

Influence of post-harvest moisture on roasted almond shelf life and consumer acceptance

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Abstract

BACKGROUND: The harvest weights of sweet almonds (*Prunus dulcis*) have significantly increased to meet consumer demand and now exceed processing facility capabilities. Crops are stockpiled for longer periods, increasing the probability of moisture exposure. Wet almonds can be mechanically dried prior to processing; however, it is unclear how this practice influences lipid oxidation, shelf-life, and consumer acceptance. To address this, almonds were exposed to 8% moisture and dried with low heat (ME). Almonds were roasted and stored under accelerated conditions for 12 months and markers of lipid oxidation, headspace volatiles, sensory attributes, and consumer liking were evaluated.

RESULTS: At 7 months of storage, light roast ME almonds had higher levels of volatiles related to lipid oxidation than non-moisture exposed almonds (NME) and were significantly higher in oxidized, cardboard and painty / solvent flavors. Although untrained consumers did not show significant preferences between the light roast ME and NME almonds, there were quality losses related to lipid oxidation that trained panelists could detect. Dark roast ME almonds demonstrated significant lipid oxidation by 5 months of storage, indicating they will have a compromised shelf life. Findings also indicate that octanal, nonanal, 2-octenal, and hexanoic acid are good indicators of consumer acceptability.

CONCLUSION: The results of this research illustrate that post-harvest moisture exposure with mechanical drying has a significant effect on the storage quality of roasted almonds and is most pronounced in dark roast products.

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Keywords: almond; moisture; HS-SPME GC/MS; descriptive analysis; sensory; shelf-life

INTRODUCTION

Sweet almonds (*Prunus dulcis*) are the seeds of a drupe in the rose family¹ and have been consumed since the early Bronze Age (1000–2000 BCE).² Almonds are an excellent source of α -tocopherol (vitamin E), high-value protein, essential minerals, and monounsaturated fats.³ The consumption of almonds is associated with lowering low-density lipoprotein (LDL) and reducing the risk of heart disease.⁴ Almonds are consumed worldwide raw and in roasted snacks, confectionary, bakery products, nut butters, and increasingly as alternative proteins in plant-based diets.

California produces more than 80% of the global almond supply and the crop more than doubled in weight between 2005–2018.⁵ Almonds are harvested mechanically using tree shakers. The fallen almonds are swept into windrows and allowed to dry to ~5% moisture. These almonds are collected and stored in stockpiles⁶ until they are processed, which involves removing the hulls and shells followed by controlled storage (i.e. indoor storage with controlled temperature). Increased harvests have surpassed processing facility capabilities and crops are stockpiled for longer periods of time, increasing the probability of moisture exposure due to rain and humidity.

Raising the moisture content of the hull and kernel after harvest can affect the quality of almonds by increasing the potential of the nutmeat to form a dark-brown discoloration upon heating (termed concealed damage) and form off flavors.^{7–10} The discoloration and formation of off flavors result from the hydrolysis of triglycerides and carbohydrates, initiated by moisture exposure. These hydrolysis products serve as precursors for the Maillard browning reaction.⁷ Zacheo *et al.* (1998)¹¹ was the first to

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demonstrate a relationship between post-harvest moisture exposure and increased lipid oxidation in almonds.

Lipid oxidation plays a vital role in the sensory attributes of food that is rich in unsaturated fatty acids.¹² Almonds are susceptible to lipid oxidation as they are 50%–60% lipid by weight and are composed primarily of oleic acid (60–70%) and linoleic acid (20–30%).¹³ Ideally, almond hulls and shells are removed at a kernel moisture content of $\leq 5\%$ as this helps prevent mechanical damage to the nutmeat (e.g. chipping and scratching) during the hulling / shelling process.¹⁴ If the kernel moisture content is $> 7\%$ (e.g. from post-harvest moisture exposure) the almonds are mechanically dried to $\sim 5\text{--}6\%$ prior to hulling and shelling.¹⁴ Rogel *et al.* (2017)⁷ demonstrated that drying raw almonds exposed to post-harvest moisture at $\leq 65^\circ\text{C}$ can reduce the degree of brown discoloration upon roasting, whereas drying above 75°C promotes the brown discoloration and the formation of volatiles related to lipid oxidation upon roasting. Although mechanical drying raw almonds below 65°C can reduce the visible browning that occurs with roasting, oxidative damage may still be present and result in a decreased shelf-life of roasted almond products.⁷ Roasted almond kernels can be stored for 18–24 months depending upon the roasting conditions and packaging used.¹⁵ Roasting promotes the formation of volatile heterocycles associated with roasted aroma (e.g. pyrazines, furans, pyrans, pyrroles)¹⁶ and volatile products arising from the oxidation of fatty acids.¹⁷ The decomposition of lipids produces a wide range of aldehydes, alcohols, ketones, and organic acids (e.g. hexenal, pentanol, acetic acid), which contribute to the off flavors associated with the development of rancidity in almonds.^{16, 18}

To date, there are no studies investigating the impact of post-harvest moisture exposure and drying have on the development of lipid oxidation in roasted stored almonds, although this practice has the potential to significantly affect product shelf life. Moreover, there are no data available evaluating whether this practice influences consumer acceptance of these almonds.

MATERIALS AND METHODS

Chemicals and reagents

Stable isotope internal standards: octanal- d_{16} , 2-methylpyrazine- d_6 , and *n*-hexyl- d_{13} alcohol were purchased from C/D/N Isotopes Inc. (Quebec, Canada). All other standards, solvents, and reagents were purchased from Sigma-Aldrich (St Louis, MO, USA) or Fisher Scientific (Hampton, NH, USA). These include high-performance liquid chromatography (HPLC) grade solvent acetic acid, chloroform, and 2,2,4-trimethylpentane; analytical grade sodium hydroxide, American Chemical Society (ACS) grade hydrochloric acid, potassium iodide (99.9%), sodium thiosulfate (99%), and the volatile compounds (95%–99%) identified with authentic standards.

Sample treatment and storage

A 300 kg sample of newly harvested raw Nonpareil almond kernels (from 2015 harvest year), which were not exposed to post-harvest moisture, was obtained from Blue Diamond Growers (Sacramento, CA, USA). The moisture content of the almonds was determined gravimetrically as 4%. The almonds were separated into a control group with no moisture exposure (NME), and a moisture exposed (ME) group. The moisture content of the ME group was increased to 8% by incubating almonds in a KMF 240 constant climate chamber (Binder Inc., Bohemia, NY, USA) at 38°C and $90 \pm 1\%$ relative humidity (% RH) for 36 h. The ME almonds were dried in a R-4 Harvest Saver Dehydrator

(Commercial Dehydrator System Inc., Eugene, OR, USA) at $50 \pm 1^\circ\text{C}$ for 12 h to reduce the moisture content to 4%. Both NME and ME almonds were dry roasted in an E32D5 Turbofan electric convection oven (Moffat Inc., Winston-Salem, NC, USA). Kernels were roasted under two different conditions: $115 \pm 3^\circ\text{C}$ for 60 min (light roast, LR) and $152 \pm 3^\circ\text{C}$ for 15 min (dark roast, DR) to achieve different nutmeat color. Almonds were cooled and divided into paper bags of 460 g each and placed into the climate-control chamber. The chamber was set at $39 \pm 1^\circ\text{C}$ and $15 \pm 1\%$ RH. Almonds were stored for up to 12 months. The location of each individual bag in the chamber was randomly assigned. Randomized samples were removed from the chamber every month, mixed thoroughly, and repackaged into vacuum sealed polyethylene bags then stored at -80°C until analyzed. A total of 52 sample types (2 treatments with 2 roasting levels) were analyzed monthly from 0–12 months for lipid oxidation markers.

Analysis of lipid oxidation

Whole almonds were ground for three 1 s pulses using a Waring laboratory grinder (Waring Laboratory Equipment, Torrington, CT, USA). The ground almonds were sieved through a size 20 Tyler standard screen (W.S. Tyler Industrial Group, Mentor, OH, USA). The oil was extracted from the ground almonds using a 12-ton Carver manual oil press (Carver, Inc., Wabash, IN, USA). The extracted oil was collected in an amber vial and stored at -20°C until analyzed. Peroxide values (PV), free fatty acid values (FFA), and conjugated dienes (CD) were measured in the extracted oil. Peroxide values were determined according to the American Oil Chemists' Society (AOCS) official method Cd 8–53, with the results expressed as peroxide milliequivalents (mEq) per kg.¹⁹ The amount of free fatty acids was determined according to the AOCS official method Cd 3d-63, with the result reported as percentage oleic acid.²⁰ Conjugated dienes level was measured according to the AOCS official method Ti 1a-64, with the results expressed as percentages.²¹ The solvents used in these protocols (e.g. chloroform, iso-octane, acetic acid) were flammable and toxic. Proper personal protection was used according to each chemical hazard class, and all work was performed in the chemical fume hood.

Color measurement

One hundred almond kernels were randomly selected from the LR samples for color analysis to correlate with the appearance attribute in the descriptive analysis. Individual almonds were sliced into identical halves using a razor blade and the color of the nutmeat was measured on one half using a ColorFlex colorimeter (HunterLab, Reston, VA, USA), with color values reported in L^* , a^* , and b^* according to the CIE Lab color scale. The port size was 0.5 in. (13 mm) with standard D65 illuminant at 10° observer angle.

Headspace volatiles analysis

Headspace volatile analysis was adapted from the method of Franklin *et al.* (2017).¹⁶ Twenty grams of almonds were ground with a Waring laboratory grinder and sieved with a size 20 Tyler sieve. An aliquot of 5 ± 0.02 g of the sieved material was weighed into a 20 mL amber headspace vial. Vials were capped and crimped immediately, then equilibrated for at least 4 h at room temperature ($23 \pm 2^\circ\text{C}$) prior to headspace sampling. An external instrument standard was analyzed in duplicate during each day to account for possible fiber and instrument changes. The external instrument standard was prepared by the same procedure as a sample but using de-volatilized almonds instead.⁹ After weighing

the de-volatilized almonds into a 20 mL headspace vial, a 400 μL vial insert containing a 0.5 μL glass capillary filled with methylpyrazine- d_6 , hexanol- d_{13} , and octanal- d_{16} in methanol, each at a concentration of 1000 $\mu\text{g mL}^{-1}$, and placed into the vial. The headspace vial was capped immediately, incubated for 4 h, and analyzed. A response factor to correct for instrument and fiber variation was calculated according to Franklin *et al.* (2017).¹⁶

The volatiles were analyzed using an Agilent 7890A gas chromatograph equipped with a GC injector 80 (Agilent Technologies, Santa Clara, CA, USA). Samples were equilibrated at 35 °C for 45 min with agitation at 400 rpm. The volatiles were extracted with a 1 cm 30/50 μm StableFlex (Supelco Inc., Bellefonte, PA, USA) divinylbenzene/carboxen/polydimethylsiloxane fiber for 45 min with agitation at 250 rpm. The fiber was desorbed using splitless injection at 250 °C. At 0.9 min the split vent opened at 50:1 ratio for a total injection time of 10 min. The fiber was cleaned in a helium-flushed needle heater for 5 min to prevent carryover. The headspace volatiles were separated using a 30 m \times 0.25 mm \times 0.25 μm DB-Wax Ultra Inert column (Agilent Technologies) at a flowrate of 1.2 mL min^{-1} . The oven program was set at 35 °C for 1 min followed by a ramp of 3 °C min^{-1} to 65 °C, followed by another ramp of 6 °C min^{-1} to 180 °C, and finally 30 °C min^{-1} to 250 °C with a 5 min hold. The mass spectra were collected using an Agilent 5975C MSD with 230 °C source temperature and 150 °C quadrupole temperature. The volatile profiles were collected scanning the range of 30–300 m/z . Tentative volatile identification was performed using the 2017 National Institute of Standards and Technology (NIST) Mass Spectral Search Program. Identification was confirmed using a retention index calculation or authentic standards when available. Relative concentrations of the headspace volatiles were calculated following the procedure described by Franklin *et al.* (2017).¹⁶

Quantitative descriptive analysis

Ten trained, experienced assessors employed by The National Food Lab, Inc. (Livermore, CA, USA) performed the descriptive analysis. Panelists participated in a 2 h orientation session to discuss the samples, develop the ballot, and review the references. The final ballot contained one appearance attribute, four aroma attributes, seven flavor attributes and nine texture attributes listed in Table S1.²² Three evaluations (replicates) were obtained from each panelist per sample with a total of 30 evaluations obtained for each sample. Almonds (57 g) were served in an 85 g opaque soufflé cup with lids coded with a random three-digit code. Panelists evaluated only LR almonds to minimize bias that can occur from advanced lipid oxidation of DR almonds. Panelists evaluated ten test samples per 2 h evaluation session, each served along with a labeled control sample (NME-LR, 0 month). Panelists used a 15-point degree-of-difference scale to indicate how different each test sample was from the control sample on an overall basis. Panelists also used 15-point intensity scales to indicate the intensity of key sensory attributes for each sample. Samples were assessed in a monadic-sequential order.

Consumer testing analysis

One hundred untrained consumers between the ages of 18 and 65, who were not pregnant, were recruited in the city of Davis, California, for hedonic testing. Consumers were served five pairs of LR almond samples, which were evaluated in descriptive analysis; each pair comprised one ME sample and one NME sample at the same amount of accelerated storage. Each sample contained six or seven almond kernels, at room temperature, identified with

randomly generated three-digit codes. Consumers were instructed to taste at least two almonds at a time and indicate their liking on a nine-point hedonic scale. After tasting both samples within a pair separately, the consumer was asked to choose a preferred sample within the pair. Consumers tasted the samples in a random and balanced order both among and within the pairs to minimize order effects. Verbal and written instructions were given to the participants, along with a tray of samples, a paper ballot, a bottle of water, an expectoration cup, and unsalted crackers for palate cleansing.

Statistical analysis

Calculated concentrations are reported as means \pm standard deviation of triplicate measurements. A two-way analysis of variance (ANOVA) with interactions, evaluating moisture exposure and sample age as main effects was performed ($P < 0.05$). When main effects were found, *post hoc* comparisons using Tukey's HSD test were applied. Binomial testing was performed on the paired preference data. Discriminant analysis and multiple factor analysis were performed as multivariate analysis using XLSTAT statistical and data analysis solution (version 2019.3.1). Data were centered prior to processing. Hierarchical clustering was performed using JMP [®] (version 14.3.0. SAS Institute Inc., Cary, NC, USA).

RESULTS AND DISCUSSION

Almonds are stored in the field in stockpiles for longer periods of time due to increased harvests and limited processing facilities. In-field storage increases the probability of post-harvest moisture exposure. Pre-processing drying is increasingly used prior to hulling and shelling without an understanding of how this practice can influence product shelf life and quality. However, previous studies in unroasted almonds demonstrate that post-harvest drying >65 °C can increase lipid oxidation and has the potential to shorten product shelf life once it is roasted. To address this, PVs, FFAs, CDs, and headspace volatiles were measured in almonds exposed to 8% moisture and subsequently dried to 4% moisture then roasted to a commercial light or dark roast (LR and DR, respectively). Roasted almonds were stored under conditions known to promote lipid oxidation and rancidity development over a 12-month period. Chemical data were correlated with sensory data (descriptive analysis and hedonic testing) to better understand the impact this has on consumer liking and acceptance.

Markers of primary oxidation in roasted almonds

The amount of FFAs, reported as a percentage of oleic acid, reflects the hydrolytic rancidity due to enzymatic or spontaneous hydrolysis of triglycerides.^{16, 23} Free fatty acids are more vulnerable to lipid oxidation than fatty acids esterified to glycerol.²³ Industry guidelines suggest any product with an FFA value $>1.5\%$ is at risk for rancidity development.¹⁵ Here, the FFA levels did not exceed 1.0% oleic over the 12 months of storage similar to other studies^{16, 24} as roasting destroys enzymes responsible for the hydrolysis of FFAs (Table 1). At 12 months of storage, ME-DR almonds ($0.61 \pm 0.02\%$ oleic acid) had significantly higher ($P < 0.05$) FFAs than the NME-DR almonds ($0.40 \pm 0.00\%$ oleic acid) (Table 1). However, there were no significant differences between the FFA values of ME-LR and NME-LR almonds at 12 months of storage (Table 1).

Table 1 Average value of chemical analyses of light roast (LR) and dark roast (DR) almonds during 12 months of accelerated storage of almonds exposed to moisture and subsequently dried (ME) and almonds with no moisture exposure (NME)[†]

Storage Month	Treatment	Free fatty acids (% oleic acid)		Peroxide value (mEq kg ⁻¹)		Conjugated dienes (%)		CIE L* value
		LR	DR	LR	DR	LR	DR	
0	NME	0.10 ± 0.02 ^{ijk}	0.09 ± 0.00 ^q	Not detected	Not detected	0.19 ± 0.00 ^{hi}	0.18 ± 0.00 ^{op}	78.63 ± 4.01
	ME	0.09 ± 0.00 ^k	0.10 ± 0.02 ^{nopq}	Not detected	Not detected	0.18 ± 0.00 ^j	0.15 ± 0.01 ^p	77.04 ± 5.34
1	NME	0.09 ± 0.00 ^{jk}	0.09 ± 0.01 ^{opq}	0.34 ± 0.11 ^{fg}	0.47 ± 0.19 ^m	0.18 ± 0.00 ^j	0.17 ± 0.00 ^{op}	79.23 ± 3.16
	ME	0.09 ± 0.00 ^k	0.10 ± 0.00 ^{nopq}	0.96 ± 0.11 ^b	0.81 ± 0.05 ^m	0.16 ± 0.00 ^j	0.18 ± 0.00 ^o	77.68 ± 4.43
2	NME	0.09 ± 0.00 ^{jk}	0.09 ± 0.01 ^q	0.21 ± 0.05 ^g	0.64 ± 0.00 ^m	0.19 ± 0.01 ^{hi}	0.21 ± 0.01 ⁿ	80.19 ± 3.05
	ME	0.10 ± 0.00 ^{hijk}	0.10 ± 0.01 ^{nopq}	0.46 ± 0.00 ^{defg}	2.17 ± 0.11 ^l	0.18 ± 0.00 ^j	0.24 ± 0.01 ^{mn}	79.58 ± 3.53
3	NME	0.10 ± 0.02 ^{hijk}	0.12 ± 0.00 ^{mno}	0.50 ± 0.00 ^{defg}	2.36 ± 0.12 ^l	0.18 ± 0.01 ^{hij}	0.27 ± 0.01 ^m	79.52 ± 2.84
	ME	0.12 ± 0.00 ^{efghij}	0.12 ± 0.01 ^{mn}	0.71 ± 0.18 ^{bcd}	4.16 ± 0.11 ^{ij}	0.19 ± 0.00 ^{ghi}	0.30 ± 0.01 ^l	78.22 ± 4.35
4	NME	0.11 ± 0.00 ^{ghijk}	0.09 ± 0.01 ^{pq}	0.70 ± 0.00 ^{bcd}	3.08 ± 0.19 ^{kl}	0.26 ± 0.02 ^{bc}	0.30 ± 0.03 ^{kl}	78.44 ± 3.06
	ME	0.11 ± 0.01 ^{hijk}	0.12 ± 0.01 ^{mno}	0.53 ± 0.06 ^{cdef}	4.09 ± 0.00 ^{ijk}	0.21 ± 0.00 ^{efg}	0.32 ± 0.00 ^{jk}	77.77 ± 3.84
5	NME	0.11 ± 0.00 ^{fghijk}	0.11 ± 0.00 ^{nop}	0.53 ± 0.06 ^{cdef}	4.95 ± 0.08 ⁱ	0.20 ± 0.00 ^{fgh}	0.35 ± 0.01 ^j	79.74 ± 3.23
	ME	0.14 ± 0.01 ^{bcdefg}	0.16 ± 0.01 ^{kl}	0.53 ± 0.06 ^{cdef}	11.95 ± 0.03 ^f	0.23 ± 0.01 ^{de}	0.45 ± 0.01 ^h	78.64 ± 4.03
6	NME	0.12 ± 0.00 ^{defghi}	0.14 ± 0.01 ^{lm}	0.64 ± 0.08 ^{cde}	3.68 ± 0.01 ^{jk}	0.22 ± 0.00 ^{def}	0.34 ± 0.00 ^j	79.80 ± 2.90
	ME	0.15 ± 0.00 ^{abcd}	0.16 ± 0.01 ^{kl}	0.56 ± 0.06 ^{cdef}	6.08 ± 0.30 ^h	0.21 ± 0.00 ^{defg}	0.40 ± 0.01 ⁱ	77.97 ± 3.62
7	NME	0.12 ± 0.00 ^{efghij}	0.17 ± 0.00 ^{jk}	0.80 ± 0.06 ^{bc}	10.09 ± 0.22 ^g	0.26 ± 0.01 ^c	0.47 ± 0.01 ^{gh}	79.61 ± 3.34
	ME	0.14 ± 0.00 ^{bcdef}	0.20 ± 0.00 ^{hi}	0.67 ± 0.00 ^{bcd}	12.71 ± 0.52 ^f	0.23 ± 0.01 ^d	0.48 ± 0.01 ^g	78.28 ± 4.27
8	NME	0.14 ± 0.00 ^{bcdef}	0.19 ± 0.01 ^{ij}	0.74 ± 0.19 ^{bcd}	12.14 ± 0.09 ^f	0.23 ± 0.01 ^{de}	0.54 ± 0.00 ^{ef}	79.33 ± 3.71
	ME	0.15 ± 0.01 ^{abc}	0.24 ± 0.01 ^f	1.41 ± 0.10 ^a	17.83 ± 0.17 ^d	0.26 ± 0.00 ^{bc}	0.56 ± 0.00 ^e	78.04 ± 3.91
9	NME	0.15 ± 0.00 ^{bcde}	0.22 ± 0.01 ^{gh}	0.68 ± 0.00 ^{bcd}	12.35 ± 0.07 ^f	0.25 ± 0.01 ^c	0.53 ± 0.00 ^f	78.73 ± 3.56
	ME	0.17 ± 0.01 ^{ab}	0.28 ± 0.01 ^e	0.62 ± 0.12 ^{cdef}	16.00 ± 0.34 ^e	0.26 ± 0.01 ^c	0.54 ± 0.01 ^{ef}	77.01 ± 3.77
10	NME	0.13 ± 0.01 ^{defgh}	0.23 ± 0.01 ^{fg}	0.50 ± 0.20 ^{defg}	12.00 ± 0.19 ^f	0.27 ± 0.00 ^{bc}	0.54 ± 0.00 ^{ef}	77.56 ± 3.29
	ME	0.16 ± 0.01 ^{ab}	0.31 ± 0.00 ^d	0.56 ± 0.11 ^{cdef}	16.06 ± 0.16 ^e	0.28 ± 0.00 ^b	0.60 ± 0.01 ^d	77.85 ± 3.43
11	NME	0.15 ± 0.00 ^{abc}	0.23 ± 0.00 ^{fg}	0.46 ± 0.06 ^{defg}	9.43 ± 0.01 ^g	0.25 ± 0.00 ^c	0.47 ± 0.01 ^{gh}	80.02 ± 3.32
	ME	0.18 ± 0.01 ^a	0.46 ± 0.01 ^b	0.58 ± 0.11 ^{cdef}	27.97 ± 0.82 ^b	0.28 ± 0.00 ^b	0.68 ± 0.00 ^c	78.25 ± 4.00
12	NME	0.16 ± 0.03 ^{ab}	0.40 ± 0.00 ^c	0.60 ± 0.14 ^{cdef}	24.48 ± 0.27 ^c	0.32 ± 0.00 ^a	0.72 ± 0.01 ^b	80.41 ± 2.85
	ME	0.16 ± 0.02 ^{ab}	0.61 ± 0.02 ^a	0.35 ± 0.05 ^{efg}	44.46 ± 1.12 ^a	0.32 ± 0.00 ^a	0.91 ± 0.00 ^a	78.53 ± 3.25

[†]Letters shared within the same column indicates there are no significant differences ($P < 0.05$) using ANOVA.

Peroxide value (PV) is commonly used as a rancidity indicator in almonds and most processors use a value of $PV < 5 \text{ mEq kg}^{-1}$ oil to ensure that kernels have not undergone significant oxidation.¹⁵ Here, PV levels were below the limit of detection at time 0 for all samples (Table 1). At 1 month, the PV levels in the ME-LR almonds ($0.96 \pm 0.11 \text{ mEq kg}^{-1}$) were significantly higher than the NME-LR ($0.34 \pm 0.11 \text{ mEq kg}^{-1}$). Starting at 2 months, PV levels were significantly higher in the ME-DR almonds as compared to the NME-DR almonds. The PV in LR almonds reached a maximum between 6–8 months for both NME ($0.80 \pm 0.06 \text{ mEq kg}^{-1}$) and ME almonds ($1.41 \pm 0.10 \text{ mEq kg}^{-1}$) (Table 1). This result is similar to the results of Franklin *et al.* (2017)⁹ in accelerated storage studies of Nonpareil almonds. The PV in DR almonds increased throughout storage and was significantly higher at 12 months in the ME-DR samples ($44.46 \pm 1.12 \text{ mEq kg}^{-1}$) as compared with the NME-DR ($24.48 \pm 0.27 \text{ mEq kg}^{-1}$) samples. Overall, ME almonds have significantly ($P < 0.05$) higher PV value than NME almonds for both LR and DR almonds. However, Tukey's post-hoc analysis indicated that most values were not significantly different between NME-LR and ME-LR almonds, whereas the ME-DR almonds were significantly higher than NME-DR after 5 months of storage. The PVs in all LR almonds remained below 5 mEq kg^{-1} throughout the 12 months of storage, whereas levels exceeded 5 mEq kg^{-1} at 7 months in the NME-DR almonds and at 5 months in the ME-DR almonds, indicating that these products would have a shorter shelf life.

Levels of CDs are not currently used as a quality marker in almonds and no industry standards exist; however, levels have been reported to correlate significantly with consumer acceptance of roasted almond products.⁹ Over 12 months of storage, CD levels in NME-LR and ME-LR increased by 68% and 78% respectively. However, at 12 months of storage, there was no significant difference ($P > 0.05$) between NME-LR and ME-LR almonds. In contrast, the ME-DR almonds showed significantly higher CD values ($P < 0.05$) than NME-DR at 12 months of storage (Table 1). The levels of CD in the ME-DR almonds were significantly higher after 4 months of storage.

These results indicate that post-harvest moisture exposure increases fatty acid oxidation and that the high temperature roasting amplifies this effect with respect to low-temperature roasting.

Headspace volatiles

Headspace volatiles are linked to sensory attributes and can be used to evaluate almond quality.¹⁸ A total of 69 volatiles were identified in the headspace of all roasted almonds and 34 were confirmed with authentic standards (Table S2). The remaining 35 volatiles were tentatively identified by comparing the MS spectra with the NIST 17 library and Kovats' retention indices with literature values listed in NIST Chemistry WebBook under comparable conditions.²⁵ Among the 69 volatiles identified, only 46 were significantly different between NME-LR and ME-LR almonds whereas

Table 2 Average volatile concentration ($\mu\text{g kg}^{-1}$ almond) measured in light roasted (LR) almond headspace at month 0, 1, 3, 5, 7, 9, and 11 months of storage of almonds exposed to moisture and subsequently dried (ME) and almonds with no moisture exposure (NME)

Months	0	1	3	5	7	9	11					
Treatment	NME	ME	NME	ME	NME	ME	NME					
3-Methyl-butanol	471.19 ± 65.63	495.51 ± 81.71	300.33 ± 35.69	306.86 ± 47.35	141.91 ± 6.66	115.02 ± 10.62	53.03 ± 4.03	45.18 ± 6.93	45.81 ± 5.28	35.60 ± 4.42	30.59 ± 3.01	26.84 ± 3.56
2-Methyl-butanol	508.72 ± 71.23	532.08 ± 91.00	303.02 ± 34.48	311.66 ± 48.76	131.70 ± 9.09	108.33 ± 10.13	50.18 ± 2.55	58.44 ± 7.99	38.29 ± 4.83	39.71 ± 14.76	28.21 ± 8.70	35.32 ± 17.31
2,4,6-Pentamethyl-heptane	10.81 ± 1.71	2.67 ± 0.34	2.47 ± 0.96	1.17 ± 0.53	6.77 ± 1.52	3.56 ± 1.19	3.37 ± 0.86	3.50 ± 0.68	5.18 ± 1.07	13.36 ± 2.22	14.86 ± 3.20	4.93 ± 1.29
Pentanal	26.47 ± 4.23	32.52 ± 4.49	76.03 ± 11.72	132.81 ± 38.82	519.75 ± 53.55	444.72 ± 61.55	454.96 ± 41.43	438.68 ± 52.56	552.09 ± 69.15	582.04 ± 69.88	416.43 ± 55.26	423.27 ± 53.03
Decane	54.13 ± 3.97	24.49 ± 6.68	15.66 ± 4.87	9.96 ± 4.81	37.48 ± 8.69	23.21 ± 8.72	18.60 ± 4.84	20.16 ± 5.55	22.55 ± 4.56	37.04 ± 13.16	11.68 ± 36.97	41.99 ± 14.47
2-Propyl-furan	0.99 ± 0.13	0.54 ± 0.12	0.84 ± 0.09	0.73 ± 0.18	3.08 ± 0.47	2.37 ± 0.46	3.43 ± 0.31	3.35 ± 0.73	4.95 ± 0.70	5.22 ± 0.78	4.17 ± 0.76	3.59 ± 0.60
Dimethyl disulfide*	1.08 ± 0.12	1.54 ± 0.22	3.73 ± 0.15	3.00 ± 0.80	2.96 ± 0.33	1.96 ± 0.34	1.18 ± 0.11	1.03 ± 0.16	1.08 ± 0.13	0.62 ± 0.10	0.57 ± 0.10	0.27 ± 0.06
Hexanal	43.59 ± 0.85	35.75 ± 2.67	133.60 ± 8.09	207.70 ± 53.08	993.06 ± 101.04	850.37 ± 136.41	968.76 ± 116.51	965.21 ± 139.20	1267.91 ± 118.17	1339.29 ± 139.68	1037.44 ± 125.16	1006.33 ± 135.69
2-Methyl-1-propanol	2.25 ± 0.20	2.07 ± 0.23	3.87 ± 0.34	3.20 ± 0.70	2.54 ± 0.18	2.31 ± 0.29	1.18 ± 0.11	1.20 ± 0.14	1.05 ± 0.10	0.83 ± 0.12	0.55 ± 0.08	0.95 ± 0.13
2-n-Butyl furan	0.45 ± 0.04	0.45 ± 0.08	1.16 ± 0.13	1.45 ± 0.34	8.96 ± 1.14	8.27 ± 1.49	11.99 ± 1.54	12.09 ± 1.71	19.36 ± 1.52	21.57 ± 2.61	18.59 ± 2.39	17.95 ± 3.18
Pentyl-oxirane*	0.05 ± 0.02	0.03 ± 0.01	0.37 ± 0.05	0.90 ± 0.23	4.39 ± 0.45	4.78 ± 0.92	6.16 ± 0.82	6.51 ± 0.86	13.12 ± 1.14	14.79 ± 1.63	14.96 ± 1.71	15.09 ± 2.46
1-Butanol	5.16 ± 0.34	4.48 ± 0.71	4.85 ± 0.34	5.26 ± 1.01	8.96 ± 0.77	8.83 ± 0.78	8.35 ± 0.79	8.77 ± 0.61	12.15 ± 1.00	12.70 ± 1.09	10.03 ± 1.01	11.08 ± 0.95
2-Heptanone*	5.85 ± 0.55	6.13 ± 0.87	9.03 ± 1.13	12.38 ± 3.02	69.87 ± 7.04	61.34 ± 10.43	115.51 ± 15.90	118.38 ± 17.23	270.42 ± 24.75	311.44 ± 35.38	338.89 ± 58.20	334.02 ± 41.55
Heptanal	6.70 ± 0.73	6.25 ± 0.44	15.22 ± 2.20	21.31 ± 3.42	109.42 ± 3.48	103.38 ± 12.26	163.28 ± 20.37	171.89 ± 21.65	331.84 ± 25.43	387.30 ± 35.63	368.96 ± 34.31	337.24 ± 33.56
o-Limonene	0.35 ± 0.04	0.37 ± 0.04	0.29 ± 0.07	0.22 ± 0.04	0.40 ± 0.03	0.45 ± 0.00	0.22 ± 0.03	0.21 ± 0.01	0.36 ± 0.03	0.33 ± 0.05	0.41 ± 0.06	0.41 ± 0.02
2-Methyl-1-butanol	53.87 ± 4.10	55.74 ± 7.65	118.39 ± 11.17	99.44 ± 21.93	121.55 ± 9.64	99.24 ± 10.31	57.57 ± 4.18	54.06 ± 5.47	60.92 ± 4.06	47.80 ± 4.59	36.68 ± 3.95	42.88 ± 5.22
3-Methyl-1-butanol	49.48 ± 4.52	55.90 ± 8.38	85.58 ± 6.24	76.39 ± 16.25	84.55 ± 6.94	76.01 ± 8.08	41.61 ± 3.25	40.20 ± 4.19	45.32 ± 3.10	36.51 ± 3.12	28.11 ± 2.80	19.39 ± 1.56
2-Pentyl-furan*	3.85 ± 0.23	3.75 ± 0.43	6.87 ± 1.42	8.57 ± 1.23	41.86 ± 2.22	37.96 ± 4.29	50.74 ± 4.95	51.17 ± 4.68	84.85 ± 5.40	95.54 ± 7.14	87.05 ± 5.09	89.81 ± 10.29
Styrene	0.93 ± 0.07	1.12 ± 0.14	1.21 ± 0.14	1.26 ± 0.16	5.03 ± 0.54	4.00 ± 0.65	4.91 ± 0.65	4.64 ± 0.67	10.76 ± 0.88	11.02 ± 1.34	14.62 ± 1.66	14.51 ± 2.61
1-Pentanol*	28.71 ± 2.84	24.61 ± 3.98	31.30 ± 2.18	44.38 ± 10.75	124.25 ± 12.43	130.45 ± 17.98	142.24 ± 13.53	149.34 ± 16.69	230.87 ± 15.08	244.27 ± 18.01	213.60 ± 20.06	218.67 ± 24.57
Methyl-pyrazine	8.50 ± 0.92	8.77 ± 2.01	4.31 ± 0.39	4.70 ± 0.61	6.19 ± 0.94	5.51 ± 1.19	3.91 ± 0.55	4.83 ± 0.74	4.93 ± 0.51	4.72 ± 0.67	4.11 ± 0.53	4.03 ± 0.71
Acetoin	74.40 ± 5.32	84.64 ± 8.08	30.17 ± 1.06	27.89 ± 1.55	15.94 ± 0.70	12.65 ± 0.78	9.08 ± 0.30	10.34 ± 0.56	9.55 ± 0.10	8.07 ± 0.15	7.36 ± 0.31	5.75 ± 0.15
2-Octanone*	0.46 ± 0.10	1.24 ± 0.22	0.77 ± 0.20	1.72 ± 0.26	5.83 ± 0.39	6.55 ± 0.56	11.06 ± 1.13	12.54 ± 1.18	30.65 ± 1.15	39.16 ± 2.45	44.69 ± 2.40	52.62 ± 6.44
Octanal*	1.69 ± 0.27	1.95 ± 0.32	5.95 ± 1.43	11.09 ± 1.46	67.18 ± 3.02	71.72 ± 5.63	120.14 ± 10.43	133.89 ± 9.71	260.48 ± 11.97	341.24 ± 17.77	320.89 ± 12.30	381.44 ± 34.26
1-Hydroxy-2-Propanone*	20.35 ± 0.71	28.69 ± 1.69	9.04 ± 0.64	9.43 ± 1.02	4.75 ± 0.13	5.50 ± 0.06	4.98 ± 0.30	5.46 ± 0.25	4.83 ± 0.52	5.39 ± 0.63	4.45 ± 0.62	4.05 ± 0.44
1-Chloro-2-propanol*	577.17 ± 44.82	438.92 ± 53.95	398.18 ± 39.68	236.02 ± 33.83	236.82 ± 15.41	166.77 ± 15.11	113.63 ± 3.73	87.72 ± 3.80	94.46 ± 3.61	85.36 ± 5.17	61.58 ± 2.84	72.48 ± 5.93
2,5-Dimethyl-pyrazine	13.47 ± 1.35	14.49 ± 2.68	8.10 ± 1.14	9.87 ± 1.02	14.96 ± 1.30	12.69 ± 2.05	9.50 ± 1.01	11.59 ± 1.36	13.60 ± 0.91	12.94 ± 1.23	12.19 ± 0.87	11.43 ± 1.70
Methylthio-2-propanone*	7.76 ± 0.45	9.08 ± 1.81	13.43 ± 1.81	9.33 ± 1.78	16.07 ± 1.88	7.16 ± 1.42	7.10 ± 0.84	4.49 ± 0.60	5.00 ± 0.33	2.54 ± 0.40	2.26 ± 0.20	1.64 ± 0.26
1-Hexanol	83.72 ± 5.72	70.92 ± 10.64	113.05 ± 14.29	126.98 ± 21.94	350.76 ± 25.35	449.04 ± 53.28	379.69 ± 30.11	353.30 ± 27.31	670.45 ± 17.78	560.57 ± 36.94	606.64 ± 30.08	486.75 ± 33.62
2-Chloro-1-propanol*	3.70 ± 0.29	3.33 ± 0.37	2.67 ± 0.22	1.84 ± 0.27	1.65 ± 0.11	1.21 ± 0.14	0.89 ± 0.04	0.73 ± 0.04	0.71 ± 0.04	0.68 ± 0.04	0.52 ± 0.06	0.63 ± 0.02
2-Ethyl-6-methyl-pyrazine*	0.46 ± 0.04	0.53 ± 0.07	0.32 ± 0.02	0.48 ± 0.01	0.63 ± 0.03	0.59 ± 0.05	0.47 ± 0.04	0.58 ± 0.02	0.63 ± 0.03	0.68 ± 0.05	0.66 ± 0.03	0.60 ± 0.07
2-Nonanone*	0.33 ± 0.02	0.44 ± 0.10	0.32 ± 0.10	0.49 ± 0.08	2.29 ± 0.17	2.68 ± 0.24	5.72 ± 0.38	6.81 ± 0.40	18.98 ± 0.62	28.53 ± 1.19	34.08 ± 1.43	48.94 ± 2.73
Nonanal*	30.24 ± 7.93	29.21 ± 6.47	56.98 ± 6.78	38.36 ± 5.92	124.21 ± 25.88	137.76 ± 29.32	124.98 ± 2.25	148.59 ± 3.77	247.35 ± 8.15	361.85 ± 12.92	311.27 ± 5.03	410.95 ± 14.36
Trimethyl-pyrazine	1.48 ± 0.06	1.64 ± 0.18	1.02 ± 0.18	1.40 ± 0.07	1.98 ± 0.12	1.69 ± 0.17	1.36 ± 0.11	1.70 ± 0.09	2.01 ± 0.08	2.05 ± 0.13	2.00 ± 0.07	1.87 ± 0.17
3-Octen-2-one*	0.52 ± 0.01	0.26 ± 0.02	1.24 ± 0.38	1.92 ± 0.25	14.19 ± 0.73	16.28 ± 1.47	22.08 ± 1.93	24.56 ± 1.65	46.96 ± 1.91	59.93 ± 3.07	56.94 ± 1.76	65.46 ± 5.28
3-Ethyl-2-methyl-1,3-hexadiene*	1.04 ± 0.01	0.99 ± 0.08	2.10 ± 0.45	3.13 ± 0.41	14.54 ± 0.85	15.06 ± 1.29	19.07 ± 1.38	21.47 ± 1.31	34.61 ± 1.55	47.27 ± 1.67	40.01 ± 1.69	47.71 ± 4.34
(E)-2-Octenal*	0.36 ± 0.01	0.45 ± 0.03	1.52 ± 0.47	2.95 ± 0.30	12.70 ± 0.54	15.60 ± 1.21	12.42 ± 0.91	14.07 ± 1.00	16.06 ± 0.50	29.18 ± 1.09	19.40 ± 0.51	30.84 ± 2.36
3-Ethyl-2,5-dimethyl-pyrazine*	0.12 ± 0.01	0.14 ± 0.01	0.08 ± 0.02	0.12 ± 0.01	0.14 ± 0.00	0.13 ± 0.01	0.10 ± 0.00	0.13 ± 0.00	0.14 ± 0.01	0.15 ± 0.01	0.14 ± 0.00	0.13 ± 0.01
2,6-Diethyl-pyrazine*	1.44 ± 0.06	1.76 ± 0.16	1.10 ± 0.19	1.73 ± 0.07	1.97 ± 0.06	1.77 ± 0.14	1.33 ± 0.06	1.84 ± 0.08	1.97 ± 0.05	2.11 ± 0.10	2.06 ± 0.04	2.04 ± 0.14
Acetic acid*	14.85 ± 0.94	45.13 ± 2.93	10.81 ± 0.88	19.01 ± 4.43	7.68 ± 0.52	20.56 ± 1.03	17.53 ± 1.92	36.42 ± 2.06	24.95 ± 2.91	48.93 ± 5.54	28.06 ± 2.88	42.89 ± 2.11
1-Octen-3-ol*	4.22 ± 0.36	3.07 ± 0.13	3.11 ± 0.49	3.19 ± 0.33	7.51 ± 0.34	8.16 ± 0.69	8.38 ± 0.54	11.05 ± 0.25	17.77 ± 0.69	28.10 ± 1.59	27.06 ± 0.13	34.50 ± 2.48
Furfural*	39.23 ± 3.37	36.35 ± 5.01	16.78 ± 0.89	14.00 ± 1.65	12.24 ± 1.31	9.32 ± 1.38	8.56 ± 0.57	8.67 ± 0.46	10.44 ± 0.36	10.52 ± 0.62	9.88 ± 0.25	8.75 ± 0.51
1-Heptanol*	2.22 ± 0.03	2.51 ± 0.24	3.14 ± 0.70	6.33 ± 0.83	16.90 ± 0.70	24.97 ± 2.06	28.89 ± 1.93	37.25 ± 2.02	64.42 ± 2.22	92.21 ± 3.82	83.77 ± 2.06	111.93 ± 7.56
2-Decanone*	0.17 ± 0.02	0.17 ± 0.02	0.15 ± 0.04	0.20 ± 0.06	0.61 ± 0.04	0.76 ± 0.11	1.30 ± 0.06	1.72 ± 0.07	3.88 ± 0.12	7.15 ± 0.39	8.11 ± 0.30	13.90 ± 0.39
Decanal*	0.58 ± 0.12	0.70 ± 0.19	0.62 ± 0.16	0.79 ± 0.05	2.67 ± 0.22	3.89 ± 0.27	5.00 ± 0.22	6.32 ± 0.10	10.61 ± 0.15	19.47 ± 1.08	16.24 ± 0.81	26.53 ± 0.26
trans-3-Nonen-2-one*	0.08 ± 0.01	0.07 ± 0.01	0.08 ± 0.03	0.10 ± 0.02	0.53 ± 0.04	0.67 ± 0.12	0.80 ± 0.06	0.94 ± 0.16	2.03 ± 0.11	3.16 ± 0.14	3.16 ± 0.18	4.59 ± 0.19
Pyroole	2.99 ± 0.25	3.44 ± 0.56	1.54 ± 0.04	1.60 ± 0.13	1.58 ± 0.08	1.22 ± 0.09	0.88 ± 0.08	1.09 ± 0.11	1.08 ± 0.06	0.99 ± 0.09	0.90 ± 0.08	0.69 ± 0.05
Benzaldehyde	8.87 ± 0.55	9.92 ± 1.10	8.79 ± 0.73	8.63 ± 0.49	15.60 ± 0.80	13.47 ± 1.39	12.64 ± 0.98	13.85 ± 0.95	20.06 ± 0.70	20.69 ± 1.36	22.19 ± 0.91	20.56 ± 2.04

Table 2. Continued

Treatment	0		1		3		5		7		9		11	
	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME
(E)-2-Nonenal*	0.49 ± 0.01	0.52 ± 0.07	0.34 ± 0.06	0.38 ± 0.05	0.79 ± 0.05	0.69 ± 0.09	0.82 ± 0.06	1.00 ± 0.05	1.21 ± 0.04	2.34 ± 0.08	1.85 ± 0.10	3.27 ± 0.11	2.52 ± 0.20	2.59 ± 0.34
2-Butyltetrahydro-furan*	0.03 ± 0.00	0.05 ± 0.01	0.60 ± 0.13	1.51 ± 0.29	5.05 ± 0.14	9.62 ± 0.52	4.75 ± 0.45	6.19 ± 0.50	4.85 ± 0.09	10.71 ± 0.42	4.80 ± 0.03	9.41 ± 0.73	4.43 ± 0.11	3.72 ± 0.60
1-Octanol*	0.60 ± 0.04	0.67 ± 0.02	0.78 ± 0.26	1.32 ± 0.13	3.41 ± 0.05	6.08 ± 0.43	5.73 ± 0.24	8.34 ± 0.42	13.51 ± 0.24	22.21 ± 0.74	19.11 ± 0.31	31.32 ± 1.05	26.16 ± 0.60	28.95 ± 1.40
2-Methyl-1H-pyrrole	0.22 ± 0.02	0.26 ± 0.04	0.11 ± 0.01	0.11 ± 0.01	0.09 ± 0.00	0.07 ± 0.00	0.06 ± 0.01	0.06 ± 0.01	0.08 ± 0.00	0.09 ± 0.00	0.05 ± 0.00	0.06 ± 0.00	0.06 ± 0.00	0.06 ± 0.01
Butyrolactone*	2.25 ± 0.18	3.11 ± 0.41	1.14 ± 0.12	1.30 ± 0.09	1.09 ± 0.05	1.67 ± 0.14	0.91 ± 0.02	1.51 ± 0.02	1.30 ± 0.02	1.67 ± 0.01	1.34 ± 0.04	1.45 ± 0.09	1.09 ± 0.07	1.52 ± 0.06
Benzeneacetaldehyde	6.223 ± 3.03	70.64 ± 6.45	10.33 ± 1.64	8.74 ± 0.20	3.80 ± 0.41	3.72 ± 0.22	3.27 ± 0.07	3.39 ± 0.07	3.46 ± 0.08	3.29 ± 0.15	3.52 ± 0.40	3.19 ± 0.19	2.35 ± 0.17	2.86 ± 0.21
(Z)-2-decenal*	0.16 ± 0.04	0.11 ± 0.03	0.15 ± 0.04	0.19 ± 0.03	0.44 ± 0.03	0.78 ± 0.08	0.60 ± 0.04	0.91 ± 0.08	0.71 ± 0.04	2.31 ± 0.21	1.10 ± 0.02	3.57 ± 0.16	1.25 ± 0.04	1.35 ± 0.22
2-Methyl- anhydride pentanoic acid*	0.17 ± 0.01	0.26 ± 0.02	0.26 ± 0.08	0.57 ± 0.05	1.96 ± 0.06	3.36 ± 0.36	3.96 ± 0.20	5.44 ± 0.30	6.57 ± 0.03	12.84 ± 0.51	8.90 ± 0.10	13.88 ± 0.66	9.67 ± 0.37	9.51 ± 1.00
2-Furanmethanol*	2.01 ± 0.12	3.02 ± 0.26	1.14 ± 0.13	1.40 ± 0.13	0.91 ± 0.02	0.92 ± 0.07	0.69 ± 0.01	1.09 ± 0.05	0.80 ± 0.02	0.98 ± 0.04	0.69 ± 0.02	0.68 ± 0.01	0.41 ± 0.03	0.46 ± 0.02
1-Nonanol*	0.26 ± 0.04	0.26 ± 0.03	0.29 ± 0.09	0.28 ± 0.02	0.63 ± 0.02	1.21 ± 0.07	0.87 ± 0.04	0.93 ± 0.10	1.89 ± 0.11	2.30 ± 0.18	2.19 ± 0.25	4.08 ± 0.03	3.12 ± 0.07	4.09 ± 0.15
3-Methyl-butanoic acid*	0.47 ± 0.05	1.00 ± 0.12	0.28 ± 0.04	0.52 ± 0.10	0.74 ± 0.07	0.91 ± 0.09	1.15 ± 0.04	1.85 ± 0.04	1.99 ± 0.07	2.30 ± 0.05	2.53 ± 0.04	3.24 ± 0.08	2.84 ± 0.07	2.60 ± 0.08
5-Ethylidihydro-2(3H)-Furanone*	2.09 ± 0.07	3.85 ± 0.28	2.80 ± 0.68	5.54 ± 0.53	19.53 ± 0.74	27.10 ± 1.80	34.09 ± 2.15	41.92 ± 1.67	72.69 ± 2.04	103.39 ± 3.32	98.00 ± 1.78	123.56 ± 6.88	123.72 ± 4.95	127.44 ± 8.45
Pentanoic acid*	0.12 ± 0.02	0.25 ± 0.03	0.20 ± 0.00	0.50 ± 0.10	2.78 ± 0.43	6.36 ± 0.17	10.39 ± 0.80	16.12 ± 0.84	28.68 ± 1.71	53.81 ± 0.64	50.47 ± 0.45	76.21 ± 4.53	72.75 ± 2.01	79.79 ± 7.37
Tetrahydro-6-methyl-2H-Pyran-2-one*	0.09 ± 0.00	0.09 ± 0.01	0.07 ± 0.01	0.08 ± 0.00	0.29 ± 0.01	0.24 ± 0.01	0.27 ± 0.01	0.34 ± 0.02	0.56 ± 0.01	0.89 ± 0.02	0.80 ± 0.00	1.13 ± 0.06	1.07 ± 0.03	1.15 ± 0.07
Dihydro-5-propyl-2(3H)-Furanone*	0.06 ± 0.01	0.18 ± 0.02	0.07 ± 0.02	0.16 ± 0.01	0.31 ± 0.01	0.50 ± 0.02	0.53 ± 0.02	0.75 ± 0.02	1.30 ± 0.03	2.15 ± 0.06	2.08 ± 0.02	3.24 ± 0.11	3.17 ± 0.05	3.38 ± 0.21
Hexanoic acid*	0.35 ± 0.04	0.64 ± 0.11	0.43 ± 0.07	0.85 ± 0.22	7.56 ± 1.97	26.81 ± 2.30	50.31 ± 4.87	85.16 ± 3.43	127.76 ± 3.51	301.10 ± 6.00	237.26 ± 4.36	420.50 ± 14.90	350.17 ± 13.76	393.04 ± 40.67
Benzyl alcohol*	0.44 ± 0.08	0.57 ± 0.03	0.40 ± 0.10	0.40 ± 0.02	0.43 ± 0.01	0.58 ± 0.03	0.37 ± 0.00	0.45 ± 0.02	0.52 ± 0.02	0.64 ± 0.01	0.58 ± 0.03	0.70 ± 0.02	0.51 ± 0.01	0.58 ± 0.01
5-Butylidihydro-2(3H)-Furanone*	0.04 ± 0.01	0.08 ± 0.00	0.07 ± 0.02	0.13 ± 0.02	0.44 ± 0.02	0.80 ± 0.04	0.86 ± 0.03	1.33 ± 0.08	1.89 ± 0.02	4.07 ± 0.22	3.50 ± 0.12	6.74 ± 0.16	5.43 ± 0.10	6.39 ± 0.38
Phenylethyl alcohol*	1.58 ± 0.05	2.28 ± 0.18	1.94 ± 0.40	1.73 ± 0.05	1.96 ± 0.07	2.54 ± 0.16	1.75 ± 0.04	2.18 ± 0.07	2.47 ± 0.06	2.40 ± 0.10	2.51 ± 0.11	3.35 ± 0.08	2.40 ± 0.01	2.80 ± 0.04
Heptanoic acid*	0.07 ± 0.01	0.10 ± 0.03	0.09 ± 0.03	0.10 ± 0.04	0.27 ± 0.07	0.43 ± 0.11	0.58 ± 0.05	1.02 ± 0.11	1.59 ± 0.12	4.85 ± 0.50	3.11 ± 0.05	9.32 ± 0.98	6.45 ± 0.36	7.74 ± 1.02
2-Vinylfuran*	0.18 ± 0.02	0.19 ± 0.03	0.25 ± 0.02	0.20 ± 0.02	0.22 ± 0.01	0.31 ± 0.01	0.22 ± 0.03	0.27 ± 0.01	0.29 ± 0.04	0.36 ± 0.02	0.35 ± 0.04	0.38 ± 0.03	0.30 ± 0.02	0.41 ± 0.04

*Significantly different between treatments across all time points ($P < 0.05$) using ANOVA. Bolded type indicates volatiles that have significant difference between treatments at 7 months of accelerated storage.

Table 3 Average volatile concentration ($\mu\text{g kg}^{-1}$ almond) measured in dark roasted (DR) almond headspace at months 0, 1, 3, 5, 7, 9, and 11 months of storage of almonds exposed to moisture and subsequently dried (ME) and almonds with no moisture exposure (NME)

Treatment	0		1		3		5		7		9		11	
	ME	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME	NME
3-Methyl-butanol	530.79 ± 90.12	576.14 ± 103.87	409.54 ± 42.72	530.23 ± 40.39	208.54 ± 19.08	167.02 ± 13.40	54.79 ± 7.64	64.86 ± 15.96	44.22 ± 1.79	45.79 ± 3.30	26.20 ± 2.01	22.42 ± 3.36	24.15 ± 4.46	22.40 ± 2.93
2-Methyl-butanol*	584.89 ± 107.50	640.85 ± 115.22	433.16 ± 47.71	572.45 ± 45.06	266.91 ± 27.33	214.95 ± 18.26	60.81 ± 10.94	71.15 ± 6.60	63.35 ± 12.08	65.95 ± 6.75	90.63 ± 26.53	78.22 ± 27.09	108.33 ± 21.78	205.12 ± 29.72
2,4,6-Pentamethyl-heptane	5.27 ± 0.67	3.32 ± 0.30	2.99 ± 1.35	6.68 ± 0.86	5.82 ± 0.19	5.29 ± 1.09	3.34 ± 1.03	2.86 ± 0.40	5.65 ± 1.66	4.92 ± 1.25	16.76 ± 3.55	10.46 ± 4.22	5.76 ± 1.95	5.53 ± 1.83
Pentanal*	47.05 ± 12.54	29.71 ± 5.59	115.96 ± 16.30	184.28 ± 16.10	599.47 ± 37.16	668.39 ± 38.62	65.51 ± 32.23	576.85 ± 42.34	939.32 ± 43.22	960.93 ± 19.14	826.49 ± 53.16	807.73 ± 13.94	983.50 ± 19.11	30.31 ± 9.77
Decane	34.72 ± 5.35	26.07 ± 3.73	18.47 ± 6.92	41.17 ± 8.06	29.61 ± 4.70	30.66 ± 5.73	14.63 ± 6.10	18.22 ± 4.60	35.31 ± 10.79	31.52 ± 6.43	118.59 ± 30.55	76.67 ± 30.82	38.33 ± 15.21	39.31 ± 9.77
2-Propyl-furan	0.39 ± 0.06	0.26 ± 0.04	0.64 ± 0.13	0.89 ± 0.09	2.41 ± 0.37	2.47 ± 0.37	2.21 ± 0.39	2.33 ± 0.16	1.68 ± 0.24	2.28 ± 0.37	2.45 ± 0.42	2.07 ± 0.26	1.90 ± 0.33	1.70 ± 0.25
Dimethyl disulfide	1.82 ± 0.19	2.42 ± 0.29	3.42 ± 0.49	4.80 ± 0.72	2.19 ± 0.33	1.53 ± 0.17	0.55 ± 0.09	0.55 ± 0.09	0.30 ± 0.04	0.34 ± 0.04	0.24 ± 0.03	0.19 ± 0.04	0.09 ± 0.03	0.06 ± 0.00
Hexanal*	65.05 ± 4.43	44.50 ± 2.39	180.73 ± 18.12	296.97 ± 16.59	1138.32 ± 86.21	1352.78 ± 59.07	1183.99 ± 90.70	1796.39 ± 61.50	1833.55 ± 27.58	1548.90 ± 26.92	1566.24 ± 50.76	1437.71 ± 18.49	1508.48 ± 8.26	1437.71 ± 18.49
2-Methyl-1-propanol	0.52 ± 0.04	0.52 ± 0.08	1.54 ± 0.09	1.82 ± 0.16	1.73 ± 0.34	1.31 ± 0.19	0.64 ± 0.15	0.71 ± 0.11	0.37 ± 0.03	0.34 ± 0.07	0.19 ± 0.04	0.20 ± 0.09	0.11 ± 0.01	0.08 ± 0.05
2-n-Butyl furan	0.43 ± 0.06	0.49 ± 0.09	1.35 ± 0.11	1.91 ± 0.08	7.97 ± 0.61	9.50 ± 1.12	9.92 ± 1.20	10.43 ± 1.22	8.99 ± 1.37	12.04 ± 1.37	15.15 ± 1.81	17.87 ± 1.51	13.28 ± 2.27	9.79 ± 1.08
Pentyl-oxirane*	0.07 ± 0.01	0.07 ± 0.02	0.43 ± 0.07	1.01 ± 0.04	5.15 ± 0.30	8.95 ± 1.97	13.31 ± 1.74	8.58 ± 0.94	38.52 ± 7.55	38.98 ± 2.97	45.00 ± 4.91	37.83 ± 9.89	35.93 ± 6.29	58.97 ± 4.28
1-Butanol*	4.00 ± 0.41	3.18 ± 0.30	4.33 ± 0.21	5.37 ± 0.06	8.97 ± 0.50	9.59 ± 0.28	9.66 ± 1.12	9.57 ± 0.44	11.42 ± 1.45	12.98 ± 1.42	13.46 ± 1.60	11.26 ± 0.85	10.06 ± 3.09	11.46 ± 3.01
2-Heptanone*	5.33 ± 0.41	4.62 ± 0.34	10.31 ± 1.22	15.22 ± 0.65	73.92 ± 5.01	110.14 ± 10.94	173.76 ± 21.75	154.89 ± 18.11	402.40 ± 75.28	468.18 ± 39.26	440.24 ± 57.23	545.06 ± 159.29	550.91 ± 92.47	969.09 ± 96.20
Heptanal*	8.38 ± 0.59	7.48 ± 1.12	20.39 ± 0.39	25.60 ± 0.98	117.32 ± 5.28	168.12 ± 10.79	234.56 ± 11.57	223.35 ± 18.57	456.64 ± 40.20	508.41 ± 18.84	426.51 ± 32.82	548.67 ± 120.19	566.42 ± 37.85	627.85 ± 3.00
D-limonene*	1.97 ± 0.09	3.23 ± 0.15	1.88 ± 0.12	2.87 ± 0.09	2.46 ± 0.04	2.89 ± 0.25	1.72 ± 0.07	1.81 ± 0.10	1.79 ± 0.18	1.92 ± 0.09	1.52 ± 0.04	1.48 ± 0.07	1.14 ± 0.05	0.94 ± 0.07
2-Methyl-1-butanol	11.49 ± 1.04	14.58 ± 2.09	44.88 ± 6.25	61.68 ± 3.49	77.43 ± 6.86	63.58 ± 3.42	33.36 ± 3.39	25.34 ± 4.72	26.21 ± 2.57	19.79 ± 2.94	15.75 ± 1.51	11.19 ± 1.57	9.80 ± 0.93	11.19 ± 1.57
3-Methyl-1-butanol	11.32 ± 0.97	16.52 ± 2.22	31.73 ± 4.69	45.30 ± 2.52	54.79 ± 4.81	46.42 ± 4.31	23.61 ± 2.71	25.32 ± 2.50	22.30 ± 4.17	23.27 ± 2.87	18.85 ± 2.79	15.77 ± 1.17	12.17 ± 1.83	14.18 ± 1.08
2-Pentyl-furan	4.26 ± 0.15	4.13 ± 0.12	7.80 ± 0.60	9.61 ± 0.35	32.85 ± 1.34	43.60 ± 3.62	47.36 ± 3.78	45.40 ± 3.26	52.28 ± 5.87	65.54 ± 5.41	81.98 ± 5.05	78.63 ± 15.26	96.43 ± 13.69	112.96 ± 10.38
Styrene	0.87 ± 0.06	1.17 ± 0.16	1.37 ± 0.19	1.78 ± 0.05	5.22 ± 0.38	5.66 ± 0.22	5.35 ± 0.56	5.37 ± 0.59	10.52 ± 2.05	11.59 ± 0.94	14.76 ± 1.94	18.21 ± 0.51	18.26 ± 3.07	17.27 ± 1.36
1-Pentanol*	20.89 ± 1.62	16.56 ± 1.97	28.31 ± 2.29	42.57 ± 2.64	134.15 ± 9.48	170.02 ± 13.65	182.77 ± 17.34	166.71 ± 13.13	256.90 ± 36.89	280.05 ± 20.10	253.72 ± 26.89	235.61 ± 26.38	205.86 ± 26.61	215.28 ± 17.26
Methyl-pyrazine	15.48 ± 1.50	12.76 ± 2.39	6.97 ± 2.41	15.15 ± 2.64	10.96 ± 1.07	10.40 ± 1.21	4.93 ± 0.50	4.32 ± 0.51	5.89 ± 0.99	5.17 ± 0.52	5.22 ± 0.65	3.46 ± 0.47	3.02 ± 0.63	2.79 ± 0.31
Acetoin*	114.75 ± 4.72	86.46 ± 7.91	32.89 ± 0.13	38.15 ± 1.37	18.61 ± 0.66	15.87 ± 0.66	7.79 ± 0.26	6.26 ± 0.46	6.41 ± 0.13	5.40 ± 0.22	4.13 ± 0.31	3.18 ± 0.27	2.64 ± 0.26	1.42 ± 0.32
2-Octanone*	0.37 ± 0.08	0.32 ± 0.07	1.02 ± 0.13	1.59 ± 0.24	5.77 ± 0.33	10.23 ± 1.07	17.33 ± 1.10	17.24 ± 1.19	53.17 ± 6.32	62.72 ± 2.44	64.73 ± 6.08	95.11 ± 29.80	106.69 ± 13.66	266.01 ± 11.37
Octanal*	2.16 ± 0.09	2.10 ± 0.49	8.37 ± 0.07	10.73 ± 0.36	66.27 ± 1.01	118.26 ± 6.55	177.94 ± 6.19	184.49 ± 9.45	397.84 ± 23.79	458.28 ± 12.65	396.54 ± 20.68	569.14 ± 154.43	578.26 ± 34.04	752.73 ± 11.05
1-Hydroxy-2-Propanone*	32.24 ± 0.88	38.82 ± 2.30	10.90 ± 0.22	12.60 ± 1.52	5.37 ± 0.10	8.85 ± 0.09	4.73 ± 0.30	3.33 ± 0.20	5.44 ± 0.59	4.33 ± 0.34	3.37 ± 0.55	2.83 ± 0.49	1.72 ± 0.29	2.04 ± 0.06
1-Chloro-2-propanol*	660.35 ± 41.18	392.46 ± 47.44	378.03 ± 14.68	274.44 ± 5.31	236.54 ± 10.83	158.88 ± 9.67	84.70 ± 3.67	75.35 ± 3.05	59.31 ± 6.06	55.24 ± 3.84	45.65 ± 4.54	30.58 ± 3.55	21.94 ± 2.68	11.45 ± 0.99
2,5-Dimethyl-pyrazine	34.02 ± 4.18	28.41 ± 3.73	17.14 ± 4.82	41.63 ± 7.36	33.04 ± 1.67	30.86 ± 2.81	15.41 ± 0.99	11.58 ± 1.03	19.68 ± 2.50	15.22 ± 0.95	17.50 ± 1.47	11.09 ± 1.95	10.37 ± 1.65	10.86 ± 0.59
Methylthio-2-Propanone	4.00 ± 0.30	5.03 ± 0.59	4.52 ± 0.63	5.11 ± 0.15	3.11 ± 0.26	2.98 ± 0.29	1.02 ± 0.07	0.75 ± 0.05	0.25 ± 0.01	0.29 ± 0.02	0.22 ± 0.01	0.16 ± 0.01	0.12 ± 0.01	0.04 ± 0.00
1-Hexanol	53.13 ± 3.16	49.70 ± 4.53	55.26 ± 4.93	70.47 ± 4.81	216.47 ± 9.83	210.78 ± 14.08	227.16 ± 14.13	225.70 ± 16.14	367.02 ± 47.41	344.67 ± 17.77	354.59 ± 28.16	313.50 ± 11.39	270.83 ± 22.46	217.75 ± 7.50
2-Chloro-1-propanol*	3.95 ± 0.23	2.92 ± 0.31	2.35 ± 0.07	2.06 ± 0.05	1.68 ± 0.06	1.24 ± 0.07	0.68 ± 0.03	0.63 ± 0.03	0.51 ± 0.03	0.37 ± 0.04	0.28 ± 0.02	0.19 ± 0.01	0.09 ± 0.01	0.09 ± 0.01
2-Ethyl-6-methyl-pyrazine	1.02 ± 0.04	0.92 ± 0.07	0.64 ± 0.16	1.60 ± 0.34	1.26 ± 0.02	1.31 ± 0.12	0.76 ± 0.02	0.56 ± 0.01	1.23 ± 0.10	0.79 ± 0.03	0.93 ± 0.06	0.62 ± 0.12	0.61 ± 0.06	0.63 ± 0.01
2-Nonanone*	0.28 ± 0.02	0.30 ± 0.05	0.38 ± 0.07	0.42 ± 0.01	2.21 ± 0.07	4.73 ± 0.27	10.72 ± 0.64	10.98 ± 0.58	45.79 ± 3.25	55.06 ± 1.32	55.87 ± 2.52	107.08 ± 36.94	127.11 ± 10.12	370.98 ± 4.77
Nonanal*	29.64 ± 5.12	32.27 ± 11.07	61.41 ± 21.79	29.24 ± 1.11	107.95 ± 11.90	166.35 ± 6.87	195.52 ± 14.05	208.50 ± 10.57	439.02 ± 21.93	482.77 ± 11.25	436.56 ± 13.38	639.35 ± 135.19	666.01 ± 14.58	877.19 ± 16.59
Trimethyl-pyrazine	5.32 ± 0.16	4.33 ± 0.20	3.10 ± 0.48	7.17 ± 1.05	5.69 ± 0.15	5.60 ± 0.29	3.32 ± 0.13	2.13 ± 0.18	4.76 ± 0.33	2.96 ± 0.11	3.55 ± 0.16	2.29 ± 0.44	2.24 ± 0.21	2.95 ± 0.05
3-Octen-2-one*	0.98 ± 0.05	0.69 ± 0.01	2.36 ± 0.09	3.65 ± 0.12	15.39 ± 0.46	25.98 ± 1.45	29.61 ± 1.44	30.93 ± 1.77	69.04 ± 6.42	74.84 ± 2.36	77.70 ± 4.21	90.08 ± 20.51	87.65 ± 8.50	164.13 ± 3.27
(E)-2-Octenal*	0.47 ± 0.04	0.47 ± 0.06	1.93 ± 0.19	3.62 ± 0.08	13.28 ± 0.26	31.54 ± 1.96	33.16 ± 1.94	26.21 ± 1.35	114.22 ± 10.72	130.24 ± 1.12	128.11 ± 4.82	145.14 ± 34.41	124.97 ± 11.62	293.77 ± 4.05
3-Ethyl-2,5-dimethyl-pyrazine	0.31 ± 0.01	0.23 ± 0.01	0.22 ± 0.02	0.45 ± 0.07	0.36 ± 0.01	0.39 ± 0.01	0.16 ± 0.01	0.16 ± 0.01	0.38 ± 0.02	0.23 ± 0.00	0.25 ± 0.01	0.17 ± 0.03	0.16 ± 0.01	0.21 ± 0.01
2,6-Diethyl-pyrazine	4.33 ± 0.08	3.27 ± 0.04	3.06 ± 0.24	6.58 ± 0.92	5.40 ± 0.04	5.68 ± 0.24	3.83 ± 0.08	2.26 ± 0.16	5.46 ± 0.09	3.29 ± 0.11	3.79 ± 0.11	2.61 ± 0.58	2.46 ± 0.20	3.29 ± 0.05
Acetic acid*	23.09 ± 0.17	31.51 ± 1.18	12.66 ± 2.03	15.73 ± 12.64	12.01 ± 1.55	33.28 ± 2.00	37.06 ± 2.44	30.06 ± 4.02	73.03 ± 8.12	62.79 ± 10.62	52.87 ± 7.51	52.52 ± 1.07	40.84 ± 1.19	89.29 ± 4.01
1-Octen-3-ol*	2.88 ± 0.07	1.85 ± 0.11	3.08 ± 0.14	4.05 ± 0.26	13.06 ± 0.73	21.55 ± 1.47	26.60 ± 1.15	22.16 ± 2.70	127.22 ± 9.24	126.94 ± 7.97	157.58 ± 4.04	150.04 ± 34.43	144.55 ± 16.00	395.23 ± 4.01
Furfural*	41.65 ± 2.68	53.02 ± 4.87	17.07 ± 2.30	21.80 ± 1.69	15.63 ± 0.55	18.05 ± 1.17	12.37 ± 0.35	9.78 ± 0.37	15.89 ± 1.63	13.29 ± 1.32	14.08 ± 0.35	12.24 ± 1.65	10.68 ± 1.04	16.31 ± 0.49
1-Heptanol*	1.75 ± 0.05	1.83 ± 0.07	2.87 ± 0.11	3.98 ± 0.07	14.84 ± 0.19	35.49 ± 1.96	49.20 ± 2.33	123.78 ± 9.35	144.37 ± 5.29	129.40 ± 6.05	181.70 ± 52.25	178.44 ± 16.12	283.18 ± 3.27	283.18 ± 3.27
2-Decanone*	0.16 ± 0.02	0.14 ± 0.03	0.23 ± 0.02	0.23 ± 0.07	0.52 ± 0.01	1.36 ± 0.05	2.63 ± 0.12	3.09 ± 0.18	12.74 ± 0.88	15.21 ± 0.54	15.89 ± 0.40	34.16 ± 11.83	40.45 ± 2.06	123.67 ± 1.19
Decanal*	0.64 ± 0.20	0.99 ± 0.20	0.90 ± 0.06	0.70 ± 0.09	2.40 ± 0.13	5.30 ± 0.37	7.88 ± 0.63	10.72 ± 0.42	24.28 ± 2.36	28.53 ± 1.58	47.60 ± 12.30	48.64 ± 6.50	80.56 ± 1.19	80.56 ± 1.19
trans-3-Nonen-2-one*	0.15 ± 0.05	0.24 ± 0.03	0.16 ± 0.03	0.17 ± 0.03	0.74 ± 0.07	1.38 ± 0.04	1.73 ± 0.19	1.72 ± 0.29	12.69 ± 0.78	11.45 ± 0.41	17.14 ± 0.33	22.53 ± 7.99	26.93 ± 1.81	103.28 ± 2.91
Pyrazole	4.16 ± 0.36	3.47 ± 0.58	1.67 ± 0.31	2.19 ± 0.31	1.13 ± 0.05	0.90 ± 0.01	0.65 ± 0.04	0.65 ± 0.04	0.42 ± 0.04	0.43 ± 0.03	0.49 ± 0.07	0.49 ± 0.04	0.65 ± 0.02	1.56 ± 0.02
Benzaldehyde*	7.24 ± 0.24	8.11 ± 0.12	9.03 ± 0.44	12.55 ± 0.65	18.86 ± 0.59	21.66 ± 1.20								

Table 3. Continued

Treatment	0		1		3		5		7		9		11	
	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME	NME	ME
(Z)-2-Decenal*	0.11 ± 0.07	0.07 ± 0.03	0.22 ± 0.06	0.15 ± 0.03	0.39 ± 0.04	2.04 ± 0.26	1.98 ± 0.20	2.20 ± 0.20	8.70 ± 0.93	9.98 ± 0.22	9.05 ± 0.67	16.38 ± 6.88	13.25 ± 0.62	31.88 ± 0.95
2-Methyl- anhydride pentanoic acid*	0.19 ± 0.01	0.24 ± 0.00	0.40 ± 0.08	0.59 ± 0.05	2.09 ± 0.27	9.56 ± 1.28	12.94 ± 0.89	10.40 ± 0.48	64.66 ± 4.80	67.31 ± 1.92	76.68 ± 1.85	86.86 ± 21.73	68.15 ± 6.36	219.57 ± 7.24
2-Furanmethanol*	2.40 ± 0.14	3.13 ± 0.19	1.40 ± 0.10	2.04 ± 0.28	1.19 ± 0.04	1.58 ± 0.06	0.94 ± 0.10	0.58 ± 0.05	0.98 ± 0.08	0.84 ± 0.03	0.77 ± 0.04	0.77 ± 0.14	0.65 ± 0.09	1.46 ± 0.16
1-Nonanol*	0.20 ± 0.03	0.24 ± 0.01	0.16 ± 0.00	0.14 ± 0.02	0.30 ± 0.03	0.43 ± 0.03	0.58 ± 0.03	0.69 ± 0.07	1.90 ± 0.46	1.97 ± 0.29	2.05 ± 0.35	3.47 ± 0.72	3.10 ± 0.11	4.84 ± 0.80
3-Methyl-butanoic acid*	0.49 ± 0.04	0.31 ± 0.05	0.28 ± 0.07	0.70 ± 0.15	1.46 ± 0.13	2.71 ± 0.25	1.94 ± 0.04	1.92 ± 0.01	5.62 ± 0.13	4.69 ± 0.04	6.85 ± 0.08	9.92 ± 4.12	4.30 ± 0.20	7.68 ± 0.04
5-Ethylidihydro-2(3H)-furanone*	1.76 ± 0.03	3.25 ± 0.06	2.77 ± 0.18	4.09 ± 0.18	16.76 ± 0.35	37.87 ± 1.67	50.39 ± 2.40	55.85 ± 2.67	135.29 ± 8.21	150.71 ± 2.38	144.68 ± 5.71	201.01 ± 48.80	201.97 ± 15.27	480.49 ± 5.64
Pentanoic acid*	0.12 ± 0.02	0.18 ± 0.02	0.39 ± 0.19	0.38 ± 0.08	2.75 ± 0.20	12.15 ± 2.14	22.70 ± 2.33	23.03 ± 1.59	99.71 ± 4.74	106.51 ± 4.03	102.62 ± 3.35	183.44 ± 65.25	183.69 ± 14.04	516.29 ± 9.83
Tetrahydro-6-methyl-2H-pyran-2-one*	0.06 ± 0.01	0.06 ± 0.00	0.06 ± 0.02	0.06 ± 0.00	0.13 ± 0.01	0.33 ± 0.02	0.39 ± 0.02	0.45 ± 0.03	1.22 ± 0.08	1.39 ± 0.01	1.28 ± 0.02	2.03 ± 0.60	2.09 ± 0.17	5.61 ± 0.01
Dihydro-5-propyl-2(3H)-furanone*	0.04 ± 0.00	0.15 ± 0.00	0.10 ± 0.02	0.10 ± 0.01	0.25 ± 0.02	0.62 ± 0.03	0.82 ± 0.04	1.04 ± 0.05	2.83 ± 0.20	3.36 ± 0.05	3.25 ± 0.18	5.69 ± 1.39	6.29 ± 0.39	15.79 ± 0.03
Hexanoic acid*	0.46 ± 0.10	0.69 ± 0.06	1.59 ± 0.98	0.76 ± 0.22	9.96 ± 1.04	69.31 ± 9.84	130.85 ± 13.78	146.35 ± 15.81	587.04 ± 46.14	619.53 ± 43.59	598.48 ± 27.79	906.51 ± 220.58	928.90 ± 38.14	1423.71 ± 11.55
Benzyl alcohol*	0.25 ± 0.03	0.61 ± 0.03	0.17 ± 0.02	0.24 ± 0.01	0.25 ± 0.01	0.46 ± 0.01	0.29 ± 0.01	0.30 ± 0.02	0.46 ± 0.03	0.49 ± 0.02	0.48 ± 0.02	0.50 ± 0.04	0.37 ± 0.01	0.47 ± 0.02
5-Butyldihydro-2(3H)-furanone*	0.04 ± 0.00	0.07 ± 0.00	0.15 ± 0.07	0.08 ± 0.00	0.39 ± 0.02	1.14 ± 0.07	1.53 ± 0.13	2.27 ± 0.16	5.97 ± 0.63	7.31 ± 0.26	6.74 ± 0.43	12.35 ± 2.69	12.78 ± 0.39	28.64 ± 0.33
Phenylethyl alcohol*	0.48 ± 0.01	0.90 ± 0.03	0.48 ± 0.07	0.65 ± 0.03	0.58 ± 0.00	1.17 ± 0.02	0.69 ± 0.05	0.88 ± 0.10	1.08 ± 0.07	1.42 ± 0.11	1.23 ± 0.02	1.42 ± 0.05	1.07 ± 0.02	1.16 ± 0.08
Heptanoic acid*	0.06 ± 0.02	0.09 ± 0.02	0.11 ± 0.06	0.09 ± 0.01	0.23 ± 0.08	0.80 ± 0.03	1.36 ± 0.20	1.81 ± 0.14	9.05 ± 1.84	12.67 ± 2.00	10.68 ± 0.27	30.17 ± 13.06	32.88 ± 1.16	123.39 ± 3.37
2-Vinylfuran*	0.19 ± 0.02	0.22 ± 0.01	0.35 ± 0.07	0.20 ± 0.01	0.24 ± 0.01	0.37 ± 0.05	0.26 ± 0.00	0.21 ± 0.04	0.37 ± 0.06	0.31 ± 0.09	0.39 ± 0.08	0.37 ± 0.10	0.48 ± 0.06	0.43 ± 0.11

*Significantly different between treatments across all time points ($P < 0.05$) using ANOVA.

49 volatiles were significantly different between NME-DR and ME-DR samples (Tables 2 and 3).

Volatile organic compounds are generated from various pathways, including the Maillard reaction, sugar pyrolysis, and via lipid oxidation. The Maillard reaction is favorable in high heat and low moisture systems.²⁶ Almonds are a low-moisture (less than 10% moisture w/w), high-fat (44–61% fat by weight) food.²⁷ Frequently reported Maillard-reaction-related volatiles found in heat-treated almonds includes Strecker aldehydes, alkylpyrazines, and furans.²⁸ Strecker degradation product of leucine (2-methylbutanal) and isoleucine (3-methylbutanal) are low-odor threshold compounds that contribute to the malty aroma in almonds.²⁹ 2,5-Dimethyl pyrazine, 2-methylpyrazine, and trimethyl pyrazine are highly correlated with clean nutty flavor / aroma and clean roasted flavor / aroma in roasted almonds.³⁰ In this study, 2,5-dimethyl pyrazine and 2-methylpyrazine were the only pyrazines detected in the headspace (Tables 2 and 3) and at levels 2–4 times lower than in other studies,^{16, 17} which can be attributed to different roasting conditions.

The major decomposition products of oleic acid alkoxy radicals include decanal, 1-decane, heptanoic acid, octanol, 2-undecenal, nonanal, octanal, heptanol, and heptanal.¹⁸ Heptanal and octanal are proposed as good indicators of rancidity in almonds due to their strong negative correlation with consumer liking and because they exist at concentrations above the aroma threshold for these compounds.¹⁶ Hexanal, the major decomposition product of linoleic acid, is a common rancidity marker in lipid rich foods. Linoleic acid is the second most abundant fatty acids in almonds and its decomposition products (e.g. hexanal, 2-heptenal, and 2-octenal) have been used to assess almond quality.^{16, 31, 32} Levels of heptanal and octanal found in this study are comparable to other studies of almonds undergoing accelerated storage.^{16, 33} At 7 months of accelerated storage, the ME-LR almonds had significantly greater levels of heptanal ($387.30 \pm 35.63 \mu\text{g kg}^{-1}$) and octanal ($341.24 \pm 17.77 \mu\text{g kg}^{-1}$) than the NME-LR almonds ($331.84 \pm 25.43 \mu\text{g kg}^{-1}$ and $260.48 \pm 11.97 \mu\text{g kg}^{-1}$) indicating a higher level of oxidation. Similar to PV values, several oxidation products of oleic and linoleic acid (e.g. hexenal and pentanal) peak around 7 months.

Levels of acetic acid, pentanoic acid, and hexanoic acid were significantly higher in all ME almond samples than the NME almonds for both DR and LR almonds (Tables 2 and 3). Organic acids (i.e. acetic, pentanoic, hexanoic, and heptanoic acid) are tertiary lipid oxidation products that increase during almond storage.^{9, 33} The concentration of these volatiles increased 55–779 times over the 12 months of storage for both ME and NME almonds (Tables 2 and 3) with levels of hexanoic acid increasing the most significantly. Rogel *et al.* (2017)³ demonstrate higher levels of acetic acid in the headspace of ME almonds. Our results indicate that organic acids, and in particular hexanoic acid, may be a useful marker for identifying almonds exposed to post-harvest moisture.

Hierarchical clustering analysis of the volatiles that were significantly different ($P < 0.05$) between ME and NME almonds (Figs S1 and S2) indicates that storage time has a greater effect on the sample clustering than moisture exposure. The shorter storage times (1–5 months) clustered together and the longer storage times (7–12 months) clustered together. Overall, pyrazines and pyrrole concentrations decreased with increased storage time and aldehydes, ketones, and organic acids increased with increased storage time.

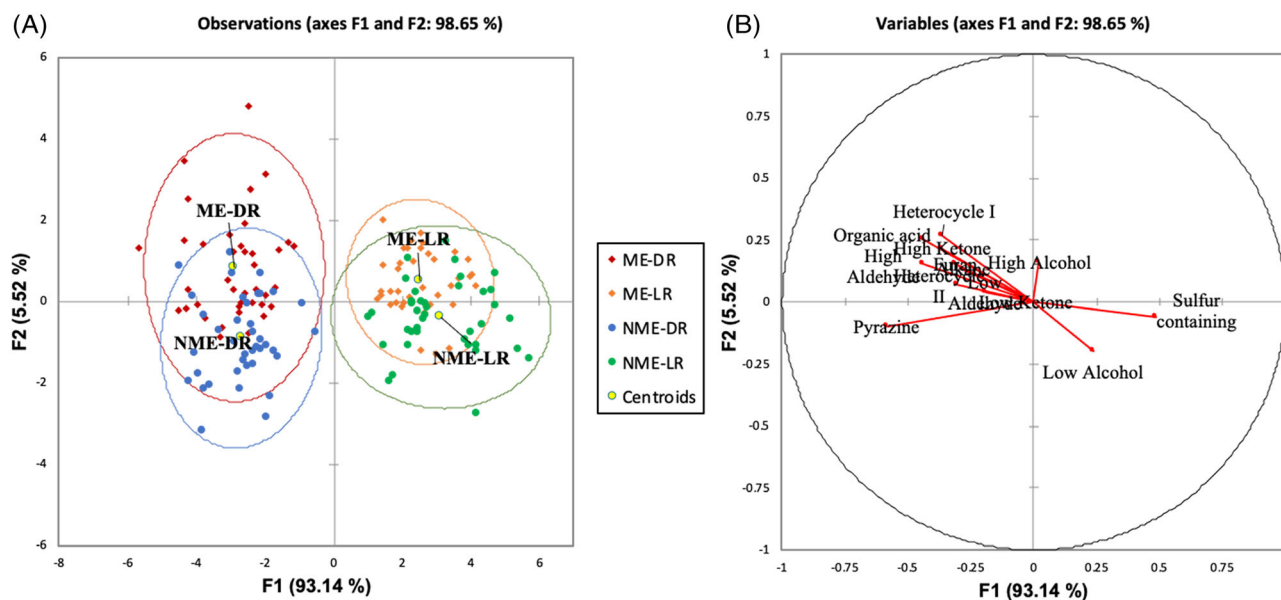


Figure 1 Discriminant analysis of volatile compounds (69), identified then grouped by chemical functionality (shown in Table S2) in almonds either exposed to 8% moisture and dried to 5% moisture (ME) or not exposed to moisture (NME) and roasted to achieve either a light roast (LR) or dark roast (DR): (a) the observation plot showing the grouping of each category, and (b) the loading plot showing the variables contributing to both factors (F1 and F2).

Table 4 Average value of hedonic testing and descriptive analysis attributes that were significantly different between treatments of light roasted almonds at 0, 1, 3, 5, and 7 months of accelerated storage

Sensory analysis	Treatment	Storage month				
		0	1	3	5	7
Hedonic testing	NME	6.68 ^a	6.43 ^{ab}	5.98 ^{abc}	5.29 ^{cd}	5.44 ^{cd}
	ME	6.36 ^{ab}	6.36 ^{ab}	5.76 ^{bcd}	5.44 ^{cd}	5.08 ^d
Degree of difference	NME	0.25 ^f	0.73 ^{de}	1.88 ^c	2.97 ^b	3.2 ^b
	ME	0.39 ^{ef}	0.94 ^d	2.1 ^c	3.32 ^{ab}	3.59 ^a
Color	NME	7.43 ^f	7.49 ^{de}	7.51 ^c	7.64 ^b	7.53 ^b
	ME	7.49 ^{ef}	7.58 ^d	7.61 ^c	7.7 ^{ab}	7.74 ^a
Clean nutty aroma	NME	4.11 ^a	3.22 ^b	2.32 ^c	1.77 ^{de}	1.56 ^{ef}
	ME	4.02 ^a	3.16 ^b	1.99 ^{cd}	1.52 ^{ef}	1.43 ^f
Clean roasted aroma	NME	3.51 ^a	2.81 ^b	2 ^c	1.59 ^{de}	1.54 ^{de}
	ME	3.43 ^a	2.76 ^b	1.78 ^{cd}	1.47 ^e	1.35 ^e
Clean nutty flavor	NME	4.41 ^a	4.1 ^{ab}	3.27 ^c	2.18 ^e	1.99 ^{ef}
	ME	4.3 ^{ab}	3.92 ^c	2.76 ^d	1.94 ^{ef}	1.71 ^f
Clean roasted flavor	NME	2.99 ^a	2.71 ^{bc}	2.16 ^d	1.67 ^e	1.6 ^{ef}
	ME	2.92 ^{ab}	2.57 ^c	1.96 ^d	1.49 ^{ef}	1.41 ^f
Total oxidized aroma	NME	0.02 ^f	0.58 ^e	1.92 ^d	2.65 ^{bc}	2.98 ^{ab}
	ME	0.05 ^f	0.67 ^e	2.58 ^c	3.1 ^a	3.2 ^a
Total oxidized flavor	NME	0.05 ^g	0.55 ^f	1.67 ^e	2.84 ^c	3.14 ^{bc}
	ME	0.1 ^g	0.62 ^f	2.1 ^d	3.35 ^{ab}	3.6 ^a
Cardboard flavor	NME	0.04 ^e	0.46 ^e	1.01 ^d	1.26 ^c	1.26 ^{bc}
	ME	0.09 ^e	0.57 ^e	1.29 ^d	1.41 ^{ab}	1.5 ^a
Painty / solvent flavor	NME	0 ^e	0.6 ^e	0.86 ^d	1.92 ^c	2.13 ^{bc}
	ME	0.01 ^e	0.12 ^e	0.98 ^d	2.34 ^{ab}	2.52 ^a

Letters shared within the same chemical measurement indicates there is no significant difference ($P < 0.05$) using ANOVA.

Volatiles identified in the headspace of almonds were placed into groups ¹² based on their structure and functional group chemistry (Table S2) and analyzed using discriminant analysis

(Fig. 1(a), (b)). Samples separate based on degree of roasting (i.e. LR or DR; Fig. 1(a)). Significant overlap was observed between the ME and NME almonds within each quadrant indicating that

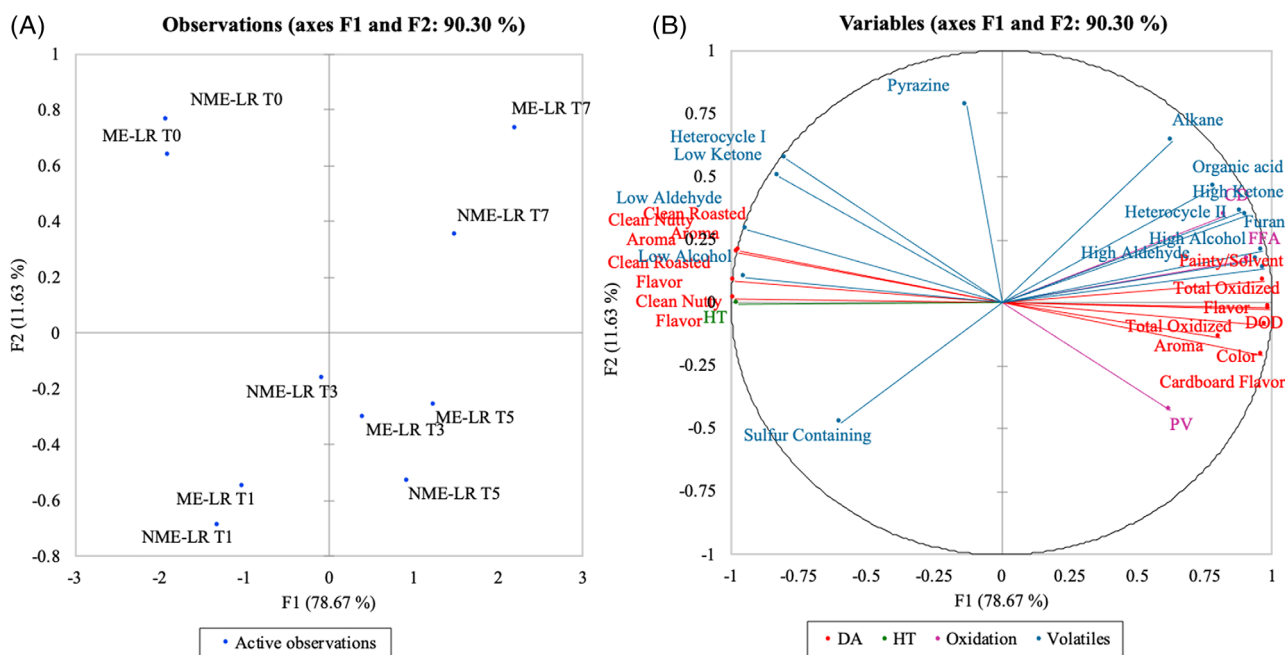


Figure 2 Multiple factor analysis of volatile compounds (69) identified then grouped (shown in Table S2), descriptive analysis (DA) attributes (Table 4), chemical analyses, and hedonic testing in almonds exposed to 8% moisture and dried to 5% moisture (ME) or not exposed to moisture (NME) and roasted to achieve light roast (LR) at 0, 1, 3, 5, and 7 months of storage: (A) the observation plot showing the separation of each sample, and (B) the loading plot showing the variables contributing to both factors (F1 and F2) with oxidation (PV, CD, and FFA) and hedonic testing (HT) included as supplementary variables.

roasting level has a greater effect on separating the samples than moisture exposure. Figure 1(b) shows the variables driving the separation observed in Fig. 1(a). The left quadrants, occupied by DR samples, separated based on Maillard reaction products (e.g. pyrazines and low molecular weight aldehydes) and lipid oxidation products (e.g. high molecular weight aldehydes and organic acids). The top quadrants, occupied by the centroids of ME-LR and ME-DR, were driven by organic acids, ketones, and high molecular weight alcohols. Discriminant analysis indicates that there is no distinct class of volatiles that can be used to differentiate between ME and NME samples and that roasting level has a greater effect on discrimination as dark roasting correlates more strongly with lipid oxidation products (e.g. high molecular weight aldehydes and organic acids).

Sensory analysis of light roasted almonds

Descriptive analysis and consumer hedonic testing were used to study differences between ME and NME almonds in LR almonds. DR almonds were not evaluated as the roasting conditions used to produce DR almonds result in significant lipid oxidation at time points past 4 months and could bias sensory evaluations. NME almonds at 0 months of storage were used as the control for all sensory analyses. The sensory evaluations were limited to 0–7 months to cover a significant part of shelf-life and to allow for the completion of hedonic testing within one sitting.

Twenty-two attributes were evaluated during the descriptive analysis (Table S1). Of these, 11 attributes were statistically different between NME and ME almonds ($P < 0.05$) across storage times (Table 4). The ME samples were significantly higher in overall degree of difference (DOD), total oxidized aroma and flavor, cardboard flavor, painty / solvent flavor, and initial hardness, and had

significantly darker color as compared to the NME almonds. The ME samples were also significantly lower in clean nutty aroma and flavor, and clean roasted aroma and flavor, which are attributes that have a positive association with fresh roasted almonds. Attributes such as total aroma and flavor, bitter flavor, and initial and secondary chewing textures were not significantly different between treatments across storage times. These data indicate that exposing almonds to moisture and drying them before roasting does not have a statistically significant effect on the texture attributes measured. When comparing the individual storage times, five attributes were significantly different ($P < 0.05$) between treatments at 7 months (Table 4). These attributes include DOD, color, total oxidized flavor, cardboard flavor, and painty / solvent flavor. A significant difference between the DOD scores occurred at 7 months of storage and indicates that the trained panelists were able to distinguish the two products from one another. The attributes that were significantly different between treatments at 7 months of storage are characteristics observed in oxidized products.^{30, 34}

Cardboard flavor is predicted by increased levels of unsaturated aldehydes, such as 2-octenal and 2-heptenal.³⁰ Herein, 2-octenal, 2-nonenal, and 2-decenal were significantly different between ME and NME almonds (Table 2) and correspond to an increase in the description of cardboard flavor in ME samples (Table 4). Pentanal and heptanal levels were not significantly different ≥ 7 months of storage, and 1-octen-3-one and dimethyl trisulfide were not detected. Total oxidized flavor and solvent/painty flavor were associated with similar volatiles as total oxidized flavor. Some proposed volatiles markers for monitoring lipid oxidation in almonds are pentanal, hexanal, 2-heptanol, heptanal, octanal, hexanoic acid, 1-pentanol, 2-octenal, nonanal, 2-heptanone, and 2-pentylfuran.^{16, 31} Among these markers, only octanal, nonanal, 2-

octanal, and hexanoic acid were significantly different between NME and ME almonds at time points when the trained panelists were able to statistically differentiate the products. These compounds have been reported to have low odor thresholds.¹⁸ Our findings suggest that octanal, nonanal, 2-octenal, and hexanoic acid may be the most sensitive indicators of almond acceptability in roasted almond products.

Almonds that are exposed to moisture after harvesting can develop a dark brown discoloration of the kernel nutmeat when heated (e.g. roasting). This discoloration is termed *concealed damage* as the color appears only after heat treatment.⁸ Browning is attributed to the hydrolysis of carbohydrates and lipids and formation of precursors that contribute to Maillard browning. Raw almonds that have *concealed damage* induced by moisture have significantly lower CIE L* color values as compared to controls.⁸ Although a previous study indicated that drying almonds below 65 °C prior to roasting can reduce discoloration in roasted almonds⁷ we found that not to be the case. Herein, the ME almonds were found to exhibit lower CIE L* color values (i.e. darker in color) than NME almonds after roasting across all time points (Table 1). This result was consistent with the descriptive analysis with ME almonds having a higher score in darkness (Table 4). The discrepancy between our study and the previous study may be explained by differences in how almond moisture was increased between studies. In the study by Rogel *et al* (2015), the almonds were sprayed with water and incubated at 45 °C for 24 h to achieve a moisture content of 8–9%; here the moisture content of the almonds was increased to 8% using a climate-controlled chamber at 38 °C and 90 ± 1% RH over 36 h.

Hedonic testing of the almonds indicated that there were no significant differences in the mean liking scores of between ME and NME almonds over all time points (Table 4). Consumer paired preference testing indicated no strong preference between the treatments (Fig. S3), with the exception of the 3 month samples where consumers preferred the NME over the ME almonds ($P < 0.05$). The average hedonic testing scores demonstrate that the storage time has a significant influence on the liking score with the highest average score of 6.68 for NME at 0 month and lowest score of 5.08 for ME at 7 months (Table 4). Although no statistically significant differences were found between the liking scores between treatments, the ME samples showed a lower average score than the NME. This suggests that a difference between the ME and NME almonds was detected by the consumers, but it was not significant enough to influence consumer preferences.

A multiple factor analysis (Fig. 2(a), (b)) was performed to show the relationship between the chemical analysis, volatile analysis, and sensory analysis of LR almonds at 0, 1, 3, 5, and 7 months of storage. The observation plot (Fig. 2(a)) demonstrates that samples separate based on the storage time in the first dimension with longer storage times in the right quadrants. Figure 2(b) shows the space generated by the grouped volatiles and the sensory attributes that were significantly different between treatments as variables. The first two dimensions explain 90% of the variables, with clean roasted flavor / aroma and clean nutty flavor / aroma correlating with low molecular weight aldehydes and alcohols (left quadrants) and total oxidized flavor / aroma correlating with high molecular weight alcohols and aldehydes (right quadrants). Peroxide value, FFA, and CD correlate with lipid oxidation volatiles and sensory attributes, whereas hedonic testing correlates only with fresh roasted sensory attributes. This demonstrates that average consumer liking correlates with fresh roasted samples. However, trained panelists are able to determine

treatment differences at 7 months of storage with the ME sample correlating with lipid oxidation attributes. At 7 months of storage, both ME and NME samples also demonstrated increased levels of volatiles related to lipid oxidation and were rated as having noticeable rancid attributes by panelists.

CONCLUSIONS

This study demonstrates that post-harvest moisture exposure and subsequent drying had a significant effect on the quality of roasted almonds during storage, and this was most pronounced in the dark roast product. The ME-DR almonds experienced significantly higher levels of lipid oxidation than NME-DR almonds at 5 months of storage and will have shorter shelf life. Although the shelf life may be similar in NME-LR and ME-LR almonds, trained panelists can detect sensory attributes related to lipid oxidation at 7 months of storage that correlate with increased levels of volatiles related to lipid oxidation. This result indicates that ME-LR almonds will have a shorter shelf life than NME-LR almonds. This information is critical for providing the industry with tools to help improve product management. For example, many almonds arriving at processors that need to be dried prior to hulling and shelling, may be better suited for product streams that undergo light roasting and / or are used in products that are consumed within 12 months.

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SUPPORTING INFORMATION DESCRIPTION

Includes clustering of headspace volatiles measured among light roasted and dark roasted samples, graph of consumer paired preference, list of sensory attributes and definitions for descriptive analysis, and a table of the headspace volatiles identified in all samples.

SUPPORTING INFORMATION

Supporting information may be found in the online version of this article.

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