Effect of Dehydration Methods on Antioxidant Activities, Phenolic Contents, Cyclic Nucleotides, and Volatiles of Jujube Fruits

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Abstract Changes in antioxidant activities, phenolic contents, cyclic nucleotides, and volatiles of jujube after dehydration using freeze-drying (FD), air drying (AD), sun drying (SD), and microwave drying (MD) were investigated. All dehydration methods caused a decrease in antioxidant activities, aldehyde, acid, and alkane contents. Esters were increased by the dehydration. FD was superior to other dehydration methods for retention of total phenolics, total flavonoids, and antioxidant activities. MD caused an increase of catechin and epicatechin levels. Although AD, MD, and SD caused declines in cyclic adenosine monophosphate (cAMP) and cyclic guanosine monophosphate (cGMP) contents, AD at 50°C was a good choice to obtain jujubes with high levels of cAMP and cGMP. Theoretical fundamentals for selection of suitable drying techniques to minimize the negative effects caused by dehydration and for meeting production requirements are provided.

Keywords: jujube, drying, volatile, antioxidant, solid phase micro-extraction

Introduction

Fruits commonly known as jujubes from *Ziziphus jujuba* belong to the buckthorn family (*Rhamnaceae*) and are cultivated in more than 30 countries around the world. Jujubes are rich in fiber, sugars, organic acids, trace minerals, proteins, and vitamins. Recognized for an attractive color and a delicious taste, jujubes have been commonly used as a Chinese traditional medicine for thousands of years and exhibit multiple beneficial effects for human health (1). Phytochemical studies have revealed that jujubes exhibit antioxidant activities that are mainly attributed to the natural antioxidants, phenolic acids, flavonoids, anthocyanins, vitamins, and cyclic nucleosides (2,3).

Fresh jujubes are perishable after harvest and deteriorate within a few days, resulting in loss of commercial value. However, dried jujubes are consumed directly and also used as raw materials for other products, such as jujube drinks, candied jujube, and jujube powder. Sun drying (SD) and air drying (AD) are widely applied for dehydration of jujubes in China. In previous studies, the drying characteristics, drying models, quality characteristics, and energy consumption of AD and microwave drying (MD) in jujubes were investigated (4–7). However, there is little information about effects

of different drying methods on antioxidant activities, phenolic compounds, and cyclic nucleotides of jujubes.

Aroma is composed of unique combinations of volatiles, and different proportions of volatiles often determine aroma properties. Wong *et al.* (8) isolated volatiles of dried *Z. jujuba* Mill. var. *inermis* (Bge.) fruits. Rehd and Said *et al.* (9) studied volatiles from fresh aerial parts of *Z. jujuba* and fruits of *Z. spina-christi*. However, the effects of different drying techniques on volatiles of jujubes have been given little attention in China. In addition, it has been reported that cyclic adenosine monophosphate (cAMP) and cyclic guanosine monophosphate (cGMP) are involved in regulation and modulation of physiological processes in the body and exhibit multiple bioactivities affecting nerve impulses and inducing phase shifts of a mammalian circadian pacemaker at night, in opposition to cAMP effects (10,11).

Hence, the objectives of this study were to investigate the effect of different dehydration methods on the antioxidant activities, phenolic compound contents, and cyclic nucleotide contents of fresh and dehydrated jujubes, and to characterize volatiles using solid phase micro-extraction (SPME) combined with gas chromatography mass spectrometry (GC-MS).

Materials and Methods

Raw materials Fresh jujubes (*Z. jujuba* cv. 'jinsixiaozao') were harvested on October 2nd, 2014, from a garden in Zhantang, Mentougou District, Beijing, China. Fruits free of visible blemishes, disease, and mechanical injury with a red peel surface color were selected.

Moisture content determination Moisture content was determined in a drying oven at vacuum degree lower than 13.3 kPa at 70°C until the jujubes weight was constant (12).

Jujubes drying AD was conducted in an air blast dryer (DHG-9053A; Shanghai Jinghong Experimental Facilities Corporation Ltd., Shanghai, China) following a previous method (4). Three levels of 50, 60, and 70°C (AD50, AD60, and AD70) were used. Drying was stopped when moisture content reached approximately 15%. MD was applied in a microwave oven (NJ07-3; Nanjing Jiequan Microwave Apparatus Co. Ltd., Nanjing, China) with a power of 45 W following a previous method (6). One kg jujubes for freeze-drying (FD) were placed at -80°C for 6 h, put into a freeze dryer (LGJ-25C; Four-Ring Science Instrument Plant Beijing Co., Ltd., Beijing, China) and dried under a 10-15 Pa vacuum at 4°C in a chamber with a -60°C of cold trap. Based on initial experiments (data not shown), both MD and FD were ended when moisture content reached approximately 15%. As a third drying method, fresh jujubes were directly exposed to sunlight in the daytime and placed indoors at night for about 17 days to reach a moisture content of 18%.

Extract preparation for measurement of total phenolics (TPs), total flavonoids (TFs), individual phenolic compounds, and antioxidant activities Extract preparation of TPs, TFs, individual phenolic compounds, and antioxidant activities followed the method of Wang *et al.* (13) with a minor modification. Approximately 2 g dried jujube or 6 g fresh jujube was extracted using 30 mL of 80% methanol in ice water in an ultrasonic bath (KD-250DE; Kun Shan Ultrasonic Instruments Co., Ltd., Kunshan, China) for 30 min. The supernatant was separated by centrifugation at 10,000×g at 4°C for 10 min and the residue was re-extracted for 3 times in the same manner. Supernatants were combined and standardized to a final volume of 100 mL using 80% methanol.

Determination of TPs and TFs TPs were measured using Folin-Ciocalteu reagent with gallic acid as a standard and TFs were measured following the NaNO₂-AlCl₃-NaOH method described by Wang *et al.* (13). TPs and TFs were expressed as mg of gallic acid equivalents (GAE)/g dry weight (DW) and mg of rutin equivalents (RE)/g DW, respectively.

Ferric reducing/antioxidant power (FRAP) assay Extracts (0.5 mL) were allowed to react with 2.0 mL of a working FRAP solution at 37°C

for 10 min in the dark. FRAP assay was conducted following the method of Zhang *et al.* (14). Results were expressed as mg of vitamin C equivalents (VCE)/g DW.

DPPH radical-scavenging assay Four mL of DPPH working solution was added to 0.4 mL of jujube extract diluted with 80% methanol, and the mixture was kept in the dark at 25°C for 20 min. Reduction of the purple DPPH radical to the yellow form was measured as described by Wang *et al.* (13). Results were expressed as mg of Trolox equivalents (TE)/g DW.

ABTS radical-scavenging assay ABTS radical-scavenging assay was conducted as described by Wang *et al.* (13) with some modification. An ABTS⁺ solution (3.6 mL) was added to 0.4 mL jujube extract diluted with 80% methanol, and the mixture was kept in the dark for 10 min. Results were expressed as mg of Trolox equivalents (TE)/g DW.

HPLC analysis of phenolic compounds Phenolic compounds were characterized and quantified following a modified method described by Zhang *et al.* (14). Waters HPLC system (2695; Waters Co., Ltd., Milford, MA, USA) was used and separation was performed using a SunfireTM C₁₈ Column (250×4.6 mm, 5 μm) at 30°C. The mobile phase was composed of A: 0.5% formic acid in water, B: acetonitrile with a gradient elution flow rate of 0.8 mL/min with 95% A for 0-5 min, 95-85% A for 5-30 min, 85-76% A for 30-44 min, 85-5% A for 44-55 min, and 5-95% A 55-60 min. The detection wavelength was set at 280 nm. Results were expressed as mg/100 g DW.

Analysis of cyclic nucleotides Eight g of fruit was extracted using 45 mL of de-ionized water at 25°C in an ultrasonic bath for 30 min. The supernatant was separated by centrifugation at 9,000×g at 4°C for 10 min, and the residue was re-extracted for two times by repetition of the above steps. cAMP and cGMP contents were characterized and quantified following the method of Zhang *et al.* (15). Shimadzu HPLC system (LC-20AT; Shimadzu Co., Ltd, Kyoto, Japan) was used and separation was performed on a Hypersil C₁₈ column (250×4.6 mm, 5 μm) at 30°C. The mobile phase was composed of methanol and 50 mM monopotassium phosphate (10:90, v/v) with isocratic elution at a flow rate of 1.0 mL/min and a detection wavelength of 254 nm. Results were expressed as μg/g DW.

Volatiles analysis Volatiles of jujubes were analyzed using SPME-GC-MS. Fresh, FD, AD, MD, and SD jujubes were homogenized for 2 min at 25°C with distilled water at proportions of 1:1, 1:0.64, 1:0.64, 1:0.64, and 1:0.77 (g/mL). Six mL homogenate was added to a 20 mL glass vial containing 1.8 g of NaCl. The vials were equilibrated 15 min with oscillation at 40°C prior to SPME analysis. The SPME device was equipped with a fused silica fiber coated with polydimethylsiloxane/divinylbenzene/carboxen. The fiber was inserted into the headspace for 40 min at 40°C. Fibers were desorbed at

250°C for 3 min in the injection port of an Agilent 7890A-5975C GC-MS (Agilent Technologies, Santa Clara, CA, USA).

Volatiles were identified using a DB-5 column (30 m, 0.25 mm, 0.25 µm) and the injection port was operated in splitless mode. The initial oven temperature was 40°C, followed by holding for 2.5 min, then increased from 40 to 180°C at the rate of 3°C/min, followed by an increase to 240°C at a rate of 10°C/min, and holding for 10 min. Helium was used as a carrier gas at a flow rate of 1.0 mL/min. MS fragmentation was performed using electronic impact at 70 eV with a source temperature of 230°C in scan mode between 50 and 550 mass units. Compound identification was performed based on comparison of mass spectra with entries in the NIST and Wiley libraries. Retention times of straight-chain alkanes (C₆-C₂₄) were used for calculation of retention indices for all identified compounds. The identities of most compounds were confirmed based on DB-5 column linear retention indices.

Statistical analysis All results were reported as mean±standard deviation (SD) values of triplicates. Statistical analysis was performed using OriginPro 8.0 software. Tukey's multiple range test was used for evaluation of significant differences.

Results and Discussion

TPs, TFs, and antioxidant activities TPs, TFs, FRAP activity, DPPH and ABTS radical scavenging activities of fresh and dried jujubes are presented in Table 1. Variations in TPs, TFs, and antioxidant activities were observed among jujubes. TPs and TFs values of fresh fruits were 13.85 mg of GAE/g DW and 9.83 RE/g DW, respectively. FD caused a significant ($p<0.05$) increase in TPs and TFs values, compared with control, while SD led to the highest and significant ($p<0.05$) reduction of TPs and TFs values, compared with control, which corresponded to significant ($p<0.05$) reductions in FRAP, DPPH, and ABTS radical scavenging activities, compared with control. AD50 also caused a significant ($p<0.05$) decrease in TPs content from 13.85 to 11.24 mg of GAE/g DW. However, MD did not produce a significant ($p>0.05$) effect on TPs and TFs values, compared with control.

Declines in TP values during AD50 and SD may be attributed to endogenous enzymatic reactions. During these treatments the

temperature was relatively low, especially in the initial process, and polyphenol oxidase (PPO) was not immediately inactivated. Zhu *et al.* (16) found that variations in TP values synchronized with changes in PPO activities in winter jujubes during post-harvest, and all values declined during the later stages of ripening.

Dehydration of jujubes also caused a significant ($p<0.05$) decrease in DPPH and ABTS radical scavenging activities, compared with controls, and SD resulted in the largest decline. FD exhibited the best retention of antioxidant activities, while other drying led to declines in antioxidant activities. The decrease in antioxidant activities could be attributed to declines in TPs and TFs and ascorbic acid content during drying (17).

All jujube extracts showed lower antioxidant activities measured using the DPPH assay than using the ABTS assay. The ABTS assay is based on generation of blue/green ABTS⁺ radical that is applicable to both hydrophilic and lipophilic antioxidant systems. The DPPH assay uses a radical dissolved in an organic medium and is applicable to hydrophobic systems (18). Similar results were also reported for comparisons of ABTS/DPPH assays in popular antioxidant-rich US foods (19). MD only caused a 4.28% decrease in the jujube FRAP value while AD and SD caused decreases of more than 47.33%. Hence, MD is a promising method for drying jujubes to retain high antioxidant activities and phenolic compounds.

Phenolic compounds Phenolic compound profiles of fresh and dried jujubes were shown in Table 2. Catechin was identified as the dominant phenolic compound in fresh and dried jujubes, ranging from 12.61 to 33.69 mg/100 g DW. FD jujubes showed better retention of phenolics than other drying methods. Six phenolic acids (chlorogenic acid, gallic acid, protocatechuic acid, caffeic acid, ferulic acid), and *p*-hydroxybenzoic acid and 3 flavonoids (catechin, epicatechin, and rutin) were identified in dried jujubes (14,20), attributed to diverse properties of jujube cultivars. Except for FD, all treatments caused a significant ($p<0.05$) decrease in phenolic acid contents, compared with controls. Caffeic acid content was low in fresh jujubes and it was not detected in AD, SD, and MD jujubes. The amount of vanillic acid in AD70 jujubes was significantly ($p<0.05$) higher than the other dried jujubes. Similarly, syringic acid content of AD70 jujubes was significantly ($p<0.05$) higher than AD50, SD, and MD jujubes.

Table 1. Antioxidant activities, TPs and TFs content profiles of fresh and dehydrated jujubes

	TPs (mg GAE/g DW)	TFs (mg RE/g DW)	FRAP (mg VCE/g DW)	DPPH (mg TE/g DW)	ABTS (mg TE/g DW)
Fresh	13.85±0.58 ^{1b}	9.83±0.34 ^b	15.17±0.53 ^a	26.27±0.41 ^a	72.10±0.66 ^a
FD	20.98±1.77 ^a	11.61±0.14 ^a	15.50±0.24 ^a	15.50±0.24 ^b	33.65±0.37 ^b
AD50	11.24±0.43 ^c	8.86±0.24 ^b	7.99±0.40 ^c	5.17±0.17 ^d	13.87±0.34 ^d
AD60	12.90±0.80 ^b	7.20±0.37 ^c	6.02±0.16 ^d	5.60±0.07 ^c	13.75±0.49 ^d
AD70	12.80±1.07 ^b	8.41±0.25 ^{bc}	7.97±0.40 ^c	5.53±0.10 ^c	14.42±0.25 ^d
SD	9.66±0.51 ^d	7.20±0.31 ^c	5.52±0.11 ^e	4.49±0.05 ^e	12.64±0.15 ^e
MD	13.64±1.20 ^b	10.50±1.16 ^{ab}	14.52±0.13 ^b	4.96±0.11 ^d	28.00±0.24 ^c

¹Values represent mean±SD of 3 replicates. Mean values in the same column for each treatment with different superscripts are significantly different ($p<0.05$).

Table 2. Phenolic compound profiles of fresh and dehydrated jujubes

	Phenolic compounds (mg/100 g DW)					
	catechin	vanillic acid	caffeic acid	syringic acid	epicatechin	rutin
Fresh	15.40±0.70 ^{1)cd}	7.97±0.56 ^a	1.56±0.06 ^a	5.14±0.24 ^a	2.84±0.32 ^b	8.54±0.13 ^a
FD	33.69±2.30 ^a	8.80±0.33 ^a	0.51±0.01 ^b	5.36±0.11 ^a	6.22±0.64 ^a	8.32±0.15 ^{ab}
AD50	14.16±0.25 ^e	5.53±0.16 ^c	ND	3.78±0.08 ^c	ND	7.80±0.38 ^b
AD60	14.38±0.52 ^d	5.38±0.14 ^c	ND	3.87±0.13 ^{bc}	0.50±0.09 ^d	8.75±0.69 ^a
AD70	16.82±0.77 ^{bc}	6.73±0.80 ^b	ND	4.03±0.11 ^b	0.17±0.01 ^e	8.30±0.29 ^{ab}
SD	12.61±0.50 ^f	4.92±0.12 ^d	ND	3.01±0.03 ^e	ND	6.49±0.22 ^c
MD	17.68±0.22 ^b	4.80±0.19 ^d	ND	3.36±0.04 ^d	4.44±0.17 ^c	8.95±0.36 ^a

¹⁾Values are reported as mean±SD, *n*=3. Means with different superscripts within a column are significantly different (*p*<0.05). ND means not detected.

SD resulted in the lowest level of vanillic acid and syringic acid, perhaps due to degradation of phenolic acids under high oxygen condition and the presence of PPO. A strong PPO activity in jujubes has been reported (16). During the dehydration process PPO activity remained high for a longer period when jujubes were exposed to SD and 50–60°C.

Compared with fresh jujubes, the catechin level of MD jujubes increased by 14.8% and the epicatechin level increased by 56.3%. However, AD50 and SD resulted in a significant (*p*<0.05) decrease in catechin content, compared with controls. In general, the epicatechin contents of AD jujubes were significantly (*p*<0.05) lower than that of fresh jujubes and it was not detected AD50 and SD jujubes. Madrau *et al.* (21) reported that AD of apricots caused significant decreases in catechin and epicatechin. AD50 and SD also led to a significant (*p*<0.05) decrease in rutin content of jujubes, compared with controls.

Unlike phenolic acids, catechin, and epicatechin, variations in rutin content were not directly correlated with PPO activity since there were no significant (*p*>0.05) differences among AD60, AD70, and MD jujubes. Thus, selection of a suitable dehydration method is critical to obtain high quality dried jujubes with antioxidant and physiological activities.

cAMP and cGMP Levels of cAMP and cGMP in fresh and dehydrated jujubes are shown in Fig. 1. Levels of cAMP and cGMP in fresh jujubes were 130.94 and 150.2 µg/g DW, respectively. FD did not cause a significant (*p*>0.05) decrease in content of cAMP or cGMP, compared with other drying treatments. Levels of cAMP in dried jujubes ranged from 12.28 to 114.69 µg/g DW and there were no significant (*p*>0.05) differences among AD50, AD60, and MD jujubes. SD caused a decrease of 90.6% in cAMP content, compared with fresh jujubes. Similarly, drying resulted in a decrease in cGMP and AD50 exhibited the best cGMP retention.

cGMP was totally degraded after MD, perhaps attributed to microwave radiation that promoted polar molecular rotation and collisions at high speed to produce a large quantity of heat. In addition, considering the convenience and cost, AD50 exhibited a good retention for cAMP and cGMP. AD50 appears to be a suitable method for production of a jujube product with high levels of cAMP and cGMP.

Fig. 1. Effect of different drying methods on cAMP and cGMP contents in jujubes. Bars represent standard deviation, *n*=3. Means with different letters between the same type columns are significantly different (*p*<0.05).

Volatile compounds Volatiles identified based on comparison of mass spectra and retention indices using a DB-5 column with libraries and previously results are shown in Table 3. A total of 46 volatiles were identified, including aldehydes, alcohols, alkenes, ketones, esters, and acids accounting for more than 90% of the chromatographic area. Impurities were probably responsible for the remaining area. The major components identified in fresh jujubes were acetic acid (26.63%), dodecanoic acid (13.27%), *n*-decanoic acid (11.76%), hexanoic acid (9.27%), 2-hexenal (7.42%), and tridecane (4.95%). Wong *et al.* (8) reported volatiles from dried *Z. jujuba* Mill. var. *inermis* (Bge.) Rehd fruits and found that aliphatic acids and carbonyl compounds accounted for 62.97 and 29.25% of total volatile contents, respectively, with the major components being decanoic acid and dodecanoic acid. Said *et al.* (9) identified 41 components in *Z. jujuba* aerial parts and *Z. spina-christii* fruits from Egypt, including borneol, dodecanoic acid, and hexadecanoic acid. Differences in volatiles reported herein and previously were probably related to geographic origin, tissue parts examined, and extraction methods used, such as hydrodistillation and distillation.

Dehydration of jujubes resulted in changes in volatile compositions. Numbers of volatile fractions in FD, AD50, AD60, AD70, SD, and MD

Table 3. Fraction (%) of volatile components of fresh and dehydrated jujubes

Compounds		Identification	RI	RT	Fresh	FD	AD50	AD60	AD70	SD	MD
Aldehydes	(E)-2-Pentenal	RI, MS	724	9.189	0.22	1.65	-	-	-	-	-
	Hexanal	RI, MS	801	10.687	3.63	12.02	0.13	0.09	0.16	3.14	0.15
	Furfural	RI, MS	829	12.676	1.89	-	7.69	7.62	21.66	2.03	29.75
	2-Hexenal	RI, MS	848	12.998	7.42	14.07	6.5	3.13	2.35	7.54	1.34
	Heptanal	RI, MS	901	15.232	0.42	2.82	-	-	-	2.58	-
	Benzaldehyde	RI, MS	960	18.203	1.23	0.31	0.31	0.52	0.32	1.04	0.04
	Nonanal	RI, MS	1107	25.200	0.85	0.51	0.13	0.19	0.20	0.71	0.47
	(E)-2-Nonenal	RI, MS	1169	27.925	-	6.22	-	-	-	-	-
	Decanal	RI, MS	1209	30.020	-	4.11	-	-	-	-	-
	5-Hydroxymethylfurfural	RI, MS	1241	31.436	0.71	-	1.22	3.21	5.32	0.91	15.34
	(E,E)-2, 4-Decadienal	RI, MS	1320	35.067	0.08	-	-	0.11	0.13	-	-
	(Z)-9,17-Octadecadienal	RI, MS	1736	53.831	1.58	-	0.14	-	-	0.25	-
Ketones	2,4-Dihydroxy-2,5-dimethyl-3(2H)-furan-3-one	RI, MS	977	19.200	0.34	-	-	-	-	-	-
	6-Methyl-5-hepten-2-one	RI, MS	986	19.489	-	3.36	-	-	-	-	-
	3,5-Dihydroxy-2-methyl-4H-pyran-4-one	RI, MS	1178	29.553	0.14	-	-	-	-	-	-
	1-(2,6,6-Trimethyl-1,3-cyclohexadien-1-yl)-2-buten-1-one	RI, MS	1362	38.009	-	-	-	0.96	0.78	-	0.27
	(E)-6,10-Dimethyl-5,9-undecadien-2-one	RI, MS	1455	40.676	-	1.13	-	-	-	-	-
Alcohols	1-(2-Furanyl)-ethanone	RI, MS	914	15.983	-	-	4.51	3.66	4.11	-	2.38
	Benzyl alcohol	RI, MS	1001	22.166	0.54	1.18	0.07	0.22	0.31	1.20	0.9
	Borneol	RI, MS	1175	28.364	0.07	-	-	-	-	0.07	-
	2-Methoxy-3-(2-propenyl)-phenol	RI, MS	1327	36.834	-	-	2.07	-	-	4.12	-
	Butylated hydroxytoluene	RI, MS	1516	41.505	-	2.14	-	0.05	-	-	-
	(Z, Z)-2-Methyl-3,13-octadecadienol	RI, MS	1565	45.461	-	-	-	-	-	0.02	-
	2,6-Bis(1,1-dimethylethyl)-4-(1-oxopropyl)phenol	RI, MS	1635	54.270	-	-	-	-	-	2.09	-
Acids	Acetic acid	RI, MS	600	5.775	26.63	18.23	22.31	20.35	21.52	26.61	24.36
	2-Methyl-propanoic acid	RI, MS	854	13.690	0.07	-	-	-	-	-	-
	3-Methyl-butanoic acid	RI, MS	858	13.716	3.87	-	2.56	3.15	3.25	1.09	0.41
	2-Methyl-butanoic acid	RI, MS	873	14.038	1.25	-	0.90	0.91	0.72	0.60	0.38
	Hexanoic acid	RI, MS	1014	20.573	9.27	-	7.08	8.12	7.53	-	-
	n-Decanoic acid	RI, MS	1385	37.450	11.76	3.47	4.94	3.07	6.93	11.11	-
	Dodecanoic acid	RI, MS	1566	45.053	13.27	4.16	9.13	7.54	4.36	12.64	4.02
	n-Hexadecanoic acid	RI, MS	1970	55.166	0.29	0.05	0.10	0.04	-	-	-
	9,12-Octadecadienoic acid	RI, MS	2101	58.082	0.99	0.12	-	0.15	-	0.25	-
Esters	Benzoic acid methyl ester	RI, MS	1062	24.882	-	-	1.27	2.46	2.16	0.03	1.02
	Benzoic acid ethyl ester	RI, MS	1140	28.532	-	-	-	3.16	1.08	-	-
	Decanoic acid ethyl ester	RI, MS	1382	38.264	-	-	3.14	4.19	2.06	-	-
	Dodecanoic acid methyl ester	RI, MS	1507	43.416	-	-	2.06	4.07	1.02	-	-
	Dodecanoic acid ethyl ester	RI, MS	1659	46.070	-	-	1.05	2.13	0.07	0.62	0.01
	Methyl myristoleate	RI, MS	1699	50.249	-	-	1.31	2.06	-	-	-
	Ethyl-9-tetradecenoate	RI, MS	1764	51.992	-	-	0.11	2.05	-	-	-
Alkanes	Dodecane	RI, MS	1200	29.645	2.35	1.25	0.75	-	-	-	-
	Tridecane	RI, MS	1300	34.128	4.95	3.56	3.78	4.32	2.83	4.63	1.94
	Tetradecane	RI, MS	1400	38.375	0.83	0.76	0.78	0.90	1.02	0.70	2.09
	Hexadecane	RI, MS	1600	46.200	0.71	0.59	-	0.63	0.37	-	-
	Heptadecane	RI, MS	1700	49.782	0.42	-	0.29	-	-	-	-
Other	Methoxy-phenyl-oxime	RI, MS	886	15.632	0.41	9.58	0.27	0.29	0.40	0.74	0.41

Fractions of components in fresh and dehydrated jujubes (%) = peak area of a component in jujubes/total peak of identified component in jujubes, -, not detected; RT means retention time; RI means retention indices, which were experimental; MS, the mass spectrum was identical with spectra in the NIST and Wiley mass databases.

jujubes were 28, 30, 25, 24, 22, and 18, respectively. Identified components of fresh and dried jujubes were 9,2-methyl-propanoic acid and 2,4-dihydroxy-2,5-dimethyl-3(2H)-furan-3-one. The compound 3,5-dihydroxy-2-methyl-4H-pyran-4-one was only identified in fresh jujubes. (*E*)-2-Nonenal, decanal, 6-methyl-5-hepten-2-one, (*E*)-6,10-dimethyl-5,9-undecadien-2-one were detected in FD samples, 2,6-bis(1,1-dimethylethyl)-4-(1-oxopropyl) phenol and (*Z,Z*)-2-methyl-3,13-octadecadienol were identified in SD jujubes.

Compared with fresh jujubes, AD, SD and MD decreased relative contents of aldehydes such as (*E*)-2-pentenal, hexanal, 2-hexenal, nonanal, and (*Z*)-9,17-octadecadienal, while FD led to increases in relative contents of (*E*)-2-pentenal, hexanal, and 2-hexenal. Bi *et al.* (22) reported that relative contents of aldehydes significantly decreased when fresh jujubes were processed into powder. It was possible that reduction of compound levels occurred during dehydration when aldehydes were deoxidized and transformed into corresponding alcohols, which promoted esterification of alcohols with acids produced during thermal decomposition. This reduction in aldehydes levels was confirmed based on reductions of acids, including acetic, 2-methyl-propanoic, 3-methyl-butanoic, 2-methyl-butanoic, hexanoic, *n*-decanoic, dodecanoic, *n*-hexadecanoic, 9,12-octadecadienoic acids, and generation of esters, including benzoic acid methyl ester, benzoic acid ethyl ester, decanoic acid ethyl ester, dodecanoic acid methyl ester, dodecanoic acid ethyl ester, methyl myristoleate, and ethyl-9-tetradecenoate present in AD, SD, and MD jujubes. The relative contents of esters were increased with AD60 and no esters were detected in FD jujubes.

Apart from newly generated esters, some of the main components identified in AD, SD, and MD jujubes were furfural and 5-hydroxy-methylfurfural. They appeared in fresh and AD, SD, and MD jujubes. Furfural and 5-hydroxymethylfurfural levels in AD, SD, and MD jujubes were much higher than those in fresh jujubes. The highest contents of them were produced using MD, followed by AD70. Similar phenomenon was also observed in dried apricots (23). Dehydration also caused loss of the alkanes dodecane, tridecane, tetradecane, hexadecane, and heptadecane. Similar alkanes were identified in the unsaponifiable fraction of *Zizyphus spina-christi*. L. seeds (24).

From the flavor of AD product, a drying temperature of 50-60°C produced the best product since AD jujubes contained maximum numbers of different volatiles with introduction of a series of esters, which are critical for the aroma of biomaterials (25). Since contributions made by the esters to the aroma of jujube is not well understood, further studies are needed to be performed.

In summary, FD was superior to other dehydration methods for retention of TPs, TFs, and antioxidant activities. HPLC analysis showed that the contents of catechin and epicatechin in FD jujubes were higher than in fresh jujubes. Considering the cost of drying, MD was the best method for preservation of flavonoids, including catechin, epicatechin, and rutin. AD, MD, and SD caused declines in cAMP and cGMP levels. However, AD50 exhibited better cAMP and cGMP retention rates than other thermal treatments. Considering

the convenience and cost of drying, AD50 is a suitable choice for production of a jujube product with high levels of cAMP and cGMP. Drying of jujubes also caused losses of aldehydes, acids, and alkanes, while esters were generated by the thermal treatments.

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