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HS-SPME-GC-MS detection of volatile compounds in *Myrciaria jabuticaba* Fruit

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Abstract

This study was performed to investigate the volatile compounds for the characteristic aroma in jabuticaba fruit distributed in southern and central regions of Brazil. The present work combines headspace solid phase microextraction (HS-SPME) and high resolution gas chromatography-mass spectrometry (GC-MS) techniques to identify and quantify the volatile compounds. The influence of different SPME fibers (CAR/PDMS and DVB/CAR/PDMS) in extraction of volatile compounds was evaluated. The effects of extraction temperature and salt concentration (NaCl) in the extraction medium were studied using the response surface methodology in order to achieve the highest extraction efficiency. The better extraction of volatile compounds was achieved by using a CAR/PDMS fiber and the optimum adsorption conditions were at 42 °C for 30 min and 5% NaCl concentration. A total of 71 compounds were identified, among these, 57% were terpenes which was the most representative class of compounds, followed by esters (19%), aldehydes (10%), alcohols (5.5%) and aromatics compounds (4.4%) and other organic compounds 2.8%. Limonene and ethyl acetate were the volatile compounds that showed highest relative concentration and these could contribute to the characteristic aroma of the jabuticaba fruit along with other compounds such as β-pinene, δ-cadinene, linalool, β-guaiene, and α-caryophyllene.

Keywords: jabuticaba; *Myrciaria*; volatile compounds; headspace solid phase microextraction; gas chromatography mass spectrometry.

1. Introduction

Jabuticaba is a Brazilian native fruit from the Atlantic rainforest that belongs to the Myrtaceae family, grape-like in appearance and texture. Its economic importance has been continuously growing in Brazil because of the sweet and slightly acidic flavor of the pulp. Jabuticaba is a small fruit, grape-like in appearance and is one of Brazil's richest sources in anthocyanins. The most common varieties are *Myrciaria cauliflora* (DC) O. Berg, *Myrciaria jabuticaba* (Vell) Berg and jabuticaba-assú. The fruit grows directly on the main trunk and branches, has a diameter verying from 1-4 centimeters, with 1 - 4 large seeds per fruit and richly colorful epicarpio covering the white sweet pulp and jelly-like in appearance (Reynertson *et al.*, 2006). It is mostly consumed as fresh fruit or turned into various industrial products are also availablesuch as juice, jellies, wines and liquors.

The interest of this fruit has grown in recent years because it has in its chemical composition phenolic constituents such as anthocyanins, flavonoids and ellagitannins. The fruit is also reported to possess well defined biological properties including antioxidant and anti-inflammatory activities (Middleton *et al.*, 2000). Although the jabuticaba is a fruit with important functional properties for health the components that give it a pleasant taste and aroma have not yet been investigated. The aroma is one of the most important quality attributes of tropical and sub-tropical fruits and numerous analytical techniques have been developed to characterize the compounds responsible

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for its aroma quality. Most of the volatile compounds of the fruit are composed of saturated and unsaturated molecules from aliphatic compounds that have functional oxygen groups such as ester, alcohol, acid, aldehyde, ketone or lactone, which originate during the maturation process by different metabolic pathways. A recent study reported that the high sugar content associated with terpenes contribute to result in sweet, acidic and slight resinous flavor of the jabuticaba fruits (Plagemann *et al.*, 2012).

The flavour impact is a complex mixture of volatile organic chemicals and taste contributors. Quality perception cannot be analysed and it is seldom possible to trace the complex composition of flavour and taste. However, one of the most interesting techniques for studying flavour quality is HS-SPME (Souza-Silva *et al.*, 2015). There is growing interest in this field to elucidate whether the flavour profiles of fresh fruits and vegetables are affected by the cultivation system (Vallverdú-Queralt and Lamuela-Raventós, 2016).

The development of fast and simple methods for volatiles extraction is important because it can reduce the sampling time and analysis can be performed quickly. A fast sampling technique that can be adapted for gas chromatography (GC) analysis of volatile compounds is the solid phase microextraction (SPME). This technique has been applied for the analysis of volatile and nonvolatile compounds in liquid and gaseous samples (Arthur and Pawliszyn, 1990) and to analyze the flavor in juices and vegetable oils (Yang and Peppard, 1994). SPME is a pretty fast and reliable technique for the determination of volatile compounds in complex matrices. It iss based on the extraction of analytes from the sample matrix using a fused silica fiber (1 - 2 cm), coated with a thin film of absorbent material which in most cases is of polymeric nature. Subsequently, in order to separate and identify the organic compounds, the desorption of analytes is carried out by increasing the temperature, where the fiber is held into the GC injection port for its instrumental analysis (Lasekan and Abbas, 2010). HS-SPME technique has been used to evaluate the pitomba fruit (*Talisia esculenta* Radlk.) (De Souza *et al.*, 2016); strawberries, raspberries, blackberries and bananas (Ibanez *et al.*, 1998); Bayberry (Cheng *et al.*, 2015); passion fruit, cashew apple, tamarind, guava and acerola (Carasek and Pawliszyn, 2006); grape berry (Parker *et al.*, 2007) and mango (Liu *et al.*, 2013).

In this study a new system was developed consisting of HS-SPME for the capture of volatile compounds on fiber containing different porous material, followed by desorption of volatile compounds onto a system of GC-MS, wherein a capillary column was used to separate the volatile compounds, identify and quantify these compounds present in jabuticaba fruit pulp. The study was performed to select the best fiber for the volatile analysis, along with the optimization of the extraction conditions such as the temperature and ionic strength of extraction media and analyzing the data obtained by response surface methodology interpretation.

2. Materials and methods

Sample

Fresh ripe jabuticaba fruits having no apparent injury or microbial spoilage were purchased from a local market of Aracaju city in the state of Sergipe, Brazil. The fruits were washed with water and disinfected using 1% sodium hypochlorite solution whereafter the fruits were again washed with water. The pulp of jabuticaba fruit was removed manually using a stainless steel knife. After the extraction of the seed, the pulp and peel were homogenized in a waring blender without addition of water, then it was filtered and the resulting homogenate was kept in polythene bags and stored in a freezer maintained at -20 °C until further analysis. For the extraction of volatiles compounds, the samples were thawed at room temperature and diluted with distilled water (Milli-Q Integral Water Purification System) in an aqueous solution of sodium chloride (Sigma Chemical Co., Aldrich Chemical Co.) at different concentrations. The final proportion of pulp and the solution was of 3.5:10.

Selection of SPME fibers in the extraction of volatile compounds

Tests were performed using different fibers such as carboxen/polydimethylsiloxane (*CAR/PDMS*; 85 µm) and divinylbenzene/Carboxen/polydimethylsiloxane (*DVB/CAR/PDMS;* 50/30 µm), both supplied by Supelco (Bellefonte, PA, USA) and these were evaluated to verify the ability to collect and concentrate the headspace volatiles of jabuticaba homogenate,mainly due to their sorption characteristics. These fibers were exposed to HS using SPME holder in 40 ml vials containing a mixture of 3.5 g of fruit homogenate and 10 ml of deionized water. The vials were sealed with a polypropylene plug and extraction perfor-

med for 30 min at 25 °C under constant magnetic stirring. After the extraction, SPME fibers were removed from the vials and inserted into the GC injection system (Agilent GC system model 7820A) - *MS* (Agilent MS model 5975), for the separation and identification of volatiles. Experiments containing only fruit homogenate with distilled water were performed between the extraction processes also to verify the results that there was no interference from earlier runs.

Extraction Conditions

After the selection of the best SPME fiber for sorption of volatiles of jabuticaba homogenate, the best experimental conditions for the extraction of volatiles by HS-SPME were determined. The Response Surface methodology (RSM) was used wherein the independent variables were salt concentration or ionic strength (IS) and extraction temperature (T). The data were interpreted on the basis of the dependent variable being the number of detected peaks (NP). A Central Composite Design (CCD) with two variables at five levels (-1, 1.41, 0, +1, 1.41) was used and the center point was replicated six times, totalizing 14 experiments. The experimental conditions in the center point were: salt concentration 15% NaCl and 35 °C. The experimental values varied were: X_1 = ionic strength (0, 5, 15, 26 and 30%) and X_2 = temperature (25, 28, 35, 42 and 45 °C). For HS-SPME analysis, 3.5 g of homogenate was mixed with 10 ml aqueous solution of NaCl at different concentrations in a hermetically sealed 40 mL vials with a polypropylene plug. Subsequently the mixture was heated at different temperatures (25, 28, 35, 42 and 45 °C) in thermostatic bath for 15 min in order to reach thermal equilibrium with constant magnetic stirring. Later the selected fiber was inserted into the vial and was exposed to HS for 30 minutes to adsorb the volatiles. The SPME fiber was immediately taken to the GC injection port and thermally desorbed for 8 min at 250 °C in split mode. The number of detected peaks in each run was used as a response variable of the experimental design and a quadratic model was constructed to describe the observed data.

Chromatographic analysis

Chromatographic analysis was performed using GC-MS system equipped with a DB-5 capillary column (inner diameter: 30 m x 0.25 mm, film thickness: 0.25 µm). The oven temperature was programmed initially at 40 °C for 4 min, then increased at the rate of 7 °C/min to 160 °C wherein held for 8 min, subsequently the temperature raised at 15 °C/min up to 250 °C and maintained for 3 min at this temperature, totalizing 38 min of chromatographic analysis. Helium was used as carrier gas at a constant rate of 1.0 mL/min. The mass spectrometer was operated in scan mode from 29 – 550 m/z, at 70 eV ionization.

The identification of volatiles compounds was performed by calculating the linear retention index (LRI) of each compound using a series of alkane standards analyzed under identical conditions as that of the samples and comparing the mass spectrum of the compound peaks with mass spectra library of NIST/EPA (NIH version 2.0). The values of LRI of each compound were also compared with those described in the literature as well as from the data base of compounds identification in our laboratory wherein standards analysis was performed in identical analytical conditions. The relative concentrations of individual components were expressed as a percentage of the relative peak area to that of the total peak area.

Statistical analysis

The statistical significance of the model was evaluated by analysis of variance (ANOVA) with significance level of 5% ($p < 0,05$). Statistica V.7 (Statsoft Inc., Tulsa, OK, USA) was used in order to generate ANOVA and response surface methodology interpretations.

3. Results and discussion

Optimization of extraction conditions using HS-SPME

The extraction time, extraction temperature, mass of the sample and sodium chloride concentration are the factors which influence the vapor pressure of the volatiles in the headspace. The optimization method evaluated only considered the effect of the salt addition associated with the adsorption temperature, while the other variables remained constant during the tests. The response was evaluated to determine the best extraction conditions for HS-SPME based on the analysis of chromatograms relating to higher number of identified volatile compounds and the intensity of the response. However, before optimization of the extraction conditions, the adsorption profile of the 3 SPME fibers was evaluated and in later experiments only the selected fiber was used.

SPME Fibers

The selection of the most suitable SPME fiber depends on the target compounds and the sample under study (Gioti *et al.*, 2007).

Two semi polar fibers - *CAR/PDMS* and DVB/*CAR/PDMS* were tested, and compared individually. The results reported in Figure 1A show that the CAR/PDMS fiber resulted in a better profile having high extraction efficiency of volatile compounds in jabuticaba as it resulted in greater number of detected compounds and their signal strength. CAR/PDMS fiber coating is most suitable for the extraction of small volatile molecules. The sensitivity of this fiber into smaller molecules, such as esters, acids and non-polar compounds, allows the extraction of a broad range of aromatic compounds (Érica *et al.*, 2017). The compounds with the highest intensity were the ethyl acetate (22.04%) and limonene (11.47%) (Figure 1B). Furthermore, the main components adsorbed were the terpenoids, including α-pinene, β-pinene, β-ocimenoe, β-caryophyllene, α-muurolene, δ-cadinene, calamenene and limonene, which could be responsible for the characteristic aroma of the fruit. Considering better results obtained by using the CAR/PDMS fiber, it was selected for further studies of the characterization of volatile compounds of jabuticaba fruit.

Effects of the combination of sodium chloride and extraction temperature

The response surface methodology (RSM) is a powerful tool that enables quickly and efficiently to interpret the data while having only a minimum number of experiments. A total of 14 experiments (Table 1) were performed in random combinations of temperature and ionic strength to check the effects of the combination of sodium chloride and the extraction temperature, the results are presented in Figure 2.

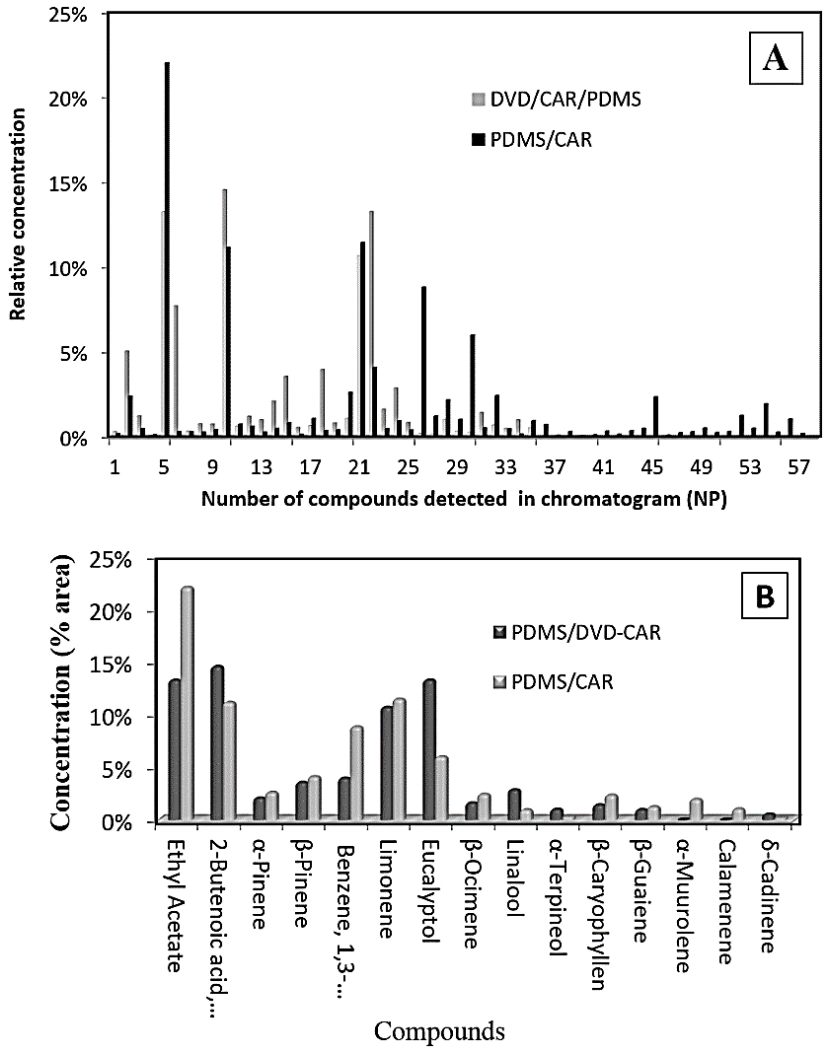

Figure 1. Influence of the fiber (PDMS/DVD/CAR and PDMS/CAR) on the extraction of volatile compounds from jabuticaba homogenate: A - Relative concentration (%) of the number of detected peaks; B - Concentration (%) of the main detected compounds.

Table 1
Description of independent variables analyzed in Central Composite Design showing the experimental and predicted values of number of peaks in chromatogram

	Coded Levels		Independent variables		Peak number	
Experiment/N°	X1	X2	IS	T / °C	Experimental	Predicted
1	-1	-1	5	28	53	55.011
2	-1	1	5	42	89	87.052
3	1	-1	26	28	46	50.700
4	1	1	26	42	81	81.741
5	0	0	15	35	63	62.167
6	0	0	15	35	63	62.167
7	0	0	15	35	62	62.167
8	-1.414	0	0	35	78	78.525
9	1.414	0	30	35	75	71.722
10	0	-1.414	15	25	44	39.824
11	0	1.414	15	45	83	84.423
12	0	0	15	35	62	62.167
13	0	0	15	35	62	62.167
14	0	0	15	35	61	62.167

(-1.41) Low level, (1.41) High level, (0) Central point, (IS) Ionic Strength and (T) Temperature.

The response surface (Figure 2A) was obtained using mathematical model expressed in coded variables by interpreting the data using Statistica software, which resulted in the following equation:

$$Y_{(NP)} = 62.1669 - 2.4050\,X_1 + 15.7700\,X_2 + 6.4800\,X_1X_1 - 0.0150\,X_2X_2 - 0.2500X_1X_2 \text{ (eq.1)}$$

Salt concentration (X_1) and temperature (X_2), as well as quadratic term ionic strength (X_1X_1) were significant ($p < 0.05$) and these influenced the volatile compounds extraction as shown in the Pareto Chart (Figure 1B). It can be observed that the temperature was the most significant parameter ($p < 0.05$), showing a strong positive influence. The model had a value R = 0.97, considered appropriate for extraction and values greater than 0.90 are considered statistically significant. It was also observed that the maximum number of detected compounds were found at the surface of the peak, this fact could indicate the best experimental conditions. According to this model, the best combination of extraction was at 42 °C with 5% NaCl, wherein 89 volatiles compounds were separated as shown in the chromatogram (Figure 3).

CCD was an important tool in the optimization of the extraction method of volatiles because traditional optimization methods evaluate only the effect of a variable while keeping the other valuables constant during the experiments. However, this type of test suffers from the limitation that it does not take into account as to what would happen if other variables also change. The experimental design (response surface + central composite design) used in this study allowed the estimation of the effects of two variables simultaneously.

The experimental design used in this work has been earlier used in studies for the extraction of volatile compounds from several fruits such as cupuassu (Oliveira *et al.*, 2004), orange juice (Mirhosseini and Tan, 2009; Charve *et al.*, 2011), cherimoya (*Annona cherimila*), soursop (*Annona muricata*) (Ferreira *et al.*, 2009; Cheong, *et al.*, 2011) and umbu (*Spondias tuberosas*) (Galvão *et al.*, 2011).

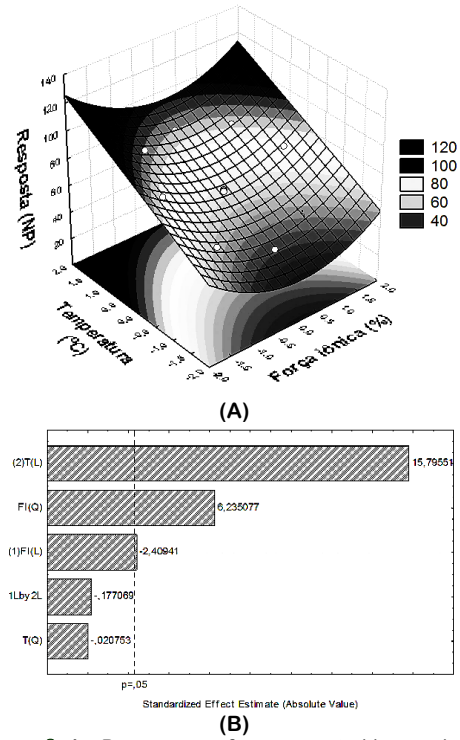

Figure 2. A - Response surface generated by quadratic model (eq. 1) in the optimization of the temperature (T, °C) and ionic strength (IS, %) in the extraction of volatile compounds of jabuticaba fruit using *HS-SPME*; B - Pareto chart showing statistically significant variables ($p < 0.05$) influencing the response during the extraction of volatile compounds.

Identification and quantification of volatile compounds

Figure 3 shows the typical total ion chromatogram obtained under best extrac-

tion conditions. The 15 compounds which are numbered in the chromatogram represent the compounds with higher intensity. Table 2 shows the data on areas of the detected peaks, expressed in percentage, for the different compounds that were used to indicate the relative concentration of each substance along with their characteristic odor note. A total of 71 compounds were identified according to the similarity of their mass spectra and by comparison with the standard values described in the literature.

Among the identified compounds, 41 (57.7%) were terpenes, 14 (19.7%) esters, 7 (9.90%) aldehydes, four (5.60%) alcohols, three (4.20%) aromatic compounds. The compounds that showed the highest concentration were: limonene (17.7%) followed by ethyl acetate (10.7%), 1,3-dichlorobenzene (9.7%), eucalyptol (5.8%) caryophyllene (5.0%), ocimene (3.6%), β-pinene (3.0%), β-guaiene (2.9%), 2-butanoic acid ethyl ester (2.5%) and α-pinene (2.2%).

Plagemann *et al.* (2012) identified only 45 volatile compounds from jabuticaba pulp, among which the β-pinene, δ-cadinene, 2-phenylethanol and linalool were the most prominent compounds in the fruit flavor. These authors also attributed to terpenes as the major contributors to the flavor, indicating that the most odorous compounds were β-caryophyllene and limonene.

In the essential oil of jabuticaba leaves (*Myrciaria cauliflora*), Duarte *et al.* (2010) identified compounds similar to those detected in this study, including α-pinene, β-pinene, limonene, δ-elemene, β-elemene, β-caryophyllene, δ-cadinene and α-muurolene, which may be responsible for the aroma of the fruit.

Comparing the results of this study with those found in other typical fruits of the Amazon of the same genus (*Myrciaria*), there was similarity in the presence of several compounds, such as in the fruit of camu-camu (*Myrciaria dúbia*) showing α-pinene (17.5), limonene (10.8), β-caryophyllene and β-pinene were reported. The β-pinene is also the most important volatile compound in umbu-cajá (*Spondias cintherea*) (Franco and Shibamoto, 2000). In pitanga (*Eugenia inflora* L.) fruit, the presence of β-pinene and β-ocimene as important constituents for aroma of the fruit was also reported (Oliveira *et al.*, 2006). While chinese bayberry had a stronger "pine" odor, which is caused mainly by α-pinene (Cheng *et al.*, 2015).

Based on the identification and quantify-cation of volatile compounds present in the jabuticaba fruit, it could be concluded that the main compounds such as limonene, ethyl acetate, eucalyptol, caryophyllene, ocimene, β-pinene and α-pinene could contribute to the characteristic aroma quality of this fruit.

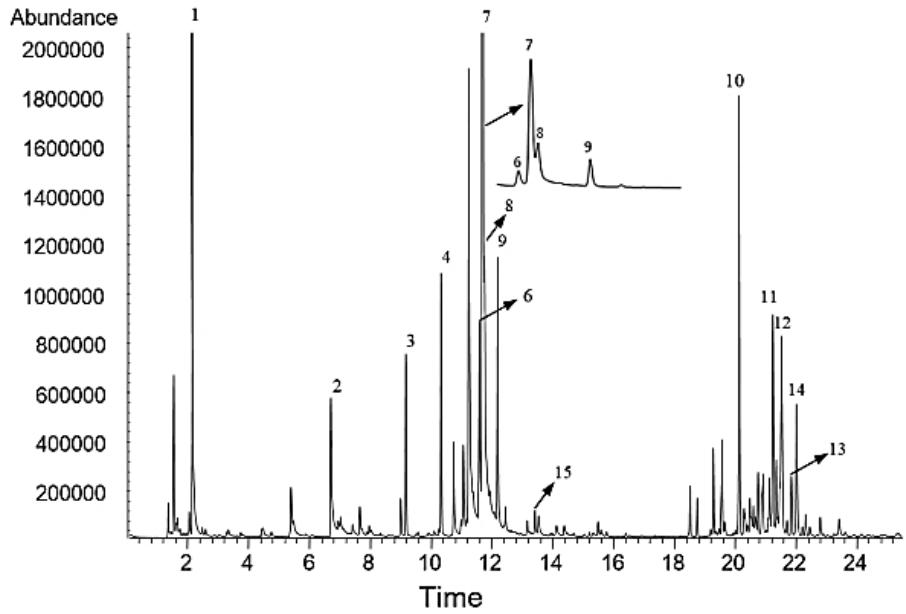

Figure 3. Representative chromatogram of the volatile compounds of jabuticaba homogenate. The predominant peaks and the most striking aroma compounds are numbered as follows: Ethyl acetate (1), 2-butenoic acid, ethyl ester, (E) - (2), α-pinene (3), β- Pinene (4), Benzene 1,3-dichloro- (5), Benzene, 1-methyl-3-(1-methylethyl) - (6), limonene (7), Eucalyptol (8), β-ocimene, (9), β-caryophyllene (10), β - guaieno (11), α-muuroleno (12), δ-cadinene (13), calameneno (14) and linalool (15).

Table 2
Volatile compounds identified in jabuticaba fruit pulp

Compounds[a]	LRI		Relative area percent	Odor description[c]	Identification[d]
	[b]DB-5	Ref.			
Alcohols					
Ethanol	369,59	----	1,75%	sweet	MS
3-methyl-1-butanol	730,45	1,2,3,7	0,05%	whiskey, malt, burnt	MS, LRI
(Z) -3-Hexenol	857,60	1,2,3,7	0,21%	grass	MS, LRI
1-Hexanol	871,34	1,2,3,10	0,18%	resin, flower, green	MS, LRI
Aldehydes					
3-Methyl-1-butanal	581,58	----	0,12%		MS
Pentanal	603,85	----	0,11%	almond, malt, pungent	MS
Hexanal	801,18	1,3,4,5,12	0,79%	grass, tallow, fat	MS, LRI
(E)-2-Hexenal	854,72	1,3,4,5	0,18%	green, leaf	MS, LRI
Nonanal	1105,58	3,4,7,10,12	0,39%	fat, citrus, green	MS, LRI
Decanal	1206,82	1,3,4,6,7,12	0,07%	soap, orange peel, tallow	MS, LRI
Aromatic compounds					
Styrene	890,20	4,10,11	0,14%	balsamic, gasoline	MS, LRI
Benzaldehyde	961,45	1,3,4,7,12	0,02%	almond, burnt sugar	MS, LRI
1,3-dichlorobenzene	1011,55	----	9,70%		MS
p-cymene	1025,73	3,6,11,16	3,13%	solvent, gasoline, citrus	MS, LRI
Esters					
Ethyl Acetate	503,21	----	10,71%	pineapple	MS
Ethyl propanoate	711,06	8	0,05%	Fruit	MS, LRI
Propyl acetate	713,12	8	0,11%	celery, ethereal, fruity, pear, powerful, raspberry	MS, LRI
Methyl (E)-2-butenoate	762,14	3	0,24%		MS
2-Methylpropyl acetate	773,63	----	0,07%	fruit, apple, banana	MS
Ethyl butanoate	803,98	2,3,6,8	0,44%	apple	MS, LRI
Ethyl (E)-2-butenoate	846,42	3,7	2,48%	Fruity	MS, LRI
3-methylbutyil acetate	879,47	----	0,39%	banana	MS
Methyl (E) -2-hexanoate	968,28	9,8,10	0,08%		MS, LRI
Ethyl hexanoate	1001,02	2,3,7,8,9	0,21%	apple peel, fruit	MS, LRI
(Z)-3-hexenyl acetate	1008,73	7,8,11	0,22%	Unripe banana, fruit, green	MS, LRI
(Z)-3-Hexenyl butanoate	1187,44	10	0,09%	wine, green	MS, LRI
Ethyl octanoate	1197,93	2,3	0,09%	fruit, fat, floral, pear	MS, LRI
Methyl 3-phenylpropenoate	1387,43	11	0,08%	strawberry	MS, LRI
Ether					
Ethyl ether	395,29	----	0,19%		MS
Ketones					
2-Heptanone	892,56	11	0,10%	soap	MS, LRI
Terpenoids					
Tricyclene	927,89	3	0,49%		MS, LRI
α-Pinene	934,01	1,4,3,6	2,15%	Terpenic, woody	MS, LRI
Sabinene	974,44	3,6,7,13	0,08%	pepper, turpentine, wood	MS, LRI
β-Pinene	976,80	1,3,4,13	2,98%	Sweet, green	MS, LRI
β-myrcene	992,14	3,6,7	1,35%	Herbaceous, woody	MS, LRI
α-Phellandrene	1004,30	3,6	1,41%	Citrus, spicy	MS, LRI
Limonene	1029,99	1,3,4,6,12,13	17,71%	Citrus, terpenic	MS, LRI
Eucalyptol	1032,28	5,13,16	5,79%	mint, sweet	MS, LRI
(E)-β-Ocimene	1050,18	3,6,4,13	3,57%	sweet, herb	MS, LRI
γ-Terpinene	1060,63	3,4,6	0,34%	gasoline, turpentine	MS, LRI
Terpinolene	1089,88	3,6,12	0,16%	Lime, terpenic	MS, LRI
Linalool	1100,45	1,3,7,10,13	0,32%	flower, lavender	MS, LRI
1,3,8-p-Menthatriene	1132,22	3,6,11	0,27%		MS, LRI
(4E,6Z)-allo-Ocimene	1143,70	5,6	0,25%	Green, fresh	MS, LRI
α-Terpineol	1193,52	3,7	0,21%	oil, anise, mint	MS, LRI
δ-Elemene	1343,45	1, 11,14	0,61%	wood	MS, LRI
α-Cubebene	1355,82	1,5,6	0,45%	herb, wax	MS, LRI
α-Copaene	1378,82	1,3,5,6	0,09%	Woody, spicy	MS, LRI
β-Cubebene	1383,31	----	0,99%		MS
β-elemene	1398,23	3,4,11,13,15	1,41%	herb, wax, fresh	MS, LRI
β-Caryophyllene	1429,10	1,4,3,5	5,01%	Spicy, woody	MS, LRI
Aromadendrene	1438,10	16	0,36%	Dusty, honey,wood	MS, LRI
α-Guaiene	1440,14	11,15	0,25%	wood, balsamic	MS, LRI
β-Gurjunene	1444,12	6	0,21%		MS, LRI
β-farnesene	1448,81	11	0,47%	wood, citrus, sweet	MS,LRI
α-Elemene	1454,94	15	0,38%	wood	MS, LRI
Allo-Aromadendrene	1459,36	5,14	0,36%	wood	MS,LRI
α-Caryophyllene	1463,50	3,5,14	0,80%	wood	MS, LRI
γ-Muurolene	1472,28	3,6,5,16	1,33%	herb, wood, spice	MS, LRI
Eremophilene	1484,32	3,6	0,84%		MS, LRI
Germacrene D	1490,50	3,6,13,14	2,92%	wood, spice	MS, LRI
Valencene	1496,47	3,11	1,00%	green, oil	MS, LRI
α-Selinene	1499,12	3,6,13	0,20%		MS, LRI
Epizonarene	1502,28	16	0,62%		MS,LRI
α-Muurolene	1505,26	13,14	3,92%	wood	MS, LRI
γ-Cadinene	1513,49	1,5,16	0,27%	wood	MS, LRI
δ-Cadinene	1520,29	3,5,6,13	0,83%	thyme, medicine, wood	MS, LRI
Calamenene	1528,06	3,11	2,20%	herb, spice	MS, LRI
Naphthalene, 1,2,3,4,4a,7-hexahydro-1,6-dimethyl-4-(1-methylethyl)-	1537,04	----	0,15%		MS
Germacrene B	1564,31	1,10,11,14	0,27%	wood, earth, spice	MS,LRI
Caryophyllene oxide	1593,21	1,3,5	0,38%	herb, sweet, spice	MS, LRI
Saturated hydrocarbons					
n-Hexane	478,80	-----	0,26%	Alkane	MS

[a] Compounds are listed in their elution order on a DB-5 column;
[b] LRI: Linear retention indices on column (DB-5) determined with n-alkanes and literature and from the data-base of our laboratory;
[c] Odor description based on the Leffingwell's Flavor-Base (Leffingwell, 2007) and Flavornet (http://www.flavornet.org/flavornet.html);
[d] Identification method: MS = mass spectrum; LRI is compared with references from standards or literature values as well as from the data base of Volatile compounds analyzed in our laboratory;
Ref: [1]Érica *et al.*, (2017); [2]Boulanger and Crouzet (2001); [3]Pino (2012); [4]Cheng *et al.* (2015); [5]Porat *et al.* (2011); [6]Liu *et al.* (2013); [7]Lasekan *et al.* (2013); [8]Christof *et al.*, (2014); [9]Márquez *et al.* (2011); [10]Liqin *et al.*, (2017); [11]Acree and Arn (2004); [12]Cuevas *et al.* (2016); [13]Plagemann *et al.* (2012); [14]Duarte *et al.* (2010); [15]Huang *et al.* (2009); and [16]Parker *et al.* (2007).

4. Conclusions

The combination of HS-SPME technique for capture and GC-MS system for identification of volatile compounds in jabuticaba fruit resulted in identification of a large number of volatile compounds belonging to the organic classes of terpenes, esters, aldehydes, alcohols and aromatic compounds, many of these have not been reported earlier. Among the 2 SPME fibers (CAR/PDMS and DVB/CAR/PDMS) evaluated for the capture of volatile compounds, the fiber CAR/PDMS resulted in higher number of compounds in jabticaba fruit. Based on this study, it could be concluded that the combination of HS-SPME with GC-MS is suitable for the extraction of volatile compounds from jabuticaba fruit. A large number of esters and terpenic compounds could be responsible for the typical aroma of the jabuticaba fruit, the principal compounds being limonene, ethyl acetate, eucalyptol, caryophyllene, ocimene, β-pinene and α-pinene.

Based on the data obtained, the authors recommended further studies taking into accounts the harvest season. In addition to chromatography gases coupled with mass spectroscopy analysis olfactory to confirm the volatile compounds responsible for the aroma of the fruit.

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