Lipid Composition of Commercially Canned Single-Strength Orange Juice

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ABSTRACT

The lipid composition of commercially canned single-strength orange juice ranged from 84-101 mg/100 ml juice (overall mean 95 ± 6). Phospholipid phosphorus, expressed as mg/100 ml juice, showed a range of from 1.56-1.95, while phospholipid phosphorus/lipid values (as μg-P/mg lipid) were within a very narrow range, 18.9 ± 1.1. The percentage distribution of lipid classes in these juices was 24-35% neutral lipids, 18-23% resin acids and glycolipids, and 43-53% phospholipids and other polar lipids. Five fatty acids, i.e. C_{16}, $C_{16:1}$, $C_{18:1}$, $C_{18:2}$, and $C_{18:3}$, accounted for over 93% of all fatty acids. The relative percentages of $C_{18:2}$ and $C_{18:3}$ differed between seasonal juices. The lipid composition does not warrant inclusion in nutritional labeling; however, lipid levels may be useful in detecting adulteration.

INTRODUCTION

Recent interest in nutrients in food products and in product quality has increased the need for information on the lipid composition of processed orange juice. There is a paucity of available information on total lipid composition of canned single-strength orange juice (SSOJ). Such information is needed to help assess the total nutritent value of orange juice products. Careful analyses of the lipid fraction also could provide information useful in assessing probability of adulteration in suspicious samples.

Early work on the lipid content of citrus juices (1,2) was limited, because these investigations lacked a quantitative procedure for the extraction of total lipids. Recognizing this limitation, Swift (3) devised a procedure for the removal of lipid containing material from orange juice, based upon the association of the lipid materials of citrus juices with the suspended matter (cloud).

Investigations by Swift and coworkers, (4-9) during the early 1950's on processed SSOJ were concerned mainly with delineating the juice's lipid components and the effects of these lipids on off-flavor development. More recent studies by other investigators on commercial SSOJ dealt with the effects of storage conditions upon the lipid composition (10) and the identification and estimation of phospholipids (11,12).

Because of intensified interest in the nutritional value of orange juice and the possible role of lipids as indicators of adulteration (12), the current study on lipid composition of SSOJ was undertaken. Lipids were monitored in SSOJ obtained from several commercial packers during the 1972-73 processing season.

EXPERIMENTAL PROCEDURES

Materials

Commercially processed, canned SSOJ was obtained from three processors during the early (November-January, mostly Hamlin orange juice) and mid- (January-March, mostly pineapple-orange juice) seasons and from four processors at two stages during the late season (April-May and May-July, mostly Valencia orange juice). The late season was divided, since there are usually flavor differences between early and late Valencias. SSOJ in 46 oz cans was taken directly from the production line in the processing plant and placed in a laboratory locker at -18 C.

Lipid Extraction, Purification and Subfractionation

Batches of 100 ml juice were taken from 46 oz cans of SSOJ, and lipids were extracted by a modification (13) of the method of Swift (3) and purified by the method of Wuthier (14). Phospholipid phosphorus was determined on the purified lipid by the method of Bartlett (15). The purified lipids were fractionated into neutral lipids, resin acids and glycolipids, and phospholipids and other polar lipids by silica gel chromatography. Columns used in separation were 30 cm long, 0.9 cm inside diameter and contained 10 g silica gel (60-200 mesh, J.T. Baker Chemical Co., Phillipsburg, N.J.). Columns were prewashed with 100 ml 0.3% acetic acid in $CHCl_3$ prior to lipid fractionation. Fractionation was conducted by percolating 70-90 mg lipid in $CHCl_3$ onto the gel column. Neutral lipids were eluted with 200 ml 0.3% acetic acid in $CHCl_3$, and resin acids and glycolipids were eluted with 350 ml acetone. After elution of these two fractions, the silica gel, containing phospholipids and other minor polar lipids, was removed quantitatively from the column and placed in a coarse fritted glass filter funnel. Polar lipids were removed from the gel by batch-wise washing with five 50 ml aliquots of MeOH. The glycolipid and polar lipid fractions were purified further of silica gel fines by Sephadex chromatography. Purity of lipid class separation was attested through thin layer chromatography (TLC) monitoring. Dry wt of the lipid fractions were determined after vacuum drying under desiccation at room temperature. Recovery of total lipid after silica gel and Sephadex chromatography ranged between 92-98%. Lipid class distributions were determined after normalizing lipid recoveries to 100%.

Fatty Acid Methyl Ester Preparation and Gas Liquid Chromatography

Methyl esters of fatty acids were prepared as previously described (10). The methyl esters were determined with a Hewlett Packard model 7610 A high efficiency gas chromatograph equipped with flame ionization detectors. The injection port and detector were at 200 C and 240 C, respectively, and the helium flow rate was 55 ml/min. The column was 3.05 m long, 4 mm inside diameter, glass U-tube packed with 3% SP-1000 coated on 100-120 mesh Gas Chrom Q. Optimum temperature program was 160-200 C at 2 C/min, then 200-240 C at 4 C/min, and held at this upper limite until the C_{26} methyl ester eluted from the column. Methyl ester percentages were calculated with an Autolab System IV computing integrator for chromatography (Autolab, Mountain View, Calif.).

RESULTS AND DISCUSSION

The total lipid extracted from 100 ml juice ranged from 84 mg (plant C, midseason) to 101 mg (plant C, early season; plant B, midseason) (Table I). Table I is divided into four principal categories, i.e., total lipid, phospholipid phosphorus, phospholipid phosphorus/lipid ratios, and percentage distribution of lipid classes. Lipid levels in early Valencia and late Valencia seasons were consistent (range

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TABLE I

Total Lipid, Phospholipid Phosphorus, and Lipid Classes of Single-Strength Orange Juice

Early season	Total lipid mg/100 ml juice	Phospholipid phosphorus mg/100 ml juice	Phospholipid phosphorus/lipid μg-P/mg lipid	Lipid classes (%) Neutral lipids	Resin acids and glycolipids	Phospholipids and other polar lipids
Plant A	86 ± 3[a]	1.75 ± .21	20.3 ± 0.8	26 ± 1	21 ± 1	53 ± 1
Plant B	95 ± 6	1.95 ± .25	20.3 ± 1.5	26 ± 2	22 ± 0	52 ± 2
Plant C	101 ± 3	1.93 ± .11	19.2 ± 0.5	24 ± 4	23 ± 1	53 ± 4
Midseason						
Plant A	99 ± 5	1.95 ± .12	19.6 ± 1.2	27 ± 1	22 ± 0	51 ± 1
Plant B	101 ± 11	1.92 ± .12	18.8 ± 1.8	33 ± 4	18 ± 1	49 ± 4
Plant C	84 ± 9	1.71 ± .22	20.5 ± 1.3	26 ± 3	21 ± 1	53 ± 2
Early Valencia season						
Plant A	89 ± 7	1.71 ± .24	19.1 ± 1.6	28 ± 1	21 ± 1	51 ± 1
Plant B	92 ± 2	1.72 ± .18	18.7 ± 1.9	29 ± 1	20 ± 1	51 ± 2
Plant C	91 ± 9	1.56 ± .12	17.4 ± 2.0	34 ± 2	21 ± 1	45 ± 1
Plant D	96 ± 4	1.86 ± .04	19.5 ± 0.8	27 ± 3	21 ± 3	52 ± 5
Late Valencia season						
Plant A	100 ± 4	1.77 ± .06	17.8 ± 0.7	32 ± 2	22 ± 1	46 ± 2
Plant B	100 ± 2	1.72 ± .20	17.2 ± 1.9	34 ± 3	23 ± 2	43 ± 2
Plant C	99 ± 9	1.82 ± .08	18.3 ± 0.9	30 ± 3	22 ± 2	48 ± 2
Plant D	95 ± 7	1.65 ± .19	17.4 ± 1.8	35 ± 1	20 ± 0	45 ± 1

[a]Values represent mean ± standard deviation of 4-6 samples.

TABLE II

Comparative Examination of Mean Values of Five Major Fatty Acids in Commercial Single-Strength Orange Juice

Fatty acid	Season Early	Mid	Early Valencia	Late Valencia
16	22.9 ± 0.4	21.5 ± 0.3	21.5 ± 0.4	21.8 ± 0.6
16:1	4.3 ± 0.2	4.2 ± 0.2	4.5 ± 0.1	4.4 ± 0.2
18:1	25.9 ± 0.5	25.1 ± 1.6	24.7 ± 0.6	25.5 ± 1.0
18:2	30.9 ± 0.0	29.6 ± 1.8	34.7 ± 0.5	32.8 ± 0.8
18:3	10.0 ± 0.4	13.1 ± 0.5	8.2 ± 0.3	8.6 ± 0.5

89-100 mg/100 ml), while levels found in early and midseasons were more variable. The overall mean lipid levels for these 14 juices were 95 ± 6 (standard deviation). Another convenient way to express lipid levels is on a wt/wt basis. The densities of these commercially processed juices ranged from 1.024-1.039 g/ml. Expressed as mg lipid/100 g juice, the range was 82-99. Therefore, the lipid percentage range on a wt/wt basis was .082-.099%, with an overall mean of .093%.

The phospholipid phosphorus content of these juices ranged from 1.56-1.95 mg/100 ml juice. These values are consistent with those reported by Vandercook, et al., (12) for 4 commercial Florida orange juices (1.4-1.7 mg lipid P/100 ml juice) and those reported by Swift and coworkers (4,8).

The phospholipid phosphorus/lipid ratios shown in Table I are consistent for all juices; expressed as μg-P/mg lipid, they range from 17.2-20.5, with an overall mean of 18.9 ± 1.1. Conversion of μg-P to μM-P produces a range of 0.53-0.64.

Citrus lipids can be grouped into three main classes, i.e. neutral lipids, glycolipids, and other polar lipids which are predominantly phospholipids. The neutral lipid fraction of orange juice is composed of monoglycerides; 1,2 diglycerides; 1,3 diglycerides; free sterols; free fatty acids; hydrocarbons; triglycerides; sterol esters; lipid pigments; and other components (10). The neutral lipid fraction of juice comprises between 24-35% of the total lipids (Table I). All early season juices (plants A, B, C) and two midseason juices (plants A, C) generally contained lower neutral lipid percentages than those from Valencia seasons. These different percentages probably reflect the predominant type orange being processed (16).

The glycolipid fraction of orange juice is composed of steryl glucosides, esterified steryl glucosides, cerebrosides, monogalactosyl diglycerides, digalactosyl diglycerides, resin acids, and other minor components (17). This group of lipids comprises between 18-23% of total citrus juice lipids and exhibits a mean percentage of 21 ± 1 (Table I).

The phospholipid and other polar lipids fraction of citrus juices are the largest and undoubtedly the most complex. The major phospholipids in citrus juice are phosphatidyl choline, phosphatidyl ethanolamine, phosphatidyl inositol, phosphatidyl serine, and phosphatidic acid (10,12). This fraction comprises between 43-53% of the total lipid content with a mean of 49 ± 3%. Of all lipid fractions, the phospholipid fraction has been shown most susceptible to breakdown during high temperature storage (8,10).

The fatty acid composition of citrus juices is extremely complex (13,18). The five major acids are palmitic, palmitoleic, oleic, linoleic, and linolenic. Stearic acid (C_{18}) is found in commercial juices at levels no higher than 1.1%. This low level of stearic acid has been confirmed in other citrus juices (13). Comparative examination of the mean values of the five major orange juice fatty acids (Table II) shows some differences in their relative percentage. Differences are mainly in the relative percentages of linoleic ($C_{18:2}$) and linolenic ($C_{18:3}$) acids.

Commercially labeled orange juice, by federal regulations (19), may contain up to 10% of juice derived from mandarins and mandarin hybrids (tangors, tangelos). Incorporation of mandarin-type fruit is carried out by citrus processors throughout the entire processing season but is more pronounced during early and midseasons (November-March) because of flavor, color, and economic factors.

The mean percentages for the five major fatty acids reported for early season juice (Table II) are comparable to percentages previously reported for pure Hamlin orange juice (18); therefore, these samples probably had little blended mandarin juice. The values reported in Table II for midseason juices are noticeably different from values of pure pineapple-orange juice. Major fatty acid values determined on hand-reamed pineapple-orange juice were: C_{16} (19.7%), $C_{16:1}$ (4.6%), $C_{18:1}$ (28.4%), $C_{18:2}$ (31.9%), and $C_{18:3}$ (8.2%). These values agree closely with values previously reported for pineapple-orange juice sacs (18). Pure pineapple-orange juice contained 8% $C_{18:3}$, while commercial midseason juices contained 13%. In fact, no orange juices examined to date had ever shown $C_{18:3}$ levels exceeding 10.5%. The higher $C_{18:3}$ percentage in these midseason juices may be due to the admixture contribution of mandarin-type fruit. Mandarin fruit show high percentages for $C_{18:3}$ (unpublished results). Support of the premise that mandarin fruit are in these midseason juices comes from organoleptic evaluations by federal inspectors. Mandarin juice has a distinctively different flavor from orange juice and is characterized by a tangerine-like aroma. Even though mandarin juice is present in orange juice at low levels, its flavor and odor are easily noticeable to citrus flavor experts.

The major fatty acid composition of early and late Valencia juices (Table II) is consistent with the fatty acid profiles previously determined for pure Valencia orange juice (13). Juice from the entire Valencia processing period (April-July) is generally free of mandarin juice, because it is not needed for color and because mandarin-type fruit are generally not harvested during this period.

As shown in Table I, the fat content of commercially processed orange juice is quite low. A typical 6 fluid oz (178 ml) orange juice serving would contain between .15 and .18 g fat. Because of this low fat level, orange juice would not qualify for nutritional labeling of its fat content according to regulations of the Food and Drug Administration on U.S. Recommended Daily Allowance (20).

One of the major applications for these data may lie in detecting adulteration which may occur in many ways. For example, concentrated orange juice may be purchased from a processor and, in turn, diluted to single-strength level (ca. 12° Brix) by out-of-state or foreign distributors. Such distributors could over-dilute the concentrate to produce more juice volumes. Also, commercial phospholipid adulterants could be added to citrus juice to function as clouding agents or emulsifiers (12). The narrow lipid range reported for SSOJ (Table I) could be used to indicate excessive dilution of juice. The phospholipid phosphorus/lipid values shown in Table I also could be used to indicate adulteration through addition of commercial lipid sources. There is also the possibility that these lipid values could be used to distinguish an orange juice from an orange drink or blends containing other cultivars.

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