

# Extraction conditions and identification of volatile organic compounds from umbu pulp by HS-SPME/GC-MS

Giovana Ribeiro Ferreira<sup>1</sup>, Raíssa Queiroz Andrade<sup>2</sup>,  
Ariane Castricini<sup>3</sup>, Flaviano Oliveira Silvério<sup>4</sup>

**Abstract**—despite the high social and economic importance of *Spondias tuberosa* Arruda, to the best of our knowledge there are very few detailed studies on the volatile compounds of this fruit popularly named umbu. Therefore, the aim of this study was to find the best extraction conditions by solid-phase microextraction in headspace mode (HS-SPME) to determine the volatile compounds from umbu fruit pulp by gas-chromatography coupled to mass spectrometry (GC-MS). The optimal conditions were obtained using 4 g of pulp and 0.2 NaCl/pulp (w/w), maintained for 10 min in incubation at 40 °C. The SPME-fiber was exposed for 20 min for extraction and then for 40 min for desorption. Thus, a total of 25 volatile compounds were detected and 16 were identified under these conditions, with 9 compounds being identified for the first time in the volatile fraction of umbu fruit pulp and two compounds were identified for the first time in the *Spondias* genus. The major chemical class was terpenes and esters, which together represent more than 90% of total chromatographic area.

**Index terms:** *Spondias tuberosa*, Headspace extraction, SPME, Caatinga fruit.

## Condições de extração e identificação de substâncias voláteis de polpa de umbu por HS-SPME/GC-MS

**Resumo** - Apesar da alta importância social da *Spondias tuberosa* Arruda, têm sido raros os estudos sobre as substâncias voláteis de frutos do umbuzeiro conhecido como umbu. Por isso, o objetivo deste estudo foi determinar as melhores condições de extração destas substâncias por microextração em fase-sólida no modo headspace (MEFS-HS) para caracterizar o perfil de voláteis na polpa dos frutos de umbu por cromatografia gasosa acoplada à espectrometria de massas (CC-EM). As condições ótimas de extração foram obtidas usando 4 g da polpa da fruta e 0,2 NaCl/polpa (m/m), mantidos por 10 min em incubação a 40 °C. A fibra de MEFS foi exposta por 20 min para a extração dos compostos e, em seguida, por 40 min para dessorção no injetor do cromatógrafo. Nestas condições, um total de 25 substâncias voláteis foram detectadas, sendo 11 delas identificadas pela primeira vez na fração volátil de polpa de umbu e 3 compostos pela primeira vez no gênero *Spondias*. As principais classes químicas foram os terpenos e os ésteres, os quais, juntos, representam mais de 90 % da área cromatográfica total.

**Termos para indexação:** *Spondias tuberosa*, extração por *Headspace*, MEFS, frutos da Caatinga.

**Corresponding author:**  
giovana.ferreira@ufvjm.edu.br

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<sup>1</sup>Doctor's degree in Materials Engineering, Universidade Federal dos Vales do Jequitinhonha e Mucuri, Janaúba-MG, Brazil. E-mail: giovana.ferreira@ufvjm.edu.br (ORCID 0000-0002-3227-7325)

<sup>2</sup>Master student in Food and Health, Universidade Federal de Minas Gerais Janaúba-MG, Brazil. E-mail: raissaq66@gmail.com (ORCID 0000-0003-3809-7648)

<sup>3</sup>Doctor's degree in Plant Science / Production Physiology, Researcher, CEGR, Empresa de Pesquisa Agropecuária de Minas Gerais, Nova Porteira-MG, Brazil. E-mail: ariane@epamig.br (ORCID 0000-0003-0677-566X)

<sup>4</sup>Doctor's degree in Organic Chemistry, Universidade Federal de Minas Gerais, Montes Claros-MG, Brazil. E-mail: flavianosilverio@ufmg.br (ORCID 0000-0002-5753-8739)

## Introduction

The Caatinga is one of the main Brazilian biomes and has stood out for its variety of species which present high importance for the population (FILIZOLA; SAMPAIO, 2015). Several authors have highlighted *Spondias tuberosa* among these species, popularly known as *umbuzeiro*, and its fruits are called *umbu* (CAVALCANTI; RESENDE; BRITO, 2000; DE LIMA; SILVA; DE OLIVEIRA, 2018; SATURNINO; SOUZA, 2019). *S. tuberosa* fruits have drawn attention for both their nutritional, cultural and socioeconomic importance for the Brazilian semi-arid region and because the current stage of scientific and technological knowledge of this species needs to be expanded (DE LIMA; SILVA; DE OLIVEIRA, 2018; MATTA; TORREZAN; RIBEIRO, 2019; SATURNINO; SOUZA, 2019).

Although the species has some different characteristics within the ecosystem, their fruits are generally reported as having an exotic and sweet flavor, greenish color and a diameter of about 3 cm (DE LIMA; SILVA; DE OLIVEIRA, 2018; SATURNINO et al., 2019). The economic exploitation of *S. tuberosa* fruits has been carried out through extractivism by Brazilian semi-arid populations, but some *umbuzeiro* orchards have been established in different locations in the North of Minas Gerais, being considered a species undergoing domestication (DONATO et al., 2019; SATURNINO; SOUZA, 2019).

Previous studies have been carried out with *S. tuberosa* aiming at better knowledge for preserving this species (DONATO et al., 2019; SALES et al., 2019; SANTOS et al., 2011). In this sense, the *Empresa de Pesquisa Agropecuária de Minas Gerais (EPAMIG)* has cultivated several *S. tuberosa* accessions for 20 years (MOREIRA et al., 2007). Nevertheless, there are few and insufficient studies on the volatile compounds of the fruits of *S. Tuberosa* (THOMAZINI et al., 1998 *apud* FRANCO; JANZANTTI, 2005; GALVÃO et al., 2010, 2011). Although there are studies on the identification of this class of compounds in umbu fruits, the technique used for extracting volatile compounds involves heating and the use of solvents (THOMAZINI et al., 1998 *apud* FRANCO; JANZANTTI, 2005; GALVÃO et al., 2010, 2011). It is important to remark that there are studies about the characterization of volatile compounds from fruits of other species of the *Spondias* genus by techniques without solvents and at close to ambient temperatures, but not of *S. Tuberosa*, one of the most important Brazilian semi-arid fruit (CEVA-ANTUNES et al., 2003, 2006; TODISCO et al., 2014).

There are currently solvent free techniques which also do not implement temperature increases, and consequently avoid contamination and chemical transformations in the analytes, such as solid phase

microextraction in the headspace mode (HS-SPME) which has been widely used for characterizing volatile compounds in fruits. Despite being a well-known technique and widespread in the scientific community, several studies have shown that the chemical composition of the volatile fraction depends on the conditions employed in the extraction method, and therefore optimizing the extraction parameters may lead to a greater content and number of substances (MEHTA et al., 2018; NONGONIERMA et al., 2006; SILVA et al., 2019).

Thus, the aim of this study was to optimize the extraction conditions by solid phase microextraction in the headspace mode (HS-SPME) for determining the volatile compounds from *S. tuberosa* fruit pulp by gas-chromatography coupled to mass spectrometry (GC-MS).

## Material and methods

### Sample preparation

*S. tuberosa* fruits with the same physiological maturity (i.e. at the beginning swelling stage and having green peels) were harvested during December of 2018 to January of 2019 from the accessions collection of EPAMIG-NORTE in Nova Porteirinha city, Minas Gerais State, Brazil. This area is delimited by the following geographic coordinates: 15° 48' 8.932" S, 43° 17' 49.445" W; 15° 48' 3.060" S, 43° 17' 43.811" W; 15° 48' 4.075" S, 43° 17' 42.648" W; 15° 48' 10.159" S, 43° 17' 48.188" W. Next, the fruits were transported to the *Laboratório de Química Instrumental* of the Universidade Federal de Minas Gerais (LQI-UFMG), Montes Claros, Minas Gerais. The fruit pulps were separated from the seeds and peels and homogenized using a domestic mixer for 30 s. Then, NaCl was added and mixed again for 1 min. Aliquots of the material were then transferred to 20 mL vials, sealed, and analyzed by HS-SPME-GC-MS.

### Extraction method

The extraction of volatile compounds was performed by the HS-SPME method using SPME-fiber of divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) from Supelco (São Paulo, Brazil). The vials with the samples were inserted in a thermostatic bath, and SPME-fiber was subsequently inserted in the vials. Finally, the fiber was transferred to the gas-chromatograph injector. The volatile compound extraction conditions by HS-SPME were chosen through univariate analysis. For the choice of extraction parameters, the effect of different sample mass,  $m(\text{NaCl})/m(\text{fruit pulp})$  ratio, incubation time, extraction time using SPME-fiber, temperature and desorption time in the chromatography injector (values of parameters was present in Table 1) on the total chromatographic area and number of volatile compounds detected was evaluated. All procedures were performed in duplicate.

**Table 1.** Optimized HS-SPME/GC-MS parameters for extracting volatile compounds from umbu fruit pulp.

Parameters	Level
Sample mass/g	1, 2 and 4
m(NaCl)/m(fruit pulp) ratio	0.050, 0.10 and 0.20
Temperature/°C	30, 35 and 40
Extraction time using SPME-Fiber/min	10, 15 and 20
Incubation time/min	10, 15 and 20
Desorption time/min	1, 5 and 40

### *Analysis and identification of volatile compounds*

After the extraction, the SPME-fiber was injected into the gas-chromatograph for compound desorption and kept in the equipment until the end of the chromatographic analysis. Static headspace analysis was performed using a PAL Syr HS 2.5 mL for combi-PAL. The chromatographic analyses were performed in a 7890A GC gas chromatograph coupled to 5975C mass spectrometer (Agilent Technologies). The capillary column was a DB-5 MS column of 30 m x 0.32 mm x 0.25  $\mu\text{m}$  from Agilent technologies. The GC oven temperature was started at 60 °C, and then increased by 3 °C  $\text{min}^{-1}$  to 240 °C. Helium (99.9999% purity) was used as carrier gas at a flow rate of 1 mL  $\text{min}^{-1}$ . All samples were injected in splitless mode with the injector temperature at 220 °C. The interface temperature was 280 °C, and the acquisition mass range was 45-600 m/z. A standard hydrocarbon solution (C7-C30) was injected under the same conditions to calculate the Van den Dool and Kratz retention index (LRI) of the sample compounds. Identification of the volatile compounds was performed using mass spectra NIST 2.0 database and Van den Dool and Kratz retention index (LRI) (Van den Dool; Kratz, 1963).

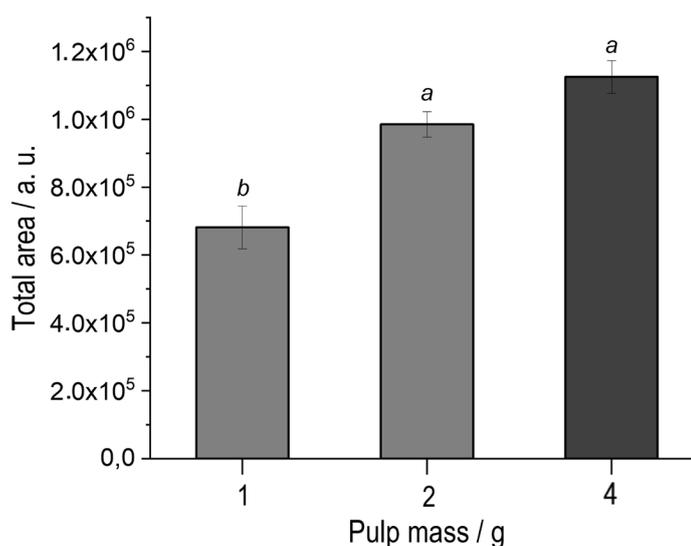
### *Statistical analysis*

The data were analyzed by one-way ANOVA with Tukey's test ( $P < 0.05$ ) using BioEstat 5.0 free software made available by the *Instituto Mamirauá* (INSTITUTO DE DESENVOLVIMENTO SUSNTETÁVEL MAMI-RAUÁ).

## *Results and Discussion*

### *Optimization of the extraction conditions*

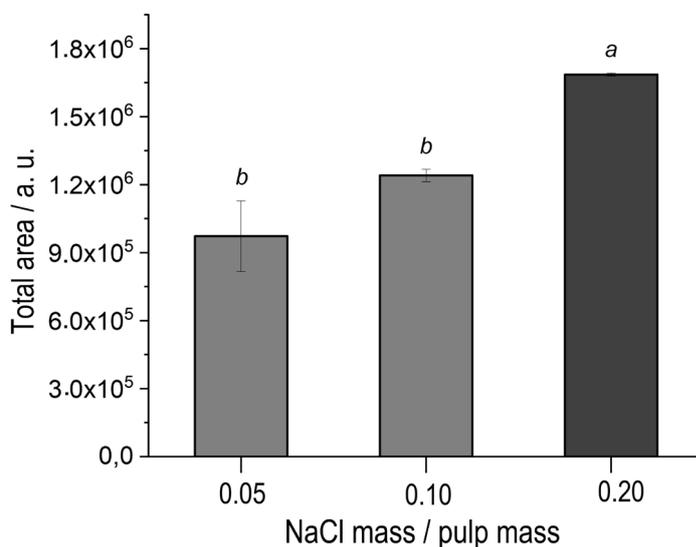
The first step was to evaluate the best sample mass for extracting volatile compounds from umbu pulp. The results obtained may be observed in Figure 1 and Table 1. The total chromatographic areas obtained were higher using 2 g and 4 g of sample. However, it was observed that 15, 15 and 16 volatile compounds were detected using 1, 2 and 4 g, respectively. Therefore, 4 g was selected as the better condition in this work. Despite having been the specific choice in this work, we emphasize that the use of a larger amount of sample does not necessarily improve the extraction of the compounds.



**Figure 1.** Total chromatographic area of volatile compounds using 1, 2 and 4 g of umbu fruit pulp. Different letters indicate significant differences ( $P < 0.05$ ) according to the Tukey's test.

The second step was to choose the best proportion between salt mass and pulp mass to be used in the analysis. The results obtained are shown in Figure 2 and Table 1. It was possible to observe that the proportion of 0.20 g of NaCl/g of pulp promoted a higher number of volatile compounds (17 compounds) and higher chromatographic

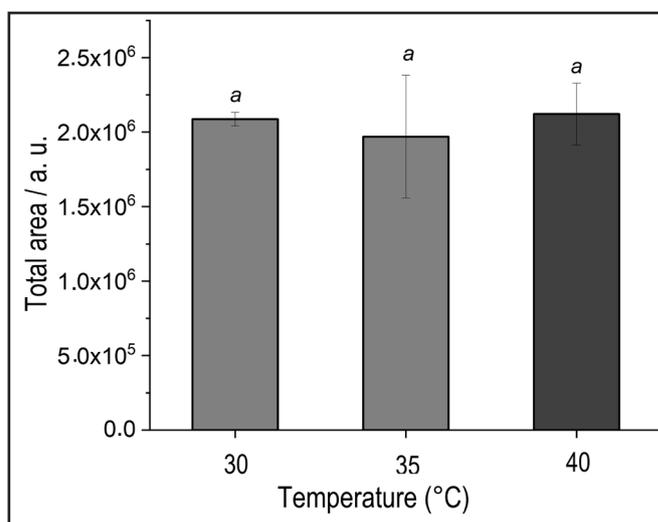
area. Therefore, the addition 0.2 g of NaCl/g of pulp was chosen for the next step of this study. The causes of this effect have been well explained in previous works (ZHANG; YANG; PAWLISZYNZ, 1994; PROSEN; ZUPANCIC-KRALJ, 1999; PORTO-FIGUEIRA et al., 2018).



**Figure 2.** Total chromatographic area of volatile compounds using 0.050, 0.10 and 0.20 g of NaCl per gram of umbu fruit pulp. Different letters indicate significant differences ( $P < 0.05$ ), according to the Tukey's test.

The third evaluated parameter was the temperature for extracting volatile compounds from umbu fruit pulp. The results obtained showed no significant differences in the total chromatographic area (Figure 3). It was observed that 15, 17 and 17 volatile compounds were detected using 30, 35 and 40 °C, respectively (Table 1). However, 40 °C promoted a minor experimental deviation in comparison

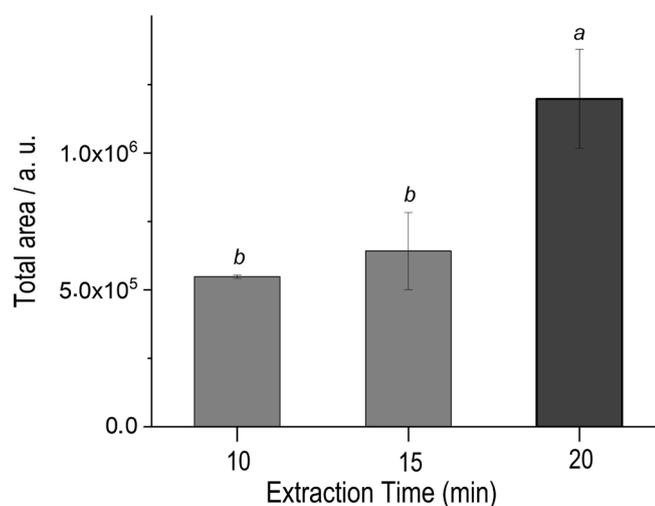
with 35 °C. Therefore, the chosen temperature was 40 °C. In addition, the choice in this case also considered that the same temperatures have been used to determine volatile compounds in other fruit matrices. (GIANNETTI et al., 2017; MAMEDE et al., 2017; PORTO-FIGUEIRA et al., 2018).



**Figure 3.** Total chromatographic area of volatile compounds from umbu fruit pulp using 30, 35 and 40 °C as extraction temperature. Different letters indicate significant differences ( $P < 0.05$ ) according to the Tukey's test.

The fourth stage of this study was to evaluate the extraction time, meaning the SPME-fiber exposure time inside the headspace vial to adsorb the volatile compounds

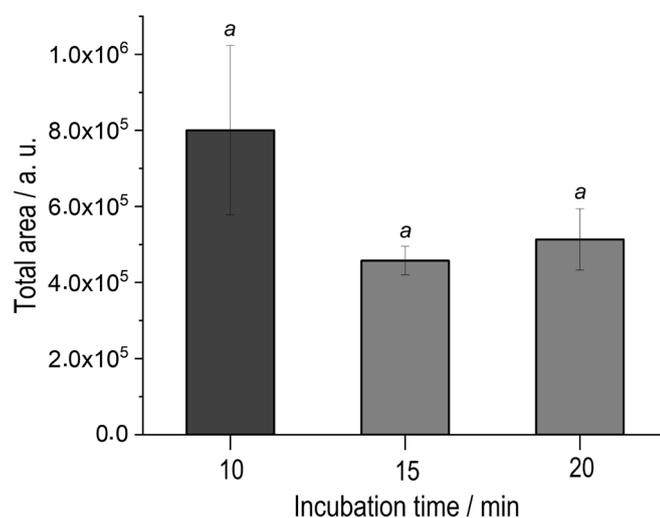
from the umbu fruit pulp. The results obtained may be observed in Figure 4 and in Table 1.



**Figure 4.** Total chromatographic area obtained from of volatile compounds from umbu fruit pulp using 10, 15 and 20 min of extraction time by Headspace-SPME. Different letters indicate significant differences ( $P < 0.05$ ) according to the Tukey's test.

It was observed that 20 min promoted a higher number of volatile compounds (18 compounds) than the extractions by 15 (17 compounds) and 10 min (16 compounds), as may be seen in Table 1. In addition, the total chromatogram area was higher using 20 min, as shown in Figure 4. Therefore, this extraction time was defined for the next steps in this study.

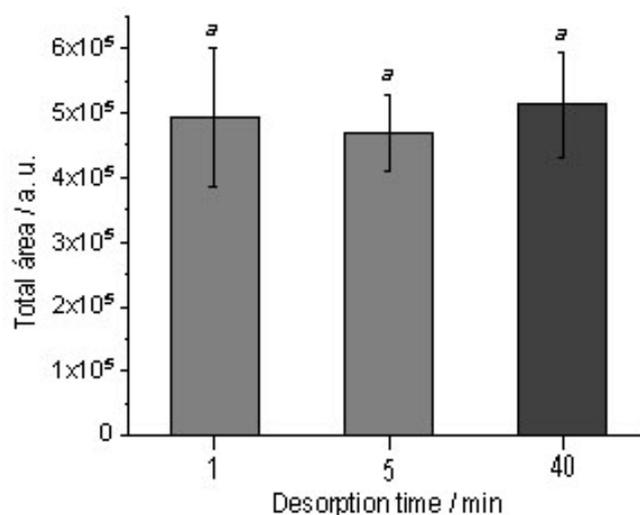
The fifth step of this study was to study the incubation time of volatile compounds from umbu fruit pulp, and the results obtained are shown in Figure 5 and in Table 1. The results revealed that the incubation time of 10 min did not promote a statistical difference in chromatographic area, but the number of detected compounds (17 compounds) was higher in relation to the other times, as may be seen in Figure 5 and in Table 1. Therefore, 10 min was chosen as the best incubation time.



**Figure 5.** Total chromatographic area of volatile compounds from umbu fruit pulp using 10, 15 and 20 min of incubation time. Different letters indicate significant differences ( $P < 0.05$ ) according to the Tukey's test.

The last evaluated parameter was the retention time of the SPME-fiber inside the chromatographic injector for desorbing the volatile compounds. The results obtained from this experiment are presented in Figure 6 and in Table 1S (Supplementary material). They showed no significant differences in the total chromatographic area and number of volatile compounds detected (16 compounds), as may be seen in Figure 6 and Table 1. Although 1 min has been

enough time to ensure satisfactory desorption (ARTHUR et al., 1992; PROSEN; ZUPANCIC-KRALJ, 1999), the permanence of the SPME-fiber for a longer period in the injector ensures the absence of interferences in the next analysis. Therefore, 40 min was chosen to desorb and condition the SPME-fiber during the analysis despite being a long time, thus avoiding contaminating an immediately following analysis.

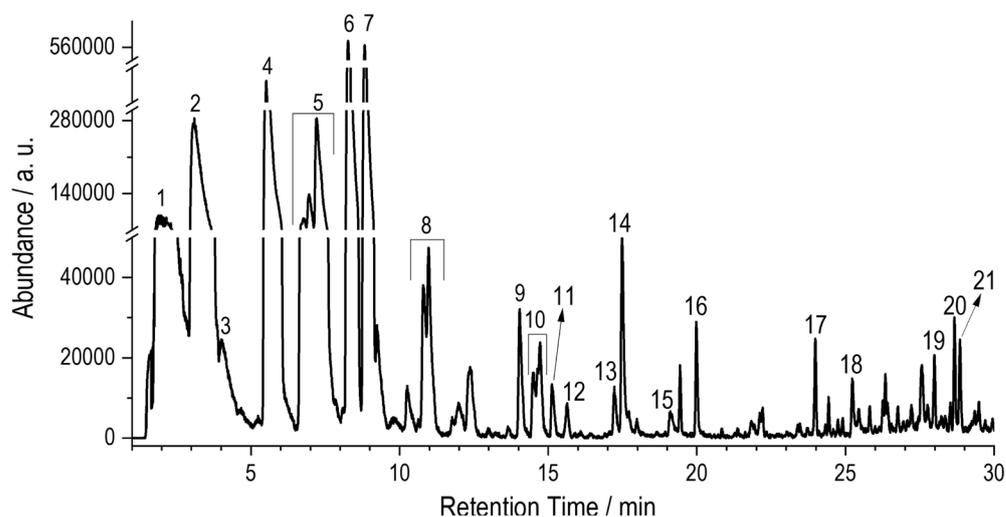


**Figure 6.** Total chromatographic area of volatile compounds from umbu fruit pulp using 1, 5 and 40 min retention time of the SPME-fiber inside the chromatographic injector. Different letters indicate significant differences ( $P < 0.05$ ) according to the Tukey's test.

#### *Volatile compounds from umbu fruit pulp*

After completing the optimization of all these parameters, an analysis was performed on the optimized extraction conditions, and the total ion chromatogram obtained is shown in Figure 7.

A total of 25 volatile compounds were detected by GC-MS from umbu fruit pulp, and 16 compounds were identified, representing about 91.5% compound abundance and 64% of the compound number (Table 2). The relative chromatographic areas of the main chemical classes identified in the volatile compounds from umbu fruit pulp may be seen in Figure 8.

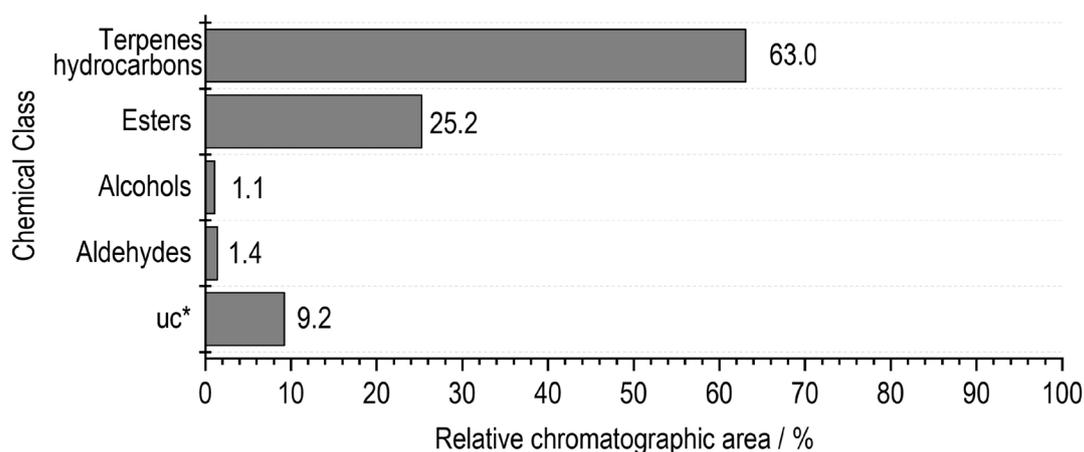


**Figure 7.** Typical total ion chromatogram of volatile compounds obtained from umbu fruit pulp under the optimized extraction conditions.

**Table 2.** Relative areas of volatile compounds from umbu fruit pulp under the optimized extraction conditions.

Pea <sup>a</sup>	Rt	LRI	LRI'	Compound	Class	Relative Area
1	1.9	<800		1,1-Dimethylprop-2-en-1-ol <sup>ii</sup>	alcohol	6.7 ± 0.3
2	3.1	811	808	Ethyl butanoate	ester	14.3 ± 0.4
3	4.0	869		unidentified compounds		0.4 ± 0.0
4	5.5	938	942	$\alpha$ -Pinene	terpene	20.4 ± 0.1
5	6.8	984	984	Mixture of $\beta$ -Pinene, Myrcene and Ethyl hexanoate	terpene	17.6 ± 0.8
	7.0	991	992		terpene	
	7.2	1001	999		ester	
6	8.3	1030	1035	Limonene	terpene	18.8 ± 1.2
7	8.8	1046	1046	$\beta$ -Ocimene	terpene	15.5 ± 1.0
8	10.8	1101	1101	Mixture of Linalool and Nonanal	alcohol/	2.0 ± 0.3
	11.0	1105	1104		aldehyde	
9	14.0	1180	1179	Terpin-4-en-1-ol	alcohol	0.6 ± 0.1
10	14.5	1191	1192	Methyl salicylate Ethyl octanoate	Ester	0.9 ± 0.0
	14.7	1196	1197			
11	15.1	1206		unidentified compounds.		0.2 ± 0.1
12	15.6	1218		unidentified compounds		0.2 ± 0.0
13	17.2	1255		unidentified compounds		0.2 ± 0.0
14	17.5	1261	1263	Dec-2-enal	aldehyde	0.7 ± 0.1
15	19.1	1299		unidentified compounds		0.1 ± 0.0
16	20.0	1319	1322	Methyl geranate	Ester	0.3 ± 0.0
17	24.0	1414	1420	Caryophyllene	terpene	0.2 ± 0.0
18	25.2	1445		unidentified compounds		0.2 ± 0.1
19	28.0	1513		Cadinene <sup>ii</sup>	terpene	0.2 ± 0.0
20	28.7	1531		unidentified compounds		0.2 ± 0.0
21	28.9	1535	1542	Selina-3,7(11)-diene	terpene	0.2 ± 0.0

Rt: retention time in minutes; u. c.: unidentified compound; <sup>a</sup>The numbers of the peaks refer to the compounds in Figure 7. <sup>i</sup>Linear retention index on DB-5 (or equivalent) column from literature (AVSAR *et al.*, 2004; BACCOURI *et al.*, 2007; BALBONTÍN *et al.*, 2007; DEGHAN *et al.*, 2007; FLAMINI *et al.*, 2007); <sup>ii</sup>tentatively identified compound.



**Figure 8.** Relative chromatographic areas of the main chemical classes identified in the volatile compounds from umbu fruits pulp. \*Including tentatively identify.

Terpenes were the main class containing representing about 64% of total chromatographic area. These compounds are important for predominantly acting in plant protection and communication (GERSHENZON; DUDAREVA, 2007). The main compound in this class was  $\alpha$ -pinene with ~20% of the total chromatographic area, also being the main compound of volatile compounds from umbu fruit pulp. Two other terpenes found in high quantities were limonene and  $\beta$ -ocimene with 19% and 15% of the total chromatographic area, respectively.

The second largest chemical class was esters, with ethyl butanoate with ~14.3%. These compounds have already been identified in other fruits (CHEN et al., 2006). The other chemical classes detected were aldehydes. Seven unidentified compounds which do not belong to any of these chemical classes were also observed in this study, representing 1.65% of the total chromatographic area.

To the best of our knowledge, 2 of the compounds identified in the volatile compounds from umbu fruit pulp were identified for the first time in the *Spondias* genus: Methyl geranato, and Selina-3,7(11)-diene. On the other hand, among the volatile compounds identified in this study, ethyl butanoate,  $\beta$ -pinene, myrcene, ocimene, linalool and caryophyllene were found in several previous works which studied the volatile compounds from other fruits of the *Spondias* genus such as *S. monbim* (cajá or Taperabá), *Spondias sp.* (caja-umbu), and *S. purpurea* (siriguela) (CEVA-ANTUNES et al., 2003, 2006; NARAIN; GALVÃO; MADRUGA, 2007; TODISCO et al., 2014; BARREIROS et al., 2018; SOSA-MOGUEL et al., 2018).

In a previous study, only seven common compounds were identified in the extracts from *S. tuberosa* fruit pulps obtained by simultaneous distillation and extraction (SDE) (GALVÃO et al., 2011). Therefore,  $\beta$ -pinene, myrcene, ethyl hexanoate, linalool, terpin-4-en-1-ol, methyl salicylate, ethyl octanoate, methyl geranate, and eudesma-3,7(11)-diene were identified for the first time in umbu fruit pulp in this study.

## Conclusions

The influence of pulp mass, salt/pulp ratio, temperature, incubation time, extraction time, and desorption time on the HS-SPME extraction were evaluated and the optimal conditions were chosen to extract volatile compounds from umbu fruit pulp. The chosen conditions were 4 g of pulp and 0.2 NaCl/pulp (w/w), maintained for 10 min in incubation at 40 °C, the extraction time was 20 min and the desorption time was 40 min. Thus, 25 volatile compounds were detected under these conditions, in which 9 compounds were identified for the first time in this fruit, and two compounds in the *Spondias* genus were characterized for the first time from vapour-phase extraction of umbu fruit pulp. Terpenes and esters were the predominant chemical classes in this volatile compound, together representing ~90% of the total chromatography area, of which the most predominant were  $\alpha$ -pinene at ~20 %.

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