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Vitamin characterization and volatile composition of camu-camu (*Myrciaria dubia* (HBK) McVaugh, Myrtaceae) at different maturation stages

Francisca das Chagas do Amaral SOUZA[1], Edson Pablo SILVA[2*], Jaime Paiva Lopes AGUIAR[1*]

Abstract

During the process of fruit development there is synthesis and catabolism of compounds that culminate with the specific characteristics of each species, such as changes in color, size, acidity, vitamins content and volatile composition. In the present study was evaluated during the development of camu-camu (*Myrciaria dubia* (HBK) McVaugh, Myrtaceae) the contents of pH, At, SS (ºBrix), Vitamin C, polyphenols, (L *, a * and b *) coloration and volatile composition using the HSPME-CGMS technique, at different stages of development. During development, an increase in the ºbrix variable was detected (6.92 to 9.02), and a decrease in the levels of vitamin C 1150 mg/100 g and total polyphenols 1280mg / 100g. Regarding the volatile composition, there was a difference of compounds according to the stage of development analyzed. Among the identified components, terpenes were the major class of compounds at all stages of maturation ($p<0.05$).

Keywords: CG/MS; volatile; maturation; food analysis; exotic fruits.

Practical Applications: Amazon fruits are an important source of nutrients acting in human metabolism, with a big interest in the chemical, food, and pharmaceutical industries. The camu camu presented a high value for vitamins and the characteristics of a volatil composition rich in terpene compounds.

1 Introduction

Natural volatile compounds include various chemical substances, such as esters, lactones, alcohols, acids, aldehydes, ketones, hydrocarbons, and some phenols, ethers, and heterocyclic compounds. These chemical substances play vital roles throughout a plant's life cycle by facilitating interactions with the environment in which they live. The substances, which are produced by plants for various reasons, form part of their immune system and determine the characteristic aroma of each plant (Janzantti et al., 2012; Simões, 2003). Aldehydes, alcohols, esters, and acids, which are important groups for the aromatic characteristics of various fruits, are likely formed via a lipid metabolism pathway (Knee, 2002).

Camu-camu (*Myrciaria dubia* (HBK) McVaugh, Myrtaceae), a typical Amazonian fruit tree, is naturally found on the banks of rivers, lakes, and igapós, both in dark and clear waters. These fruits have an agreeable appearance and taste (Yuyama et al., 2002). Camu-camu has high nutritional value because of the high vitamin C (1380-1490 mg/100 g pulp and 2050 mg/100 g peel) and potassium content in this fruit, in addition to the presence of carotenoids, such as anthocyanins (Zanatta & Mercadante 2007). However, although these substances have important physiological functions, such as mediating interactions between other plants and defending against microorganisms, they can also be volatile, because many of these substances are monoterpenes (Pontes et al., 2009; Aguiar et al., 2019). There are no studies related to the volatile composition of these substances. Some volatile compounds, such as d-limonene, can treat sputum and have anticancer activity. Β-Linalool is used to treat a variety of diseases and has anti-inflammatory properties, while (E)-α-bergamotene and (E)-β-farnesene indirectly defend the plants against lepidopteran larvae (Rodríguez et al., 2011). The analysis of the volatile compounds is of extreme importance for the characterization of new genetic materials, because the identification of these allows the discovery of new compounds that can be used in the food industry. Additionally, studying the formation or changes of the volatile compound composition during the maturation, processing, and storage of the fruits and their products can increase the understanding of how flavor is formed (Cozzolino et al., 2001). Moreover, the aroma is one of the most expressive characteristics of a food, and the characterization of the aromatic profile can represent a useful tool to evaluate the organoleptic properties, as well as can be used to guarantee the authenticity of the fruit (Janzantti et al., 2012). In the early 1990s, headspace solid-phase microextraction (HSPME) emerged as a promising alternative for conventional techniques in the isolation of headspace volatile compounds (Ma et al., 2013).

HSPME, which combines the extraction and preconcentration in a single step, is one of the most modern techniques used for extraction. It has many advantages, such as high sensitivity, low-cost, solventless, and simplicity, over the conventional extraction techniques (Ma et al., 2013; Pontes et al., 2009). Because of the high sensitivity of this assay, it can be used to identify the volatile fruit profile, especially in fruits without a present study on their volatile composition. In this study, we

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[1]*Laboratório de Fisico-química de alimentos – LFQA, Instituto Nacional de Pesquisas na Amazônia, Manaus, AM, Brasil*
[2]*Centro de Biotecnologia da Amazônia – CBA/SUFRAMA, Manaus, AM, Brasil*
*Corresponding author: edsonpablos@hotmail.com; jaguiar@inpa.gov.br

determine the chemical and volatile composition of camu-camu (*Myrciaria dubia* (H.B.K.) McVaugh, Myrtaceae) in CG / MS at different stages of development using HSPME.

2 Material and methods

2.1. Plant material and evaluation of fruit

Fruits were harvested at random in the morning from January 2019 to March 2019 at the INPA (National Institute for Research in the Amazon) Campus in Manaus, Brazil (latitude 3º092 '820 "S, 59º994`046" O longitude). The fruits were divided into three equal batches according to the stages of development immature green, mature green and ripened representing the fruits of the day. The fruits were frozen in liquid nitrogen and stored in a freezer until the time of analysis.

2.1.2 Chemical characterization

Total soluble solids (TSS) of the pulp were measured with a digital refractometer 'Palette' PR-100 (ATAGO U.S.A., Inc.), with an automatic temperature compensation at 25 °C (Association of Official Agricultural Chemists, 2010) and the results were expressed as °Brix. The results for titratable acidity were expressed as 100 g-1 pulp *in natura*, considering citric acid as the predominant acid, during the development stages. The pH of the pulp was measured with a pH meter, Tec-3P-MP (TECNAL – Sao Paulo, Brazil) according to the methods described Association of Official Agricultural Chemists (2010). All the experiments were repeated 6 times independently. Colour of the peel (L *, a *, b *) was determined at different points using a colour measuring spectrophotometer (HunterLab ColorQUEST II Sphere) – Sao Paulo, Brazil.

2.1.3 Total polyphenols

The content were reference measured by a photometric Folin-Ciocalteu assay according to a proposed international standard method (Association of Official Agricultural Chemists, 2010). Absorbance (E) at 540 nm of the reaction solution is determined in a 1 cm light-path cell by a Lengguang-752 spectrophotometer (Shimadzu Optical Instrument Sao Paulo, Brazil). The calibration standard is gallic acid.

2.1.4 Vitamin C

L-Ascorbic acid (vitamin C) and metaphosphoric acid were purchased from Merck (A Coruña, Spain). All analyses were carried out in duplicate on tissue vegetable homogenized using a pestle and mortar. The vitamin C was extracted as described by Association of Official Agricultural Chemists (2010). Twenty grams of homogenate was mechanically stirred in 60 ml of a 4É5% (w/v) solution of metaphosphoric acid for 15 min. The mixture was filtered (Whatman No 541), and the was diluted to100 ml with HPLC grade water. An aliquot of the acid extract was then filtered through 0.45μm Millipore filter prior to injection into the chromatographic column. The HPLC apparatus used consisted of a Spectra-Physics liquid chromatograph equipped with anSP8800 ternary pump and a Rheodyne 20 μL injection loop and a Spectra Focus UVÈVis forward optical scanning detector controlled by Spectra Focus software. The column was a Tracer ODS2 C18 column (4 id x250 mm) of particle size 5μm (Tecknocromc,Vigo, Spain) ; and was used with a precolumn (Tecknocromc TR-015326) packed with the same material. The mobile phase was HPLC grade water brought to pH 2.2 with metaphosphoric acid; the flow rate was 0.5ml.min^{-1}, the detection wavelength was 245 nm. Quantitation used the external standard method. This method was developed in our laboratory and it is described in detail (including repeatability, recovery and limit of detection) in a previous paper Association of Official Agricultural Chemists (2010).

2.2 Volatile compounds

2.2.1 Fiber conditioning and extraction of volatile compounds

Volatile compounds were extracted by the HS-SPME technique. Pulp (sample, 1 g) was transferred to a 10 mL glass vial (suitable for volatile retention) which was continuously stirred at 50 °C for 15 min. The 50/30 μm DVB/CAR/PDMS fibre (divinylbenzene/carboxen/polydimethylsiloxane) (Supelco, Bellefonte, PA, USA) was used to separate the volatile compounds present in the sample. The fibre was packed at a temperature of 270 °C for 1 h prior to use. The pre-conditioning time for the analytes was 25 min. The fibre was exposed to the headspace of glass vial containing the sample at 50 °C for 15 min, the syringe was immediately taken to the CG-MS injector, wherein, the volatile compounds were desorbed at 250 °C for 2 min resulting in a splitless injection.

A spectrometer, CGMS-2010 Plus (Shimadzu) Tokyo, Japan with a mass selective detector model QP2010 Plus was used to detect the volatile compounds. A capillary column of fused silica (30 m × 0.25 mm and 0.25 μm thick) with 5% of diphenyl-, 95% polydimethylsiloxane polymer (DB5) acting as a stationary phase. For best separation, temperature gradient was established in the column initiating from 60 °C, with an increase of 3 °C per min until the maximum temperature of 270 °C was attained. The carrier gas was helium and the flow rate was adjusted to 1.8 mL min^{-1} for splitless injection with initial pressure of 100 KPa in the column. The conditions adjusted in the mass spectrometer (MS) were: mass selective detector operating by electronic impact and impact energy of 70 eV; scanning speed of 1000 m/z s^{-1}; scan interval of 0.5 fragments/sec and filter for mass of the detected fragments being 29 Da and 600 Da. Each component was identified by comparing its mass spectra with already existing information present in the spectrometer databases (Willey229.lib and FFSC1.3.Lib) and the component identification book by Adams (2007). For comparing and calculating the indices, standards of the saturated alkanes (C7-C30) (Supelco, Sigma-Aldrich, Bellefonte, PA, USA) were used as a reference to determine the retention index (RI).

2.4 Statistical analysis

Statistical analysis of the chemical and physical variables was performed with the support of SISVAR program (Ferreira, 2010). Volatile compound composition profile data were analyzed using a multivariate statistical analysis using the PCA and HCA techniques using the Sensomaker software, version 1.91 (Nunes & Pinheiro, 2017).

3. Results and discussion

The fruit ripening process includes a series of biochemical steps, where the compounds within the fruit are catabolized or synthesized. For example, the degradation of chlorophyll and synthesis of the phenolic compounds results in color changes. The chemical characterization of the camu-camu (Table 1) throughout the development, detected significant ($p<0.05$) levels of vitamin C and total polyphenols, compounds with effective bioactive activity, besides influencing the metabolism of other secondary compounds such as volatiles. The increase observed in vitamin c and polyphenols compounds antioxidant during the development of fruit demonstrate the capacity these fruit with source of nutrients with high aggregate value biologic, that can be important in your maintenance and utilization for food human nutrition. Observed that changes in polyphenols content are similar with the change in the volatile compounds during of development stages. The degradation and synthesis of new volatile compounds occur through the end of the physiological process. Silva et al. (2013) reports that volatile compounds are of great importance during the development process, because they facilitate the maintenance and perpetuation of the species. These compounds are responsible for the characteristic aroma and can be synthesized during development, post-harvest, and storage. The overall composition in the fruit is determined by several factors, such as the species, variety, and technological treatments used (Rizzolo et al., 1992).

For the color results, a significant increase was observed in all variables (L *, a * and b *) (Table 1), highlighting the values detected in a * and b *, which reflect the intensification of the red color (Figure 1), which may be correlated with carotenoid deboning and synthesis and chlorophyll degradation. These values may also be correlated with the results obtained for volatiles, since this variation is closely correlated with terpenoid synthesis. According to Yuan et al. (2015), tetraterpenes or carotenoids, are widely distributed pigments in nature, responsible for checking the coloration of different plants, vegetables and foods with a spectrum of colors ranging from yellow to red. Among the very important functions performed by this class of terpenes in nature

Table 1: Chemical composition of the camu-camu in three stages of maturation.

Variables		Green fruit	Mature green	Ripened
pH		2.23b±0.31	3.68a±0.25	2.41b±0.22
At(g de citric acid.100g-1)		2.75a±0.21	2.77a±0.12	2.4a±0.18
SS (°Brix)		6.92b±0.82	7.73b±0.52	9.02a±0.75
Vitamin C (mg/100g ascorbic acid)		1230b±2.56	1264a±2.63	1150c±1.35
Polyphenls Total (mg/100g gallic acid)		1380a±2.72	1352a±1.61	1280b±1.21
Colour values	L*	63.83a±3.2	54.20b±2.35	26.77c±1.52
	a*	8.56c±1.23	13.32b±1.36	20.78a±1.70
	b*	35.30a±2.32	25.36b±1.85	6.79c±0.85

Means followed by the same letter in the line do not differ from each other ($p <0.05$).

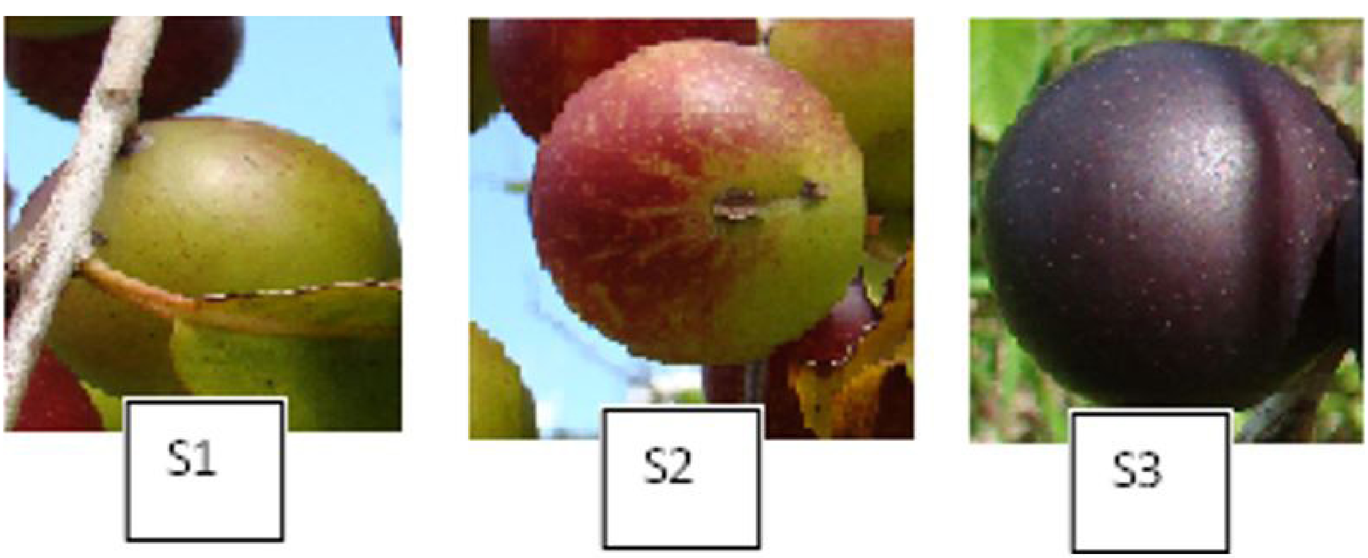

Figure 1. Camu-camu at different ripening stages. 1: Green camu-camu fruit; 2: camu-camu fruit beginning the ripening; 3: camu-camu fruit at intermediate stage of ripening;

are: (i) are considered accessory pigments of photosynthesis in plants, presenting maximum absorption in the range of ultraviolet and blue; (ii) in animal tissues, some of them are precursors of vitamin A; (iii) certain carotenoids have significant antioxidant activity (Souza et al., 2013).

The results obtained in this study corroborate this difference, as the volatile compound composition of camu-camu changed in different phases of ripening, along with the color of the fruits (Table 2, Figure 2). A large percentage of volatile compounds was identified for fruits in the immature green stage (90.9% were identified), the fruit stage mature green (92.30% were identified), and the mature fruit (84.61% were identified).

The compounds were identified including hydrocarbons (heptane 6.11%, decane 5.77%), alcohols (Hepten-1-ol (4Z) 3.74%), esters (Methyl butyl- 2-methyl butyrate <2-> 9.05%), and terpenes (caryophyllene 31.02%, tricyclene 14.02%, limonene 10.01%, sabinene 7.21%, isocitrinellene 4.78%, pinene 3.39%); terpenes represented the highest percentage of volatile compounds (Figure 2, Table 2). Zhang et al. (2017) also found that terpenes were the major class of volatile compounds in different citrus species. In plants, monoterpenes exert a fungicidal role and attract pollinators (Chitarra & Chitarra, 2005). The high content of terpenes, such as caryophyllene, which is a sesquiterpene compound found in the essential oils of many different species (Molina-Jasso et al., 2009), found in green fruit may also

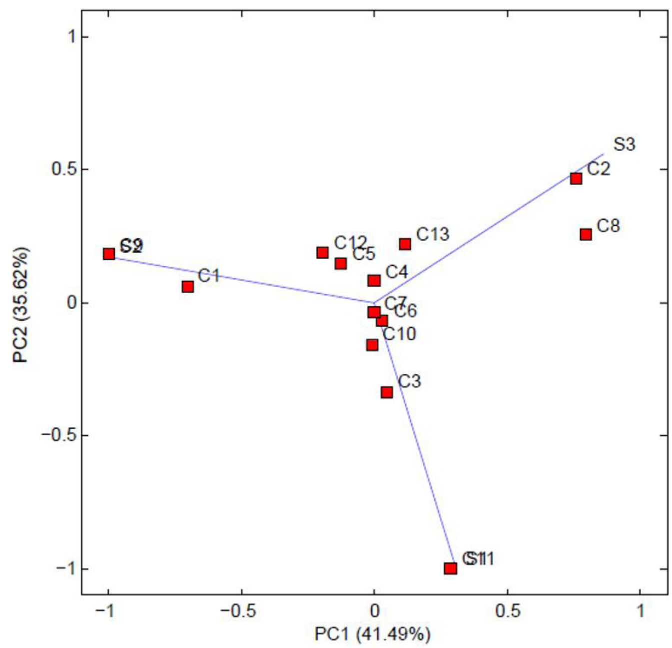

Figure 2. Principal component analysis (PCA) for the volatile profile of camu-camu fruit at different ripening stages. S1, S2 and S3. C: Identification of the compounds according to Table 2.

Table 2. Volatile compounds of the camu-camu in different ripening stages.

ID	*Compounds	**RI	Stage (% area)			±
			S1	S2	S3	Odors***
1	Carene α-3	1011	0	9.04	6.98	Citrus
2	Carophyllene-E-	1419	31.02	5.98	6.15	Citrus
3	Cymene -p-	1024	0	2.8	3.05	Floral
4	Decane (n)	1000	5.77	0	0	Fruit
5	Isocitronellene	923	4.78	0	0	Fruit
6	Heptane	700	6.11	0	0	Fruit
7	Hepten-1-ol	966	3.74	0	0	Fruit
8	Limonene	1029	10.01	32.1	27.54	Citrus
9	Methyl-2-butyrate	1100	9.05	0	0	Fruit
10	Myrcene	990	0	3.2	4.56	Spicy
11	Pentanoic acid 4 methyl	940	0	2.74	0	Fruit
12	Pinene β	979	3.39	4.96	3.75	Fruit
13	Phellandrene β	1029	0	2.73	0	Fennel
14	Sabinene	975	7.21	0	0	Citrus
15	Sylvestrene - iso-	1008	0	3.38	4.25	Fruit
16	Terpinen-4-ol	1177	0	0	2.63	Herbal
17	Terpinene α	1059	0	4.01	3.92	Woddy
18	Thujene-α-	930	0	2.8	3.11	Fruit
19	Tricyclene	926	14.2	23.26	28.35	Green

S1, S2 and S3: stages green fruit, mature green and ripened respectively. * Identification of compounds according by bibliography Willey229.Lib, FFSC1.3.Lib and Adams, 2007. ** RI = retention index on DB-5 capillary column. *** (The Good Scents Company, 2019).

be associated with the plant's defense system. In addition, caryophyllene is associated with anti-inflammatory activities, gastric cytoprotective effects, antioxidants, and other functions (Tambe et al., 1996). An increase in the concentration of tricyclene and limonene was observed during development; this increase was associated with the synthesis of phenolic compounds in the fruit. According to Aguiar et al. (2019), limonene is the major component of lemon and orange peel oils, as well as alkaline essential oils, and is effective in preventing dehydration and inhibiting microbial growth in plants.

Similar to the green fruit stage, terpenes were the dominant class of identified compounds (Figure 2, Table 2). However, a change was observed in the percentage of the major compounds; a significant increase was detected in tricyclene (from 14.2% to 23.26%) and limonene (from 10.01% to 32.1%). A drastic reduction of caryophyllene, which was the major compound in the green fruit, was observed (from 31.02% to 5.98%) in the fruit at this stage. These same compounds were detected by Caprioli et al. (2016), Kupska et al. (2014) and Dabbou et al. (2016) in Concomitant to these changes, there is an accentuation of the purple color in the fruits, a factor that may be related to the degradation of chlorophyll and synthesis of pigments. The route of formation of these compounds occurs via the mevalonate route and 1-deoxylylulose 5-phosphate (DXP) pathway, which give rise to the different terpenes (Kitaoka et al., 2015). According to Yuan et al. (2015), terpenes and carotenoids have a wide distribution in nature responsible for conferring the color and aroma of different plants, vegetables, and foods. The color spectrum ranges from yellow to red. There is an extensive variety of tetraterpenes, which are divided into carotenes (terpenes) and xanthophylls (terpenoids).

In the ripened fruits, peaks were identified, which represents a percentage of 84.61% (Figure 2, Table 2). Similar to the other stages of development, the terpene class predominated the volatile compounds; unlike the other stages of development, there was a significant increase in tricyclene, which increased from 14.2% in the immature green camu-camu to 23.26% mature green, reaching 28.35% in ripened camu-camu. A high limonene content of 27.54% was also observed. The high content of terpenoids detected with the advancement of ripening is directly related to the degradation of chlorophyll and synthesis of carotenoids, a class which comprises the terpenes and their homologs. Aguiar et al. (2019), evaluate the chemical and volatile composition of an Amazonian fruit, detected similar results in relation to terpenes as major compounds, with emphasis on alpha-pinene and limonene. Martineli et al. (2013), and Franco et al. (2004) also detected terpenes as the major class when analyzing the volatile compounds in persimmon and mango cultivars, respectively. Mono- and sesqui-terpenes, phenolic derivatives, lipid derived compounds, compounds derived from amino acids, and compounds derived from the breakdown of carotenoids, among others, are the most important compounds responsible for aroma (Lewinsohn et al., 2005). Kupska et al. (2014) observed a similar results in your studied about comprehensive two-dimensional gas chromatography for determination of the terpenes profile of berries. According to Edagi & Kluge (2009), during the fruit ripening process, ethylene induces the formation of compounds responsible for the aroma of the fruit. According to Chitarra & Chitarra (2005), in the post-harvest phase, the aroma changes based on the degree of maturation, harvest season, nutrition, handling, storage, and artificial maturing.

3 Conclusion

Terpenes were the dominate class of volatile compounds identified by HSPME-CG/MS in the maturation stages evaluated. In the immature green stage, the compound with the highest percentage of area was caryophyllene 31.02%; in the mature green stage, limonene 32.01% had the highest percentage of area, and in the ripened stage, tricyclene 28.35% had the highest percentage of area. This identification provides important information for the food, flavor, and pharmaceutical industries, because these compounds have proven antioxidant and antimicrobial roles, demonstrating their viability in food, cosmetics, and perfumes. In the chemical composition, vitamin C contents and total polyphenols found in mature fruits

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