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Identification of predominant aroma components of raw, dry roasted and oil roasted almonds

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ABSTRACT

Volatile components of raw, dry roasted and oil roasted almonds were isolated by solvent extraction/solvent-assisted flavor evaporation and predominant aroma compounds identified by gas chromatography-olfactometry (GCO) and aroma extract dilutions analysis (AEDA). Selected odorants were quantitated by GC–mass spectrometry and odor-activity values (OAVs) determined. Results of AEDA indicated that 1-octen-3-one and acetic acid were important aroma compounds in raw almonds. Those predominant in dry roasted almonds were methional, 2- and 3-methylbutanal, 2-acetyl-1-pyrroline and 2,3-pentanedione; whereas, in oil roasted almonds 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone, 2,3-pentanedione, methional and 2-acetyl-1-pyrroline were the predominant aroma compounds. Overall, oil roasted almonds contained a greater number and higher abundance of aroma compounds than either raw or dry roasted almonds. The results of this study demonstrate the importance of lipid-derived volatile compounds in raw almond aroma. Meanwhile, in dry and oil roasted almonds, the predominant aroma compounds were derived *via* the Maillard reaction, lipid degradation/oxidation and sugar degradation.

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1. Introduction

Among tree nuts, almond (*Prunus dulcis* (Mill) D.A. Webb) ranks third in worldwide production behind cashew and walnut, with the US being the largest producer. The most common almond produced in the US is the Nonpareil variety, which has numerous applications in the food industry and also wide appeal among consumers. Almond consumption has grown in recent years due in part to reports indicating the potential health benefits of almond consumption (Kamil & Chen, 2012).

Aside from the health-related studies, there are numerous reports on the volatile flavor components of raw and roasted almonds. Volatile aldehydes, ketones, alcohols, alkanes and heterocyclic compounds have been reported in raw almonds (Agila & Barringer, 2012; Beck, Mahoney, Cook, & Gee, 2011; Manzano, Diego, Bernal, Nozal, & Bernal, 2014; Mexis, Badeka, Chouliara, Riganakos, & Kontominas, 2009; Wirthensohn et al., 2008; Xiao et al., 2014). Most studies published on raw almonds reported aldehydes, such as hexanal, nonanal and benzaldehyde, as the main volatile components (Mexis et al., 2009; Xiao et al., 2014). Likewise, aldehydes also were reported as the major volatile components of almond oil (Sanahuja, Santonja, Teruel, Carratalá, & Selva, 2011).

Previous studies on dry roasted almonds reported aldehydes, ketones, alcohols, aromatic hydrocarbons, terpenes, and linear hydrocarbons as the main volatile constituents. Takei, Shimada, Watanabe, and Yamanishi (1974), and Takei and Yamanishi (1974) identified 17 pyrazines and 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (HDMF) as the main volatile compounds in roasted almonds. Vázquez-Araújo, Enguix, Verdú, García-García, and Carbonell-Barrachina (2008) and Vázquez-Araújo, Verdú, Navarro, Martínez-Sánchez, and Carbonell-Barrachina (2009) reported that pyrazines, pyrroles and furans comprised the main volatile compound classes in toasted almonds. In more recent studies, aldehydes were reported as the major volatiles in roasted almonds (Agila & Barringer, 2012; Manzano et al., 2014; Valdés et al., 2015; Xiao et al., 2014; Yang et al., 2013).

There are only a few reports on the volatile components of oil roasted almonds. Agila and Barringer (2012) reported that aldehydes and alcohols were the main volatile compounds and these compounds were present at lower concentrations in oil roasted almonds than in oven (dry) roasted almonds. Valdés et al. (2015) analyzed commercially-available fried (oil roasted) almonds and reported that the main volatile compounds were aldehydes.

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Various techniques have been used to extract or isolate the volatile compounds from almonds. Most studies employed some type of headspace method, with only a few exceptions in which solvent extraction techniques were used (Takei & Yamanishi, 1974; Takei et al., 1974; Vázquez-Araújo et al., 2008). In general, headspace methods alone are not suitable for the comprehensive aroma characterization of a food product because they do not allow for the isolation and analysis of semi-volatile aroma compounds. Realizing this limitation, the present study utilized solvent extraction combined with solvent-assisted flavor evaporation (SAFE) for the careful and exhaustive isolation of both volatile and semi-volatile compounds from raw and roasted almonds. This approach has been reported to be suitable isolation of aroma compounds from high fat containing foods (Engel, Bahr, & Schieberle, 1999), such as almonds. In addition, this study is the first to report the use of gas chromatography-olfactometry (GCO) for the identification of compounds in raw and roasted almonds. The objective of this study was to identify the predominant aroma components of raw, dry roasted and oil roasted almonds by application of solvent extraction-SAFE combined with GCO and aroma extract dilution analysis (AEDA) and by determination of odor-activity values (OAVs).

2. Material and methods

2.1. Samples

Whole shelled almonds (*Prunus dulcis* (Mill) D.A. Webb), Nonpareil variety, harvested in the 2011 season, were obtained from Blue Diamond®, California. Upon arrival, almonds were stored at ~4 °C until needed.

2.2. Roasting of almonds

Almonds were either dry roasted in an air ventilated oven (DN-61 Constant Temperature Oven, American Scientific Products, Ocala, FL) at 165 °C for 15 min or oil roasted in a Cayenne® electric fryer (The Vollrath Co., L.L.C., Sheboygan, WI) at 152 ± 3 °C for 8 min in canola oil (almond to oil ratio 1:4 w/w). Roasting conditions were chosen based on published literature and industrial practices (Lukac et al., 2007; Perren & Escher, 2007).

2.3. Chemicals

Diethyl ether (anhydrous, 99.7%), hydrochloric acid (HCl, 36.5%), sodium hydroxide (NaOH, 98.7%), sodium carbonate (Na_2CO_3), sodium chloride (NaCl) and sodium sulfate (granular, anhydrous 99%) were purchased from Fisher Scientific (Fairlawn, NJ). *n*-Alkane standards (C7–C30) and internal standards; 2-ethylbutanoic acid (acid fraction), 2-methyl-3-heptanone (neutral fraction) and 2,4,6-trimethylpyridine (collidine) (basic fraction) were purchased from Sigma-Aldrich Chemicals Co. (St. Louis, MO). Deodorized water was prepared by boiling deionized-distilled water until one-third of the original volume was evaporated.

Authentic reference compounds listed in Tables 1–5 were obtained from commercial sources, as follows: Nos. **1–7**, **11**, **17–20**, **24–26**, **28–31**, **36**, **39**, **40**, **42**, **46**, **47**, **54**, **55**, **57–59** (Sigma-Aldrich Chemicals Co.); No. **9** and **38** (Bedoukian, Danbury, CT); and No. **12** (Lancaster, Windham, NH). The following compounds were synthesized using published procedures: Nos. **14**, **22**, **33** and **34** (Fuganti, Gatti, & Serra, 2007), No. **16** (Ullrich & Grosch, 1988), No. **53** (Lin, Fay, Welti, & Blank, 1999) and No. **48** (Schuh & Schieberle, 2005).

2.4. Aroma compound extraction

Almonds (300 g) were ground for 5 s in a glass blender, sieved (No. 12; SA standard testing sieve; W.S. Tyler Inc. Mentor, OH) and divided equally into two Teflon (FPE) centrifuge bottles. Diethyl ether (100 mL) was added to each bottle. The bottles were sealed with FPE caps, shaken at 200 rpm (DS-500 orbital shaker; VWR International, Radnor, PA) for 16 h, and then centrifuged at 2000*g* for 15 min (IEC HN-SII Centrifuge; Damon/IEC Division; Needham, MA). The ether layer was collected. Extraction was repeated two more times as above, except 50 mL ether were used for each extraction and extraction time was 30 min. The three extracts were combined, concentrated to 200 mL using a Vigreux column (43 °C) and stored at −70 °C until subjected to solvent-assisted flavor evaporation (SAFE) as described by Rotsatchakul, Chaiseri, and Cadwallader (2007).

2.5. Fractionation of aroma extracts

The SAFE extract from above was subjected to compound class fractionation. First, the extract was washed with aqueous sodium carbonate ($NaCO_3$) (5% w/v; 3 × 20 mL) to separate the acidic compounds (aqueous phase) from the neutral/basic compounds (ether phase). The aqueous phase was acidified with HCl (~4 N) to pH 2 and extracted with ether (3 × 20 mL) to yield the acidic (A) fraction. The neutral/basic fraction from above was extracted with HCl (0.5 M, 3 × 20 mL) to separate the neutral (N) compounds (ether fraction) from the basic (B) compounds (aqueous fraction). The aqueous phase was made alkaline (pH 9) with NaOH (~2 N) and then basic volatiles were extracted into ether (3 × 20 mL).

Each fraction from above was washed with saturated NaCl solution (2 × 10 mL), condensed to 10 mL using a Vigreux column (43 °C) and then dried over anhydrous Na_2SO_4 (2 g). Extracts were then further concentrated to 2 mL using a Vigreux column (43 °C) and stored at −70 °C. Extracts were concentrated to 0.2 mL under a gentle stream of nitrogen prior to analysis.

2.6. Gas chromatography-olfactometry (GCO)

The GCO system consisted of a 6890 GC (Agilent Technologies Inc., Santa Clara, CA) equipped with a flame ionization detector (FID), a sniff port (DATU Technology Transfer, Geneva, NY) and cool on-column injector (+3 °C oven tracking). Each extract (2 μL) was injected into a capillary column (either Stabilwax®-DA, 15 m length × 0.32 mm i.d. × 0.5 μm film; or RTX®5, 15 m length × 0.32 mm i.d. × 0.5 μm film; Restek, Bellefonte, PA). GCO oven temperature was programmed from 40 to 225 °C at a rate of 10 °C/min with initial and final hold times of 5 and 40 min, respectively. Carrier gas was helium at a constant flow of 2 mL/min. Column effluent was split 1:1 between sniff port and FID using 0.15 mm i.d. deactivated capillary columns of equal length (1 m). FID and sniff port block temperatures were 250 °C.

2.7. Aroma extract dilution analysis (AEDA)

AEDA was performed on 1:3 v/v dilutions using the GCO conditions described above according to Zhou, Wintersteen, and Cadwallader (2002). GCO was performed for 25 min by three trained panelists. Panelists had received at least 40 h of training in the GCO technique by exposure to standard solutions of aroma compounds as well as experimental aroma extracts. Flavor dilution (FD) factors reported in Table 2 are the results of one panelist who completed all dilutions. Only compounds with FD-factors ⩾3 for raw almonds and FD-factors ⩾9 for dry and oil roasted are reported.

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