Hindawi
Journal of Food Quality
Volume 2018, Article ID 9242675, 7 pages
https://doi.org/10.1155/2018/9242675

Research Article

Effect of Thermal Treatments on Quality and Aroma of Watermelon Juice

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Received 12 September 2017; Revised 4 January 2018; Accepted 16 January 2018; Published 20 February 2018

Academic Editor: Hüseyin Erten

The effect of thermal treatments on the quality and aroma of watermelon juice was evaluated. Watermelon juice was pasteurized via ultrahigh temperature (UHT, pasteurized at 135°C for 2 s), low temperature long time (LTLT, pasteurized at 60°C for 30 min), and high temperature short time (HTST, pasteurized at 100°C for 5 min), respectively. UHT and LTLT reduced the total flora count and maintained the color of the pasteurized juice, while the HTST led to a significant color difference. A total of 27, 21, 22, and 21 volatiles were identified in the unpasteurized juice, UHT, LTLT, and HTST, respectively. The typical watermelon aroma, including (3*Z*)-3-nonen-1-ol, *(E)*-2-nonen-1-ol, 1-nonanal, (2*E*)-2-nonenal, and *(E,Z)*-2,6-nonadienal, was abundant in the LTLT. Consequently, the aroma of the LTLT was similar to that of unpasteurized juice. Moreover, the shelf life of the LTLT reached 101 and 14 days at 4 and 25°C, respectively. Hence, the LTLT was the best way to maintain the quality and aroma of watermelon juice.

1. Introduction

Watermelon *(Citrullus lanatus)* juice is welcomed all over the world [1], which provides consumers with both moisture and physiological benefits. Intake of watermelon juice increases plasma concentrations of lycopene, β-carotene [2], and arginine [3, 4], reduces chemical-induced hepatotoxicity [5] and proliferation of cancer cell line [6], and even relieves muscle soreness of athletes [7]. Moreover, the production of watermelon juice will reduce the seasonal excess of watermelons in summer.

Watermelon juice is sensitive to heat, oxygen, light, and ions [1, 8–10]. Several techniques have been applied to maintain the original quality and aroma of watermelon juice. High pressure treatment less influences the aroma and color of watermelon juice [1, 10]; high pressure carbon dioxide treatment inactivates polyphenol oxidase, peroxidase, and pectin methylesterase of watermelon juice, thus maintaining their original properties [11]; pulsed electric field treatment maintains the typical aroma of watermelon juice for 21 days [8] and also maintains the total antioxidant capacity of watermelon juice [12]. However, the mentioned techniques were intermittent, which maintained qualities and aroma of watermelon juice at the expense of high cost and limited production capacity. In 2012, the production of watermelons reached 68,000,000 tons in China alone. Even 0.1% of the production (68,000 tons) would pose a significant challenge on the current production capacity of intermittent techniques.

Conventional and continuous processing techniques are more feasible for the production of watermelon juice, such as ultrahigh temperature (UHT), low temperature long time (LTLT), and high temperature short time (HTST). LTLT and HTST are the most conventional ways to inactivate bacteria for centuries [13]. UHT is widely used in milk production; however, the technique is also used for fruit juices, cream, soy milk, yogurt, wine, soups, honey, and even stews [14]. UHT treatment has effectively inactivated watermelon juice bacteria [15] and maintained the typical aroma of sugarcane [16]. However, to the best of our knowledge, the effect of three thermal treatments on the quality and aroma of watermelon juice had not been compared. Hence, the UHT, LTLT, and HTST were applied to watermelon juice, and the quality and aroma of the pasteurized juice were compared.

2. Material and Methods

2.1. Processing of Watermelon Juice. Mature watermelon (*Citrullus lanatus* var. Jingxin number 3) was purchased from a local fruit market. The fruits were round with regular green stripes and an average weight of 3.3 kg. The flesh of the fruits was red and their central soluble solid content (SSC) was 11.5%~13.5%. The fruits were peeled and squeezed via Philips juicer after storage at 4°C for 24 h (HR1861, Philips Co., Beijing, China). The initial pH and SSC of the juice were 5.2 and 7.6%, respectively. The juice was mixed with a complex food additive, including carboxymethylcellulose sodium, ascorbic acid, xanthan gum, ethylene diamine tetraacetic acid, carminum, and sodium pyrophosphate, at a ratio of 100 : 1. After mixing for 10 min, the pH and SSC of the mixture were adjusted to 4.10 via citric acid and 8.0% via high fructose corn syrup (4502504-01, Fresh Juice Industry (Kunshan) Co. Ltd.). The resulting juice was further homogenized at 50 MPa (NS101L2 K, GEA Niro Soavi S.p.A., Parma, Italy). In succession, the juice was pasteurized via UHT, LTLT, and HTST treatments, respectively. Specifically, the UHT treatment heated the juice in a tubular heat exchanger to 135°C for 2 s (Model Number FT74 UHT/HTST Processing System, Armfield Technical Education Co. Ltd., Ringwood, UK). The LTLT treatment heated the juice in a water bath to 60°C for 30 min. The HTST treatment heated the juice in a tubular heat exchanger to 100°C for 5 min (Model Number FT74 UHT/HTST Processing System, Armfield Technical Education Co. Ltd., Ringwood, UK). Finally, the pasteurized juice was sterile filled into 300 mL PET bottles via aseptic filling machine (number 741, Shanghai Triowin Intelligent Machinery Co. Ltd., Shanghai, China).

2.2. Total Flora Counts. Juice was serially diluted and plated in total count agar for total flora counts according to national standard GB 4789.2-2016 [17]. The plates were incubated at 37°C for 48 h and counted manually.

2.3. Determination of Color and Soluble Solid Content. Color L^*, a^*, and b^* values of the sample were measured in a reflectance mode for 6 times at 23°C (Chromameter CR-300, Minolta, Japan). The total color difference (ΔE) was calculated by

$$\Delta E = \left[(L - L_0)^2 + (a - a_0)^2 + (b - b_0)^2\right]^{1/2}, \quad (1)$$

where ΔE is the total color difference between a sample and the control; L and L_0 are the lightness of a sample and the control, respectively; a and a_0 are the redness of a sample and the control, respectively; b and b_0 are the yellowness of a sample and the control, respectively.

SSC was measured via a pocket refractometer (Pal-α, ATAGO, Japan) with distilled water as a blank.

2.4. Electronic Nose Analysis. Aroma of the sample was compared via an electronic nose PEN3 (Airsense Analytics GmbH, Schwerin, Germany). The electronic nose was turned on for 30 min and was cleaned by flushing the testing system with filtered air at a flow rate of 400 mL/min for 180 s. The electronic sensor was immediately inserted into the testing tube after the sample of 2 mL was put into the tube at 25°C for 30 min. The responses of the sensor were collected for 60 s with an interval of 1 s. During the data collection, the chamber flow was 400 mL/min with filtered air. The resulting data were further analyzed by principal component analysis (PEN3 & WinMuster, Version 1.6.2, Airsense Analytics GmbH, Schwerin, Germany).

2.5. Headspace Purge and Trap. An aliquot of 10 mL of the sample was put into a 20 mL glass vessel. The glass vessel was placed in a water bath at 30°C for 30 min. The headspace was purged with a constant flow of nitrogen at 20 mL/min for 10 min. The volatile compounds were trapped in a glass capillary tube (3 mm internal diameter) containing 100 mg of Tenax® adsorbent. The Tenax trap was then disconnected from the system and placed in a Chrompack TCT/PTI 400 injector.

2.6. Determination, Quantification, and Classification of Volatiles. The determination, quantification, and classification of volatiles followed the recent method [18] with a few modifications. Specifically, volatiles of the sample were evaluated on an Agilent 6890 gas chromatograph coupled to an Agilent 5973I mass selective detector (Agilent Technologies, Palo Alto, CA). The volatile compounds were separated on a DB-Wax column (30 m × 0.25 mm i.d., 0.25 μm film thickness, Agilent Technologies). The injection was performed in splitless mode (0.7 mm splitless inlet liner, Supelco) and injector temperature was 220°C. The purge valve was opened at 0.5 min at a flow rate of 50 mL/min. Helium (99.999%) was used as the carrier gas with a constant starting flow rate at 0.7 mL/min. The oven temperature was programmed as follows: 35°C for 1 min, 5°C/min to 100°C, and 20°C/min to a final temperature of 250°C with a final holding time of 5 min. The detector was fitted with an electron impact ionization source set at 230°C. The quadrupole temperature was set to 150°C and the transfer line temperature was kept at 250°C. The solvent delay was set to 3 min. Total ion chromatograms were collected by scanning from m/z 30 to 150 at a rate of 3.06 scans/s.

Volatile compounds were identified via comparison of their mass spectra and retention times with those of authentic standards or via comparison of Kovats' retention indexes and mass spectrum with those reported in the NIST Mass Spectral Search Program (version 2.0a) with <80% as a cutoff to match compounds. The Kovats index was calculated from the retention times of C6–C40 n-alkanes following the recent method [19].

Quantification of the volatile compounds was carried out by calibration curves in the selected ion monitoring mode. 2-Methyl-3-heptanone with a concentration of 45 mg/L in the analyzed samples was used as internal standard to monitor the instrument response and retention time stability. Quantitative analysis was performed using software Agilent Mass Hunter (USA).

The volatiles were classified into six groups based on their chemical structure, including alcohol, aldehyde, ketone, acid, ester, and others. Specifically, the volatile content of each

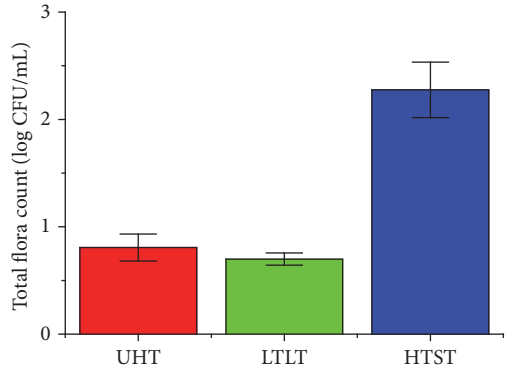

Figure 1: Effect of thermal treatments on the total flora counts of the watermelon juice. UHT: ultrahigh temperature; LTLT: low temperature long time; HTST: high temperature short time.

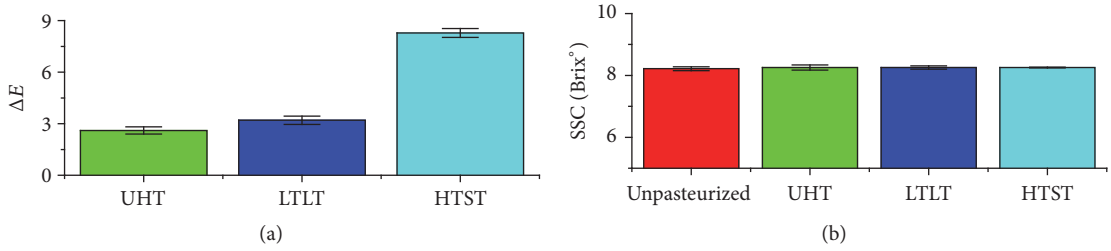

Figure 2: Effect of thermal treatments on the color (a) and soluble solid content (b) of the watermelon juice. UHT: ultrahigh temperature; LTLT: low temperature long time; HTST: high temperature short time.

group was accumulated and ranked, and ranked content was normalized with the biggest one in each group as the 100% and the 0 mg/L as the 0%. The normalized content was used to express distribution of the volatiles via a radar chart.

2.7. Statistical Analysis. All experiments were done in triplicate or more. The data were expressed as average ± standard deviation of at least three repetitions. Analysis of variance (ANOVA) was used to compare mean differences of the results. If differences among means were detected, multiple comparisons were performed using Duncan's Multiple Range Test. Directional difference tests were employed to enable direct comparison of the results of the sensory evaluations. All analyses were conducted using SPSS (Windows Version 19).

3. Results and Discussion

3.1. Total Flora Count and Shelf Life of Watermelon Juice. The effect of thermal treatments on total flora count of watermelon juice is shown in Figure 1. The initial total flora count of watermelon juice was (5.8 ± 0.2) log CFU/mL. The total flora count of the UHT and LTLT was below 2.0 log CFU/mL, which met the requirement of most of the national beverage and juice regulations, while that of the HTST was higher than 2.0 log CFU/mL. The total flora count of the UHT was statistically similar to that of the LTLT; however, it was significantly lower than that of the HTST. Remarkably, the HTST reduced total flora count for 3.5 log CFU/mL, while heating to 90°C for 30 s inactivates *Lactobacillus brevis* and *Saccharomyces cerevisiae* in apple juice for 5.0 log CFU/mL [20]. This difference could result from the low acidity of apple juice.

The shelf life of the watermelon juice was evaluated based on a threshold of total flora count at 2.0 log CFU/mL. The shelf life of the UHT and LTLT was 90 and 101 d at 4°C and was 10 and 14 d at 25°C, respectively. Hence, the LTLT achieved a longer shelf life than the UHT at both 4 and 25°C. The shelf life of LTLT was also longer than the juice pasteurized by high-intensity pulsed electric field (91 days at 5°C) [21].

3.2. Color and Soluble Solid Content of Watermelon Juice. The effect of thermal treatments on color and SSC of watermelon juice is shown in Figure 2. The initial color of the juice was L^* (of 28.38 ± 0.11), a^* (of 4.75 ± 0.10), and b^* (of 2.43 ± 0.02). A normal person will identify a color difference when ΔE is above 6.5. ΔE of the UHT and LTLT was below 6.5 and was statistically similar. Consequently, the UHT and LTLT well maintained the color of the pasteurized juice. Being similar to our results, the heating at 76.6°C for 17 s leads to a darker but redder juice when the taste panelists rate the pasteurized watermelon juice similarly to raw juice in color [22]. However, ΔE of the HTST was 8.27 ± 0.25 and was significantly higher than that of the UHT and LTLT. Consequently, the HTST led to a significant color difference. Each treatment resulted in no significant influence on SSC of watermelon juice, which was statistically similar to recently reported results [1, 22, 23].

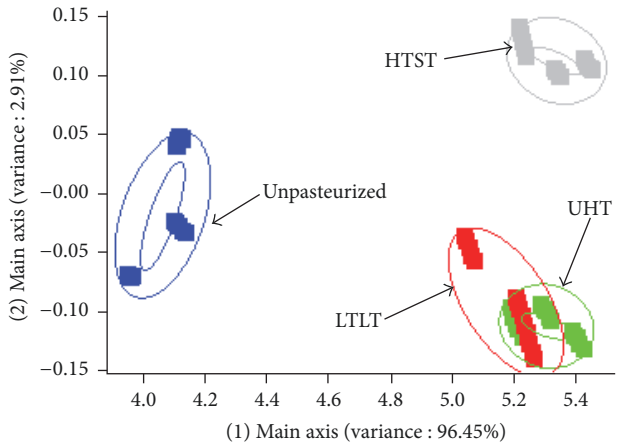

Figure 3: Effect of thermal treatments on the aroma of the watermelon juice. UHT: ultrahigh temperature; LTLT: low temperature long time; HTST: high temperature short time.

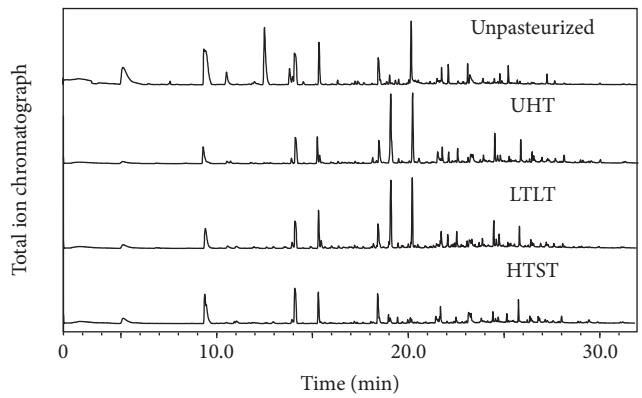

Figure 4: Total ion chromatographs of the watermelon juice. UHT: ultrahigh temperature; LTLT: low temperature long time; HTST: high temperature short time.

3.3. Aroma of Watermelon Juice. The aroma of the pasteurized juice was compared to unpasteurized juice via the sensor array of 10 electrodes of an electric nose. The dimensions of the electrical response were reduced via principal component analysis. Main components 1 and 2 contributed 96.45% and 2.91% of the total watermelon aroma, respectively (Figure 3). Specifically, the aroma of the three treatments was statistically similar based on main component 1 but significantly different to that of the unpasteurized juice. The aroma of the UHT was similar to that of the LTLT based on main component 2, while being different to that of the HTST. Furthermore, the distance from the UHT and LTLT to the unpasteurized juice was shorter than that from the HTST. Consequently, the aroma of the UHT and LTLT was more similar to that of unpasteurized juice. In contrast to our results, heating to 80°C for 5 min or heating to 90°C for 28 s leads to a significant aroma change in berry juice [24] or apple cider [25], respectively. Consequently, a lower heating temperature was the key to maintain the aroma of the juice.

3.4. Volatiles of Watermelon Juice. The volatiles of watermelon juice were assigned via GC-MS analysis by comparing their mass spectra and retention times with those of authentic standards or via comparison of Kovats' retention indexes and mass spectrum. The total ion chromatograph profiles are shown in Figure 4. A total of 27, 21, 22, and 21 volatiles were identified in the unpasteurized juice, UHT, LTLT, and HTST, respectively (Table 1). The volatiles were further classified as alcohol, aldehyde, ketone, acid, ester, and others based on their chemical structures (Figure 5). Unpasteurized juice presented the highest aldehyde content, while the HTST showed the highest acid and ester contents. This phenomenon resulted from the contact of the juice with oxygen during thermal processing [26].

Alcohol and aldehyde constituted 87.5%, 74.0%, 72.0%, and 69.3% in unpasteurized juice, UHT, LTLT, and HTST, respectively. Among alcohol and aldehyde, C9 alcohol and aldehyde present the typical aroma of watermelon. Specifically, (3*Z*)-3-nonen-1-ol, *(E)*-2-nonen-1-ol, 1-nonanal, (2*E*)-2-nonenal, and *(E,Z)*-2,6-nonadienal have been designated as the typical watermelon aroma [19, 27]. The typical volatile content of unpasteurized juice was significantly higher than that of pasteurized juice (Figure 5), which was in accord with the results of the electrical nose. Remarkably, the LTLT was the best to maintain the typical volatile content of pasteurized watermelon juice.

Remarkably, acetic acid, 2-methyl butyric acid, and octanoic acid were only identified in pasteurized watermelon juice, which could result from the thermal treatments. In contrast to our results, heating to 90°C for 30 s leads to a complete loss of acetic acid in apple juice [20].

4. Conclusion

The total flora count of the UHT and LTLT was below 2.0 LogCFU/mL, which met the requirement of most national safety regulations on beverage. The UHT and LTLT well maintained the color of the pasteurized juice, while the HTST led to a significant color difference. A total of 27, 21, 22, and 21 volatiles were identified in unpasteurized juice, UHT, LTLT, and HTST, respectively. Alcohol and aldehyde were the main volatiles in each watermelon juice, which constituted 87.5%, 74.0%, 72.0%, and 69.3% in unpasteurized juice, UHT, LTLT, and HTST, respectively. The typical watermelon aroma contents of the LTLT, including (3*Z*)-3-nonen-1-ol, *(E)*-2-nonen-1-ol, 1-nonanal, (2*E*)-2-nonenal, and *(E,Z)*-2,6-nonadienal, were the highest in all pasteurized juice. Consequently, the aroma of the LTLT was more similar to the unpasteurized juice than that of other treatments. Moreover, the shelf life of the LTLT reached 101 and 14 d at 4 and 25°C, respectively. Hence, the LTLT was the best way to maintain qualities and aroma of watermelon juice.

Additional Points

Practical Application. Watermelon juice is sensitive to heat, oxygen, light, and ions. The LTLT (pasteurized at 60°C for

Table 1: Volatiles detected in the watermelon juice.

Type	Volatiles	Unpasteurized (mg/L)	UHT (mg/L)	LTLT (mg/L)	HTST (mg/L)
Alcohol	2-Methyl-1-propanol	2.99 ± 0.18	-	-	-
	3-Methyl-1-butanol	0.58 ± 0.04	-	-	-
	Hexyl alcohol	2.78 ± 0.43	-	-	-
	2-Octanol	3.13 ± 0.25	-	-	-
	2-Ethylhexanol	1.10 ± 0.14	-	-	0.93 ± 0.32
	(3*Z*)-3-Nonen-1-ol[§]	10.3 ± 0.84	7.54 ± 1.20**	9.37 ± 0.58*	5.68 ± 1.20**
	(E)-2-Nonen-1-ol[§]	13.0 ± 1.35	10.6 ± 0.87**	10.9 ± 0.57**	9.95 ± 0.51**
	Geraniol	0.75 ± 0.04	-	-	-
	Phenethyl alcohol	9.19 ± 0.91	11.6 ± 0.59**	-	-
	1-Tridecanol	2.41 ± 0.14	26.3 ± 1.28**	20.9 ± 1.59**	15.8 ± 1.21
	Dodecyl alcohol	0.58 ± 0.03	-	-	-
	1-Tetradecanol	-	3.97 ± 0.64	3.16 ± 1.04	-
	Subtotal	*46.81*	*60.01*	*44.33*	*32.32*
Aldehyde	2-Hexenal, *(E)*[§]	13.0 ± 1.20	3.77 ± 0.25**	11.8 ± 0.75*	7.08 ± 0.72**
	Octanal	15.7 ± 1.39	10.2 ± 0.58**	6.50 ± 0.74**	13.25 ± 1.20*
	(E)-Hept-2-enal	3.68 ± 0.68	-	1.21 ± 0.24**	-
	1-Nonanal[§]	25.4 ± 2.39	22.5 ± 1.55*	23.7 ± 2.04*	28.07 ± 0.87*
	Decyl aldehyde	18.3 ± 0.88	17.3 ± 1.24	4.29 ± 0.47**	6.21 ± 0.49**
	(2*E*)-2-Nonenal[§]	30.4 ± 2.89	24.3 ± 1.89*	26.88 ± 1.47*	29.38 ± 2.41
	(E,Z)-2,6-Nonadienal[§]	77.6 ± 5.21	60.5 ± 2.80**	69.4 ± 4.24*	66.1 ± 0.89**
	(E)-2-Dodecenal	23.0 ± 2.41	15.9 ± 1.14**	20.7 ± 2.45*	18.8 ± 2.08**
	Subtotal	*207.08*	*154.47*	*164.48*	*168.89*
Ketone	6-Methyl-5-hepten-2-one	11.92 ± 1.52	14.7 ± 1.22**	7.57 ± 1.07**	10.1 ± 0.88*
	6,10-Dimethyl-5,9-undecadien-2-one	8.50 ± 1.14	17.8 ± 1.38**	26.8 ± 1.52**	18.7 ± 0.58**
	2-Pentadecanone	0.99 ± 0.07	5.89 ± 0.38**	8.09 ± 1.03**	7.89 ± 0.74**
	Subtotal	*21.41*	*38.36*	*42.46*	*36.69*
Acid	Acetic acid	-	8.27 ± 1.71	3.65 ± 0.28	11.17 ± 1.51
	2-Methyl butyric acid	-	1.04 ± 0.17	0.67 ± 0.02	2.41 ± 0.18
	Octanoic acid	-	6.09 ± 0.47	0.64 ± 0.09	9.80 ± 1.42
	Subtotal	*0*	*15.4*	*4.96*	*23.38*
Ester	Octyl formate	-	-	-	2.38±
	Diisobutyl phthalate	0.78 ± 0.03	7.05 ± 0.71**	9.54 ± 0.85**	9.16 ± 0.17**
	Isopropyl palmitate	-	2.52 ± 0.21	0.73 ± 0.03	2.41 ± 0.17
	Subtotal	*0.78*	*9.57*	*10.27*	*13.95*
Other	Tetradecane	1.94 ± 0.17	-	8.41 ± 1.30**	-
	1-Hexadecene	2.09 ± 0.28	-	-	-
	2-Pentylfuran	9.25 ± 0.84	12.1 ± 0.98**	15.0 ± 1.17**	14.9 ± 1.07**
	Phenol	0.84 ± 0.04	-	-	-
	Subtotal	*14.12*	*12.1*	*23.41*	*14.9*

-: not detected; ∗: significant difference with the unpasteurized juice at $p \leq 0.05$; ∗∗: significant difference with the unpasteurized juice at $p \leq 0.01$; [§]typical volatiles of watermelon; UHT: ultrahigh temperature; LTLT: low temperature long time; HTST: high temperature short time.

30 min) reduced the total flora count to 2.0 LogCFU/mL and showed no significant influence on the color of the pasteurized juice. Moreover, LTLT maintained the typical aroma of watermelon, including (3Z)-3-nonen-1-ol, (E)-2-nonen-1-ol, 1-nonanal, (2E)-2-nonenal, and (E,Z)-2,6-nonadienal. The shelf life of the LTLT reached 101 and 14 days at 4 and 25°C, respectively. Hence, LTLT was the best way to maintain the quality and aroma of watermelon juice.

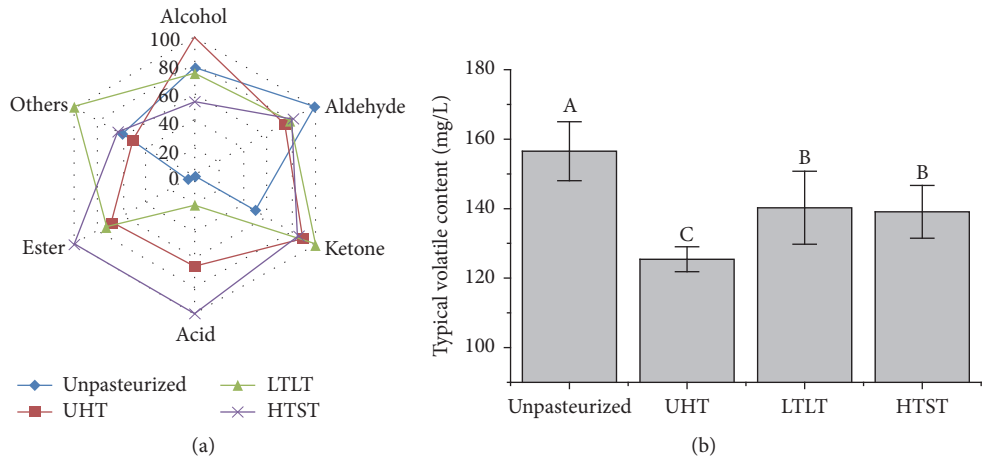

Figure 5: Effect of treatments on the distribution of the volatile chemical structure (a) and the content of the typical volatiles (b) and of the watermelon juice. UHT: ultrahigh temperature; LTLT: low temperature long time; HTST: high temperature short time.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

The authors acknowledge financial support of China Agricultural Research System (CARS-25), Beijing Natural Science Foundation (6172013), Beijing Key Laboratory of Fruits and Vegetable Storage and Processing (Z141105004414037), and the National Key Research and Development Program of China (2016YFD0400302-5).

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