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Volatile compounds captured through purge and trap technique in caja-umbu (*Spondias* sp.) fruits during maturation

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Abstract

This work identifies the volatile compounds present in the pulp of caja-umbu (*Spondias* sp.) fruits harvested at two different stages (half-ripe and ripe) of maturation. The volatiles were captured through purge and trap technique. The half-ripe caja-umbu fruit pulp contained 67 components among which the principal compounds, representing an area of 71.7% of chromatogram, were identified as β-caryophyllene (22.2%), 2-methyl butanal (19.3%), 2-hexanol (18.6%), ethyl butyrate (7.6%) and α-caryophyllene (3.9%). However, in the ripe caja-umbu fruit pulp, 70 compounds were detected among which 2-methyl butanal (28.4%), 2-hexanol (15.0%), β-caryophyllene (14.1%), ethyl butyrate (6.1%) and α-caryophyllene (2.4%) were prominent compounds. There were notable quantitative differences in prominent compounds such as β-caryophyllene and 2-hexanol which were quantitatively higher in half-ripe fruits while 2-methyl butanal known to possess characteristic pungent fresh fruit aroma increased with maturation, being relatively higher in its content in ripe fruits.

Keywords: Dynamic headspace; Volatiles; Flavor; Aroma; Gas chromatography; Mass spectrometry

1. Introdution

The northeast region of Brazil is known for large scale production of various tropical and sub-tropical fruits as in this region, the growth conditions are favorable for their cultivation such as higher temperatures, light effects and adequate humidity. Among the exotic fruits pertaining to the genus *Spondias*, which are very much appreciated in the region are yellow mombin (*Spondias mombin* L.), umbu (*Spondias tuberosa* Arruda Camara) and caja-umbu fruits.

Although the fruit's origin is unknown, caja-umbu is considered to be a natural hybrid between cajá or yellow mombin (*S. mombin* L.) and umbu (*S. tuberosa* Arruda Camara) fruits (Giacometti, 1993). It possesses some xerophytic characters and is widely spread in some northeastern Brazilian states such Rio Grande do Norte, Ceará, Piauí, Pernambuco e Bahia. In some earlier reports, the fruit was cited as umbu-cajá (Franco & Janzantti, 2005; Franco & Shibamoto, 2000; Lima, Lima, Aldrigue, & Gondim, 2002), however, of late, the fruit is standardized for citation as cajá-umbu (Lira Junior et al., 2005). Furthermore, the use of *Spondias cytherea*, characterizing specie of the caja-umbu fruit, is misnomer as it represents the fruit ambarella (Leon & Shaw, 1990).

The caja-umbu production, although unquantified in Brazil, is considered to be of minor scale. The fruit is known for its attractive appearance, nutritional quality, pleasing aroma and flavor characteristics which are very much appreciated for its consumption as a fresh fruit or in processed form such as pulp, juice, nectars and ice-cream products (Souza, 1998). There is very little work done on post-harvest aspects of caja-umbu fruits. Lima et al. (2002) assembled data on physical, physico-chemical and chemical characteristics of fruits at different stages of mat-

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uration. However, they did not report any data on volatile aroma compounds present in the fruit.

In order to avail full potential of commercialization of caja-umbu fruits it is important to know the maturation and ripening process during which changes occur in their color, texture and flavor specially in its volatile compounds. There is only one work published on the identification of volatile compounds in caja-umbu fruit pulp wherein Franco and Shibamoto (2000) identified only 26 volatile compounds utilizing the dynamic headspace isolation technique. They reported a volatile profile representing a large majority (87%) of terpenic compounds along with 5% esters. However, their work was limited to mature ripe fruits. The objective of this work was also to use the dynamic headspace technique, being in particular, the purge and trap concentration of volatiles and to identify the compounds present in pulp of caja-umbu fruit at two different maturation (half-ripe and ripe) stages.

2. Material and methods

2.1. Fruits and other materials

Fresh half-ripe and ripe caja-umbu fruits were obtained from a Experimental Station Farm, administered by IPA (*Empresa Pernambucana de Pesquisa Agropecuária*), situated in the city of Itambé, in the Pernambuco state of Brazil. Fruits at half-ripe stage of maturation were classified as those possessing either predominant or total yellow color while those at ripe stage possessed totally orange–yellow color. The fruits were transported to the laboratory in the city of João Pessoa in small cardboard boxes and did not have any application whatsoever of inhibitor or accelerator for the control of maturation. Fruits free from any apparent skin damage were selected for analysis. The solvents and authentic standard flavor compounds used in volatiles identification were of pure grade (purity >97.7%) of Merck and Sigma–Aldrich companies, respectively.

2.2. Volatiles isolation

The fruit, after being washed with distilled water, were cooled to 2 °C by submerging it in ice-cooled water. The skin and kernel were separated manually by using a stainless steel knife and the pulp macerated. The capture of volatile components was undertaken in a dynamic headspace system consisting of purge and trap concentrator (Make Tekmar, model 3000). The trap contained a polymer mixture of tenax/silica gel/charcoal (Trap No. 3 of Tekmar). Fifteen millilitres of fruit pulp was taken in the fritless sparger which served as sampler unit and its heating was programmed with an initial temperature of 15 °C for 15 min, followed by heating until 80 °C at which temperature it was maintained for 35 min. Helium gas was utilized as a purge flowing at 40 ml/min. The other analytical conditions were:

Trap temperature: purge 30 °C; desorption 180 °C; bake 225 °C.
Time: purge 50 min; injection 1 min; desorption 1.5 min; trap bake 20 min.

The purge and trap concentrator was interfaced with a system containing a gas chromatograph coupled with a mass spectrometer.

2.3. High-resolution gas chromatography/mass spectrometry

A combined system of Varian gas chromatograph (GC 3800) coupled with mass spectrometer (Saturn 2000R) was used and the analytical data were processed by using software "Saturn GC/MS Workstation, version 5.5" (Varian Inc., Palo Alto, USA). Capillary GC investigations were carried out on a 30 m (length) × 0.25 mm (internal diameter) innophase bondable polyethylene glycol polar capillary column (HP-INNOWax; 0.25 μm film thickness; Hewlett–Packard, Inc., Palo Alto, USA) (Narain & Galvão, 2004). The carrier gas used was helium and column head pressure was maintained at 11.5 psi having a flow rate of 1 ml/min. The oven temperature was programmed: initiation at 30 °C for 5 min, increased at 7 °C/min to 100 °C, maintained at 100 °C for 5 min, increased at 1 °C/min to 130 °C, increased at 10 °C/min to 195 °C wherein maintained for 45 min. The temperatures of the injection port and the GC/MS interface were 175 °C and 195 °C, respectively. The mass spectrometer was operated in the electron ionization mode with an electrical energy of 70 eV and an ion source temperature of 250 °C. The mass spectrum was scanned between 35 and 450 a.m.u. at 0.31 s intervals.

2.4. Compound identification

The linear retention index (RI) values for unknowns were determined based on retention time data obtained by analyzing a series of normal alkanes (C_8–C_{21}). Volatile components were positively identified by matching their RI values and mass spectra with those of standards, also run under identical chromatographic conditions in the laboratory. The identification was also based on matching an unknown mass spectrum with a spectrum available on the National Institute of Standards and Technology, USA (NIST) mass spectral data system or the literature (Adams, 1995; Jennings & Shibamoto, 1980; Kondjoyan & Berdagué, 1996).

3. Results and discussion

Table 1 lists the principal volatile compounds identified in the pulp of caja-umbu fruits at two different stages (half-ripe and ripe) of maturation while Table 2 presents the principal compounds which were found to have major differences between the fruits of half-ripe and ripe stages. The data lists the retention indices and peak area percent values