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Sensory and volatile profiles of monofloral honeys produced by native stingless bees of the brazilian semiarid region

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ABSTRACT

Monofloral honeys produced by stingless bees *M. subnitida* Ducke and *M. scutellaris* Latrelle in typical flowering of the Brazilian semi-arid *Ziziphus juazeiro* Mart (juazeiro), *Croton heliotropiifolius* Kunth (velame branco) and *Mimosa arenosa* willd Poir (jurema branca) were characterized in relation to volatile and sensorial profile. It identified 11 sensory descriptors and 96 volatile compounds. It was noticed a strong effect of flowering in sensorial profile and volatile of honeys. Juazeiro honey stood out with a higher characteristic aroma, taste sweet, caramel flavor and levels of aromatic aldehydes; jurema honey has been described with herb and beeswax aroma and the presence of sulfur compounds and ketones; volatile acids associated with acid taste, medicinal taste and clove aroma characterized the velame branco honey. These results demonstrate that the knowledge of the sensory and aroma profile of these honeys can contribute to characterization of its floral and geographical identity.

1. Introduction

Brazil has a diverse fauna of social bees known as Brazilian native bees, stingless bees, indigenous bees, or simply “meliponini” or “Meliponinae” bees. Approximately 192 species of stingless bees are found in Brazil, including *Melipona subnitida* Ducke (jandaira) and *M. scutellaris* Latrelle (uruçu), which are species endemic to the Brazilian semiarid region. In addition to producing honeys with excellent sensory qualities that are highly appreciated for their distinctive flavors, these bees also offer ecological benefits, including the conservation of native plants through pollination (Silva et al., 2013; Biluca et al., 2014; Sousa et al., 2016; Chuttong, Chuttonh, Chanbang, Sringarm, & Burget, 2016).

The Brazilian semiarid region is noted for environmental conditions that favor meliponiculture. The region has intense and diverse natural flowering, a vast land area and climate variability that enable honey production throughout the year. Its biome has distinct typical vegetation in both the rainy and dry seasons; together, they promote nectar and pollen flow and enable bee colony maintenance throughout the year (Silva et al., 2014; Sousa et al., 2016). These aspects differentiate the region because honey production is restricted to specific seasons in other Brazilian regions and countries.

The climate variability and extremely rich flora of the Brazilian semiarid region have enabled the production of a wide variety of honeys from stingless bees with unique sensory qualities. However, studies characterizing these honeys are relatively recent and are insufficient to establish their “identities” and quality standards. The lack of data has undermined the competitiveness of the honeys in national and international markets.

Honey composition is closely associated with the botanical origin and geographic area of production because the soil and climate determine the bee flora (Silva, Gauche, Gonzaga, Costa, & Fett, 2016; Silva, Lima, Caetano, & Torres, 2017). Monofloral honeys differ from one another in their volatile fraction compositions, which in turn greatly affect the individual sensory characteristics of each type of honey, among other characteristics. Volatile compounds, which primarily account for food aroma and flavor, are present in honey at very low concentrations as complex mixtures of different chemical classes, including monoterpenes, norisoprenoids, sesquiterpenes, benzenoids, alcohols, esters, ketones and aldehydes (Manyi-Loh, Anip, & Clarke, 2011; Silva et al., 2016).

The volatile compounds present in honey usually come from flower nectar and may be considered markers of bee-visited plants. Thus,

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characterization of the volatile compound profile of monofloral honeys has been used for product classification (floral origin characterization), and some volatile compounds may be used as specific chemical markers for the botanical origin of the honey (Castro-Vázquez, Díaz-Maroto, & Pérez-Coello, 2007; Fuente, Sanz, Martínez-Castro, & Sanz, 2007; Jerkovic, Tuberoso, Marijanovic, Jelic, & Kasum, 2009; Castro-Vázquez, Leon-Ruiz, Alañon, Pérez-Coello, & González-Porto, 2014; Karabagias, Badeka, Kontakos, Karabournioti, & Kontominas, 2014; Seisonen, Kivima, & Vene, 2015; Silva et al., 2017).

The honey sensory profile is another tool that enables the identification of the honey's botanical origin. This approach has been used to complement the results from chemical and physical analyses of honey and to assess the compliance of monofloral honeys because it may detect botanical components untraceable by analytical methods that change the characteristic sensory traits of the products (Castro-Vázquez, Díaz-Maroto, & Pérez-Coello, 2012; Piana et al., 2004; Tahir, Xiaobo, Xiaowei, Jiyong, & Mariod, 2016).

Thus, the progress of knowledge on the sensory and chemical aspects of the aroma and taste of monofloral honeys from the Brazilian semiarid region has made key contributions to the establishment of the floral and geographical identities of these honeys and the improvement of their trade competitiveness. Accordingly, the present study aimed to characterize the volatile and sensory profiles of honeys produced by jandaira and uruçu meliponini in juazeiro, velame branco and jurema branca blossoms, which stand out among the typical Brazilian semiarid region vegetation.

2. Materials and methods

2.1. Samples

Six honey samples from native stingless bees produced by the jandaira (Melípona *subnitida* Ducke) and uruçu species (*Melipona scutellaris* Latrelle) from three different blossoms *Ziziphus juazeiro* Mart (juazeiro), *Croton heliotropiifolius* Kunth (velame branco) and *Mimosa arenosa* willd Poir (jurema branca)] juazeiro (*Ziziphus juazeiro* Mart), velame branco (*Croton heliotropiifolius* Kunth) and jurema branca (*Mimosa arenosa* willd Poir)] were analyzed in a complete block design to assess “bee species” and “blossom type” effects. Samples were collected from meliponaries located in the Seridó region of Rio Grande do Norte state (− 06°46′14″S, − 36°44′00″W) and the Agreste region of Paraíba state (7°10′15″S, 35°51′14″W), both in the Brazilian semiarid region. Honeys were directly collected from the hives by suction using syringes (one per colony) and then stored at a temperature of approximately 7 °C in sterile and properly labeled glass jars prior to the tests. All samples were produced in 2014 in the seasons specific for each blossom; thus, juazeiro blossom honey was collected in the dry season of 2014, and the other blossom honeys were collected in the rainy season of the same year.

2.2. Melissopalinological analysis

The melissopalinological analysis was performed to confirm the classification and monofloral origin of the honey samples. For this purpose, 10 g of each sample was dissolved in 20 ml of distilled water and centrifuged at 4000 rpm for 20 min. The sediment was dried at 40 °C and then mounted on a slide with Entellan (Merck, 1.07961.0500). Pollen characterization was determined by the 500-pollen grain count and identification in at least 4 different fields of the slide using an optical microscope (Nikon Optiphot II microscope; 400 × and 1000 ×). The pollen grains were compared with reference images from the University of São Paulo (Universidade de São Paulo - USP), São Paulo (SP), Brazil. All samples contained > 65% pollen grains derived from the same botanical origin (Table 1).

Table 1
Melissopalinological composition of honey produced by jandaira and uruçu meliponini in the Brazilian semiarid region.

Bee	Melissopalinological composition of honey	Pollen %
Uruçu	Velame branco (*Croton heliotropiifolius*)	65
Melipona scutellaris Latrelle	Jurema Branca (*Mimosa arenosa*)	22
	Vassourinha de botão (*Polygala violacea*)	5.3
	Chanana (*Turnera subulata*)	7.4
Jandaíra	Velame branco (*Croton heliotropiifolius*)	69
Melípona *subnitida* Ducke	Jurema Branca (*Mimosa arenosa*)	21
	Marmeleiro (*Croton sonderianus*)	4.8
	Vassourinha de botão (*Polygala violacea*)	5
Uruçu	Jurema Branca (*Mimosa arenosa*)	67.5
Melipona *scutellaris* Latrelle	Amargosa (*Pavania Cancellata*)	24.46
	Velame (*Croton heliotropiifolius* Kunth)	8
Jandaíra	Jurema Branca (*Mimosa arenosa*)	72.4
Melípona *subnitida* Ducke	Marmeleiro (*Croton sonderianus*)	20
	Velame (*Croton heliotropiifolius*)	7.6
Uruçu	Juazeiro (*Ziziphus juazeiro* Mart.)	69
Melipona *scutellaris* Latrelle	Vassourinha de botão (*Polygala violacea*)	21.3
	Sabiá (*Mimosa caesalpinifolia*)	9.7
Jandaíra	Juazeiro (*Ziziphus juazeiro* Mart)	71
Melípona *subnitida* Ducke	Cajá (*Spondias mombin* L.)	26.7
	Sabiá (*Mimosa caesalpinifolia*)	2.3

2.3. Extraction and volatile compound analysis

Volatile compounds were extracted by Head Space Solid Phase Micro-Extraction (HS-SPME) using polydimethylsiloxane/divinylbenzene (PDMS/DVB) fibers (Supelco, Bellefonte, PA, USA). The samples were prepared by mixing 10 g of honey with 10 ml of Milli-Q water in 60-ml headspace vials sealed airtight with polytetrafluoroethylene silicone septa (Supelco, Bellefonte, PA, USA). The system was subjected to heating at 45 °C in a water bath for 15 min. After cooling, the fiber was exposed to the sample headspace for 45 min under 700 rpm magnetic stirring and then transferred to the gas chromatograph injector wherein the analytes were desorbed for 5 min.

A Varian Saturn 3800 gas chromatograph coupled to a Varian Saturn 2000R mass detector and a VF-5MS capillary column (60 m × 0.25 mm × 0.25 μm) was used to separate and identify the honey volatile compounds. The gas chromatograph oven temperature was set to and maintained at 40 °C for 2 min, followed by a 2 °C/min ramp to 60 °C, a 3 °C/min ramp to 90 °C and 4 °C/min ramp to 240 °C; this temperature was maintained for 10 min. The temperature was maintained at 250 °C in the injector and detector. Helium was used as the carrier gas at a constant flow rate of 1.0 ml/min. The mass spectrometer was operated in electron impact with a 200 °C ion source temperature and 70 V ionization energy with a scan-to-scan variation from 29 *m/z* to 400 m/z at 3.33 scans/s.

The compounds were identified using the following methods: (1) comparing their experimental mass spectra with the spectra of compounds supplied by the National Institute of Standards and Technology/Environmental Protection Agency/National Institutes of Health virtual library (NIST/EPA/NIH Mass Spectral Database, version 2.0, 2008), (2) comparing the linear retention indices (LRI) assessed using the retention time of a homologous series of *n*-alkanes (C_8 - C_{25}) analyzed under conditions previously described in the literature for columns with the same polarity (Adams, 2008; Jerkovic & Kus, 2014; Karabagias et al., 2014; Rivellino et al., 2013) and (3) comparing the mass spectra with the spectra of pure standards analyzed in the same device under the same methodological conditions.

Compounds with mass spectra and linear retention indices compatible with the injected pure standards analyzed under the same conditions as the isolate were considered positively identified. Compounds

Table 2
F and p ANOVA values of the sources of variation in the "blossom", "bee species" and "blossom ∗ bee" interaction for each descriptor and mean intensity of the aroma and flavor/taste descriptors of honeys produced by two "Melipona" species from three blossoms of the Brazilian semiarid region.

Sensory descriptors		Blossom		Bee species		Blossom ∗ bee		Velame branco		Juazeiro		Jurema branca	
		F	p	F	p	F	p	Uruçu	Jandaíra	Uruçu	Jandaíra	Uruçu	Jandaíra
Aroma[1]	Characteristic[3]	324.10	< 0.0001	10.31	0.0016	6.82	0.0014	3.9 c	3.1 d	6.6 a	6.6 a	4.9 b	4.9 b
	Caramel[3]	315.86	< 0.0001	0.41	0.5239	1.55	0.2154	1.6 c	1.3 c	4.4 a	4.3 a	2.9 b	3.0 b
	Beeswax[2]	615.43	< 0.0001	0.06	0.8092	0.62	0.5381	0.9 b	1.0 b	0.9 b	0.8 b	4.9 a	5.0 a
	Clove[2]	282.89	< 0.0001	138.33	< 0.0001	121.20	< 0.0001	1.2 b	3.9 a	0.4 c	0.5 c	0.5 c	0.5 c
	Floral[3]	152.12	< 0.0001	110.54	< 0.0001	118.02	< 0.0001	2.4 c	4.9 a	3.1 b	3.0 b	2.0 d	2.1 d
	Herbaceous[2]	272.05	< 0.0001	15.60	0.0001	3.65	0.0279	1.7 c	0.8 d	0.7 d	0.6 d	4.3 a	3.8 b
	Medicinal[3]	494.41	< 0.0001	8.40	0.0042	8.35	0.0003	4.3 b	5.3 a	0.9 c	1.0 c	1.1 c	1.1 c
Flavor/Taste[1]	Acid[3]	225.00	< 0.0001	0.23	0.6319	1.27	0.2836	7.1 a	6.6 b	1.3 d	1.5 d	3.5 c	3.4 c
	Caramel[3]	423.31	< 0.0001	1.19	0.2765	0.43	0.6524	0.5 c	0.3 c	4.4 a	4.2 a	2.1 b	2.1 b
	Sweet[3]	703.12	< 0.0001	0.30	0.5819	0.21	0.8104	5.1 c	5.1 c	8.8 a	8.8 a	6.4 b	6.5 b
	Medicinal[3]	621.56	< 0.0001	0.04	0.848	4.73	0.0099	3.9 b	4.3 a	1.3 c	1.0 c	0.7 d	0.6 d

[1] Means with the same letters in the same row are not different from one another at $p < 0.05$,
[2] For the beeswax, clove and herbaceous trait: 0 = none and 9 = strong.
[3] For the caramel aroma, floral aroma, medicinal aroma, acid taste, sweet taste, and caramel flavor traits: 0 = weak and 9 = strong.

with mass spectra compatible with the spectra provided by the device library and linear retention indices similar to those found in the literature were considered identified compounds. Compounds with no linear retention index available in the literature whose identification was based on the data generated by the mass spectrometer alone were considered tentatively identified compounds. The volatile compounds were grouped into chemical classes by calculating the average abundances for each peak present in the chromatogram and the area percentage for each chemical class. Each sample was injected in triplicate.

2.4. Sensory profile

The aroma and flavor profile of each honey sample was developed according to the fundamentals of the Quantitative Descriptive Analysis (QDA®) proposed by Stone, Sidel, Oliver, Woosley, and Singleton (1974). The study was previously submitted to the Research Ethics Committee of the Federal University of Paraíba (Universidade Federal da Paraíba – UFPB) and approved under Certificate of Presentation for Ethical Consideration (Certificado de Apresentação para Apreciação Ética – CAAE) number 06371012.8.0000.5188.

2.4.1. Recruitment of judges and development of descriptive terminology

Students and staff of the Federal University of Paraíba (Universidade Federal da Paraíba) were initially recruited. We selected 20 volunteers who showed the highest interest and willingness to participate in the sensory tests, product familiarity, ability to use intensity scales, memory of 25 aromas from the honey Aroma Wheel suggested by Bruneau, Barbier, Gallez, and Guyot-Declerck (2000), and ability to differentiate the aroma of honey samples of different origins produced by stingless and stinging bees using a series of triangle tests (ASTM, 2004; Meilgaard, Civille, & Carr, 2006).

The 20 judges selected using the previously described tests developed the descriptive terminology for honey samples using the Repertory Grid Keily's Method reported in Moskowitz (1983). All samples were served in disposable cups coded with random three-digit numbers at 25 °C. The samples were offered to the judges to allow them to indicate the terms that best described the similarities and differences in the aroma and flavor traits between samples. Then, the judges met in groups under the supervision of a moderator and consensually defined the terms describing the samples. This process generated 7 aroma descriptors, 2 flavor descriptors and 2 taste descriptors and a list with the definitions of each term and references. A sample descriptive assessment form was also prepared wherein the descriptors consensually generated by the sensory panel were associated with 9-cm non-structured scales anchored with the intensity terms "none/weak" and "strong" on the left and right ends of the scale.

2.4.2. Training and selection

Several judge training sessions were conducted using the references generated in the previous step, the descriptor definitions, stingless bee honey samples and a fact sheet. All judges were asked to evaluate six honey samples in four replicates at the end of the training step using the fact sheet. The samples were evaluated in different sessions, with only three samples served in each session.

The data generated by each judge in each trait were evaluated by a two-factor (sample and replicate) analysis of variance (ANOVA). Judges with suitable discriminative powers (pF samples < 0.1), good reproducibility in trials (pF replicates > 0.05) and a consensus with the team in at least 80% of the descriptors were selected to form the final descriptive team (ASTM, 2004).

2.4.3. Sample evaluation

The 8 judges selected and trained as described in the previous steps evaluated the honey samples of interest in the present study in 3 replicates using the descriptive evaluation form developed in the previous step. Only 3 samples were evaluated in each session, which balanced the order of presentation between judges and sessions.

2.5. Statistical analysis

The sensory data were evaluated by ANOVA with the following sources of variation: bee, blossom and interaction between the bee ∗ blossom effects. Tukey's test was used to compare the means. Principal component analysis (PCA) was applied to the chemical and sensory data using the statistical software Statistical Analysis System (SAS®, 2014).

3. Results

3.1. Sensory profiles of "Melipona" honey

Table 2 outlines the F and pF values of each sensory descriptor of honey regarding the "blossom", "bee species" and "blossom*bee" interaction effects. The "blossom" variable had a significant ($p < 0.0001$) effect on all aroma and flavor sensory descriptors, whereas the "bee" variable only had a significant effect on the following descriptors: characteristic ($p = 0.0016$), floral ($p < 0.0001$), medicinal ($p = 0.0042$), clove ($p < 0.0001$) and herbaceous aroma ($p = 0.0001$).

Table 2 indicated that the caramel aroma and beeswax, acid and sweet tastes, and caramel flavor scores were only affected by the

blossom because both pFbee and pFblossom*bee were higher than 0.05 for these descriptors. The juazeiro blossom honeys were significantly ($p \leq 0.05$) sweeter and had stronger aromas and caramel flavors than the other blossom honeys. In contrast, the velame blossom honeys were more acidic and less sweet ($p \leq 0.05$).

The aforementioned results corroborate the results reported by Sousa et al. (2016), who examined monofloral honeys produced by jandaira and uruçu bees in juazeiro, sensitive plant, velame branco and jurema branca blossoms from all Brazilian semiarid regions and verified that the bee species had no significant ($p \leq 0.05$) effect on the sweet taste, characteristic honey flavor, and aroma and acid taste intensity of honeys of the same floral origin. These researchers also observed that the juazeiro honeys had a stronger honey aroma and sweeter taste than the other honeys, whereas the sensitive plant and velame branco blossom honeys had stronger aromas and acid tastes in both bee species (jandaira and uruçu).

Table 2 indicates the occurrence of a significant interaction between the blossoms and bee species regarding the following descriptors: characteristic ($p = 0.0014$), floral ($p < 0.0001$), medicinal ($p = 0.0003$), clove ($p < 0.0001$) and herbaceous aroma ($p = 0.0279$), and medicinal flavor ($p = 0.0099$). This finding indicated that the blossom had a different effect on each bee species for these sensory descriptors, as shown in Fig. 1.

Fig. 1 clearly shows that the blossom had a much stronger effect on the honey aroma and flavor scores, even for scores significantly ($p \leq 0.05$) affected by the bee species. The only exception was the floral aroma, which was stronger in the velame blossom honey produced by the jandaira bees than in the honey produced by the uruçu bees.

Table 2 outlines the average intensity of each descriptor for each sample evaluated by the sensory panel. Table 2 and Fig. 1 show that the juazeiro blossom honeys had a significantly ($p \leq 0.05$) stronger characteristic aroma, caramel aroma, sweet taste and caramel flavor than the jurema branca and velame branco blossom honeys regardless of the bee species. The jurema branca blossom honeys had a significantly ($p \leq 0.05$) stronger beeswax and herbaceous aroma, whereas the velame blossom honeys had significantly ($p \leq 0.05$) higher medicinal aroma, medicinal flavor and acid taste scores than the honeys produced from the other blossoms, regardless of the bee species. The herbaceous aroma of the jurema branca blossom honey produced by the uruçu bees was significantly ($p \leq 0.05$) stronger than the herbaceous aroma of the honey produced by the jandaira bees.

Fig. 2 shows the results from the principal component analysis (PCA) of the sensory data, which accounts for 93.5% of the total sensory profile variation between samples. In this figure, sensory descriptors are represented by vectors, which indicate their importance for sample

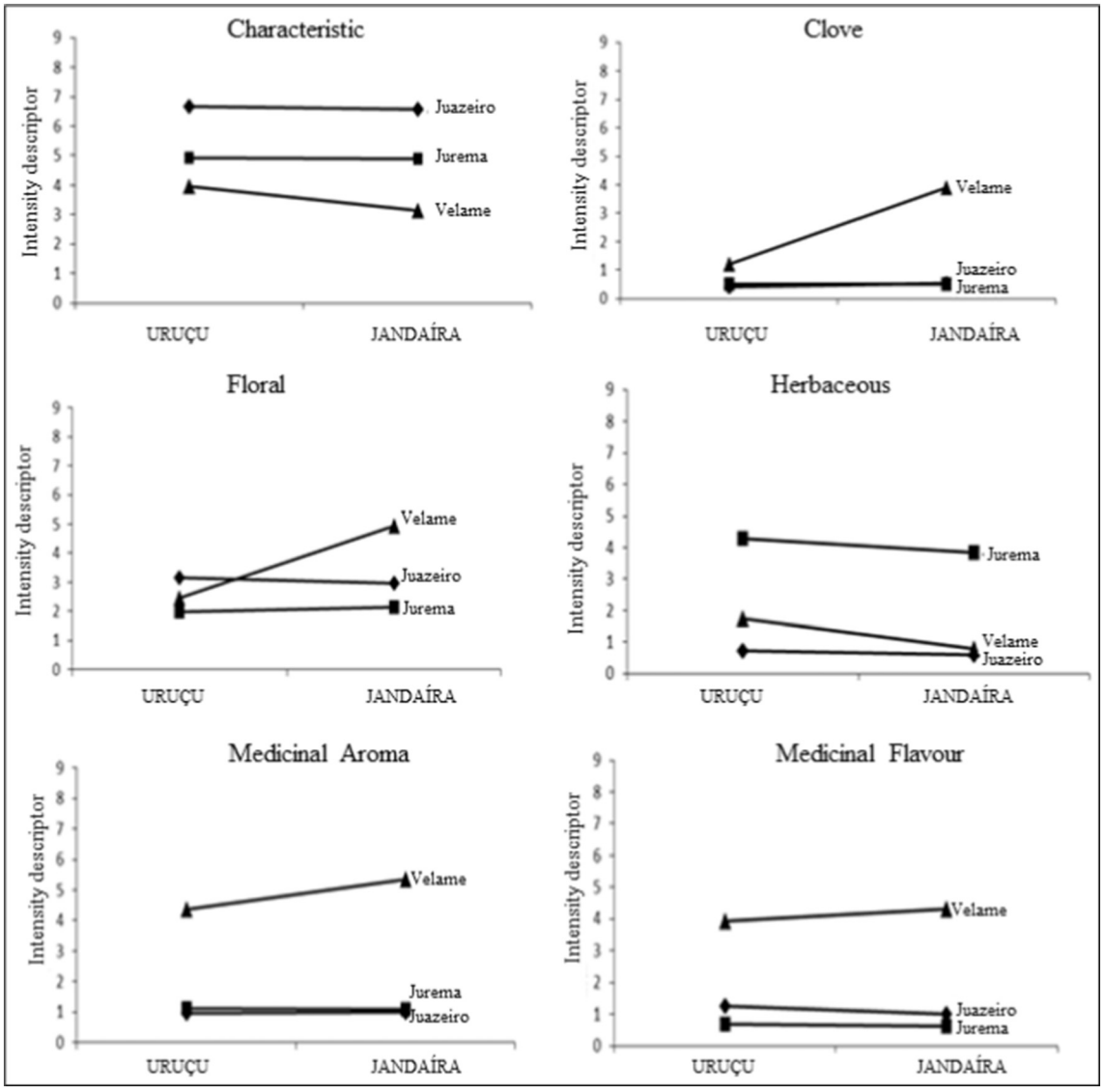

Fig. 1. Effects of bee and blossom species on descriptors with significant blossom*bee interactions.

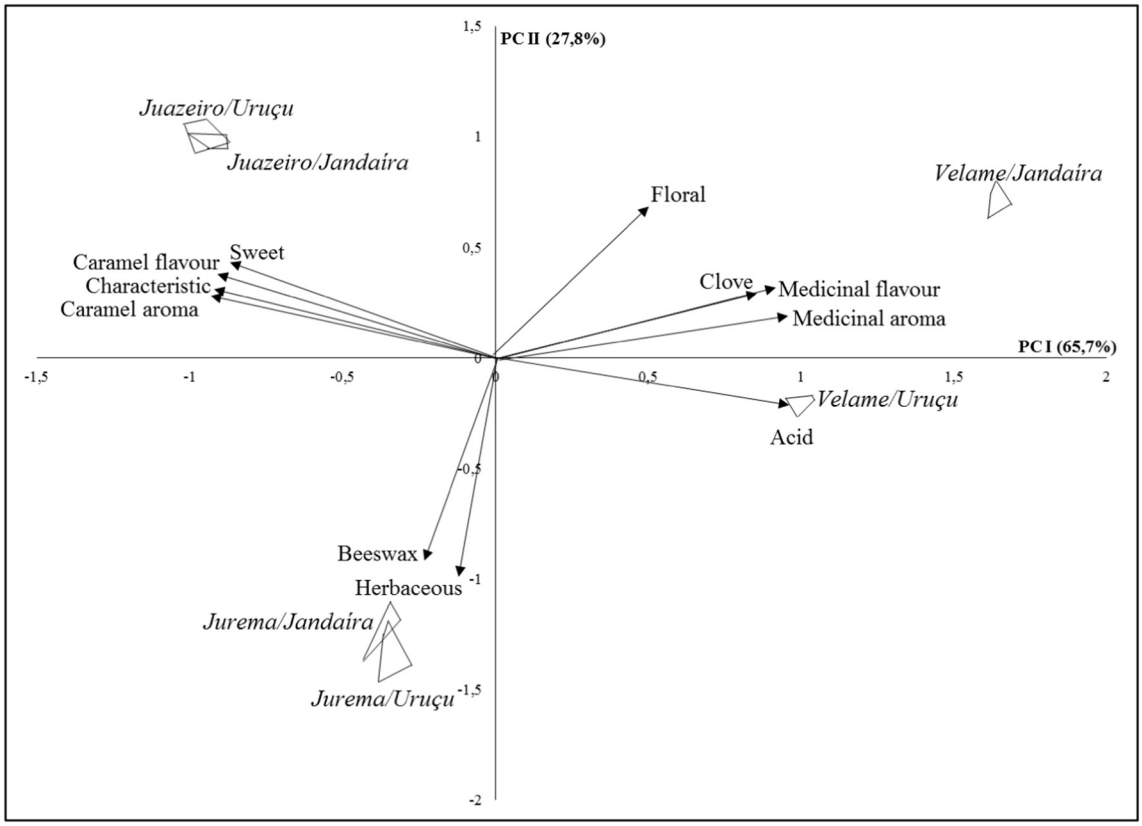

Fig. 2. Principal component analysis of the aroma, flavor and taste descriptors of the elegant mimosa, jua and velame blossom honeys produced by the jandaira and true uruçu stingless bees.

segmentation and the direction of the increase in descriptor intensity when decomposed in each axis. The samples are represented by triangles whose vertices correspond to each replicate performed by the sensory panel. Similar samples occupy nearby regions, whereas samples with different sensory profiles occupy regions farther apart from one another. The sample position in relation to the axis and vectors indicates descriptors with higher or lower scores in each sample compared with the other samples (Biasoto, Netto, Marques, & Silva, 2014).

The analysis in Fig. 2 shows a visible separation between the honeys of three botanical origins. The honeys produced by the same blossom occupy regions close to one another regardless of the bee species. The juazeiro blossom honeys had sensory profiles that were quite similar to one another and were different from the samples associated with the other blossoms, as shown in Fig. 2. Their positions in Fig. 2 suggest that they typically have a stronger characteristic and caramel aroma, sweet taste and caramel flavor. These differences from the other samples were significant at $p \leq 0.05$, as shown in Table 2.

Fig. 2 also suggests that the jurema branca blossom honeys had sensory profiles that were very similar to one another and were different from the profiles of the other blossoms regardless of the bee species; these honeys showed higher herbaceous aroma and beeswax scores. The significant differences at $p \leq 0.05$ are outlined in Table 2.

The velame branco blossom honey samples also occupied regions close to one another in Fig. 2, which indicated that they had sensory profiles that were similar to one another and different from the other samples. Fig. 2 suggests that these honeys typically have a significantly ($p \leq 0.05$) stronger clove aroma, medicinal aroma and flavor and acid taste than the other samples, as outlined in Table 2. However, the distance between the velame blossom honey produced by jandaira bees and the honey produced by uruçu bees suggested that the bee species had a significant effect on the honey sensory profile, as shown in Fig. 2. The sample produced by the jandaira bees occupied the rightmost position in axis 1 of Fig. 2, which indicated that this sample had a stronger floral and clove aroma, medicinal aroma and flavor, and acidity than the honey produced by the uruçu bees. All differences are significant at $p \leq 0.05$ with the exception of the acid taste, as shown in Table 2.

3.2. Volatile profile of "Melipona" honey

A total of 96 different volatile compounds belonging to the following chemical classes were identified in the six samples of stingless bee honey tested: terpenes (28 compounds), esters (17), norisoprenoids (9), acids (9), alcohols (6) hydrocarbons (5), benzene compounds (5), furans (5), sulfur compounds (5), ketones (4) and aldehydes (3; Table 3). Only two of the 96 compounds (hotrienol monoterpene and safranal norisoprene) were present in all of the tested honey samples.

A total of 44 compounds were detected in the honey samples from the jurema branca blossoms produced by the uruçu species, whereas 33 compounds were detected in the honey produced by the jandaira bees; thirty-two of the compounds were detected in both honeys as shown in Table 3. All of the compounds detected in the honey produced by the jandaira bees were also present in the uruçu honey with the exception of the α-pinene monoterpene. A total of 38 and 29 compounds were detected in the velame blossom honey samples produced by the uruçu and jandaira bees, respectively, including 21 compounds detected in both honeys. A total of 26 and 28 compounds were detected in the blossom juazeiro honey samples produced by the uruçu and jandaira bees, respectively, including 19 compounds detected in both honeys.

The volatile compound profiles mostly varied from sample to sample (Table 3); the qualitative diversity was clearer in honeys of different botanical origins than in honeys produced by different bee species from the same blossom. However, these profiles were not uniform for samples from the same blossom produced by different bee

Table 3
Volatile compositions of the velame, elegant mimosa and jua blossom honeys produced by the true uruçu and jandaira species.

Compound	LRI[2]	"Peak Area Count × 10^6"[1]					
		Velame branco		Juazeiro		Jurema branca	
		Uruçu	Jandaíra	Uruçu	Jandaíra	Uruçu	Jandaíra
Acids							
Ethanoic acid[c]	< 800	11.20	6.72	–	1.56	2.26	0.63
Propanoic acid[c]	< 800	–	1.11	–	–	–	–
Octanoic acid[b]	1186	–	–	–	–	0.30	–
Decanoic acid[b]	1375	0.22	–	–	–	–	–
Dodecanoic acid[b]	1530	0.45	–	–	–	–	–
Tetradecanoic acid[b]	1764	2.02	0.80	–	–	–	–
Pentadecanoic acid[b]	1862	0.17	–	–	–	–	–
Hexadecanoic acid[b]	1967	6.88	5.91	–	–	–	–
Octadecanoic acid[b]	2167	1.36	–	–	–	–	–
Total area		22.3	14.54	–	1.56	2.56	0.63
% area		35.40	12.13	–	0.70	3.82	3.20
Number of compounds		n = 7	n = 4	n = 0	n = 1	n = 2	n = 1
Alcohols							
2-Methyl-1-Butanol[c]	< 800	0.70	–	–	–	0.32	–
Pentanol[c]	< 800	0.65	–	–	–	0.65	0.35
2,3-Butanediol[b]	807	–	–	0.89	9.39	0.33	–
Hexanol[b]	850	–	–	–	–	3.91	0.27
Octanol[b]	1075	1.25	0.47	–	–	1.71	0.46
Nonanol[b]	1162	0.72	–	–	26.3	1.55	0.50
Total area		3.32	0.47	0.89	35.69	8.47	1.5
% area		5.27	0.39	1.87	15.91	12.62	8.04
Number of compounds		n = 4	n = 1	n = 1	n = 2	n = 6	n = 4
Aldehydes							
Benzaldehyde[a]	961	–	–	0.14	0.84	–	–
Benzeneacetaldehyde[b]	1031	–	–	0.29	2.53	–	–
Lilial[b]	1522	–	–	–	1.69	–	–
Total area		–	–	0.43	5.06	–	–
% area		–	–	0.90	2.26	–	–
Number of compounds		n = 0	n = 0	n = 2	n = 3	n = 0	n = 0
Benzenoid compounds							
Toluene[c]	< 800	0.36	–	–	–	–	–
p-Xileno[b]	850	0.15	–	–	–	–	–
Styrene[b]	875	–	–	–	–	0.54	0.73
4-Methyl-1-methoxybenzene[b]	1007	1.62	15.40	–	–	–	–
2,5-dimethy-phenoll[b]	1020	–	–	0.81	8.64	–	–
Total area		2.13	15.40	0.81	8.64	0.54	0.73
% area		3.38	12.85	1.70	3.85	0.80	3.71
Number of compounds		n = 3	n = 1	n = 1	n = 1	n = 1	n = 1
Ketones							
2-Heptanone[b]	881	–	–	–	0.49	–	–
4-Undecanone[b]	1202	–	–	–	–	0.24	0.07
2-Tridecanone[b]	1492	0.94	–	1.35	–	–	–
2-Pentadecanone[b]	1688	0.58	0.31	–	–	–	–
Total area		1.52	0.31	1.35	0.49	0.24	0.07
% area		2.42	0.26	2.84	0.22	0.36	0.36
Number of compounds		n = 2	n = 1	n = 1	n = 1	n = 1	n = 1
Esters							
Methyl Etanoate[c]	< 800	1.42	–	–	–	–	–
Ethyl acetate[c]	< 800	–	–	0.31	–	–	–
Ethyl Propanoate[c]	< 800	0.19	7.25	–	–	0.36	0.46
Propyl Acetate[c]	< 800	0.88	8.75	–	–	–	–
Ethyl Butanoate[b]	802	–	–	–	–	0.28	0.08
Ethyl Pentanoate[b]	891	0.41	0.63	–	–	0.34	0.38
Ethyl Benzoate[b]	1158	–	–	–	–	2.61	0.53
Ethyl Octanoate[b]	1186	–	–	–	–	0.73	0.80
Ethyl 2-phenylethanoate[b]	1223	0.15	0.19	–	–	1.36	1.02
Phenylethyl Acetate[b]	1236	–	–	–	–	2.03	0.82
Ethyl Nanoate[b]	1280	–	–	–	–	0.58	–
Nonanyl Acetate[b]	1296	–	–	–	–	2.30	1.29
Ethyl Decanoate[b]	1396	0.23	5.95	–	–	–	–
Ethyl 4-methoxybenzoate[b]	1420	–	–	–	–	0.68	–
Ethyl Dodecanoate[b]	1595	0.18	4.89	–	–	2.19	0.76
Ethyl Tetradecanoate[b]	1782	–	–	–	–	0.32	–
Ethyl Hexadecanoate[b]	1994	0.34	–	–	–	0.47	0.33
Total area		3.8	27.66	0.31	–	14.25	6.47
% area		6.03	23.08	0.65		21.24	32.91

(*continued on next page*)

Table 3 (*continued*)

Compound	LRI[2]	"Peak Area Count × 10^6"[1]					
		Velame branco		Juazeiro		Jurema branca	
		Uruçu	Jandaíra	Uruçu	Jandaíra	Uruçu	Jandaíra
Number of compounds		n = 8	n = 6	n = 1	n = 0	n = 13	n = 10
Furans							
Furfural[b]	836	–	8.18	–	–	–	–
2-Furanmethanol[b]	860	–	5.01	2.50	5.18	–	–
Isomaltol[b]	989	–	–	–	1.09	–	–
Furaneol[b]	1052	0.68	3.39	2.57	4.34	–	–
5-Hydroxymethylfurfural[b]	1234	–	2.50	–	–	–	–
Total area		0.68	19.08	5.07	10.61	–	–
% area		1.08	15.92	10.66	4.73	–	–
Number of compounds		n = 1	n = 4	n = 2	n = 3	n = 0	n = 0
Hydrocarbons							
Pentane[b]	< 800	–	–	–	2.58	0.23	–
Heptane[b]	< 800	–	0.49	–	–	–	–
Octane[b]	800	0.54	–	0.09	2.11	0.12	0.06
Hexadecane[a]	1598	–	–	–	1.61	–	–
Tricosane[b]	2398	0.28	–	–	–	–	–
Total area		0.82	0.49	0.09	6.3	0.35	0.06
% area		1.30	0.41	0.19	2.81	0.52	0.31
Number of compounds		n = 2	n = 1	n = 1	n = 3	n = 2	n = 1
Norisoprenoids							
α-Isophorone[b]	1123	4.32	2.58	–	–	–	–
Safranal[b]	1184	4.75	6.31	7.04	33	3.87	1.24
Edulan II[b]	1247	–	–	0.78	6.5	–	–
Edulan I dihydro[b]	1267	–	–	–	10.2	–	–
α-Ionene[b]	1274	–	–	2.42	25.1	–	–
1,1,6-Trimethyl-1,2-dihydronaphthalene[b]	1336	–	–	1.44	–	0.57	0.53
β-Damascenone[b]	1368	–	–	4.80	25.2	–	–
β-Ionone epoxide[b]	1458	–	–	2.00	5.27	–	–
β-Ionone[b]	1477	1.01	–	–	–	–	–
Total area		10.08	8.89	18.48	105.27	4.44	1.77
% area		16.00	7.42	38.86	46.93	6.62	9.00
Number of compounds		n = 3	n = 2	n = 6	n = 6	n = 2	n = 2
Sulfur							
Thioacetic S-acid[c]	< 800	–	–	8.32	2.47	–	–
Methanethiol[c]	< 800	1.55	–	–	–	–	–
Thiazolidine[b]	902	–	–	–	–	6.33	–
2-Propylthiazole[b]	980	–	–	–	–	5.07	–
2-Butylthiazole[c]	1040	–	–	–	–	7.60	–
Total area		1.55	–	8.32	2.47	19	–
% area		2.46	–	17.49	1.10	28.32	–
Number of compounds		n = 1	n = 0	n = 1	n = 1	n = 3	n = 0
Terpenoids							
α-Pinene[b]	926	–	–	–	–	–	0.17
α-Terpinene[a]	1004	–	–	1.25	–	–	–
D-Sylvestrene[b]	1024	–	–	–	5.53	–	–
Limonene[a]	1028	–	–	–	–	3.57	2.61
Ocimene[b]	1035	–	0.31	–	–	–	–
γ-Terpinene[b]	1045	–	–	0.21	5.98	–	–
Cis-Linalool oxide[b]	1076	0.14	–	0.49	–	–	–
Trans-Linalool oxide[b]	1088	–	–	–	–	0.44	–
Linalool[a]	1091	0.07	9.32	–	–	–	–
Hotrienol[a]	1092	3.73	0.59	1.58	8.35	0.93	0.14
Isothujol[b]	1128	1.31	4.12	–	–	–	–
2,6-Dimethyl-1,3,5,7-octatetraene[b]	1134	–	–	–	–	0.98	0.32
Nerol oxide[b]	1143	–	0.00	0.46	–	0.39	0.20
1-Adamantanol[b]	1159	–	–	0.15	6.97	–	–
α-Terpineol[b]	1196	1.84	1.69	–	–	–	–
Pulegone[b]	1238	–	–	–	–	2.32	0.30
2,6-Diemtyl-3,7-Octadiene-2,6-diol[b]	1330	–	2.65	–	–	–	–
α-Copaene[b]	1357	–	5.00	1.71	4.24	0.23	0.08
Cedrene[b]	1393	–	–	–	–	0.39	0.16
Methyl Eugenol[b]	1341	7.30	8.81	–	–	–	–
α-Caryophyllene[a]	1418	2.40	0.52	3.28	13	–	–
β-Caryophyllene[a]	1421	–	–	1.33	–	2.80	0.33
α-Aromade-rene[b]	1439	–	–	–	–	0.50	0.78
β-Selinene[b]	1440	–	–	–	–	1.00	0.17
α-Farnesene[b]	1498	–	–	–	–	2.00	2.59
δ-Cardinene[b]	1503	–	–	–	–	1.26	0.50

(*continued on next page*)

Table 3 (*continued*)

Compound	LRI[2]	"Peak Area Count × 10^6"[1]					
		Velame branco		Juazeiro		Jurema branca	
		Uruçu	Jandaíra	Uruçu	Jandaíra	Uruçu	Jandaíra
Eremophylene[b]	1526	–	–	–	–	0.44	–
β-Guaiene[b]	1485	–	–	1.35	4.15	–	–
Total area		16.79	33.01	11.81	48.22	17.25	8.35
% area		26.66	27.54	24.83	21.50	25.71	42.47
Number of compounds		n = 7	n = 10	n = 10	n = 7	n = 14	n = 13

-: Undetected compound.
[1] Peak area count values, mean data from triplicate sample injection.
[2] LRI: Retention index in the VF-5MS column.
[a] Positively identified compounds.
[b] Identified compounds.
[c] Tentatively identified compounds.

species, possibly because by definition honey could be considered monofloral when it contained 45% pollen from the same plant. Thus, the honey can maintain the same melissopalinological classification even if 55% of the pollen composition varies, which will result in different compositional profiles (Jerkovic & Kus, 2014).

The sources of variation for the "blossom", "bee" and "blossom*bee" interaction effects on the sample volatile compound profiles are outlined in Table 4. The analysis of the table showed that the "bee*-blossom" interaction had a significant ($p \leq 0.05$) effect on nearly all of the chemical classes identified in the honeys, including acids ($p = 0.0027$), alcohols ($p = 0.0306$), aldehydes ($p = 0.0109$), esters ($p \leq 0.0001$), hydrocarbons ($p \leq 0.0001$), norisoprenoids ($p \leq 0.0001$), terpenes ($p \leq 0.0001$) and sulfur compounds ($p \leq 0.0001$). This result indicates that the effect of the blossom on the chemical classes in the honey volatile profile is uneven and varies according to the bee species and vice-versa for almost all chemical classes (Table 4).

The ketone and furan classes were affected by the bee species and blossoms, albeit without the bee*blossom interaction. In this case, the "bee" and "blossom" variables were independent [i.e., the effect of one variable on the honey volatile profile (e.g., the bee species) was not affected by variation in the other variable (e.g., the blossom type)]. Indeed, Table 3 shows that the honeys produced by uruçu bees had a higher ketone content than the honeys produced by jandaira bees in all botanical origins.

Most compounds belonging to the furan class (particularly furfural and 5-hydroxymethylfurfural) have been used as classic indicators of heating, inadequate storage and honey adulteration with inverted sugar because these compounds are found at very low quantities in fresh honeys (Amri & Ladjama, 2013; Karabagias et al., 2014; Risner, Kiser, & Dube, 2006). However, tropical honeys may be naturally rich in these compounds without honey overheating or adulteration. This effect results from the unique climatic characteristics of the tropics region (Marchini, Moreti, & Otsuk, 2005), where the ambient temperatures reach up to 40 °C. In honey, the formation of furans (particularly the aforementioned furans) depends on the type of sugar, pH, water activity and beehive conditions (Rizelio et al., 2012). Castro-Vázquez et al. (2012) found a significant increase in the furaneol and 2-furanmethanol concentrations in honeys stored at 40 °C when studying volatile profile changes in honeys stored at different temperatures.

The chemical class of benzene compounds was unaffected by the bee species ($p = 0.1171$) or "blossom" type ($p = 0.3222$).

The juazeiro blossom honey produced by jandaira bees had higher alcohol, aldehyde, hydrocarbon and norisoprenoid contents than the other samples (Table 3). According to Moreira, De Maria, Pietroluongo, Luiz, and Trugo (2010), the hydrocarbons present in honey may derive from flower nectar, insect exudates collected by the bees and transformed into honey, or even beeswax. The aldehyde chemical class was only found in juazeiro blossom honeys and was present at higher concentrations in the samples produced by jandaira bees. Benzaldehyde and benzeneacetaldehyde, which are present in both samples, reportedly have a pleasant "honey" aroma according to several studies. Benzeneacetaldehyde is a volatile compound with a strong odorous power and low threshold (4 ppb) (Blank, Fischer, & Grosch, 1989; Castro-Vázquez et al., 2007; Karabagias et al., 2014). The occurrence is relevant because the juazeiro blossom honeys had a stronger "characteristic aroma" according to the trained sensory panel, suggesting a relationship between these aromatic aldehydes and the perception of the characteristic honey aroma in the present study.

The honey produced by the uruçu species in jurema branca blossoms had a higher concentration of sulfur compounds, which differentiated this honey from the other samples. The 2-propyl thiazole and 2-butyl thiazole compounds present in the honey produced by the uruçu species and the presence of safranal, hexanol, limonene, α-farnesene and δ-cardinene may have contributed to the herbaceous aromas perceived by the sensory panel in the honeys produced in the jurema branca blossoms. These compounds reportedly have an "herbaceous", "green", or "grass" aroma (www.odour.org.uk).

Velame blossom honeys typically have higher concentrations of acids than other honeys (Table 3), which may explain why these honeys have a stronger acidic taste than other honeys (Table 2). In honeys, acids have different aromas that range from spicy to rancid depending on the length of the molecule's carbon chain. Short-chain acids, including acetic acid, have spicy flavors and aromas, whereas long-chain acids are associated with a rancid aroma (Barra, Ponce-Díaz, & Venegas-Gallegos, 2010; Manyi-Loh et al., 2011). Honey acidity derives from two sources: organic acids of different nectar origins and D-glucose oxidase enzymatic activity, which catalyzes the conversion of D-glucose

Table 4
F and pF ANOVA values of the sources of variation for the "blossom", "bee" and "blossom ∗ bee" interaction.

Atributos	Blossom		Bee		Blossom ∗ bee	
	F	*p*	F	*p*	F	*p*
Acids	169.05	< 0.0001	10.00	0.0082	10.05	0.0027
Alcohols	2.71	0.1072	1.87	0.1970	4.73	0.0306
Aldehydes	9.48	0.0034	6.74	0.0234	6.74	0.0109
Benzenoids	1.25	0.3222	2.85	0.1171	0.82	0.4650
Ketones	5.78	0.0175	12.45	0.0042	2.04	0.1721
Esters	65.78	< 0.0001	21.86	0.0005	71.72	< 0.0001
Furans	3.95	0.0481	6.92	0.0219	3.24	0.0752
Hydrocarbons	19.13	0.0002	19.17	0.0009	26.02	< 0.0001
Norisoprenoids	53.82	< 0.0001	29.73	0.0001	34.01	< 0.0001
Sulfur	66.13	< 0.0001	129.81	< 0.0001	83.35	< 0.0001
Terpenoids	47.97	< 0.0001	95.85	< 0.0001	79.52	< 0.0001

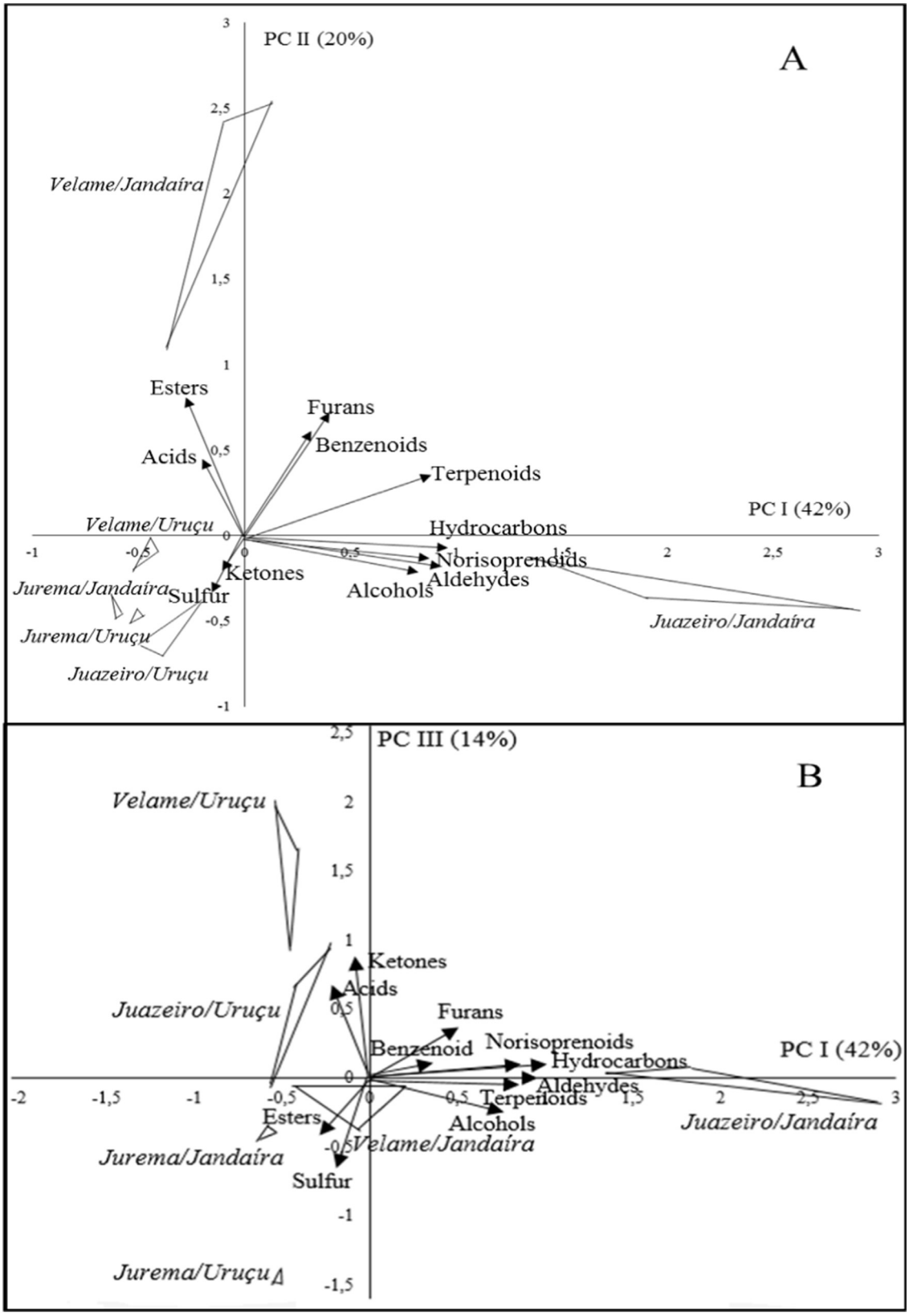

Fig. 3. Principal component analysis of the chemical classes of the aroma components of the elegant mimosa, jua and velame blossom honeys produced by the jandaira and true uruçu stingless bees. (A) Sample variation regarding principal components I and II and (B) sample variation regarding principal components I and III.

into gluconic acid (Abadio, Moura, & Silva, 2010; Belay, Solomon, Bultossa, Adgaba, & Melaku, 2013). Honey acidity may also be caused by bacterial activity during the product maturation stage and its mineral content (Alqarni, Owayss, & Mahmoud, 2012; Pasini, Gardini, Marcazzan, & Carboni, 2013).

The sensory analysis of the velame honeys showed that they

typically had a stronger clove aroma than the other samples in addition to a stronger acidic taste (2). The volatile profile outlined in Table 3 showed that the velame honeys were the only honey with methyl eugenol in their compositions. This volatile compound is considered a characteristic impact compound that accounts for the "clove", "spicy" and "hot" aromas of several products (www.odour.org.uk). Although the data on the chemical composition of flower extracts or essential oils of the *Croton heliotropiifolius* (velame) species are scarce, several species of the same genus, including *Croton adamantinus*, *Croton zehntneri*, and *Croton malambo*, have shown that this phenyl terpenoid is one of the main chemical constituents of essential oils (Cabral et al., 2014; Colorado, Duarte, Munoz, & Stashenko, 2010; Ximenes et al., 2013).

Fig. 3A and B show the results from the quantitative descriptive analysis that was applied to the abundance data on the chemical classes of the volatile compounds of the honey samples in the present study. Together, both figures account for 76% of the variation between samples. Fig. 3A shows the position of the velame blossom honey produced by jandaira bees and suggests that this honey stands out among the other honeys due to its higher concentration of esters, as shown in Table 3. In turn, the honey produced by jandaira bees from juazeiro blossoms differed from the other honeys because it had a higher concentration of aliphatic hydrocarbons, norisoprenoids, aldehydes and alcohols, as shown in Table 3. The differences between the sensory profiles of both honeys produced by jandaira bees highlight the blossom effect on the product's volatile composition.

Fig. 3B shows the variation of samples based on principal component III (PC III) of the PCA. In the present study, PC III is strongly associated with the sample's acid, ketone and sulfur compound concentrations. Fig. 3B shows that the more positive PC III values are indicative of higher sample acid and ketone concentrations. Thus, the results shown in Fig. 3B indicate that the velame and juazeiro samples produced by the uruçu bees have higher acid and ketone concentrations, as shown in Table 3. Similarly, Fig. 3B also suggests that the Jurema blossom honey produced by the uruçu bees has a higher sulfur compound concentration, as shown in Table 3.

The maintenance of the same volatile compound profiles in honeys from the same floral origin was previously reported by Bicchi, Belliardo, and Frattini (1983), who observed the same chromatographic profile of volatile compounds of honeys from the Piedmont region in different harvest years. However, the accumulation of phytochemicals and the precursors of volatile components, including carbohydrates, phenols and volatile organic compounds, depends on the climatic conditions and soil characteristics. Thus, differences between honeys with the same botanical origin produced by different species in different regions are presumably associated with different nectar or pollen compositions, which have the strongest effects on the chemical composition of the honey (Castro-Vázquez et al., 2014; Jerkovic & Kus, 2014).

Few studies have correlated the chemical constituents of flower extracts or nectar with their compositions in honeys. Generally, only partial similarities between the volatile constituents of nectar, flower extracts, and honeys have been found. Differences between honey and flower extracts are expected because the honey aroma compounds are constituents of various flower and plant parts. Partial qualitative similarities were also found in studies evaluating the nectar and essential oil compositions in honey (Jerkovic & Kus, 2014).

4. Conclusion

In the present study, the blossom botanical origin had a stronger effect on the aroma and flavor profile of the honeys than the producing bee species. The juazeiro blossom honeys had a higher sweetness and stronger caramel aroma and flavor. These honeys also had a more characteristic honey aroma, which might be associated with the benzaldehyde and benzeneacetaldehyde compounds. The velame blossom honeys typically showed a strong medicinal taste, clove aroma and higher acid concentration than the other honeys. The compounds hexanol, limonene, α-farnesene and δ-cardinene may have contributed to the generation of the herbaceous aromas perceived with higher intensity by the sensory panel in the jurema branca blossom honeys. Future studies should corroborate that these volatile compounds may be markers of these blossoms and desirable compounds in meliponini honey.

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