacuum evaporation jam, *DW* Dry weight basis

$^{a\text{-}b}$Statistical differences between 0 months jam samples in the same column ($p < 0.05$) (n = 3)

$^{A\text{-}B}$Statistical differences between after 4 months stored jam samples in the same column ($p < 0.05$) (n = 3)

higher HMF content. Due to the ongoing browning reactions, HMF concentration increased in both jams with storage, resulting in 22.63 and 1.66 mg/kg for OPJ and VEJ, respectively ($p < 0.05$). This increase in HMF was lower in VEJ than OPJ.

Rada-Mendoza et al. [28] reported that HMF concentration increased from 0.6 to 35.2 mg/100 g in commercial peach jams stored for 12 months at 35 °C. In another study, Aslanova et al. [30] stored strawberry, apricot, and cherry jams for 6 months at 10, 20, and 37 °C and reported that storage time and temperature had a major impact on HMF formation. They reported that HMF increased by 1.1–9.8% in jams stored at 10 °C for 4 months, 5.5–30.63% in jams stored at 20 °C for 4 months, and 424.77–583.43% in jams stored at 37 °C for 4 months. Tomruk et al. [26] reported HMF concentrations of 129 and 21.4 mg/kg in strawberry jams produced under atmospheric pressure (traditional method, 100 °C, 101.325 kPa) and under vacuum (75 °C, 39.5168 kPa), respectively. These HMF increases during excess heat treatment (in atmopheric, open pan concentration) and storage were similar with our findings.

Phenolic compound content

Phenolic compound contents of the samples are shown in Table 2. According to the results of our analysis, epicatechin was the main phenolic component. In fresh jujube fruit contents of epicatechin, catechin, rutin, chlorogenic acid and ferulic acid were 254.47, 113.57, 88.33, 37.85 and 1.66 mg/kg (in DW), respectively. Du et al. [31] reported catechin, chlorogenic acid, epicatechin, ferulic acid and p-hydroxy benzoic acid concentrations of 10.84, 8.93, 49.26, 5.2 and 2.53 mg/kg in jujube fruit, respectively (in DW).

In the present study, losses of epicatechin, catechin, rutin, and chlorogenic and ferulic acid in VEJ and OPJ were 88.6 and 90.94%; 68.7 and 74.69%; 72.49 and 75.8%; 83.14 and 84.6%; 100% and 100%, respectively. The phenolics loss in OPJ was more than VEJ. Because, low temperature and

Table 2 Phenolics, organic acids, mineral contents of jujube fruit and jams (Initial and after 4 months storage, mg/kg in DW)

Compounds	Samples, time								
	Jujube Fruit	0 months				4 months			
	Fresh	OPJ		VEJ		OPJ		VEJ	
	Amount	Amount	% Loss	Amount	% Loss	Amount	% Loss	Amount	% Loss
Phenolic Compounds									
Catechin	113.57 ± 2.21	28.75 ± 0.89^{a}	74.69	35.55 ± 0.96^{b}	68.7	18.47 ± 0.71^{A}	83.74	26.11 ± 0.94^{B}	77.01
Chlorogenic acid	37.85 ± 1.07	5.83 ± 0.42^{a}	84.6	6.38 ± 0.39^{b}	83.14	4.30 ± 0.27^{A}	88.64	5.41 ± 0.37^{A}	85.71
Epicatechin	254.47 ± 3.66	23.06 ± 0.51^{a}	90.94	29.02 ± 1.42^{b}	88.6	6.52 ± 0.29^{A}	97.44	9.58 ± 0.41^{B}	96.24
Ferulic acid	1.66 ± 0.07	0	100	0	100	0	100	0	100
Rutin	88.33 ± 2.27	21.38 ± 0.76^{a}	75.8	24.30 ± 0.53^{b}	72.49	17.77 ± 0.97^{A}	79.88	20.74 ± 0.43^{A}	76.52
p-hydroxy benzoic acid	2.11 ± 0.05	0.7 ± 0.02^{a}	66.83	0.9 ± 0.03^{b}	57.35	0	100	0	100
Organic Acids									
Oxalic	459.30 ± 13.41	384.72 ± 7.39^{a}	16.24	287.22 ± 10.73^{b}	37.47	191.59 ± 7.62^{A}	58.29	263.02 ± 9.91^{B}	42.73
Tartaric	1561.11 ± 46.67	845.05 ± 31.27^{a}	45.87	1187.44 ± 44.21^{b}	23.94	781.25 ± 19.05^{A}	49.96	1028.47 ± 42.77^{B}	34.12
Malic	4758.61 ± 100.78	1710.34 ± 57.39^{a}	64.06	1901.38 ± 54.57^{b}	60.04	1218.29 ± 30.38^{A}	74.4	1734.84 ± 55.28^{B}	63.54
Ascorbic	3681.48 ± 69.61	498.05 ± 11.41^{a}	86.47	982.36 ± 18.44^{b}	73.32	243.45 ± 8.21^{A}	93.39	666.29 ± 22.07^{B}	81.9
Succinic	455.69 ± 17.52	151.25 ± 4.21^{a}	66.81	100.97 ± 2.76^{b}	77.84	92.49 ± 2.16^{B}	79.7	86.48 ± 3.18^{A}	81.02
Minerals									
Fe	130.65 ± 4.42	42.34 ± 1.25^{a}	67.59	47.18 ± 1.95^{b}	63.89	2.77 ± 0.12^{A}	97.88	3.54 ± 0.10^{B}	97.29
Ca	2579.16 ± 69.06	604.02 ± 16.49^{a}	76.58	511.66 ± 18.84^{b}	80.16	328.33 ± 9.57^{A}	87.27	352.77 ± 10.28^{B}	86.32
Mg	570.97 ± 21.45	90.83 ± 2.38^{a}	84.09	95.01 ± 3.09^{b}	83.36	72.22 ± 2.96^{A}	87.35	89.65 ± 4.01^{B}	84.3
K	11,476.38 ± 279.65	1800.02 ± 56.58^{a}	84.32	1818.05 ± 66.08^{a}	84.16	1691.66 ± 68.22^{A}	85.26	1761.11 ± 61.17^{B}	84.65
Na	170.73 ± 7.01	133.62 ± 5.19^{a}	21.74	97.20 ± 3.91^{b}	43.07	103.16 ± 4.73^{A}	39.58	84.84 ± 3.08^{B}	50.31
Zn	14.75 ± 0.62	10.92 ± 0.43^{a}	25.97	11.45 ± 0.46^{b}	22.37	7.71 ± 0.31^{A}	47.72	8.08 ± 0.32^{A}	45.22
Se	4.42 ± 0.19	2.30 ± 0.13^{a}	47.96	2.84 ± 0.02^{b}	35.75	1.14 ± 0.02^{A}	74.21	1.82 ± 0.01^{B}	58.82
Cu	4.68 ± 0.02	2.17 ± 0.01^{a}	53.63	2.23 ± 0.01^{b}	52.35	1.12 ± 0.01^{A}	76.07	1.26 ± 0.02^{B}	73.07

OPJ Open pan evaporation jam, *VEJ* Vacuum evaporation jam, *DW* Dry weight basis

[a-b]Statistical differences between 0 months jam samples in the same row ($p < 0.05$) ($n = 3$)

[A-B]Statistical differences between after 4 months stored jam samples in the same row ($p < 0.05$) ($n = 3$)

short heating time were applied in VEJ. Fruits are generally rich in bioactive compounds like phenolics which are heat-sensitive components where the rate of deterioration varies with the process conditions applied. The phenolic compounds decreases significantly depending on processing conditions (temperature, time of processing) and storage conditions [32]. Rababah et al. [17] reported phenolic compound losses of 93.2, 87.95, 72.31, 76.19 and 68.58% during the jam-making processes for strawberry, sour cherry, apricot, fig, and orange. Poiana et al. [33] reported 25–42% loss in total phenolic content during jam processing from frozen strawberry, sweet cherry, and sour cherry. Poiana et al. [34] reported 42–51% loss of total phenolic content in bilberry jam, and Renna et al. [35] reported 46–56% loss of total phenolic content in carrot jam.

In our study, epicatechin, catechin, rutin, chlorogenic acid, and ferulic acid losses in VEJ and OPJ after 4 months of storage were 96.24 and 97.44%; 77.01 and 83.74%; 76.52 and 79.88%; 85.71 and 88.64%; and 100 and 100%, respectively. Kopjar et al. [36] reported that in strawberry jams, 6 weeks of storage at 4 °C and room temperature caused phenolic content loss of 16–31% and 48–56%, respectively. Rababah et al. [17] reported 21, 4, 61, 55 and 27% loss in total phenolic content respectively in strawberry, sour cherry, apricot, fig, and orange jams after 5 months of storage at room temperature. Poiana et al. [33] reported 9–11% and 18–25% loss of total phenolic content in strawberry, sweet cherry, sour cherry jams after 1 and 3 months of storage, respectively. In another study Poiana et al. [34] reported 41–57% loss of total phenolic content in bilberry jam after 7 months of storage at 20 °C. Renna et al. [35] reported that there was approximately 32% loss of total phenolic content in carrot jam after 90 days of storage.

In jujube concentrates (65 °Brix) produced by microwave, traditional, and vacuum methods, Najafabadi et al. [37] reported 25.94 and 29.71% loss of total phenolic content in the vacuum and traditionally processed concentrates after 90 days of storage at 4 °C. After 4 months of storage, total phenolic content losses in VEJ and OPJ were 35.68 and 40.96%, respectively in our study.

Organic acid content

Organic acids are substances that give fruit their traditional aroma and flavor. Initial and post-storage organic acid contents of fresh jujube fruit and the jujube jams are shown in Table 2. The concentrations of mean organic acids in the jams were found to be statistically significant ($p < 0.05$). Malic acid was identified as the major acid in jujube fruit with a concentration of 4758.61 mg/kg (in DW), while other components were ascorbic, tartaric, oxalic and succinic acid 3681.48, 1561.11, 459.30 and 455.69 mg/kg (in DW), respectively. Uddin and Hussain [16] reported the concentration of ascorbic acid in jujube fruit as 2350.29 mg/kg (in DW). We measured a higher concentration of ascorbic acid in the fresh jujube fruit in our study. Gao et al. [25] analyzed 10 different jujube fruits and detected 4 different organic acids: malic, ascorbic, citric and succinic acids 407.76–1128.74, 329.57–553.23, 57.68–280.85 and 20.29–243.36 mg/kg (in DW), respectively.

In the present study, the greatest organic acid loss while producing jam from jujube fruit was in ascorbic acid, which diminished by 73.32 and 86.47% in VEJ and OPJ, respectively. This may be due to reason that long-time heating and high temperatures reached to the point 104.5 °C in during open pan processing of jam results in more loss of ascorbic acid in open pan processing. This has led to ascorbic acid being the acid most affected by heat treatment. Losses of other organic acids in VEJ and OPJ were 37.47 and 16.24% for oxalic acid; 23.94 and 45.87% for tartaric acid; 60.04 and 64.06% for malic acid; and 77.84 and 66.81% for succinic acid, respectively. Uddin and Hussain [16] reported approximately 98.19% loss of ascorbic acid as a result of open-pan jam processing of jujube fruit. Compared to their results, we observed less ascorbic acid loss in our study, especially with the vacuum method (73.32%). Jawaheer et al. [38] stated 62.5% loss of ascorbic acid for guava fruit during jam processing. Poiana et al. [33] reported that thermal jam processing of strawberry, sour cherry, and sweet cherry resulted in vitamin C losses of 78, 70, and 54%, respectively.

After storing the VEJ and OPJ for 4 months, we observed losses of 42.73 and 58.29% for oxalic acid; 34.12 and 49.96% for tartaric acid; 63.54 and 74.4% for malic acid; 81.9 and 93.39% for ascorbic acid; and 81.02 and 79.7% for succinic acid, respectively. Jawaheer et al. [38] found that ascorbic acid concentration decreased by 70.2% in guava jam after 3 months of storage. Patras et al. [39] stored strawberry jam at 4 °C and 15 °C for 7, 14, 21, and 28 days and demonstrated ascorbic acid loss after each storage period. After 28 days of storage, ascorbic acid concentrations were reduced by 50% in jams stored at 4 °C and by 70% in those stored at 15 °C. Mazur et al. [40] stated that jams produced using red raspberry genotypes had ascorbic acid losses of 91% and 100% after 3 and 6 months of storage at 20 °C, respectively.

Najafabadi et al. [37] produced jujube concentrates (65 °Brix) using microwave, traditional, and vacuum methods and observed a reduction in organic acid content after 90 days of storage at 4 °C, with 35.94–39.21% loss of ascorbic acid after storage. Losses in our study ranged from 32.17 to 51.11% in our study.

Because ascorbic acid readily degrades at high temperatures, the results of our study are evidence that the vacuum method retains vitamins more than the open-pan method. Our findings indicate that there was greater organic acid degradation during storage in jams produced using the

traditional cooking method. Substantial changes in organic acids occur as a result of heat-accelerated reactions between non-nitrogenous carboxylic acids and sugars [41].

Mineral content

The mineral content of fruit can be affected by climate, soil nutrient content, harvest season, and the soils used. Minerals detected in the fresh jujube fruit and jams are shown in Table 2. In our study, concentrations of Fe, Ca, Mg, K, Na, Zn, Cu and Se in jujube fruit were 130.65, 2579.16, 570.97, 11,476.38, 170.73, 14.75, 4.68 and 4.42 mg/kg (in DW). In jujube fruit collected from 5 different regions, İmamoğlu [42] reported Fe, Na, Mg, K, Ca, Zn and Cu concentrations of 12.7–22.3, 45.2–83.2, 271.2–470.9, 2692.4–8333.6, 209.2–1166, 5–9.1 and 2.5–5.2 mg/kg (in DW), respectively.

In the jujube jams produced in our study, concentrations of Fe, Ca, Mg, K, Na, Zn, Cu and Se were 42.34–47.18, 511.66–604.02, 90.83–95.01, 1800–1818.05, 97.2–133.62, 10.92–11.45, 2.17–2.23 and 2.30–2.84 mg/kg (in DW), respectively. As seen in Table 2, all results are clearly lower in the jams than in fresh fruit, due to the dilution of fruit pulp with sugar and pectin during jam preparation. Similar results were reported previously by Plessi et al. [43]. Because fiber and minerals are generally more resilient nutrients, there tend to be less notable changes in their contents. High temperatures achieved during processing cause to changes in minerals. Vitamins, minerals, phenolic compounds, flavonoids and antioxidants are affected by light, temperature, and type of pectin added during processing [44].

In 6 different studies investigating the mineral content of 41 different jams (Plessi et al. [43], Mushumbusi [45], Sallam et al. [46], Okudu and Umahi [47], Naeem et al. [48] and Kántor et al. [49]) the highest Fe content was found in hibiscus jam, at 46.3 mg/kg (in DM) (Sallam et al. [46]), whereas the Fe concentration in the VEJ in the present study was higher than all 41 of those samples, at 47.18 mg/kg (in DM). It was noted that the OPJ sample in to the study had a Ca concentration of 604.02 mg/kg in DM, which was the third highest concentration after soursop jam (1481.39 mg/kg in DM) and pawpaw jam (1382.32 mg/kg in DM) [47]. When compared with the mint, papaya + mango, hibiscus jams made by Sallam et al. [46], it was determined that jujube jam was within the range of these jam values in terms of Zn content. Although Se was not detected in any of the jams in these studies, its concentration was determined as 2.84 mg/kg in the jujube VEJ in the present study. In addition, the highest concentration of Cu reported in these studies was 3.80 mg/kg in DW in mint jam (Sallam et al. [46]), after which is the concentration in jujube jam, 2.23 mg/kg in DW. Based on these comparisons, jujube jam was determined to be a good source of the minerals Fe, Ca, Zn, Se, and Cu.

Wani et al. [50] stored jams they produced from caronda fruit for 40 and 80 days and reported that Fe content decreased from an initial concentration of 36–37 mg/100 g to 30.8–32.7 mg/100 g after 80 days or storage, which is similar to the post-storage mineral losses observed in our study. The mineral loss observed during storage is in agreement with the findings of Kuşçu and Bulantekin [12] in apple molasses and Kuşçu and Yıldırım [51] in caper jams. Toker et al. [52] described numerous reactions involving minerals and divalent cations, which were implicated as a factor in HMF formation.

Aroma compounds

Flavor is one of the main factors influencing consumers' decisions to buy food products. In addition to tastants (e.g., organic acids and sugars), volatile aromatic compounds are a major determinant of food flavor. The different varieties of fresh fruits each have a distinct volatile profile consisting of numerous compounds derived from phytonutrients such as amino, fatty acids, phenols, terpenoids, and carotenoids [53].

We determined 40 different aroma compounds in the jujube fruit and jam samples in our study (Table 3). Aroma compounds in jams were found to be statistically significant ($p < 0.05$). Our comparison of fresh fruit and jams before and after storage showed that some aroma compounds in the fruit were lost due to thermal processing, that some new aroma compounds formed during jam processing, and that different aroma compounds not found in jujube fruit formed after storage. Due to the high temperatures used in the open-pan method, the amount of furfural in OPJ was much higher compared to VEJ, about 28.4-fold before storage and 23.41-fold after storage. This showed that in our study, thermal processing conditions had a greater effect on furfural formation than storage conditions. Similar results were reported previously by Kuşçu and Bulantekin [12]. We also found that furfural content increased with storage in both jam samples. As with furfural formation, we found that acetone was formed due to sugar degradation resulting from thermal processing and increased in concentration during storage of the jams. We attribute the 10-fold increase in 2-octenal concentration in jams compared to fresh fruit to thermal processing. Ultimately, the formation of by products with thermal processing is a known fact. In fresh jujube fruit, 2-hexenal was the most prominent aroma compound, followed by n-pentanal, n-hexanal, and benzaldehyde (Table 3). According to the literature, 2-hexenal and heptanal are associated with fruity, green, and apple-like aroma while benzaldehyde is a volatile compound responsible for the typical sweet cherry, raspberry and marzipan-like aroma. Acetaldehyde has been identified to contribute to apple aroma [53–55]. In our study, we found that benzaldehyde and acetaldehyde completely degraded due to the

Table 3 Aroma compounds found in jujube fruit and jams

Aroma Compound		Fresh Jujube	Area (% of total area)			
			0 months		4 months	
			OPJ	VEJ	OPJ	VEJ
1	Acetaldehyde	1.91	–	–	–	–
2	n-Pentane	2.78	–	–	–	–
3	Crotonaldehyde	0.12	1.33	–	–	–
4	3-Methylbutanal	0.26	1.51	–	–	–
5	Methyl propyl ketone	3.30	–	–	–	–
6	n-Pentanal	15.37	4.72^{b}	3.31^{a}	6.17^{B}	2.44^{A}
7	Methyl isothiocyanate	0.09	–	–	–	–
8	4-Methyl-pent-1-en-3-one	0.15	0.86^{b}	0.35^{a}	0.67^{B}	0.34^{A}
9	3-Pentanone, 2-methyl	0.12	–	–	–	–
10	2-Pentenal	1.90	0.54^{b}	0.45^{a}	0.55^{B}	0.49^{A}
11	Isopropenyl ethyl ketone	0.32	0.33^{b}	0.19^{a}	0.43^{B}	0.22^{A}
12	2-Hexanone	0.12	–	–	–	–
13	n-hexanal	5.87	11.27^{a}	12.80^{b}	9.27^{A}	11.55^{B}
14	2-Hexenal	46.9	32.13^{a}	33.25^{b}	30.77^{A}	33.69^{B}
15	2-Heptanone	1.53	–	0.31	–	0.21
16	n-heptanal	0.52	1.36^{b}	0.80^{a}	1.18^{B}	0.90^{A}
17	Alpha-Pinene	0.32	0.76	–	0.85^{B}	0.31^{A}
18	2-Heptenal	0.81	5.25^{b}	3.51^{a}	4.76^{B}	3.46^{A}
19	Benzaldehyde	5.70	–	–	–	–
20	Hept-5-en-2-one <6-methyl->	0.80	–	–	–	–
21	Hexyl methyl ketone; Octan-2-one	0.43	–	–	–	–
22	Capronate	1.92	–	–	–	–
23	n-Octanal	0.08	0.76^{b}	0.59^{a}	–	0.67
24	l-Limonene	4.98	0.43^{b}	0.27^{a}	0.53^{B}	0.38^{A}
25	Eucalyptol	0.26	1.16^{b}	1.06^{a}	0.58^{A}	0.67^{B}
26	2- Octenal	1.86	17.14^{b}	16.31^{a}	12.94^{A}	15.67^{B}
27	Methyl heptyl ketone	1.59	–	–	–	–
28	Acetone	–	6.90^{b}	3.12^{a}	9.47^{B}	4.50^{A}
29	Ethyl vinyl ketone	–	0.57^{b}	0.40^{a}	0.56^{B}	0.36^{A}
30	Acetoin	–	3.57	–	4.74	–
31	Furfural	–	6.52^{b}	0.23^{a}	10.30^{B}	0.44^{A}
32	Vinyl amyl ketone	–	1.22^{b}	1.08^{a}	1.04^{B}	0.99^{A}
33	6-Methyl-5-Hepten-2-One	–	0.37	–	0.69	–
34	2,4 Heptadıenal	–	0.38	–	0.36	–
35	2-Butenal	–	–	0.89	1.31^{B}	0.59^{A}
36	3-Methylbutanal	–	–	0.89	1.86^{B}	0.57^{A}
37	n-Butanal	–	–	19.11	–	20.84
38	n-Butanol	–	–	1.39	–	0.94
39	Benzene	–	–	0.05	–	0.14
40	2-Decenal	–	–	0.53	–	0.23
	TOTAL	100	100	100	100	100

OPJ Open pan evaporation jam, *VEJ* Vacuum evaporation jam, *DW* Dry weight basis, – not detected

[a-b]Statistical differences between 0 months jam samples in the same row ($p < 0.05$) (n = 3)

[A-B]Statistical differences between after 4 months stored jam samples in the same row ($p < 0.05$) (n = 3)

thermal processing used when processing jujube fruit into jam. L-limonene is one of the major aroma components in citrus fruits [56]. Jam processing resulted in a substantial reduction in the L-limonene content of the jujube fruit in the present study.

Acetoin, 6-methyl-5-heptane-2-one, and 2,4 heptadienal compounds were not found in fresh jujube fruit or VEJ, but were detected in OPJ. We hypothesize that these compounds formed as a result of excess heat processing and oxidation. In contrast, n-butanal (volatile), n-butanol (fermentation-alcohol), benzene (volatile), and 2-decenal (volatile) were not found in fresh jujube or OPJ, but were detected in VEJ. We believe these compounds formed during vacuum evaporation since high temperatures are not used.

Conclusion

Comparison of jams produced by traditional and vacuum concentration methods showed that bioactive components (total phenolic content, organic acids, etc.) were better preserved with the vacuum method. Furfural content increased in both jams during storage; however, thermal processing conditions had a greater effect on furfural formation than storage conditions. Due to the low-temperature and oxygen-free environment created in vacuum processing, this method reduced the HMF content of jujube jam. Total sugar content was better preserved in the vacuum method because it does not use high temperatures. Sugar content decreased in both types of jams in our study, which may be a result of reducing sugars entering Maillard reactions and increasing HMF. It was determined that jujube jam was a particularly good source of the minerals Fe, Ca, Zn, Se, and Cu. Although we detected varying degrees of phenolic compound and organic acid loss during the jujube jam-making process, we recommend jujube jam as a good source of these nutritional components. Our comparison of fresh fruit and jams before and after storage showed that some aroma compounds in the fruit were lost due to thermal processing, that some new aroma compounds formed during jam processing, and that different aroma compounds not found in jujube fruit formed after storage. In light of our results, further studies are recommended to better determine the time/temperature relationship in jams that are produced by vacuum method and heated for pasteurization. For future studies, storing the both of room and refrigerator conditions and determining the changes with periodically analyzes and storing for 6 months may give a better idea.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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