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Sugar Composition Analysis of Commercial Citrus Juice Products

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Citrus juice is one of the most popular fruit juices in the market. From 2013 to 2015, under a citrus juice nutrition and quality research program, the sugar composition and concentrations of 286 and 38 samples of commercial 100% orange and grapefruit juice products, respectively, collected from food stores located in Florida, California, New Jersey, and Texas, were analyzed using a high performance anion exchange chromatography system coupled with pulsed amperometric detection. The average concentration of glucose, fructose and sucrose in the orange juice was 2.1, 2.4, and 4.4 g/100 mL, respectively. The average concentration of total sugars in orange juice was 8.9 g/100 mL with a range of 6.6 to 11.1 g/100 mL. There were no significant differences in average total sugar concentrations between not-from-concentrate and from-concentrate orange juice samples, and among orange juice samples collected from Florida, California, New Jersey and Texas. For tested 100% grapefruit juice, the average concentration of glucose, fructose and sucrose was 2.5, 2.7 and 2.8 g/100 mL, respectively. The average concentration of total sugars in grapefruit juice was 8.0 g/100 mL with a range of 6.8 to 9.7 g/100 mL. The lower total sugar levels in grapefruit juice were largely due to lower sucrose levels, compared to the orange juice. The glucose:fructose:sucrose ratio was about 1:1:2 for orange juice, and about 1:1:1 for grapefruit juice tested in this study. However, the content of fructose was slightly higher than that of glucose in both orange and grapefruit juice. The results from this study provide further information regarding the nutrition and quality of commercial orange and grapefruit juice products.

Citrus juice is one of the most popular fruit juices in the market and is an important component of a healthy diet (O'Neil et al., 2012). Sugars are the major components of citrus juice soluble solids. The sweetness of orange juice is related to its sugar content. In oranges, sugars account for about 80% of the total soluble solids (expressed as °Brix), with the remainder consisting of organic acids, free amino acids, inorganic ions, vitamins, and a few other minor components (Grierson, 2006a; Lee and Coates, 2000). The °Brix:acid ratio serves as an indication of juice palatability, with higher values corresponding to a sweeter taste. Very high values may indicate insipid tasting juice (Echeverria, 1990). Sugar content of citrus juice is not only a nutrition and quality issue, but are also of economic interest because growers' monetary returns are based not only on yield (boxes of fruit), but also on pound-solid (weight of sugar in the juice).

Common orange and grapefruit juice are sold in U.S. markets as from-concentrate (re-constituted juice) and not-from-concentrate. Commercial citrus juice products include sugar content information on their labels with those values varying slightly depending on the brand. There is no sugar composition information on the labels. Glucose, fructose, and sucrose are the main sugars present in commercial citrus juice. The ratio is about 1:1:2 based on weight (Lee and Coates, 2000). Orange juice is susceptible to adulteration by blending corn syrup or sugar syrup with orange juice concentrate and other cheaper ingredients (Lee and Coates, 2000; Villamiel et al., 1998). Therefore, additional information on orange juice composition would help identify orange juice nutrition, quality, and authenticity of commercial products. Further study is needed because there is currently limited information regarding sugar composition, ratios of individual sugars, and their changes in commercial citrus juices from different market locations in the United States.

Various methods and approaches are available for citrus juice sugar composition analysis such as high-performance liquid chromatography (HPLC) (Cancalon, 1994; Lee and Coates, 2000; White and Widmer, 1990), gas chromatography (Villamiel et al., 1998) and capillary electrophoresis (Lee and Coates, 2000). However, the use of high-performance anion-exchange chromatography with pulsed amperometric detection (HPAE/PAD) has been reported to be a rapid, simple, accurate and sensitive approach for the quantitative and routine analysis of sugar composition in citrus juice (White, 1990; White and Widmer, 1990).

The objective of this study was to analyze the sugar composition and concentrations of commercial 100% orange and grapefruit juice products collected from four different market locations in the United States using a HPAE/PAD system as a part of a monitoring program for citrus juice nutrition and quality.

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Materials and Methods

Sample collection. From June 2013 to April 2015, 286 and 38 samples of commercial 100% orange and grapefruit juice products, respectively, were collected from food stores located in Florida, California, New Jersey, and Texas. The number of orange juice samples collected from Florida, California, New Jersey, and Texas was 167, 47, 35, and 37, respectively. All samples were stored at –29 °C if they were not processed on the day obtained.

Sample preparation. Twenty grams of each juice sample was placed in a 50-mL centrifuge tube and centrifuged at 17,136 x g for 5 min at 10 °C, then the supernatant was collected and the pellet was discarded. Two grams of supernatant was diluted ten-fold with HPLC-grade water in a 50-mL tube. About 10 mL of each sample was run through an anion exchange column (AG 1-x8 resin, 100–200 mesh, chloride form) (Bio-Rad, Hercules, CA) after the column was washed with 10 mL of HPLC grade water. The initial 4 mL of eluent was discarded and the remaining eluent collected, filtered through a 0.45-μm nylon filter, and then further diluted another 200-fold with HPLC grade water. The diluted sample was transferred to a Dionex HPLC vial (1.8 mL in volume) and used for sugar analysis with the HPAE/PAD system. The sample dilution factor was about 2000 with this sample preparation procedure. The exact dilution number was calculated for each sample based on weight.

HPAE/PAD analysis. A Dionex ICS-3000 ion chromatography system (Sunnyvale, CA) with Dionex Carbopac PA200 column (3 x 250 mm) and a PA200 guard column (3 x 50 mm) and an electrochemical (EC) detector was used in this study. The EC detector used a disposable gold electrode, a standard quad carbohydrate waveform, IntAmp setting, 0.50 second rise time, and data collection rate at 1.0 Hz. An AgCl reference electrode was used, and pH with present mobile phase was 12.4–13.2. The compartment temperature was at 35 °C; each injected sample volume was 25 μL. A simple sugar isocratic program with a mobile phase containing 99.4% 150 mM NaOH and 0.6% 150 mM NaOH plus 250 mM NaOAc was run for 7 min. The elution order was inositol, glucose, fructose, sucrose, and then maltose. The standard individual sugars at various concentrations were run at the same time as the samples to establish standard curves to calculate the concentrations of the individual sugars in the samples.

Data analysis. The resulting data of HPAE/PAD analysis were calculated and standardized based on standard curve. Three runs were conducted for each sample. Average concentrations and standard deviations for each of the individual sugars and the total sugar content were calculated. Analysis of variance of total sugar content data was performed using the JMP statistical package (SAS Institute Inc., Cary, NC). The means of total sugar content of orange juice among four geographic sample collection locations were compared using the Turkey-Kramer multiple range test ($P \leq 0.05$). The *t*-test was used to compare the concentration differences ($P \leq 0.05$) of the individual sugars between orange and grapefruit juice.

Results and Discussion

In this study, 286 commercial orange juice samples (172 not-from-concentrate, and 114 from-concentrate) were collected and analyzed for sugar composition and concentration using an HPAE/PAD system. The results are shown in Fig. 1 and Table 1. The concentration of fructose was slightly higher than glucose in orange juice. The ratio of glucose:fructose:sucrose based on weight in orange juice was about 1:1:2. This ratio was considered typical for commercial orange juice and was consistent with other reports of commercial orange juice sampled from Florida processing plants and analyzed using the HPLC method (Lee and Coates, 2000). The sugar composition, concentration and ratios in orange fruit can be affected by many factors such as citrus cultivars, fruit maturity, climate, juice processing, and storage conditions (Cancalon, 1994; Grierson, 2006b). Cancalon (1994) studied sugar composition changes of 'Valencia' oranges in Florida during fruit maturation and found that glucose, fructose and sucrose were present at a 1:1:2 (by weight) ratio from December to April, but from May to June of the same season, the ratios could reach 1:1:3. The glucose:fructose ratio was about 0.9 in fresh fruit, the same value as was reported by Lee and Coates (2000) in commercial orange juice. The ratio of glucose:fructose was suggested as being useful way to investigate potential orange juice adulteration (Lee and Coates, 2000) since in most fruit, glucose exceeds fructose; the ratio is about three-fold in apple and pear (Widdowson and McCance, 1935). The average total sugar content of from-concentrate orange juice was the same (8.9 g/100 mL) as that of not-from-concentrate orange juice. The average total sugar content of orange juice samples collected from Florida, California, New Jersey and Texas was 8.9, 8.9, 8.7, and 9.0 g/100 mL, respectively, with no significant differences ($P \leq 0.05$) among sample locations.

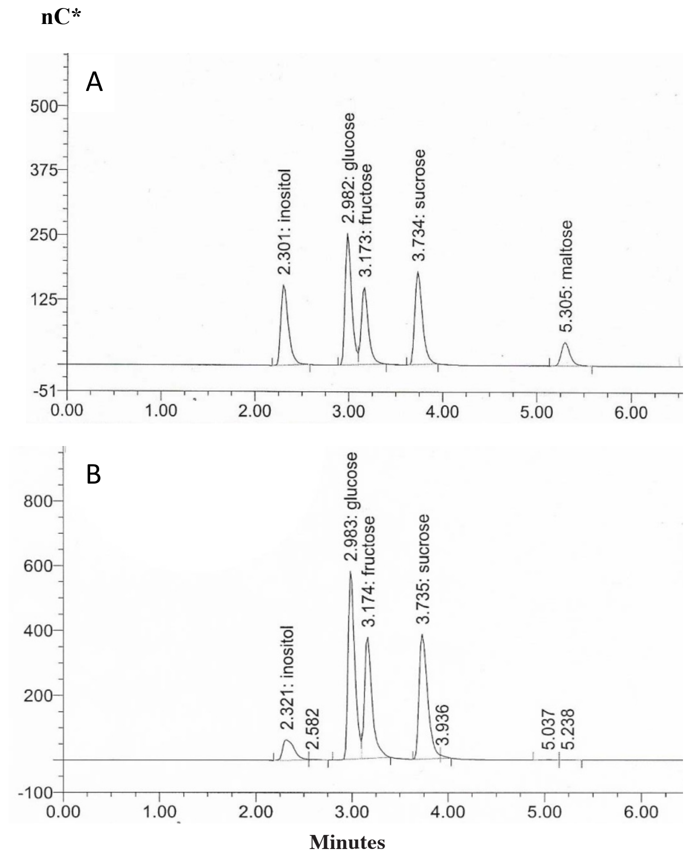

Fig. 1. The sugar profiles from high performance anion-exchange chromatography with pulsed amperometric detection for (**A**) aqueous solution of standard sugars and inositol, and (**B**) a commercial 100% orange juice product.
* nC = Nano Coulombs for electrical charges.

The sugar composition and concentrations of 38 samples of commercial 100% grapefruit juice (34 not-from-concen-

Table 1. Sugar composition and average concentrations of commercial 100% orange and grapefruit juice products collected from California, Florida, New Jersey, and Texas. The analysis was conducted using a high-performance anion-exchange chromatography with pulsed amperometric detection system.

	Glucose (g/100 mL)	Fructose (g/100 mL)	Glucose:fructose ratio	Sucrose (g/100 mL)	Total sugar (g/100 mL)	Glucose:fructose: sucrose ratio
Orange juice (286 samples)						
Mean	2.1 ± 0.2	2.4 ± 0.2	0.9	4.4 ± 0.3	8.9 ± 0.6	1.0:1.1:2.1
Min.	1.5	1.7	0.9	3.3	6.6	1.0:1.1:2.2
Max.	3.1	3.3	0.9	5.3	11.1	1.0:1.1:1.7
Grapefruit juice (38 samples)						
Mean	2.5 ± 0.2	2.7 ± 0.2	0.9	2.8 ± 0.2	8.0 ± 0.5	1.0:1.1:1.1
Min.	2.1	2.2	1.0	2.3	6.8	1.0:1.0:1.1
Max.	3.1	3.2	0.9	3.4	9.7	1.0:1.0:1.1

trate and 4 from-concentrate) are shown in Table 1. The glucose:fructose:sucrose ratio (by weight) was about 1:1:1. Fructose content was also slightly higher than that of glucose with a glucose:fructose ratio of about 0.9 (Table 1), which was similar to that for orange juice. The average total concentration of sugars in grapefruit juice (8.0 g/100 mL) was significantly lower ($P \leq 0.05$) than that of orange juice (8.9 g/100 mL). This was largely due to the significantly lower ($P \leq 0.05$) sucrose level in grapefruit juice compared to orange juice. Similar values for both juices were reported by others (White and Widmer, 1990). There were no significant differences in concentrations of glucose or fructose between orange and grapefruit juice. However, limited numbers of grapefruit juice samples were collected in this study, and more samples are needed for more reliable results.

Maltose was included in the current analysis but was not detected in any orange or grapefruit juice samples tested. Maltose has been reported in citrus leaves (Cancalon et al., 2011; Fan et al., 2010) but not in citrus fruit or juice. Inositol was also included in the current analysis, although it is not a sugar or carbohydrate, but is a cyclic polyol or sugar alcohol. Inositol was detected in both orange and grapefruit juice samples at a range of 0.14% to 0.40% in this study. The presence of inositol in orange and grapefruit juice is consistent with reports by others (Lee and Coates, 2000; Villamiel et al., 1998; White, 1990). The presence of inositol in citrus juice deserves further attention since unusually low inositol levels in commercial citrus juice may indicate possible juice dilution. The use of HPAE/PAD to analyze free sugars in citrus juice has been reported as a low-cost and simple-to-perform method that yields rapid, selective and sensitive results (White, 1990; White and Widmer, 1990). Results from the current study agree with that assessment. Sample preparations were simple, and the analysis times were less than 10 min per sample cycle (Fig. 1). Therefore, the HPAE/PAD can routinely be used to quantify sugars such as glucose, fructose and sucrose in citrus juice products.

Conclusions

In conclusion, the sugar composition in both commercial 100% orange and grapefruit juice consisted of glucose, fructose and sucrose. The average total sugar content was 8.9 and 8.0 g/100 mL, respectively, for orange and grapefruit juice. The lower total sugar levels in grapefruit juice compared to those in orange juice were largely due to the lower sucrose levels in grapefruit juice. There were no significant differences in total sugar content between from-concentrate and not-from-concentrate orange juice, and among orange juice samples collected from four states in the United States. The glucose:fructose:sucrose ratio in commercial 100% orange juice was 1:1:2. The results regarding sugars and inositol from this study are consistent with those of commercial citrus juice reported in the literature.

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