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Chemical and sensory comparison of fresh and dried lulo (*Solanum quitoense* Lam.) fruit aroma

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ABSTRACT

The odour-active volatile compounds of lulo fruit (*Solanum quitoense* Lam.) were isolated by solvent extraction followed by solvent-assisted flavour evaporation (SAFE). GC–O and GCMS analyses as well as quantitation by internal standard method showed that (*Z*)-3-hexenal, ethyl butanoate, 3-sulphanylhexyl acetate, and ethyl hexanoate were key aroma compounds in this fruit. Other odorants with relevance because their contribution (high *OAVs*) to the overall aroma were 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone, methyl benzoate, (*E*)-2-hexenal, and hexanal. Lulo fruit pulp in presence of maltodextrin DE-20 was dried by using four different types of drying methods: hot air-drying (HD), spray drying (SD), lyophilisation (LD), and ultrasonic convective hot air-drying (HUD). LD sample exhibited the highest sensory rank (lulo-like) in comparison with fresh fruit pulp. Hot-air drying processes (HD and HUD) changed adversely the aroma of lulo fruit pulp.

1. Introduction

Tropical fruit consumption is increasing in international markets due to growing recognition of its nutritional and therapeutic value, as well as its attractive exotic and characteristic flavour (Lasekan & Abbas, 2012; Rufino et al., 2010). Lulo (*Solanum quitoense* Lam.) is a tropical fruit characterised by its refreshing and intense aroma, with potential interest for international markets. This fruit, also known as “naranjilla”, is cultivated at altitudes between 1600 and 2450 m above sea level in humid Andean forests. It is a bush that produces spherical fruits (4–6 cm diameter) exhibiting an attractive yellow–orange peel colour when ripe. The flesh is green, sour, and juicy with many small seeds inside and it is commonly consumed in juices or jellies. This fruit has high fibre and organic acid content (citric acid), minerals (phosphorous, calcium, iron), and also vitamins, such as niacin, thiamin, riboflavin, and vitamins A and C (29.4 mg/100 g) (Gancel, Alter, Dhuique-Mayer, Ruales, & Vaillant, 2008).

Some studies regarding volatile composition of *S. quitoense* fruit has been done. Brunke, Mair, and Hammerschmidt (1989) studied the volatile composition of Ecuadorian lulo, reporting several methyl and ethyl esters as main constituents. Afterwards, Suárez and Duque (1991) characterised the volatiles from liquid–liquid extraction and SDE (simultaneous distillation–extraction) extracts of *S. quitoense* fruit by GC and GCMS, identifying several aliphatic esters and alcohols as main constituents. Labile compounds, such as hydroxyesters, lactones, C_{13}-norisoprenoids, and terpenols, were not found in the SDE extract (Osorio, Morales, & Duque, 2005). Osorio, Duque, and Batista-Viera (2003) also studied the influence of endogenous fruit enzymes on the release of volatiles from leaf glycosides. Acosta, Pérez, and Vaillant (2009) identified ten volatile compounds in *S. quitoense* fruit pulp from Costa Rica, with methyl butanoate being the major constituent. However, up to now there is no report that specifies the identity of odour-active volatiles of *S. quitoense* fruit, among the overall volatile compound mixture.

According to Colombian Ministry of Agriculture, *S. quitoense* is a promising fruit to be exported; production increased from 49,777 tons in 2009 up to 57,712 tons during 2011 with 6810 hectares planted in several states of the country (Ministerio de Agricultura y Desarrollo Rural de Colombia, 2012). However, lulo fruit is so perishable that its postharvest handling is a difficult challenge. Drying has been the most important strategy to overcome this problem, since fruit shelf life is increased and spoilage is avoided after this process.

One of the biggest problems in fruit dehydration is to preserve the natural active ingredients and prevent the development of unpleasant off-flavours that damage the sensory quality of final products (Osorio, Carriazo, & Barbosa, 2011). As such, proper

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selection of drying method for the preservation of fruits is very important and vital to the process economics (Fernandes, Rodrigues, Lim Law, & Mujumdar, 2011). The simplest and cheapest technique among different drying methods is hot air drying; nevertheless sensory quality of final product is affected by this thermal process, due to volatile compound loss, degradation or transformation. Non-thermal drying techniques, such as lyophilisation (Ceballos, Giraldo, & Orrego, 2012; Marques, Silveira, & Freire, 2006), osmotic dehydration (Osorio et al., 2007), and refractance window system (Abonyi et al., 2001), have been used in fruits with acceptable sensory results. The application of ultrasound during drying processes increases the water diffusivity of the fruit in most cases. This phenomenon may occur because of the formation of microchannels during the application of ultrasound (Fernandes & Rodrigues, 2008; Mulet, Cárcel, Sanjuán, & Bon, 2003). Spray drying is a common drying technique used to preserve aroma and other sensitive compounds in the food industry (Risch & Reineccius, 1993). By encapsulation of active compounds into a coating material, they are protected from the outside environment, increasing their shelf-life. Recently, mixtures of lulo pulp with different concentrations of gum arabic and maltodextrin were spray-dried. Retention of ascorbic acid, flavonoids and antioxidant capacity were determined with different conditions of processing (Igual, Ramires, Mosquera, & Martínez-Navarrete, 2014).

This paper presents the study of sensory and chemical changes (odour-active volatiles) of fresh and dried lulo fruit. Dried lulo fruit powders were obtained by four different methods: hot air-drying, lyophilisation, spray-drying, and ultrasonic-assisted hot-air drying, from mixtures of lulo pulp with maltodextrin DE-20. The aroma and taste of these powders as well as their odour-active volatile compositions were compared.

2. Materials and methods

2.1. General

The water activity (A_w) of microencapsulates was measured using a hygrometer HygroPalm AW1 (Rotronic Instruments, Huntington, NY), at 20 °C using 1 g of each solid. The moisture content of microencapsulates was determined using an electronic moisture balance MOC-120H (Shimadzu, Columbia, MD).

2.2. Plant material

Fresh lulo (*S. quitoense* Lam.) fruits were obtained from a local market in Bogotá, Colombia and processed immediately upon arrival at the laboratory. Ripe fruits were classified as “extra” quality and their ripening stage selected according to their peel colour (100% orange equals #5 on the colour scale; Icontec, 2004). The pH of fruit pulp was 3.10 ± 0.05 and soluble solids content (SS) was 7.8 ± 0.2 °Brix (data are given as average ± standard deviation $n = 5$).

2.3. Chemicals

Dichloromethane, pentane, sodium sulphate (anhydrous), and *n*-alkane mix (C_8–C_{26}) were acquired from Merck (Darmstadt, Germany). All solvents were freshly distilled prior to use. Pure reference standards of methyl butanoate, 2-methyl-2-butanol, ethyl butanoate, butyl acetate, hexanal, (*Z*)-3-hexenal, methyl hexanoate, (*E*)-2-hexenal, ethyl hexanoate, (*Z*)-3-hexen-1-ol, acetic acid, methyl benzoate, butanoic acid, ethyl benzoate, 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone, and 2-methyl-3-heptanone were purchased from Sigma–Aldrich (St. Louis, MO). 3-Sulphanylhexyl acetate was synthesised in the laboratory according to the procedure reported by Steinhaus, Sinuco, Polster, Osorio, and Schieberle (2008).

2.4. Quantitation of key-aroma compounds in lulo fruit

Aroma extract of lulo was obtained by solvent extraction followed by solvent-assisted flavour evaporation (SAFE distillation) (Engel, Bahr, & Schieberle, 1999), as described by Forero, Sinuco, and Osorio (2014). Briefly, lulo fruit pulp (250 g) was blended and then mixed with portions of anhydrous sodium sulphate to obtain a dry power. The homogenate was extracted with a dichloromethane-ethyl ether (7:3, *v*/*v*) mixture, and after SAFE, the organic phase was dried over anhydrous sodium sulphate and concentrated to 1 mL using a Vigreux column. The final extract exhibited the characteristic odour of lulo fruit when a drop of the solution was vaporised on a strip of filter paper.

Quantitative analyses of lulo odour-active volatiles exhibiting dilution factors higher than 8 (aroma extract dilution analysis (AEDA) results; Forero et al., in press), was done by internal standard (IS) method. For that purpose, 2-methyl-3-heptanone (0.3–50 μg) was selected as standard, dissolved in the extraction solvent, and added to homogenised fruit pulp before extraction. Calibration curves were constructed using a series of solutions of varying nominal concentrations containing each analyte (IS:analyte from 1:5 to 5:1), where the slope was assumed as the response factor. An identical amount of internal standard was added to each solution and the corresponding chromatograms obtained (IOFI Working Group on Methods of Analysis, 2011). All data were obtained in triplicate. The concentration of each analyte was calculated by comparison of GC-FID signals with those of standards, taking into account the relative response factor, according to the following equation:

$$[\ ]x = \frac{A_x}{A_{istd}} * \frac{\mu\text{g } istd}{\text{kg } Fruit} * RF \tag{1}$$

where [ ]*x* is the analyte concentration in mg/kg fruit, A_x is the analyte area, A_{istd} is the internal standard area, and *RF* is the response factor.

Linear retention indices (LRI) were calculated according to the Kovats method using a mixture of normal alkanes C_6–C_{26} as external references. Structural assignments were confirmed by comparison of retention indices (on two columns), mass spectrum and odour notes with those exhibited by solutions of standards (50 μg/mL). Key-aroma compounds were determined by their *OAV* (odour activity value = concentration divided by odour threshold; Grosch, 1994).

2.5. Gas chromatography (GC-FID) and gas chromatography–mass spectrometry (GC–MS)

GC–MS analyses were carried out on a 5973 mass selective detector coupled to an Agilent 6890 gas chromatograph (Agilent Technologies Inc. Wilmington, DE), by using a DB-FFAP capillary column (30 m × 0.25 mm, 0.32 μm film thickness; J&W Scientific, Chromatographie-Handel Müller, Fridolfing, Germany). The carrier gas was helium with a flow of 1 mL/min. The column oven was programmed from 40 °C (after 1 min) to 180 °C at 4 °C/min, then at 12 °C/min until 250 °C. MS data were recorded from *m*/*z* 30 to *m*/*z* 350, with electron energy of 70 eV, and processed by ChemStation software (Rev. E01). GC-FID analyses were performed using a gas chromatograph HP 5890 Series II (Hewlett–Packard) equipped with an FID and operated in split mode (1:10; injected volume, 1 μL). The injection port was set at 240 °C and helium was used as carrier gas at 1.0 mL/min. Chromatographic conditions were the same as for the GC–MS analysis.