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Discrimination of mango fruit maturity by volatiles using the electronic nose and gas chromatography☆

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Abstract

Mango fruit (*Mangifera indica* L.), cv. 'Cogshall', 'Kent' and 'Keitt' were harvested at different maturities (61–115 d past flowering and 80–307 average g fresh weight for 'Cogshall') and at different sizes (364–1563 and 276–894 average g fresh weight for 'Keitt' and 'Kent', respectively). Immediately after harvest (green) or after 1 week of ripening at room temperature (ripe), fruit were homogenized or left intact and evaluated by electronic nose (enose) or by gas chromatography (GC) for aroma and other volatiles as well as for soluble solids and acids. Volatile data from the different harvest maturities and ripening stages were discriminated by using multivariate statistics (discriminant factor analysis). Both the enose and GC were able, in most cases, to separate fruit from different harvest maturities, especially for 'Cogshall' mangoes, at both the green and ripe stages as well as discriminate green from ripe fruit and fruit from the different varieties within a maturity stage. Solids and acids data indicated that later harvest maturities resulted in sweeter fruit and later-harvested fruit had a different volatile profile from earlier-harvested fruit. Mango fruit volatiles may be useful as maturity markers to determine optimal harvest maturity for mango fruit that results in full quality upon ripening.
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Keywords: Mango; Electronic nose; Aroma volatiles; Harvest maturity

1. Introduction

Mango fruit, *Manguifera indica* L. originated in Burma and India and are grown in most tropical regions of the world. There are 49 species and thousands of cultivars. Mango fruit are climacteric (Pantastico, 1984) and mature between the eleventh and fourteenth week after fruit set. Disorders are observed when fruit are harvested too early (Sy et al., 1989), yet the appropriate harvest maturity stage for optimal postharvest quality is difficult to determine, and varies by cultivar. Normally, fruit are harvested at the not clearly defined "mature green" stage for export markets, but subsequently ripen with poor quality if harvested immature. Biochemical measurements that are used as a maturity index for other fruit crops include titratable acidity, total soluble sugars, starch content, carotenoids, and physical measurements such as fruit weight, firmness and color, but are not always correlated with optimal quality (Cristo, 1994), and often require destruction of the fruit. One report successfully used dry matter and starch to predict soluble solids content using near infared spectroscopy (Saranwong et al., 2004) as an indication of quality. Nevertheless, other methods of measuring maturity for optimal postharvest flavor quality are still needed, especially if non-destructive.

Mango is a climacteric fruit, and as such, important biochemical changes occur during the respiratory climacteric, just before ripening. Most volatile compounds, such as terpene alcohols, nor-isoprenoid derivatives, and aromatic alcohols are glycosidically bound, and are liberated during ripening (Sakho et al., 1985). Harvest maturity can affect this process and affect the final flavor/aroma quality of the ripened fruit (Bender et al., 2000).

A review of the publications identifying volatile compounds in mango fruit reported a total of 267–435 compounds (Maarse,

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1991; Nijssen et al., 1999). Terpene hydrocarbons are the major class of compounds in mango, with contents of 16–90%. δ-3-Carene is the major compound in most mango cultivars, with limonene, β-ocimene, myrcene and α-terpinolene having importance in some cultivars. δ-3-Carene is believed to be the compound responsible for the typical mango aroma (MacLeod and Pieris, 1984; MacLeod and Snyder, 1985), and sesquiterpene hydrocarbons may also be in amounts as high as 10% in some cultivars, with large variability between cultivars (Sakho et al., 1985). Oxygenated compounds vary among mango varieties including alcohols, ketones, and esters. Along with the terpene and sesquiterpene hydrocarbons, they all contribute to the characteristic mango flavor.

Volatile (often aroma) compounds are traditionally analyzed by gas chromatography (GC) analysis with flame ionization (FID) or mass spectrophotometer (MS) detectors. Headspace sampling allows identification of aroma volatiles in the vapor phase in equilibrium with the solid or liquid sample matrix (Bicchi and Joulain, 1990). The static headspace technique is easy to implement, but has its limitations due to the low concentration of volatiles in the headspace, and small volumes that one can inject in a GC and be detected by an FID or MS detector. Another detection system coupled to headspace sampling is the electronic nose (enose). The use and optimization of an enose with fruit has been studied on orange juice (Shaw et al., 2000), tomato by Maul et al. (1997, 1998, 2000), and on apples (Bai et al., 2004). While many industries rely on the classical analytical techniques of gas or liquid chromatography, or on sensory analyses to evaluate product flavor and aroma, enose allows differentiation between products based on the volatile compounds.

In this study, volatile compounds were investigated in the context of finding new maturity markers for mango (Ackerman and Torline, 1984) in whole mango fruit and fruit homogenate in a joint project with the French Agricultural Research Center for Agricultural Development (CIRAD) and the USDA/ARS Citrus and Subtropical Products Laboratory (USCSPL) using enose and GC. In addition, harvest maturity was investigated for effect on mango flavor compounds.

2. Materials and methods

2.1. Fruit material

Reunion Island mangoes, cv. Cogshall, were harvested from a commercial orchard every 7–14 d from fruit set to commercial maturity (61–115 d after fruit set, mature green stage, Table 1), and were air-shipped to Marseille, France and transported to CIRAD, Montpellier, France. One batch of four fruit was immediately homogenized individually upon receipt (green fruit), while the remaining 6–8 fruit were ripened in air at 20 °C for 1 week prior to homogenizing (ripe fruit). Each homogenized fruit was frozen individually, stored at −20 °C, and was considered a single sample unit for later electronic nose or gas chromatography (GC) analysis, both of which were performed in duplicate.

Florida mangoes, cvs. Keitt and Kent, were harvested at different sizes from a commercial grove in Homestead, FL, transported to the USCSPL and sorted by weight into five lots of 6–18 fruit/lot, ranging from 364 to 1563 g for 'Keitt', and 10–23 fruit, ranging from 276 to 894 g for 'Kent', to get a range of harvest maturities. After ripening, 'Keitt' and 'Kent' fruit from each lot were divided into three replicates of 2–7 fruit each and homogenized after ripening for GC analysis.

2.2. Gas chromatography

For 'Cogshall' mango, volatile analysis was performed at CIRAD in France. A sampling flask containing 2 g mango homogenate was diluted with 20 mL distilled water, and placed in a water bath at 37 °C. Helium was swept through the homogenate at 20 mL/min, and adsorbed on a trap made of a mixture of activated charcoal and graphite, for 1 h at 37 °C. Desorption was by a MW-1 microwave sampler (Rektorik, 1982). The trap was subjected to microwaves for 7 s, allowing for a split 1:20 flash injection. Compound separation was on a Varian 3400 GC equipped with a Flame Ionization detector (FID) and using a DB-Wax column (60 m, 0.32 mm i.d., 0.25 μm film thickness, J&W Scientific, Folsom, CA). Injector and detector temperatures were 190 and 230 °C, respectively. The temperature program was 50 °C for 6 m, increased to 220 °C at 6 °C m^{-1}, then held for 16 m. Compounds were identified by transferring the portable microwave unit onto a GC 8000 FISON (Thermo Separation Products) equipped with a quadrupole TRIO 1000 FISON MS. Mass spectra data acquisition conditions were: positive electron impact, 35–400 *m/z*, 70 eV, transfer temperature 190 °C, source temperature 180 °C, electron multiplier detector at 500 V. Data are shown as intensity of GC detector signal (mV).

For 'Keitt' and 'Kent' mango, volatile analysis was performed at the USCSPL in Florida. Homogenate, diluted 50% with deionized water (v/v) (2 mL), was placed in a 6 mL sealed vial and fast frozen by immersion in liquid nitrogen and stored at −20 °C until analysis. The vial was equilibrated at 80 °C for 15 m in a static headspace sampler (Perkin-Elmer HS6, Boston, MA) coupled to a Perkin-Elmer 8500 GC equipped with a FID. The column used was a DBWAX (J&W Scientific, Folsum, CA) with a polar coating (30 m, 0.53 mm i.d., 1 μm film thickness). Carrier gas was He at 56 cm s^{-1}. The temperature was held at 40 °C for 6 m then increased to 180 °C at 6 °C m^{-1}. Compound identification was by retention time comparison to known standards as well as by spiking deodorized homogenate with five levels of known compounds to form calibration curves (Malundo et al., 1997). Compound identities were confirmed by analyzing samples from the same fruit by GC/MS. In this case, mango homogenate, 600 mL, was diluted with 600 mL DI water and then centrifuged at 6000 × *g* for 15 m. Organic compounds were extracted from the supernatant using methylene chloride and examined using a GC–MS (MSD 5973, Agilent, Palo Alto, CA), fitted with a DB5 column (30 m, 0.32 mm i.d, 1 μm film thickness, J&W Scientific) (Malundo et al., 1997; Lebrun et al., 2004).

2.3. Electronic nose

2.3.1. Fruit pulp

The enose FOX 4000 (Alpha MOS, Toulouse, France) was equipped with an automatic headspace sampler HS100 (Alpha