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Analytical Methods

Sensory and instrumental volatile flavor analysis of commercial orange juices prepared by different processing methods

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ABSTRACT

The objective of the current study was to correlate the sensory and instrumental flavor analysis results of commercial orange juice (OJ) products prepared by different processing methods. Descriptive analysis was conducted using a highly trained panel (n = 6) to evaluate four OJs in triplicate. Volatile compounds associated with the four OJs were quantitatively and qualitatively identified using a Dynamic Headspace Sampling, followed by Gas Chromatography–Mass Spectrometry analysis. The sensory characteristics of the four commercially available OJs were significantly different ($p < .05$). OJs requiring refrigeration storage (OJ3 and OJ4) had high intensity of orange peel flavor, and shelf-stable OJs (OJ1 and OJ2) had high intensity of cooked orange flavor ($p < .05$). Similarly, differences in volatile flavor profiles of 4 OJs were documented. The shelf-stable OJs (OJ1 and OJ2) had desirable volatile flavor compounds, such as β-pinene, dl-limonene, linalool, nonalool, and decanal, and OJs requiring refrigeration had high levels of α- and β-terpineol.

1. Introduction

Orange juice (OJ) is one of the most widely consumed fruit beverages in the world. Its consumption has increased steadily to the point that about 672 metric tons were consumed in the United States in 2013, and 2145 tons were drunk worldwide in 2013 (CITRUSBR, 2016). In the regional consumption distribution for that year, North America ranked the highest (839,000 tons), followed by Europe (778,000 tons) and Asia (264,000 tons; CITRUSBR, 2016). South Korea ranked 13th highest OJ consuming countries in the world (Kim, Kim, & Lee, 2016). OJ has a unique taste that is well liked by consumers (Kim, Lee, Kwak, & Kang, 2013). In addition, the nutritional benefits of consuming OJ, such the reduced risk of obesity among the US population according to the National Health and Nutrition Examination Survey 2003–2006 (O'Neil, Nicklas, Rampersaud, & Fulgoni, 2012), decreased risk of contracting urinary stones (Wabner & Pak, 1993), and high vitamin C content (Kim et al., 2016), have been reported. Unsurprisingly, with the combination of its distinctive flavor and positive health effects, OJ consumption continues to rise.

The typical OJ manufacturing process involves squeezing fresh oranges, followed by the centrifugation and heat processing steps. Typical process of manufacture 100% OJs from concentrates requires the rehydration step after water removal of freshly squeezed orange juice. Regardless of types of OJs, almost all commercial juices are thermally processed, as this is the most cost-effective method discovered to date for reducing the microbial contamination and enzymatic activities in the juice matrix (Perez-Cacho & Rouseff, 2008). The temperature selected for the heat processing steps varies according to the storage temperature requirements: the thermal treatment of freshly squeezed juice requiring refrigeration is typically conducted at 75 °C for 30 s, whereas the pasteurization temperature for shelf-stable products made from frozen, concentrated juice is set to 95 °C for 30 s (Gil-Izquierdo, Gil, & Ferreres, 2002). Different thermal treatment temperatures affect the flavor profile of OJ from the sensory and flavor chemistry perspectives. Previously, key volatile compounds responsible for "good" quality juice flavor were identified using descriptive sensory and instrumental flavor analysis (Bettini, Shaw, & Lancas, 1998; Petersen, Tonder, & Poll, 2009). Various studies were documented using instrumental volatile compound analysis on juices treated with different processing methods (Baxter, Easton, Schneebeli, & Whitfield, 2005; Bettini et al., 1998; Kim et al., 2016; Min, Jin, Min, Yeom, & Zhang, 2003; Rega, Fournier, & Guichard, 2003). In addition to instrumental flavor compound analysis, the sensory and physiochemical characteristics of juices treated with various processing techniques were also well documented (Gil-Izquierdo et al., 2002; Kim et al., 2013). Based on the current literature review, the sensory characteristics and the volatile

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flavor profile of OJ are greatly influenced by the temperature of thermal treatment during processing and by storage conditions.

A recent study revealed the distinctive differences among the sensory characteristics of seven commercially available OJ brands (Kim et al., 2013). Such characteristics can be grouped into two categories: one being high in "natural" orange flavor and the other having a high intensity of "processed" orange flavor. The "natural" orange flavors are described as high in "orange peel", "orange flesh", "citrus fruit", and "grapefruit" while "processed orange" flavors are described as high in "over-ripe", "cooked orange", and "yogurt-like" flavors (Kim et al., 2013). These flavor differences are mainly derived from the varied processing steps during the manufacturing stage. The limitation of this study is the lack of instrumental flavor analysis on juices with different sensory characteristics, and thus only partial information was provided on the flavor characteristics of juices manufactured under different processing techniques. Accordingly, the objective of the current study is to conduct the sensory and instrumental volatile flavor analyses of OJs prepared by different processing methods. In addition, the correlation between the sensory and flavor characteristics of commercial juices was determined.

2. Materials and method

2.1. OJ samples and chemical reagents used in the study

Four juices were carefully selected based on market share, processing type, and flavor characteristics according to the author's previously published work. The descriptive sensory analysis results from the previous study were used for representative sample selection (Kim et al., 2013). Based on their study, commercial OJs were grouped into two categories by flavor: natural orange flavor group and processed orange flavor group. This study selected two OJ samples from the processed orange flavor group (OJ1 and 2) and two samples from the natural orange flavor group (OJ3 and 4). Among the four OJs selected, two required room temperature storage (OJ1 and OJ2) and the others refrigeration storage (OJ3 and OJ4). All samples were reconstituted from frozen concentrates; a detailed description of these samples can be found in Table 1. All samples were purchased at a local grocery store and had at least a week before the expiration date to assure the freshness of the products. Samples were kept at 4 °C until further analysis was conducted. All chemical reagents used in the study were purchased from the Sigma-Aldrich Corporation (St. Louis, MO, USA).

2.2. Dynamic Headspace Sampling (DHS) and Gas Chromatography–Mass Spectrometry (GC–MS) analysis

Flavor extraction from OJs was conducted using Gerstel MPS 2 with DHS (Gerstel, Mülheim an der Ruhr, Germany). The flow diagram of DHS analysis for OJ samples can be found in Fig. 1. About 3 mL of OJs was prepared in a 10 mL headspace screw cap glass vessel with 100 µL of tetradecane (10 mg/L, internal standard). The sample was incubated in an MPS-2 incubator with agitation (400 rpm) at 60 °C for 10 min. Then, the headspace above the OJs was purged with nitrogen gas at 70 mL/min for 3 min, and the flavor compounds from the purged headspace were trapped onto the Tenax TA packing in a glass tube (Gerstel, Mülheim ander Ruhr, Germany). After purging and trapping, the Tenax TA packing was dried with an additional purge flow at 40 mL/min for 2.5 min to remove residual water. The tube with trapped flavors was introduced into a thermal desorption unit (TDU, Gerstel, Germany), in which the compounds were thermally desorbed. The temperature programming of TDU was maintained at 25 °C (0.2 min) and then ramped to 250 °C at 720 °C/min. The desorbed compounds were transferred to a cooled injection system 4 (CIS4, Gerstel, Germany), in which the compounds were cryofocused to improve peak sharpness. The temperature of CIS4 was maintained at −10 °C with liquid nitrogen and then ramped to 250 °C at 12 °C/s and held for 5 min.

The separation and detection of the flavors from OJs were conducted using a 6890 GC instrument coupled with a 5973 mass-selective detector (Agilent, Palo Alto, CA, USA). The compounds were separated using a DB-WAX column (60 m length × 0.25 mm i.d., 0.25 µm film thickness, J & W Scientific, USA). The GC injector was a split mode (50:1) and maintained at 250 °C. The GC oven temperature program was set to 50 °C (held for 2.5 min) and then increased to 200 °C at 4 °C/min. The carrier gas was helium, which was delivered at constant linear velocity of 30 cm/s. The mass spectrometer was operated in the positive electron impact ionization mode using automatic gain control, with 70 eV of electron energy and a scan mode (scan range: 35–500 m/z). Volatile compounds were identified by the retention index on DB-WAX, and mass spectrum was assessed using the Wiley mass databases and co-injection with authentic chemicals. The quantification of volatile compounds was expressed by the peak area ratio (PAR), which was calculated by the GC peak area divided by the peak area of internal standard. The analysis was conducted in triplicate. The volatile compounds from OJs were tentatively identified by mass spectra comparison and retention indices. Retention indices were calculated using C_7–C_{40} alkanes in *n*-hexane (Sigma-Aldrich, Steinheim, Germany), and the mass spectra of each compound were compared with the information provided in the Wiley 9th and NIST08 library database.

2.3. Descriptive sensory analysis of OJs

A trained sensory descriptive panel was used for the descriptive sensory profile of the four OJs. The panel consisted of four females and two males aged 24–37. Each panelist had over 200 h of prior experience in the evaluation of fruit-flavored beverages, fermented soybean products, and coffee beverages using the Spectrum™ method. In addition, the panel leader had more than 5000 h of experience in the descriptive analysis of various food products using the same method. Prior to evaluation, a 10 h calibration session was initiated. During this session, the sensory lexicon used in previous studies (Kim et al., 2013, 2016) was re-evaluated and modified using sensory references, and the use of the 15-point universal scale in the Spectrum™ method with basic taste solution was re-calibrated. On the day of evaluation, samples were presented in 210 mL disposable paper cups (Jinkwang Papers, Ghimhae, South Korea) labeled with a random three-digit code. The flavor profiles of the juices were created by each panelist (n = 6) in triplicate in a randomized balanced design. On each day, a maximum of four samples were evaluated in one sitting to minimize the panelists' fatigue. During the evaluation, a 2 min rest was enforced between samples, and the panelists were instructed to use water to cleanse their palate between tastings. All samples were kept at 4 °C until the day of evaluation and were served to the panelists at 8 °C. Paper ballots were

Table 1
Sample Description of OJs included in the study.

Sample	Ingredient list	Storage requirement
OJ1	Concentrated orange juice, Orange juice extraction, Purified water, Natural flavoring substances (orange)	Room temperature
OJ2	Concentrated orange juice, Purified water, High fructose corn syrup, Calcium lactate, Citric acid, Vitamin C	Room temperature
OJ3	Concentrated orange juice (100%), Purified water	Refrigeration
OJ4	Orange squash (100%), Purified water, Vitamin C	Refrigeration