Contribution of Volatile Compounds to the Characteristic Aroma of Baked ‘Jewel’ Sweetpotatoes

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ABSTRACT. Breeding sweetpotatoes [*Ipomoea batatas* (L.) Lam.] for improved flavor would be greatly facilitated by understanding the flavor chemistry of the crop. To ascertain the chemical composition of the aroma, an aroma extract of baked ‘Jewel’ sweetpotatoes was obtained using a cold solvent trap system and analyzed by gas chromatography (GC), gas chromatography–mass spectrometry (GC–MS) and gas chromatography olfactometry (GCO) using aroma extract dilution analysis (AEDA). GC with a flame ionization detector (GC–FID) revealed ≈60 compounds presented in the aroma extract, of which 48 were identified. Olfactory evaluation of the eluted compounds using GC with a thermal conductivity detector (GC–TCD) indicated the presence of 37 odor-active peaks in the aroma extract. Three compounds, phenylacetaldehyde (perfume), maltol (caramel), and methyl geranate (2,6-octadienoic acid, 3,7-dimethyl-, methyl ester) (sweet candy) possessed the highest flavor dilution (FD) values (1500) via AEDA. 2-Acetyl furan (baked potato), 2-pentyl furan (floral), 2-acetyl pyrrole (sweet, caramel), geraniol (sweet floral), and β -ionone (violet) had FD values of 1000. These compounds are thought to be the most potent odorants in baked ‘Jewel’ sweetpotatoes. Additionally, 1,2,4-trimethyl benzene, 2-furmethanol, benzaldehyde, 5-methyl-2-furfural, linalool, isopulegone, n-decanal, 2,4-decadienal, octyl ketone, α -copaene, 4-decanolide, and one unidentified compound were also contributors to the aroma. There was not a character impact compound that comprised the basic baked sweetpotato aroma. The aroma appeared to be made up of a relatively complex mixture of compounds. Maillard and/or caramelization reactions, Strecker degradation of phenylalanine, lipid and carotenoid degradation, and the thermal release of glycosidically bound terpenes appear to be involved in the formation of the characteristic aroma of baked ‘Jewel’ sweetpotatoes.

The sweetpotato (*Ipomoea batatas*) represents the seventh most important food crop in the world (Food and Agr. Organization, 1997). Despite exceptional nutritional attributes (Kays and Kays, 1998) and distinct agronomic advantages (e.g., yield potential and geographical range), worldwide production has not increased significantly in recent decades and in the United States, per capita consumption has declined tremendously since the early 1930s (Edmond and Ammerman, 1971). It has been proposed that the very dominant flavor of the cooked product limits its popularity and has been a critical factor mediating the decline in consumption (Kays, 1985, 1988). In addition to the dominant flavor, the availability of only one flavor-type in the United States has impeded the development of new, uniquely flavored processed products.

There is a tremendous range in flavor diversity within the sweetpotato gene pool (Kays and Horvat, 1984; McLaurin and Kays, 1992), such that integration of an analytical technique for the rapid screening of clones for flavor into sweetpotato breeding programs would enhance the rate at which flavor quality could be improved. The availability of sweetpotatoes with a lower flavor impact would increase the level of acceptance of the crop as a staple food and increase its adoption in new geographic areas and by additional social groups. Determining the primary compounds that make up the characteristic flavor of cooked sweetpotatoes is an essential step in facilitating the development, via plant breeding, of improved conventional sweetpotatoes and new flavor types selected for specific consumer populations and uses.

The flavor of cooked sweetpotatoes is derived from the combination of taste and aroma. The predominant taste is sweetness, due primarily to maltose and sucrose (Horvat et al., 1991; Kays and Horvat, 1984). Likewise, a cross-section of baked sweetpotato volatile compounds have been identified (Horvat et al., 1991; Kays

and Horvat, 1984; Purcell et al., 1980; Sun et al., 1993, 1994, 1995; Tiu et al., 1985). The contribution of critical volatile constituents to the overall aroma of baked sweetpotatoes, however, has not been established, although maltol was found to be an important contributor to the aroma of baked North American-type sweetpotatoes (Sun et al., 1995).

With technological advances in analytical instrumentation, it is not unusual to find several hundred volatile compounds emanating from fruits and vegetables. Since the characteristic aroma of a number of crops is often derived from only a very small number or even a single volatile compound (Kays, 1991), the correlation of volatile constituents with sensory analysis is essential to determine which odor-active compounds are important. Gas chromatography olfactometry (GCO) is a powerful tool for identifying and assessing the importance of odorants. Three methods are commonly employed to assess the contribution of individual compounds: aroma extract dilution analysis (AEDA), CharmAnalysis, and Osme (Acree, 1997). With AEDA, the contribution of odor-active compounds to the overall food aroma is estimated by ranking the odor activities of single odorants on the basis of their odor thresholds in air (Grosch, 1993). AEDA has been used to determine critical odorants in a diverse array of food products [e.g., wheat and rye bread crust (Schieberle and Grosch, 1987), soybean oil (Guth and Grosch, 1990), fresh and stored lemons (Schieberle and Grosch, 1988), beer (Schieberle, 1991a), cooked popcorn (Schieberle, 1991b), coffee brews (Semmelroch and Grosch, 1996), and roasted chicory roots (Baek and Cadwallader, 1998)].

The objectives of this study were to identify the odor-active compounds and ascertain their relative contribution to the aroma of baked ‘Jewel’ sweetpotatoes using dilution analysis. These results, combined with the quantitative analytical technique developed previously (Sun et al., 1993), are designed to provide objective indices for the development of new cultivars with improved flavor and in turn, increase consumer acceptance and consumption of sweetpotatoes.

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Materials and Methods

SAMPLE PREPARATION. 'Jewel' sweetpotatoes, grown at the University of Georgia Horticultural Farm (Athens, Georgia) using standard sweetpotato production practices (Granberry et al., 1990), were harvested, cured at 29 °C and 95% relative humidity (RH) for 7 d, and stored at 15 °C, 85% RH until analysis. Cross-sectional cores (1.7 cm i.d.) from five representative roots (7 to 9 cm in diameter) were removed and peeled immediately before baking. Three hundred and fifty grams of sweetpotato cores were placed inside a specially constructed 1-L glass container in an electric convection oven at 204 °C and held at that temperature for a total baking and volatile collection time of 70 min.

VOLATILE COLLECTION. The volatile collection apparatus and procedures were used as described previously (Sun et al., 1993) with minor modification. Volatiles produced during baking were collected with a cold solvent trap system in a glass sample container (3 cm i.d. × 75 cm high) containing ice-cold redistilled methylene chloride (J.T. Baker, Phillipsburg, N.J.) as the solvent. Helium carrier gas transported the volatiles emanating from the sample to the cold solvent trap. One hundred and forty milliliters of methylene chloride containing 2.25 µg ethyl benzoate as an internal standard was used to trap the volatiles. An additional 10 mL of methylene chloride was used to rinse the trapping chamber at the end of the collection period. The solvent was concentrated using a Kuderna-Danish evaporator/concentrator in which the sample flask was heated using a steam bath. The sample was reduced to 5 mL, and then was concentrated to 0.2 mL by slowly flushing it with purified nitrogen gas. Five microliter aliquots were immediately chromatographed.

GAS CHROMATOGRAPHY ANALYSIS. Analyses were performed with a gas chromatograph (GC) (HP 5890; Hewlett-Packard, San Fernando, Calif.) with a split-splitless injection port temperature of 200 °C and the flame ionization detector (FID) at 250 °C. Separations were made on a 15 m × 0.53 mm i.d. fused silica column coated with DB-1 (Alltech Associates, Deerfield, Ill.). The samples were injected in the splitless mode (35 °C) with a purge time of 0.5 min and held at that temperature for 2 min, after which the oven temperature was increased to 220 °C at 3 °C·min⁻¹ and held at that temperature for an additional 10 min. Signal output was integrated using an integrator (HP 3396 Series III, Hewlett-Packard) to quantify the area under each peak.

GAS CHROMATOGRAPHY-MASS SPECTROSCOPY ANALYSIS. A GC-mass spectrometer (MS) system (HP 5985B; Hewlett Packard) was used to identify the volatile compounds. MS conditions were: ion source 200 °C; electron energy 70 eV; multiplier voltage 220 V; GC-MS interface zone 300 °C; and a scan range of 40 to 400 atomic mass units. Identities when possible were confirmed using authentic standards for retention time and odor description.

GAS CHROMATOGRAPHY OLFACTORY. An aroma extract dilution analysis (AEDA) (Grosch, 1993) was used to create an aromagram of the aroma extract of the baked sample. A series of increasingly diluted solutions of the aroma extract were analyzed by GCO evaluation at the GC-thermal conductivity detector (TCD) exhaust port with the same column and conditions as the FID analysis. Using standards, the retention times were determined to be identical between TCD and FID techniques. Since the dilution value obtained for each compound is proportional to its odor activity value (OAV), those compounds persisting at the higher dilutions were considered to be major contributors to the overall aroma of the extract. The flavor dilution (FD) value is the last dilution at which an odor-active compound was detected by at least two of the three panelists. A

series of dilutions (10%, 1%, 0.5%, 0.25%, 0.125%, 0.1%, 0.065%, 0.05%, and 0.025%) were prepared from the original extract using redistilled methylene chloride.

Three trained panelists evaluated the aroma of the individual components in the GC-TCD effluent giving descriptors of the odor active compounds. All odors reported were detected in the original undiluted extract. Three replications of each dilution were used to ascertain the odor activity level.

Results and Discussion

The volatile extract obtained had a pleasant baked sweetpotato odor similar to the cooked product. A chromatogram of the volatile extract is illustrated in Fig. 1, with peak numbers corresponding to the compounds listed in Table 1. At least 60 compounds were present in the extract, of which 48 were identified. The area for the 48 peaks represented >96% of the total peak area of the chromatogram (excluding solvent). Quantitative estimates for individual compounds (Table 1) were calculated from the ratio of the peak area to an internal standard (ethyl benzoate) and related to the fresh weight (FW) of sweetpotato. The volatiles identified in this study included heterocyclic compounds, aldehydes, alcohols, ketones, aromatic hydrocarbons, terpenoids, and acids and included 24 compounds not reported previously from baked or fresh sweetpotato storage roots.

GCO evaluation demonstrated the presence of 37 odor-active peaks in the aroma extract for which the aroma descriptors are listed in Table 1. Singly, none of the peaks were representative of the characteristic aroma of a baked sweetpotato. The aroma appeared to be an integration of a relatively complex mixture of aroma compounds. In several instances, compounds were given odor descriptors that differed depending upon the dilution, indicative of the well known effect of concentration on aroma. Odor perception is also known to be affected by numerous physiological and environmental factors (Kays and Wang, 2000). Sensory panelists were consistent, however, in their ability to detect compounds in diluted solutions and assign the appropriate descriptor.

Of the odorants present, phenylacetaldehyde, maltol, and methyl geranate possessed the highest FD factors (i.e., 1500). Methyl geranate eluted just before the germacrene D. Due to the intensity of methyl geranate, a small portion of the odor carried over into the GC effluent for germacrene D. Substantiation of the odor of germacrene D and β -caryophyllene with authentic standards indicated they were essentially odorless at the concentrations present in the extract (Table 1). Compounds which had FD factors of 1000 included 2-acetyl furan, 2-pentyl furan, 2-acetyl pyrrole, geraniol, and β -ionone. In contrast, 1,2,4-trimethyl benzene, 2-furmethanol, benzaldehyde, 5-methyl-2-furfural, linalool, 2,4-decadienal, octyl ketone, α -copaene, 4-decanolide, and an unknown had FD factors between 100 to 800. All of these compounds appear to contribute to the aroma of baked 'Jewel' sweetpotatoes.

Maltol, a well-known Maillard reaction product with a caramel-like aroma, was demonstrated previously to be an important component in the aroma of baked sweetpotatoes (Sun et al., 1995) and the current study conformed its importance. Maltose is an efficient precursor of maltol during heating when in the presence of amino acids (Tressl et al., 1981, 1989), however, maltol can also be formed via the caramelization of maltose and certain maltooligosaccharides (Specht and Baltes, 1994). Maltol enhances the flavor of soft drinks, fruit juices, and carbohydrate-rich foods and is generally recognized as a food flavoring substance (Hodge, 1967).

Maltose also represents a primary precursor for a cross-section

of other volatile compounds emanating from baked sweetpotatoes [e.g., 2-furmethanol, 5-methyl-2-furfural, 2-acetyl furan (Sun et al., 1994) and 2-acetyl pyrrole] which are thermally generated via the Maillard reaction. A generalized pathway for Maillard reaction volatiles in baked sweetpotatoes has been presented by Sun et al. (1995).

Phenylacetaldehyde gives a perfume aroma note and is formed via Strecker degradation of phenylalanine. It is found widely in many flavors including fruit, chocolate, and honey (Scarpellino and Soukup, 1993). Phenylacetaldehyde has a high FD factor in cooked beef (Gasser and Grosch, 1988) and has been found in a cross-section of other foods (Gasser and Grosch, 1990; Munch et al., 1997). In contrast, benzaldehyde has a strong nutty, almond odor (FD = 800) though it may also originate from the same amino acid.

Methyl geranate, a relatively little known monoterpene ester in foods, had a pronounced influence on the aroma of baked sweetpotato. Likewise, several other terpenes are also important contributors. For example, the monoterpene alcohols, geraniol and linalool, provide sweet and floral notes, respectively. They are thought to be key compounds contributing to the aroma of Kansho-shochu, a distilled sweetpotato spirit (Ohta et al., 1990) and are found in the fusel oil of crude ethanol made from sweetpotato (Taira, 1963a, 1963b). The sesquiterpene, α -copaene, was responsible for an

earthy, baked potato aroma and has been reported previously in both baked roots (Horvat et al., 1991) and fresh leaves (Nottingham et al., 1989). Additionally, β -farnesene, α -bisabolene, and nerolidol were odor-active, giving sweet, woody, and rose-like aromas, respectively. These terpene compounds have also been found to be important odor-active volatiles in a number of fruits and vegetables.

Both free and nonvolatile glycosidically bound terpene compounds are present in fruits and vegetables (Williams, 1993), including raw sweetpotato (Ohta et al., 1990). In the raw storage roots, terpene alcohols are present primarily in the form of β -glucoside conjugates which can be readily hydrolyzed by β -glucosidase. A fungal-derived, immobilized endo- β -glucosidase, for example, has been used to increase the concentration of volatile terpenes in wine and passion fruit juice (Shoseyov et al., 1990). The glucosidic bond can also be broken with exposure to high temperatures, e.g., tomato (*Lycopersicon esculentum* Mill.) (Buttery et al., 1971). Kinugasa and Takeo (1990) demonstrated that terpenes in tea when infused with pectinase were derived from their respective glycosides. Since two of the important odor-active monoterpene alcohols in cooked 'Jewel' sweetpotatoes do not emanate from raw roots (unpublished data), they appear to be liberated from nonvolatile glycosides by enzyme hydrolysis and/or heating.

Carotenoids, of which β -carotene predominates in the orange-

Fig. 1. Gas chromatographic (top) and aroma dilution analysis aromagram (bottom) of the headspace volatile extract from baked 'Jewel' sweetpotatoes. (I.S. = internal standard, peak numbers correspond to compounds listed in Table 1).

flesh sweetpotato cultivars grown in North America (Kays, 1992; Simonne et al., 1993), degrade via oxidation and subsequent isomerization, leading to the formation of several odorants with generally low odor thresholds in most products (Kanasawud and Crouzet, 1990; Weeks, 1986). In the sweetpotato, however, β -ionone, a

degradation product of β -carotene, contributes significantly to the aroma (i.e., FD = 1000). β -Ionone with a violet-like aroma has been identified in a wide range of processed food and fresh fruits and vegetables and is used in licorice candy and chewing gum (Arctander, 1969). 1,2,4-Trimethyl benzene, cyclohexanol, 2,2-dimethyl-1,3-

Table 1. Volatiles emanating from baked 'Jewel' sweetpotatoes.

Peak no.	Volatile compound	Concn ($\mu\text{g}\cdot\text{kg}^{-1}$ fresh wt)	Odor descriptor	FD factor
1	Pyridine	5.6	Fishy	10
2	1,2,4-Cyclopentanetriol	1.2	Nt ^z	
3	1,2,4-Trimethyl benzene	2.7	Flowery	800
4	3-Furaldehyde	14.5	Coconut	10
5	Xylene	0.3	Earthy	1
6	2-Furmethanol	14.1	Burnt	100
7	2-Furancarboxaldehyde	1.3	Burnt	1
8	2-Acetyl furan	4.4	Baked potato	1000
9	Benzaldehyde	2.1	Nutty, almond	800
10	5-Methyl-2-furfural	0.9	Sweet	400
11	2-Pentyl furan	1.2	Floral	1000
12	2,3-Pentanedione	0.7	Buttery	10
13	Phenylacetaldehyde	29.7	Perfume	1500
14	Limonene	Tr ^y	Lemon	10
15	3,4-Dihydropyran ^x	2.1	Roasted peanut	1
16	2-Acetyl pyrrole ^x	0.3	Sweet, caramel	1000
17	Maltol	10.8	Caramel	1500
18	Linalool	0.8	Sweet, flower	800
19	Isopulegone ^x	0.8	Cooked starchy	100
20	4,5-Dimethyl-4-hexen-3-one ^x	Tr	Nt	
21	Geraniol	0.4	Sweet floral	1000
22	2,4-Nonadienal	1.2	Watermelon	400
23	2-Naphthalenone ^x	1.2	Nt	
24	Cyclohexanol	5.4	Fruity, sweet	1
25	n-Decanal	Tr	Hot apple	100
26	2,2-Dimethyl-1,3-cyclohexanediol ^x	0.4	Fruity, sweet	1
27	2,3-Nonadecanediol ^x	0.5	Musty, decay peanut	10
28	2,4-Decadienal	0.6	Musty, cooked starchy	400
29	Octyl ketone ^x	Tr	Musty, cereal	100
30	Methyl geranate	Tr	Sweet candy	1500
31	Germacrene D ^x	0.9	Nt	
32	β -Caryophyllene	0.3	Nt	
33	β -Farnesene	0.3	Hot apple, sweet	10
34	α -Copaene	0.3	Earthy, baked potato	800
35	α -Bisabolene	0.3	Balsamic odor	10
36	Bohlmann 176 ^x	1.5	Rose-like	1
37	2(4H)-benzofuranone ^x	1.3	Rose-like	1
38	β -Ionone	1.6	Violet	1000
39	Nerolidol	0.2	Rose-like	10
40	4-Decanolide ^x	Tr	Fruity, peach-like	100
41	Unknown	1.0	Burnt starch	100
42	Tetradecanoic acid	4.2	Waxy, oily	1
43	10-Heneicosene ^x	0.5	Nt	
44	Palmitic acid	54.1	Nt	
45	Octadecanol	2.6	Nt	
46	1-Nonadecanol	24.0	Nt	
47	9,12-Octadecadienoic acid	2.4	Nt	
48	Silane ^x	2.3	Nt	
49	Squalene	0.2	Nt	

^zNt = odor not detected at the concentration present.

^yTr = < 1% based on GC peak areas.

^xTentatively identified by MS.

cyclohexanediol, n-decanal, and benzofuranone can also be formed via the fragmentation of β -carotene (Kanasawud and Crouzet, 1990; Onyewu et al., 1989).

The lipid oxidation products, 2,4-nonadienal and 2,4-decadienal, had watermelon and musty, cooked starch aromas, respectively, and had FD values of 400. Both compounds are decomposition products of linoleic or linolenic acids and have been identified previously in potato chips, cooked rice, cooked beef, corn oil, and wheat bread (Baltes and Song, 1994). 2,4-Decadienal is an important aroma compound in baked white potato (*Solanum tuberosum* L.) (Buttery et al., 1973). The presence of both 2,4-nonadienal and 2,4-decadienal is not surprising since linoleic is the most abundant fatty acid in sweetpotatoes (Bogges et al., 1970). Other lipid oxidative degradation products, such as n-decanal and 4-decanolide, had relatively low FD values (i.e., 100). 2-Pentyl furan, with a high FD factor of 1000, could be formed from 2,4-decadienal (Mandin et al., 1999), which was also identified in boiled potato aroma (Nursten and Sheen, 1974).

Tiu et al. (1985) conducted GCO sensory evaluation of baked 'Jewel' sweetpotato volatiles and reported the presence of two unidentified compounds that were described as having the characteristic sweetpotato aroma. We were unable to identify a specific region of the chromatogram which had a baked 'Jewel' aroma and conclude that there is not a primary character impact compound (i.e., one comprising the characteristic aroma of baked sweetpotato). The aroma appears to be a delicate balance of different classes of chemical compounds.

A detailed study of the mechanisms involved in the synthesis of these critical volatiles would be invaluable in the development of improved traditional U.S. type sweetpotatoes as well as new flavor types. Generally, carbohydrates, amino acids, fatty acids, β -carotene, and storage-forms of terpenoids are precursors for the odorants identified. Among carbohydrates, maltose appears to be the most important for formation of volatiles since the other primary monosaccharides and disaccharides remain essentially unchanged during baking and if maltose production is inhibited, volatile synthesis is greatly repressed (Sun et al., 1994). In addition to monoterpenes, fatty acids and shikimate derived aroma compounds, as well as norisoprenoids derived from carotenoids by oxidative degradation, have been found to accumulate as glycoconjugates and act as flavor precursors in certain fruits (Gerhard et al., 1991; Williams, 1993). The extent to which critical odorants in sweetpotato are derived from glycosides remains to be elucidated.

GCO sensory evaluation indicated that several quantitatively significant components contributed little if at all to the overall aroma. For example, palmitic acid and 1-nonadecanol comprised nearly 20% and 10% of the total peak area, respectively, but were not perceived at the concentrations present. In contrast, several compounds (e.g., methyl geranate and geraniol) that made up only small fractions of the total GC profile were major contributors to the overall aroma.

In sweetpotato breeding programs, flavor is generally given a relatively low priority in the selection process due to the difficulty and subjectivity of the current means of flavor measurement (typically sensory analysis) and a general lack of understanding of flavor chemistry (Kays and Wang, 2000), even though it is considered to be one of the most important traits (Martin and Jones, 1986). An analytical selection protocol for flavor, interfaced with subsequent sensory analyses, is a viable way to greatly increase selection accuracy and the number of clones that can be assessed. This would allow moving flavor earlier in selection sequence and thus imposing a substantially increased selection pressure for a desired flavor type.

The sweetpotato gene pool provides a diverse mosaic of flavors (McLaurin and Kays, 1992) that await use. Qualitative and quantitative characterization of the chemistry of preference of target consumer populations, coupled with improved selection methods will allow exploiting this natural resource, and expand acceptability and use of sweetpotatoes in the diet.

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