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Comparison of Volatile Flavor Components in Fresh and Processed Orange Juices

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Fresh juices from Hamlin, Pineapple, and Valencia oranges and different commercial brands of processed orange juices were analyzed for volatile flavor components by a headspace analysis technique. Twenty components including eight alcohols, four aldehydes, three esters, and five hydrocarbons were identified and quantified. Unpasteurized and pasteurized single-strength juices not made from concentrate did not show marked changes in the profile of flavor components when compared to fresh juice. In contrast, pasteurized reconstituted juices from concentrate showed decreases in acetaldehyde, methyl acetate, methyl butyrate, and ethyl butyrate with increases in decanal, octanal, and linalool. Aseptically packaged single-strength juice, canned juice, and a 10% juice drink exhibited increased α -terpineol. Canned juice and the 10% juice drink also exhibited low levels of ethyl butyrate, acetaldehyde, hexanal, and limonene and total disappearance of ethyl acetate. This procedure has potential for routine monitoring of quality of processed citrus products.

The delicate fresh flavor of orange juice is easily changed by heat treatment during processing or by storage (Shaw, 1986). The juice undergoes compositional changes that invariably cause an alteration in the original flavor and aroma of the fresh juice. In order for processors to better understand the changes that take place during processing and storage of orange juices, quantitative information on the important volatile flavor components present in both fresh and processed orange juices is needed. Such information will enable processors to alter processing conditions and amounts of volatile flavor fractions added to produce products with flavor profiles more closely resembling those in fresh juice than is currently possible.

Of the volatile components important to flavor, esters and aldehydes are the primary contributors to fresh orange flavor (Bruemmer, 1975), although other components could also be important (Shaw, 1977). Other factors that influence the flavor are correct proportions of the different compounds (Shaw, 1979), taste threshold values of volatiles (Patton and Josephson, 1957), synergistic effects between volatiles (Shaw and Wilson, 1980), and the interaction of nonvolatile with volatile flavor components (Ahmed et al., 1978b).

Only recently have analytical methods become available to accurately quantify trace volatile constituents in citrus juices. Schreier (1981) quantified 29 volatile constituents of one fresh orange juice and showed quantitative changes in some constituents as well as the appearance of two new constituents after heat treatment of the juice. Solvent extraction and column chromatography

were necessary steps prior to gas chromatographic (GC) analysis. Moshonas and Shaw (1987) quantified 24 volatile components of one fresh sample each of Valencia and Temple orange juices that had been distilled prior to GC analysis. Marsili (1986) used headspace analysis of a diluted orange juice sample to quantify nine volatile components of one processed juice sample. Rodriguez and Culbertson (1983) quantified eight volatile components of one fresh, one freeze-concentrated, and one heat-concentrated orange juice sample by GC analysis using a radioactive detector. Because only a single fresh or processed juice sample was analyzed in each of the above cases, none of those studies showed a range of quantitative values for individual flavor components present in either fresh or processed juices. Such a range of values determined on a variety of juice samples is needed to assess effects of quantitative changes due to processing on the loss of fresh flavor quality.

In the current report, quantitative values for 20 volatile flavor components of 15 fresh orange juices and 14 juices from major types of processed orange juice products were compared. By determining quantitative values for volatile components from several juice samples, we now have a better perspective on the quantities present in both fresh and processed juices.

MATERIALS AND METHODS

Juice Samples. Fresh juice samples were hand-extracted from Pineapple, Hamlin, and Valencia oranges (*Citrus sinensis* (L.) Osbeck) with use of a domestic mixer fitted with a reamer.

The extracted juice was screened to separate the seeds and pulp from the juice. Commercial processed juice samples used as indicated in Table I were as follows: fresh-squeezed = commercial unpasteurized juice; pasteurized (1 and 2) = pasteurized single-strength juice (not from concentrate) in cardboard cartons; frozen concentrated (1-3) = frozen concentrated orange juices (FCOJ) reconstituted to single-strength juice for analysis; aseptic concentrate = concentrated juice packaged aseptically in flexible multilayered carton and reconstituted to single-strength juice for analysis; reconstituted from concentrate = pasteurized reconstituted juice from concentrate in glass, cardboard carton, aseptic flexible multilayered cartons (1 and 2), and aluminum or tin cans; juice drink = a 10% orange juice drink packaged aseptically in flexible carton.

All commercial processed juice and drink samples were purchased from local markets except for aseptic juice 1, which was obtained directly from a processing line and kept at -18°C until analyzed.

Analysis of Volatile Components. Juice samples (2 mL each) were transferred to 10-mL vials equipped with crimp-top caps with TFE/silicone septa seals. Volatile flavor components were determined by headspace analysis on a Perkin-Elmer Model 8500 gas chromatograph with an FID detector and a Model HS-6 headspace sampler. A 0.53 mm \times 30 m polar Durowax column (1.0- μm film thickness) (J&W Scientific, Folsom, CA) was used with a 6.0-psi helium head pressure (81 cm/s linear gas velocity). Juice samples were equilibrated in the headspace sampler for 15 min at 80°C prior to injection. Injection parameters for the headspace sampler were 0.5-min vial pressurization time followed by 0.02-min injection time. Column oven temperature programming was 40°C for 6 min and then raised at $6^{\circ}\text{C}/\text{min}$ to 180°C . The FID detector amplifier range setting was for high sensitivity, and the temperature was 250°C . The different components were identified by comparison of retention times with those of standards and by enrichment of juice with authentic samples. Concentrations were calculated with use of regression equations, determined by injecting five different concentrations of each component added to a juice base to obtain a peak height calibration curve. The standard concentrations (ppm) were the following: acetaldehyde, 1.6-8.0; ethyl acetate, 0.4-2.0; methanol, 20-100; ethanol, 100-1500; methyl butyrate, 0.02-0.10; α -pinene, 0.6-3.0; ethyl butyrate, 0.2-1.0; hexanal, 0.04-0.20; isobutyl alcohol, 0.06-0.30; sabinene, 0.06-0.30; γ -terpinene, 0.08-0.40; octanal, 0.12-0.60; hexanol, 0.2-1.0; *cis*-3-hexenol, 0.3-1.5; *trans*-2-hexenol, 0.03-0.15; decanal, 0.4-2.0; linalool, 0.8-4; α -terpineol, 1-5; valencene 1.4-7. All determinations were carried out in triplicate. The juice base was prepared by reconstitution to 11.8° Brix of concentrated juice (pumpout) from an evaporator that contained no added flavor fractions.

Statistical Analysis. Data for the different components were analyzed by analysis of variance using the General Linear Model (GLM) procedure, a package program of the Statistical Analysis System (SAS Institute Inc., Cary, NC). Specific differences were determined by least significant difference (LSD). All comparisons were made at a 5% level of significance.

RESULTS AND DISCUSSION

Quantitative values determined for 20 components of fresh and processed orange juices are listed in Table I. Fifteen samples were used to establish a mean and range of values for each component in fresh juice (columns 1 and 2 of Table I). Juices from mature Hamlin, Pineapple, and Valencia cultivars harvested at different times during a season were used to determine this data base for fresh juice volatile components. Literature values for these components, where available, are listed in column 3 for comparison. Thirteen processed commercial orange juice samples and one 10% juice drink were analyzed, and the quantities of the same 20 volatile components were determined for comparison with fresh juice values. The processed juice samples are listed in Table I from left to right in decreasing order of fresh juice flavor, as judged subjectively by us. Several volatile aldehydes,

esters, alcohols, and hydrocarbons known to contribute to the desirable flavor of orange juice (Ahmed et al., 1978a) were quantified in this study.

Aldehydes. Variation in aldehyde content among the processed products is shown in Table I. Generally, the better flavored products (as judged by us), unpasteurized juice and juice not made from concentrate (fresh-squeezed and pasteurized 1 and 2), showed total aldehyde and acetaldehyde levels most closely resembling those in fresh juice. Samples with flavors least like that of fresh juice, canned juice (tin) and a 10% juice drink, had the lowest levels of acetaldehyde and total aldehydes. The three frozen concentrated juices showed considerable variability in total aldehyde levels, reflecting the variable flavor quality in these products. Hexanal is not believed important to fresh orange juice flavor, except for some possible contribution to a green flavor note (Arctander, 1969) mainly provided by hexenols (see Alcohols).

Octanal and decanal are generally considered important contributors to orange flavor (Arctander, 1969; Boelens and van Gemert, 1987), and one of the standards of identity for orange peel oil is its aldehyde content (mostly octanal and decanal). However, Ahmed et al. (1978a) found decanal to make a negative contribution to orange juice flavor at the level tested (0.72 ppm). In the current study, fresh orange juice had relatively low levels of both octanal and decanal, with most processed juice products having considerably higher levels of both aldehydes than found in fresh juice. The relatively high levels of peel oil in most processed juices (see Hydrocarbons) probably account for the high levels of octanal and decanal in those samples.

Aseptically packaged juices often have relatively low quantities of volatile flavor components after storage required for normal distribution to the consumer (Moshonas and Shaw, 1989a,b). The polyethylene liner of the package can absorb some of the flavor components, and because of this, oil is usually added to the juice at a level of 0.020-0.025% to achieve a desired level of 0.010-0.015% when the juice is consumed (Flora, 1988). The two aseptically packaged single-strength juices reconstituted from concentrate (aseptic 1 and 2 in Table I) had high levels of acetaldehyde and total aldehydes for reconstituted juice products. Aseptic 1 juice was obtained from the processor the same day it was packaged and kept frozen until analyzed. Aseptic 2 juice was a product of the same brand purchased from a local market. This product, which had been stored for 9 weeks, contained levels of volatile constituents similar to those present in sample 1. The two juices were not from the same lot, however.

Esters. The three esters quantified in this study, ethyl acetate, methyl butyrate, and ethyl butyrate, are known to contribute to the "top-note" of fruit flavors, including citrus (Arctander, 1969). Ethyl butyrate is generally the major volatile ester in orange juices and orange flavor fractions and is an important contributor to desirable flavor in orange products (Ahmed et al., 1978a). Reports on the composition of freeze-concentrated orange juice have suggested the ethyl butyrate content in fresh orange juice varies widely, perhaps as much as 400-fold (Strobel, 1983, 1984). Our data indicate a variation of approximately 4-fold for the fresh juice samples reported in Table I. In processed juices, less than half of the samples analyzed contained amounts of ethyl butyrate within the range found for fresh juice, with all other samples having smaller amounts. A general decrease in ethyl butyrate, as well as in total esters, was observed with decreasing quality

of the product type. These differences could be a major factor in the fruity top-notes present in fresh juice that are often missing in processed orange juice products. Ethyl acetate and methyl butyrate were present in most processed juices at levels within the range found in fresh juice. The one notable exception was the unusually high level of ethyl acetate present in the commercial unpasteurized sample (fresh squeezed in Table I). Enzyme activity, still present in this unpasteurized juice sample, may be responsible for the high level of this ester.

Alcohols. Quantities of eight alcohols in fresh juice and in the different commercial brands of processed juices are depicted in Table I. The fresh-squeezed juice contained the highest level of methanol followed by one aseptically packaged reconstituted juice (aseptic 1), pasteurized single-strength juices not from concentrate (1 and 2), one FCOJ sample (1), and two of the pasteurized reconstituted juices from concentrate (carton and aseptic 2). The rest of the samples contained very small amounts (<10 ppm) of methanol. The methanol level was highest in the fresh-squeezed juice, undoubtedly because the pectinmethylesterase enzyme, still active in this unpasteurized juice, demethylated some pectin and liberated methanol in the process.

The highest level of ethanol was detected in one of the aseptically packaged reconstituted juices from concentrate (aseptic 1). Variable amounts were present in the other juice samples, the lowest being detected in the tin-canned juice from concentrate. In all except two samples (FCOJ 3 and tin-canned) the ethanol contents were within the rather broad (>10-fold) range found in fresh juice. The main function of ethanol in synthetic flavorings and perfumes is to act as a solvent and to provide a lift to other aromas (Arctander, 1969), and it probably performs a similar function in orange juice products.

Trace levels of 2-methyl-1-propanol were determined in most samples except for the aseptic concentrate, juice drink, and the two canned juices. Only the fresh-squeezed juice contained an appreciable level of hexanol. These two alcohols at the low levels present probably do not contribute appreciably to orange flavor.

Two unsaturated alcohols, *cis*-3-hexenol and *trans*-2-hexenol, are important contributors to the green, leafy top-note in fresh orange flavor and in other fruit flavors. *trans*-2-Hexenol has a more desirable sweet, fruity flavor than does *cis*-3-hexenol (Arctander, 1969). The fresh and processed juices in this study generally contained a much higher level of *cis*-3-hexenol than of *trans*-2-hexenol. The fact that a preponderance of *cis*-3-hexenol over *trans*-2-hexenol was maintained in most processed juice samples is probably because aqueous orange essence recovered from fresh juice during the concentrating process is used to provide these volatile flavor components that add the green flavor and other top-notes to processed juices. Commercial unpasteurized fresh-squeezed juice was the one exception where *trans*-2-hexenol was present in slightly greater quantity than was *cis*-3-hexenol.

Linalool is present in varying amounts in orange peel oils (Shaw, 1979). In fresh juice and in two of the highest quality processed juices (pasteurized 1 and 2 of Table I), only traces of this alcohol were found. In other processed juices, the linalool content, ranging from 0.37 to 4.3 ppm, was much higher than that found in fresh juice.

α -Terpineol was not detected in fresh juice nor in most processed juices. It is a degradation product of limonene, the major orange oil constituent, and is a known contributor to off-flavor in orange juice at levels of 2 ppm or higher (Tatum et al., 1975). It was found in appreciable

quantity in the juices (aseptic sample 2 and canned) and 10% juice drink stored at room temperature. Only in the 10% orange juice drink did it exceed the detectable level of 2 ppm.

Ethanol was by far the major alcohol present in all samples, and its amount varied widely and inconsistently with product quality. Thus, the quantities of total alcohols in fresh and processed juices and drinks showed no consistent changes.

Hydrocarbons. Four hydrocarbons were quantified in Table I by headspace gas chromatography, and a fifth hydrocarbon, limonene, was quantified by the Scott oil determination method (Scott and Veldhuis, 1966). Since limonene was not soluble in the standard aqueous ethanolic mixture used for GC calibration at the relatively high level needed, its value had to be determined independently. The presence of a sixth hydrocarbon, myrcene, was also detected in all juice samples. This component, however, was not quantified due to its insolubility in the ethanol mixture of standards.

α -Pinene was judged by Ahmed et al. (1978a) to make a positive contribution to flavor in processed orange juice. Since it is a constituent of peel oil and of juice oil, its level should depend on the oil content in the juice. Values in Table I generally confirm this trend. Thus, juices with high oil contents, as indicated by limonene contents in Table I, had high α -pinene contents, while those with low oil contents had generally low levels of α -pinene. Fresh juice, with a low total oil content, showed only a trace of α -pinene.

Sabinene and γ -terpinene are also components of orange peel oil. They were both present at consistently low levels in most juice samples. Their importance in orange flavor has not been determined. γ -Terpinene has a citruslike aroma, while sabinene has a warm, spicy aroma and flavor (Arctander, 1969). Valencene is a trace component of orange peel oil but is found in much higher quantities in orange juice oil (Hunter and Brogden, 1965). Its quantity in fresh juice was highly variable as was its level in processed juices and did not correlate with peel oil content. In Table I, the valencene content was highest in fresh juice and in processed juices not made from concentrate, reflecting the contribution of juice oil to total oil components present in these juices. Valencene possesses a weak, citruslike aroma and may contribute to orange flavor, but its flavor threshold level has not been reported.

Limonene is the second most abundant volatile component in orange juice after ethanol. Its amount in hand-reamed orange juices (Table I) was lower than that found in any of the 100% juice samples analyzed. Ahmed et al. (1978a) found limonene to be an important contributor to orange flavor when added at a level of 190 ppm (0.019%) to processed juice. The optimum level in processed juice is 0.015–0.020% peel oil (>95% limonene) (Carter, 1985). From the current study and an earlier report (Rice et al., 1952), the peel oil content of hand-extracted juice is lower than that in most commercial orange juice products. Increasing the peel oil content also increases the levels of other oil-soluble flavor constituents, e.g., octanal, decanal, linalool, and α -pinene.

Profiles of Volatile Components. The GC profiles obtained for the different types of orange juices demonstrate the quantitative variability of volatile flavor components in processed orange juice. The fresh-squeezed unpasteurized juice exhibited a profile of volatile components closest to that of fresh orange juice. Pasteurization or mild heat during processing of single-strength juice

(pasteurized 1 and 2) caused relatively small changes in the volatile flavor composition. The most profound changes in volatile components occurred in juices that were reconstituted from concentrates, packaged by aseptic means or in cans, and stored at room temperature. Most reconstituted juices showed reductions in acetaldehyde, ethyl acetate, methyl butyrate, and ethyl butyrate with slight increases in decanal and octanal. These compounds are thought to be primary contributors to fresh orange flavor (Flora, 1988; Bruemmer, 1975). The increased levels of linalool in the reconstituted juices could reflect addition of high amounts of peel oil by processors and could also contribute to objectionable flavor if present at levels higher than 8 ppm (Murdock et al., 1967).

Canned juices were low in many of the important volatile components. Acetaldehyde and ethyl butyrate were reduced substantially, accompanied by almost total losses in ethyl acetate and methyl butyrate and large increases in α -terpineol. These differences can be ascribed to the fact that canned juices receive more heat input during pasteurization, they remain at relatively high temperatures for extended periods of time (Varsel, 1980), and aqueous essence, which contains many desirable volatile flavor constituents, is not added to these products.

The composition and flavor of the 10% orange juice drink was markedly different from that of any of the processed 100% orange juice products. The absence of important flavor components and the presence of high levels of linalool and α -terpineol in the orange juice drink account for its poor flavor quality relative to fresh orange juice.

The headspace analysis technique used in this study is fast and reproducible and could be used for routine monitoring of citrus product quality. Data presented herein could serve as a guide to the citrus juice industry where production of orange juice with optimum flavor quality is of increasing importance.

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