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Potential Typicality Marker of Volatile Composition of Commercial Sparkling Wines from the Caatinga Biome

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Abstract
The present research aimed to identify the volatile profile of sparkling wines from São Francisco Valley, which products will have soon the Geographical Indication requested by the producers. Volatile organic compounds from muscat, brut, brut rosé and demi-sec sparkling wines produced in the São Francisco Valley, located in the Caatinga region of Brazil, were extracted, separated and identified using the HS-SPME/GC–MS technique. The results reached the identification of a total of 109 compounds, classified in 13 chemical groups, being the main esters, terpenes, and alcohols. It was found that some compounds with expressive area are unique to each type of sparkling wine: a total of 23 in muscat, 9 in brut, 5 in brut rosé, and 4 in demi-sec. This suggests a strong association between the grape varieties and the technological processes of winemaking. The volatile profiles of each commercial sparkling wine in the São Francisco Valley present possible chemical markers of typicity which can be used to distinguish the commercial wines from the region.

Keywords Volatile compound · São Francisco Valley · Geographical indication · Typicality marker · Gas chromatography

Introduction

Global wine (made with *Vitis vinifera* grapes) production in 2019 reached 260 million hectolitres (mhL), which 2.0 mhL were produced by Brazil, where it has been outstanding in wine production in the southern hemisphere, occupying the fifth position with high-quality products, among red, sparkling, and also white wines (Ibravin, 2020a; OIV, 2020). Brazilian wineries have been investing in Geographical Indications (GI) in the last 20 years, a quality seal that attests the quality due to the specificity of the *terroir* of each producing region. Nowadays there are seven regions with GI, mainly in the South, in Rio Grande do Sul and Santa Catarina States (Embrapa, 2020).

Geographical Indication is able to boost territorial development in its social, economic, political and cultural aspects, adding a differential value to products and services, and giving notoriety to the region (Siedenberg et al. 2017). In

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this regard, the São Francisco Valley (VSF) is a traditional winegrowing region, producing tropical wines since 30 years ago, actually with 4 million liters of wines (*Vitis vinifera* grapes) per year, which sparkling wines represent about 70% of the total production (Embrapa, 2020; Pereira et al. 2018). The VSF is exclusively located in the northeastern Brazil, in the caatinga biome (exclusively Brazilian vegetation), presenting a tropical semi-arid climate. In these conditions, with high annual average temperatures (26.5ºC), high solar radiation, and water availability for irrigation, it is possible to produce grapes every day of the year, by scheduling the plots. The soils are classified as yellow eutrophic argisol/typical plintustalf (soil taxonomy alfisol), usually with low natural fertility, and a vine is pruned once a year and the grapes are harvested twice (Benedetti et al. 2011; Ibravin, 2020b). In this region, the sparkling wines are mainly produced by the *asti* (single fermentation in pressure tanks) and *charmat* (second alcoholic fermentation in the pressure tanks or autoclaves) methods (Brasil, 2004; Pereira et al. 2018; Soares et al. 2015).

In addition to winemaking protocols and grape variety, other factors such as soil and climatic conditions, as well as viticultural practices influence the chemical composition of wines, mainly volatile compounds (Fernandes et al. 2018). The aromas present varied intensity and complexity and are associated with the expression of a single grape variety or its mixture/blend, being considered decisive factors in the sensory quality, typicality, and acceptability of wines (Sánchez-Palomo et al. 2017).

The aromas are directly influenced by the volatile composition of the wines. The extraction of the compounds by solid phase microextraction (SPME) associated with separation by high efficiency gas chromatography (GC) and the mass spectrophotometric detector represents a reliable tool for analysis (Olegário et al. 2019; Ruiz et al. 2019). These techniques have been used for wine analysis (Muñoz-Redondo et al. 2020; Tufariello et al. 2019; Ubeda et al. 2019) in order to differentiate cultivars, determine the quality and typicality of wines (Sánchez-Palomo et al. 2017), and differentiate the geographical origin of this beverage (Ziółkowska et al. 2016).

A study by Nascimento et al. (2018) with experimental sparkling wines from the Chenin Blanc and Syrah cultivars produced in the São Francisco Valley showed that the grape variety used can significantly influence the volatile profile. Recently, de Macedo Morais et al. (2022) described the volatile profile of different commercial tropical red wines produced in this region, with the detection of unique compounds. Barbará et al. (2020) studied the volatile profile of wines produced with Syrah grapes and it was shown that ten days of maceration and 19° Brix were significant for the volatile composition. Fernandes et al. (2018) analyzed wines from the VSF, Minas Gerais, and Rio Grande do Sul in Brazil, and showed that the main volatile profile markers of red wines were esters and alcohols. The above-mentioned studies indicate that factors such as cultivar, technological winemaking process, and geographical origin influence on the volatile composition of experimental wines produced in the VSF. However, in our knowledge, there is no studies to date characterizing the volatile composition of commercial sparkling wines from this region.

In this context, this study aimed to identify the volatile profile of the main sparkling wines produced in the region, which products will take part of the Geographical Indication São Francisco Valley, in the way of recognition by the Brazilian Government. The characterization of the products will help the scientific community, as well as the producers and consumers, to know in details which are the main aroma present in the sparkling wines.

Material and Methods

Samples

Twelve sparkling wines indicated to compose the Vale do São Francisco Geographical Indication, and were grouped into four commercial categories of sparkling wines from the Caatinga biome. All wines came from the same winery (9° 15′ S and 40° 50′ W), because this one is the highest of the region, with most different wines with 120 hectares of vineyards (60% for sparkling wines). They represent around 65% of total sparkling wines produced in the region. The details of four groups of sparkling wine are detailed in Table 1. These sparkling wines selected represent in terms of volume produced/marketed and in types, approximately 50–60% of the total of the region, being thus, quite representative. The samples were composed by three bottles (750 mL) from the same batch and the experiments were carried out in triplicate. Sparkling wines were stored at 16ºC ± 1ºC until the analysis for a period not exceeding 6 months.

Solid-phase microextraction

An aliquot of 30 mL of sparkling wine was transferred to a 100-mL glass vial with a screw cap containing a Teflon-lined septum (Supelco®, Bellafonte, PA, EUA). Extractions were carried out with an SPME device (Supelco®, Bellafonte, PA, EUA) containing a fused-silica fiber coated with a 65-μm layer of Polydimethylsiloxane/Divinylbenzene (PDMS/DVB, Supelco, Bellefonte, USA). The stainless steel needle housing the fiber penetrated the septum of the glass recipient, and after equilibration at 45ºC for 15 min, the fiber was then exposed to the headspace above the wine for 30 min, under continuous stirring (250 rpm) according to the method adapted from Barros et al. (2012). After extraction,

Table 1 Description of the samples of Sparkling wine from the São Francisco Valley, production process and composition

Sparkling wine	Varieties	Method	Elaboration process	Filtration	Alcohol content	Sugar content
Moscatel	*Muscat*	*Asti*	One fermentation (30 days at 16 ± 2ºC)	0.45 µm membrane	7.24%	62.32 g/L
Brut rosé	*Grenache*	*Charmat*	First fermentation in steal stainless (30 days at 16 ± 2ºC); second fermentation inside autoclave/ pressure tanks (30 days at 16 ± 2ºC)		11.57%	10.36 g/L
Demi-sec	*Chenin Blanc; Sauvignon Blanc;* and *Verdejo* (1:1:1)	*Charmat*			10.87%	24.61 g/L
Brut		*Charmat*			11.80%	8.02 g/L

the fiber was collected and the SPME device was removed from the wine sample vial and inserted directly into the GC–MS injection port. The fiber was conditioned before the extraction by heating it in the gas chromatograph injection port at 250 ºC for 30 min. Blank analyses were carried out before the analysis of each sample, with the same methods of samples analyses.

Gas chromatography–Mass Spectrometry Conditions

An Agilent® Technologies 5977B (Little Falls, ME, USA) mass spectrometer coupled to a 7890B gas chromatograph was used to separate and identify the volatiles collected by SPME. GC separation of the collected volatiles was performed on a VF-5MS (30 m × 0.25 mm × 0.25 µm) column (Agilent J&W Scientific). The temperature program employed was 10 min at 40 ºC, a ramp of 7 ºC/min to 250 ºC, and held for 5 min, according to the method adapted from Barros et al. (2012) and Arcanjo et al. (2015). Helium (analytical purity of 99.9999%) was used as the carrier gas at a flow rate of 1.2 mL.min^{-1}. The injection port was in splitless mode at a temperature of 250ºC.

Mass spectrometry detector was operated in electron impact mode with a source temperature of 250 ºC, an ionizing voltage of 70 eV, and a scan range from 35 to 350 m/z at 3.33scans/s. The transfer line was held at 250 °C. The SPME data were acquired and analyzed using Mass Hunter software (Agilent®, Version 10.0, 2008).

The linear retention index was calculated for each volatile compound using the retention times of a homologous series of C8-C20 n-alkanes, of which the linear retention index below 800 were expressed as < 800. Volatile compounds that have spectral similarity to those of the NIST/EPA/NIH Mass Spectral Database were considered identified (Version 2.2 2014) showing the Match > 600 and RMatch > 700 coefficients of linear retention confirmed by the scientific literature (Kondjoyan and Berdagué, 1996). All identified compounds were quantified using total ion chromatogram (TIC) peak areas. The data were represented in terms of total chromatographic area and percentage of area and discussed according to their chemical classes. A search for antecedence was carried out in bibliographic databases to track the volatile compounds identified in red and sparkling wines from the world published in the journals available at *Portal Periódicos Capes* that contain more than 50,000 national and international journal titles. All results were compared and their similarities and differences discussed.

Statistical Analysis

The Shapiro–Wilk test was performed to check whether the results had a normal distribution. However, the data did not follow a normal distribution ($p < 0.05$), so the Kruskal–Wallis non-parametric test and the Dunn's post hoc multiple comparison test ($p < 0.05$) were used to verify the difference between the total area averages of the volatile compound groups, and subsequently displayed in the form of graphs.

The data were self-scaled and multivariate analyzes (principal component analysis and hierarchical cluster analysis with color map) were used to group the samples according to the sparkling wine categories according to their volatile composition.

The XLSTAT® version 5.03 (Addinsoft, New York, USA, 2014), MATLAB® version 7.10.0.499 (The Mathworks, Inc., Natick, MA, R2010a) and GraphPad Prism® version 6.01 (Graphpad Software Inc., San Diego, California, USA) software programs were used for these statistical analyses.

Results and Discussion

Characterization and Comparison of the Volatile Composition of Sparkling Wines from the São Francisco Valley

The identification of volatile compounds resulted in a total of 109 compounds classified into 13 groups according to their chemical characteristics. The average of chromatography area for each sparkling wine category are reported in the Table 2.

The total number of compounds identified for each category of sparkling wine was different: 83 for moscatel,

Table 2 Volatile compounds tentatively identified in the sparkling wines of the São Francisco Valley by HS–SPME–GC–MS

Code	Volatile compound*			"Peak area count" ± SD ($\times 10^{6}$)				Odor description
		LRI (lit)	LRI (cal)	MOSCATEL	BRUT ROSÉ	BRUT	DEMI-SEC	
	Acids (8)							
Ac 14	Hexanoic acid	990	995	nd	nd	0.09 ± 0.08	nd	Fatty, rancid, rubber/ferric, green[1,2,3,4]
Ac 37	Octanoic acid	1180	1176	0.02 ± 0.02	nd	nd	nd	Fatty, muddy/cooked vegetables, sweat, cheese, rancid[1,2,3,5,6]
Ac 66	Decanoic acid	1383	1385	$2.99^{b} \pm 1.94$	$27.13^{a} \pm 18.35$	$7.00^{a,b} \pm 0.46$	$8.19^{a,b} \pm 4.51$	Rancid[1]
Ac 77	Undecanoic acid	1475	1477	0.11 ± 0.06	nd	nd	nd	Oil[1]
Ac 96	Tetradecanoic acid	1763	1768	$0.20^{a} \pm 0.07$	$0.99^{a} \pm 0.19$	$0.26^{a} \pm 0.13$	$0.33^{a} \pm 0.09$	Waxy, fatty, soapy, coconut[16, 17]
Ac 104	Pentadecanoic acid	1867	1866	$0.07^{a} \pm 0.06$	$0.05^{a} \pm 0.00$	$0.05^{a} \pm 0.02$	nd	Waxy[16,17]
Ac 107	(*E*)-9-Hexadecenoic acid	1938	1940	nd	$0.23^{a} \pm 0.14$	$0.07^{a,b} \pm 0.01$	nd	Waxy, creamy, fatty, soap[16,17]
Ac 108	Hexadecanoic acid	1968	1960	$0.54^{a} \pm 0.45$	$1.31^{a} \pm 0.01$	$0.59^{a} \pm 0.22$	$0.48^{a} \pm 0.14$	Waxy, fatty [16,17]
	Total acids			3.94^{b}	29.71^{a}	$8.05^{a,b}$	$9.00^{a,b}$	
	Alcohols (14)							
Al 4	3-Methyl-1-butanol	736	$<$ 800	$6.85^{a} \pm 3.88$	$26.08^{a} \pm 11.95$	$39.28^{a} \pm 12.81$	$29.13^{a} \pm 2.42$	Alcoholic, whiskey, fruity, banana, fusel, oil solvent [1,16,17]
Al 3	2-Methyl-1-butanol	739	$<$ 800	nd	nd	nd	6.43 ± 2.05	Roasted, winey, onion, fruity, fusel, Alcoholic, whiskey, burnt, nail polish [6,16,17]
Al 2	2,3-Butanediol	788	$<$ 800	$1.41^{a,b} \pm 1.27$	$0.85^{a,b} \pm 0.00$	$0.24^{b} \pm 0.01$	$3.93^{a} \pm 0.34$	Fruity, creamy, buttery [1,7]
Al 6	1-Hexanol	868	865	$1.04^{a} \pm 0.65$	$1.25^{a} \pm 0.45$	$0.65^{a} \pm 0.50$	$2.43^{a} \pm 0.15$	Ethereal, fusel, oil, fruity, alcoholic, sweet, green, vegetative, grass just cut, fresh [2,7,8, 16,17]
Al 8	3-Methyl-2-hexanol	906	898	$0.06^{a} \pm 0.05$	nd	nd	nd	
Al 16	3-Methyl-4-heptanol	997	999	nd	nd	$0.11^{a} \pm 0.04$	nd	
Al 27	1-Octanol	1075	1076	$0.27^{a,b} \pm 0.21$	$0.67^{a} \pm 0.15$	nd	$0.17^{a,b} \pm 0.01$	Waxy, green, orange, aldehydic, rose, mushroom, solvent[3,16,17]
Al 31	Phenylethyl alcohol	1116	1114	$5.03^{a} \pm 4.92$	$4.75^{a} \pm 1.11$	$16.48^{a} \pm 1.78$	$8.26^{a} \pm 1.98$	Floral, rose, honey, flower, woody [2,5]
Al 36	1-Nonanol	1173	1172	nd	nd	$0.04^{a} \pm 0.00$	nd	Resin, vegetable, raspberry[3,9] Fresh, clean, fatty, floral, rose, orange, dusty wet, oily[16,17]
Al 51	1-Decanol	1273	1272	$0.16^{b} \pm 0.12$	$1.56^{a} \pm 0.14$	$1.26^{a,b} \pm 0.97$	$0.33^{a,b} \pm 0.05$	Fatty, waxy, floral, orange, sweet, clean, watery [16,17]
Al 71	Tyrosol	1444	1444	nd	$0.66^{a} \pm 0.01$	nd	$0.21^{a,b} \pm 0.00$	Sweet, floral, fruity, toasted bread, smoky, clove [10, 16,17]
Al 76	1-Dodecanol	1473	1474	$0.25 \pm ^{a}$ 0.21	$0.25^{a} \pm 0.03$	$0.81^{a} \pm 0.13$	$1.03^{a} \pm 0.26$	Earthy, soapy, waxy, fatty, honey, coconut, unpleasant, flowery [1,16,17]
Al 100	2-Heptadecanol	1802	1799	0.01 ± 0.01	nd	nd	nd	
Al 105	1-Hexadecanol	1884	1880	$0.18^{a} \pm 0.16$	$0.10^{a} \pm 0.01$	$0.08^{a} \pm 0.00$	nd	Waxy, clean, greasy, floral, oily [16,17]

Table 2 (continued)

Code	Volatile compound*	LRI (lit)	LRI (cal)	"Peak area count" ± SD (× 10^6) MOSCATEL	BRUT ROSÉ	BRUT	DEMI-SEC	Odor description
	Total Alcohols			15.23^{b}	$36.16^{a,b}$	58.95^{a}	51.91^{a}	
	Aldehydes (6)							
Ald 11	Benzaldehyde	962	960	nd	nd	0.18 ± 0.00	nd	Sharp, sweet, bitter, cherry, almond (1,16,17)
Ald 42	Decanal	1205	1206	21.69 ± 12.41	$3.62^{a,b} \pm 0.13$	nd	nd	Sweet, waxy, floral, grassy, orange, arugula (1,5,16)
Ald 58	Undecanal	1307	1307	nd	nd	$0.42^{a} \pm 0.15$	$0.77^{a} \pm 0.51$	Waxy, soapy, aldehydic, citrus, green fatty, cloth, laundered cloth, floral (1,16,17)
Ald 69	Dodecanal	1409	1410	$0.25^{b} \pm 0.20$	$0.53^{a,b} \pm 0.19$	$0.73^{a,b} \pm 0.39$	$1.28^{a} \pm 0.39$	soapy, waxy, aldehydic, citrus, green, floral (1,16,17)
Ald 75	(*E*)-2-Dodecenal	1468	1470	nd	0.25 ± 0.08	nd	nd	Citrus, metallic, mandarin, orange, waxy aldehydic (16,17)
Ald 103	10-Octadecenal	1863	1857	$0.11^{a,b} \pm 0.07$	$0.30^{a} \pm 0.05$	$0.04^{b} \pm 0.01$	$0.19^{a,b} \pm 0.05$	
	Total aldehydes			22.05^{a}	$4.70^{a,b}$	1.37^{b}	2.24^{b}	
	Aromatics compounds (7)							
Ar 84	4-Phenyl-decane	1546	1550	$0.22^{a,b} \pm 0.06$	$0.07^{b} \pm 0.05$	$0.17^{a,b} \pm 0.00$	$0.31^{a} \pm 0.01$	
Ar 85	3-Phenyl-decane	1568	1567	$0.35^{a} \pm 0.22$	$0.51^{a} \pm 0.11$	$0.41^{a} \pm 0.14$	$0.59^{a} \pm 0.10$	
Ar 92	6-Phenyl-undecane	1628	1635	nd	nd	$0.13^{a,b} \pm 0.05$	$0.26^{a} \pm 0.06$	
Ar 93	5-Phenyl-undecane	1632	1638	$0.32^{a,b} \pm 0.08$	$0.18^{b} \pm 0.04$	$0.39^{a,b} \pm 0.02$	$0.42^{a} \pm 0.02$	
Ar 94	4-Phenyl-undecane	1643	1649	nd	nd	nd	0.57 ± 0.12	
Ar 98	2-Phenyl-undecane	1708	1711	nd	nd	0.09 ± 0.06	nd	
Ar 81	5-Phenyl-dodecane	1730	1737	nd	nd	nd	0.10 ± 0.02	
	Total Aromatics Compounds			0.89^{b}	0.76^{b}	$1.20^{a,b}$	2.25^{a}	
	C13-Norisoprenoid (2)							
No 62	1,1,6-Trimethyl-1,2-dihydronaphthalene	1350	1351	nd	nd	0.62 ± 0.01	nd	Licorice, burned, tabac, herb, petrol (3,11,16)
No 63	1,2-Dihydro-1,5,8-trimethyl-naphthalene	1354	1352	$2.66^{a,b} \pm 1.18$	$5.90^{a} \pm 0.07$	nd	$0.98^{a,b} \pm 0.10$	
	Total C13-Norisoprenóide			$2.66^{a,b}$	5.90^{a}	0.62^{c}	$0.98^{b,c}$	
	Esters (37)							
Es 5	Ethyl butanoate	802	801	$0.02^{b} \pm 0.00$	$0.39^{a,b} \pm 0.11$	$0.36^{a,b} \pm 0.12$	$0.56^{a} \pm 0.06$	Juicy, pineapple, cognac, fruity, sweet, tutti frutti, apple, aromatic (8,12,16,17)
Es 7	Isoamyl acetate	876	875	$0.57^{a} \pm 0.38$	$1.20^{a} \pm 0.05$	$2.53^{a} \pm 2.52$	$1.75^{a} \pm 0.16$	Sweet, fruity, solvente, banana (4,12)
Es 9	2-Methyl-1-butyl acetate	880	881	nd	0.43 ± 0.36	nd	nd	Fruit, overripe fruit sweet banana juicy fruit fruity (16,17)
Es 17	Ethyl hexanoate	1000	1003	$21.79^{a} \pm 15.54$	$26.66^{a} \pm 4.72$	$18.70^{a} \pm 0.82$	$14.8^{a} \pm 1.14$	Pineapple, waxy, sweet, fruity, green apple, banana, strawberry, floral, candy (1,2,4,5,6,8)

Table 2 (continued)

Code	Volatile compound*			"Peak area count" ± SD (× 10^6)				Odor description
		LRI (lit)	LRI (cal)	MOSCATEL	BRUT ROSÉ	BRUT	DEMI-SEC	
Es 18	Hexyl acetate	1011	1017	0.82 ± 0.01	nd	nd	nd	Fruity, banana, sweet, green, apple, pear, ripe fruit [1,5,6,12,16]
Es 24	Ethyl furoate	1047	1055	nd	nd	nd	0.07 ± 0.04	
Es 30	Ethyl 2,4-hexadienoate	1097	1100	20.89 ± 15.51	nd	nd	nd	Warm, fruity, anise, licorice, ether [16,17]
Es 35	Ethyl benzoate	1171	1168	$0.20^{a} \pm 0.19$	$0.33^{a} \pm 0.21$	$0.14^{a} \pm 0.00$	nd	Dry, musty, sweet, wintergreen, fruity [13, 16,17]
Es 38	Diethyl succinate	1182	1182	$1.77^{b} \pm 1.17$	$20.96^{a} \pm 0.74$	$10.18^{a,b} \pm 0.20$	$16.38^{a,b} \pm 5.02$	Mild, cooked, apple, ylang, fruity, over-ripe melon, lavender, sugary, floral, fermented, lactic [1,3,5.12, 16,17]
Es 41	Ethyl octanoate	1196	1200	$171.32^{a} \pm 116.24$	$267.08^{a} \pm 3.42$	$144.77^{a} \pm 19.77$	$114.08^{a} \pm 6.59$	Wine, waxy, apricot, fruity, fresh, pineap-ple, banana, apple, strawberry, pears, floral, sweet [1,2,3,4,5, 16]
Es 43	Isopropyl octanoate	1218	1217	nd	nd	17.44 ± 13.03	nd	Fruity, banana, coconut, cognac [16,17]
Es 46	Ethyl 2-phenylacetate	1246	1244	$2.59^{a} \pm 1.58$	$2.40^{a} \pm 0.57$	nd	nd	Sweet, floral, honey, bal-samic, cocoa, rose, fruity [1,2,3, 16,17]
Es 47	Isoamyl hexanoate	1252	1251	$0.22^{a,b} \pm 0.00$	$0.54^{a} \pm 0.08$	$0.15^{b} \pm 0.01$	$0.36^{a,b} \pm 0.17$	Fruity, banana, apple, pineapple, green [3,16,17]
Es 49	β-Phenethyl acetate	1258	1256	$0.38^{b} \pm 0.15$	$3.47^{a} \pm 0.02$	$2.82^{a,b} \pm 0.03$	$1.57^{a,b} \pm 0.33$	Floral, rose, sweet, honey, fruity tropical [3,16,17]
Es 52	Ethyl glutarate	1283	1283	nd	nd	0.06 ± 0.00	nd	
Es 53	Bornyl acetate	1285	1286	0.02 ± 0.00	nd	nd	nd	Woody, pine, herbal, cedar, spice [16,17]
Es 54	Isobornyl acetate	1286	1289	$1.02^{a} \pm 0.70$	$3.73^{a} \pm 2.82$	nd	nd	Balsam, camphor, herbal, woody, sweet [16,17]
Es 55	Propyl octanoate	1290	1291	$0.30^{a,b} \pm 0.24$	$0.92^{a} \pm 0.53$	$0.07^{a,b} \pm 0.02$	nd	Coconut, caco, gin, fruit [14, 16,17]
Es 56	Ethyl nonanoate	1296	1296	$0.16^{a,b} \pm 0.12$	$0.90^{a} \pm 0.57$	$0.18^{a,b} \pm 0.12$	nd	Floral, fruity, rose, waxy, rum, wine natural tropical [1,3, 16,17]
Es 57	Isomenthol acetate	1305	1306	$0.08^{a} \pm 0.06$	$0.12^{a} \pm 0.01$	nd	nd	
Es 60	Methyl decanoate	1325	1325	$0.11^{a,b} \pm 0.08$	$0.21^{a} \pm 0.00$	$0.07^{a,b} \pm 0.02$	nd	Oily, wine, fruity, floral [3, 16,17]
Es 61	Isobutyl octanoate	1348	1349	$0.78^{a} \pm 0.69$	$0.47^{a} \pm 0.01$	$0.11^{a} \pm 0.01$	nd	Fruity, green, oily, floral, white flowers, anise, herb [3,16,17]
Es 64	Propyl 2,4-hexadienecarboxylate	1366	1363	0.63 ± 0.44	nd	nd	nd	Sweet, fruity [16,17]
Es 67	Ethyl 9-decenoate	1387	1390	$9.50^{a} \pm 6.86$	$38.24^{a} \pm 0.90$	$8.06^{a} \pm 1.12$	$9.43^{a} \pm 0.96$	Fruity, fatty [1,16,17]
Es 68	Ethyl decanoate	1396	1401	$71.51^{a} \pm 42.64$	$157.40^{a} \pm 6.39$	$92.50^{a} \pm 0.32$	$72.32^{a} \pm 8.47$	Sweet, waxy, apple, oily, floral, grape, fruity [1,2,6, 16,17]
Es 70	Ethyl isopentyl succinate	1432	1431	nd	$1.13^{a} \pm 0.06$	$0.77^{a} \pm 0.11$	$1.15^{a} \pm 0.13$	
Es 72	3-Methylbutyl octanoate	1446	1448	$0.41^{b} \pm 0.21$	$2.35^{a} \pm 0.20$	$0.78^{a,b} \pm 0.24$	$0.71^{a,b} \pm 0.33$	Sweet, oily, fruity, green, soapy, pineap-ple coconut [6,16,17)]
Es 73	2-Methylbutyl octanoate	1449	1451	$0.05^{a,b} \pm 0.05$	$0.28^{a} \pm 0.00$	$0.10^{a,b} \pm 0.04$	nd	

Table 2 (continued)

Code	Volatile compound*	LRI (lit)	LRI (cal)	"Peak area count" ± SD (× 10^6) MOSCATEL	BRUT ROSÉ	BRUT	DEMI-SEC	Odor description
Es 82	Ethyl 3-hydroxytridecanoate	1539	1539	$0.10^{a} \pm 0.06$	$0.15^{a} \pm 0.00$	$0.17^{a} \pm 0.06$	nd	
Es 89	Ethyl vanillate	1579	1590	nd	0.12 ± 0.06	nd	nd	Phenolic, burnt, guaiacol, smoky, powdery metallic, creamy, vanilla [6, 16,17]
Es 90	Ethyl dodecanoate	1595	1596	$0.52^{a} \pm 0.27$	$0.80^{a} \pm 0.09$	$0.81^{a} \pm 0.03$	$0.28^{a} \pm 0.02$	Mango, waxy, soapy, clean, floral, fruity, sweet [1,3,12, 16,17]
Es 91	Geranyl iso-valerate	1606	1600	$0.16^{a,b} \pm 0.14$	nd	$0.20^{a,b} \pm 0.11$	$0.32^{a} \pm 0.03$	Sweet, fruity, green, oily, herbal, fruity Melon [16,17]
Es 95	Isoamyl decanoate	1646	1651	$0.06^{a,b} \pm 0.05$	$0.29^{a,b} \pm 0.08$	$1.67^{a} \pm 1.33$	nd	Waxy, banana, sweet, cognac, green, fruity [3,16,17]
Es 97	2-Ethylhexyl octanoate	1688	1694	$0.09^{a} \pm 0.07$	$4.58^{a} \pm 4.15$	$0.07^{a} \pm 0.04$	$0.05^{a} \pm 0.00$	
Es 99	Ethyl tetradecanoate	1794	1794	$0.35^{a} \pm 0.04$	$0.27^{a} \pm 0.05$	$0.54^{a} \pm 0.05$	$0.30^{a} \pm 0.04$	Sweet, waxy, violet, orris, floral, caramel, sweet fruit, butter, fatty odor [3,12,16,17]
Es 106	Ethyl pentadecanoate	1894	1894	nd	nd	0.53 ± 0.19	nd	Honey, sweet [16,17]
Es 109	Ethyl hexadecanoate	1993	1993	$0.27^{a} \pm 0.18$	$0.27^{a} \pm 0.01$	$0.71^{a} \pm 0.18$	$0.37^{a} \pm 0.13$	Mild, waxy, creamy, milky, balsam, fatty, fruity, rancid, sweet [1,12,16,17]
	Total Esters			$306.70^{a,b}$	535.66^{a}	$304.51^{a,b}$	234.56^{b}	
	Furans (6)							
Fu 10	Methoxy phenyl oxime	E	947	19.04 ± 18.94	nd	nd	nd	
Fu 13	2,4-Dihydroxy-2,5-dimethyl-3(2H)-furanone	989	992	$0.44^{a} \pm 0.38$	$0.75^{a} \pm 0.33$	$0.11^{a} \pm 0.01$	$0.82^{a} \pm 0.23$	Fruity, green, earthy, beany, vegetable metallic [16,17]
Fu 15	2-Pentyl furan	993	999	0.03 ± 0.02	nd	nd	nd	Sweet, citrus, herbal, green, celery, spicy minty, woody [16,17]
Fu 22	Tetrahydro-2,2-dimethyl-5-(1-methyl-1-propenyl) furan	1047	1048	0.25 ± 0.20	nd	nd	nd	
Fu 29	2-Furyl hydroxymethyl ketone	1087	1087	$0.53^{a,b} \pm 0.22$	$1.35^{a} \pm 0.41$	$0.19^{b} \pm 0.03$	$0.51^{a,b} \pm 0.06$	Fatty, buttery, musty, waxy, caramellic, almond [11,16,17]
Fu 45	5-Hydroxymethylfurfural	1233	1235	$2.51^{a,b} \pm 1.82$	$1.11^{b} \pm 0.47$	$5.71^{a,b} \pm 4.03$	$17.38^{a} \pm 5.57$	
	Total furans			22.81^{a}	3.21^{b}	$6.01^{a,b}$	18.71^{a}	
	Hydrocarbons (4)							
Hy 1	1-Methoxy-2-ethylbutane	778	‹ 800	$0.04^{a,b} \pm 0.03$	$1.16^{a} \pm 0.06$	nd	$0.05^{a,b} \pm 0.02$	
Hy 83	2,6,10-Trimethyltetradecane	1541	1543	$0.01^{a,b} \pm 0.01$	$0.04^{a,b} \pm 0.01$	$0.24^{a} \pm 0.02$	nd	
Hy 78	3-Butyl-1,2,4-cyclopentanetrione	1486	1485	$0.15^{a,b} \pm 0.11$	$0.35^{a} \pm 0.06$	nd	nd	
Hy 79	Pentadecane	1500	1496	nd	nd	$0.12^{a} \pm 0.11$	$0.24^{a} \pm 0.21$	Waxy [17]
	Total hydrocarbons			0.20^{b}	1.55^{a}	$0.37^{a,b}$	0.29^{b}	
	Ketones (4)							
Ke 65	3-Dodecanone	1380	1384	3.75 ± 2.65	nd	nd	nd	Fatty, soapy waxy fruity [17]
Ke 87	3-Tetradecanone	1573	1585	$0.52^{a} \pm 0.35$	$0.09^{a} \pm 0.03$	$0.18^{a} \pm 0.00$	$0.06^{a} \pm 0.00$	
Ke 101	2-Hexadecan-2-one	1806	1806	0.51 ± 0.31	nd	nd	nd	Fruity [16,17]

Table 2 (continued)

Code	Volatile compound*			“Peak area count” ± SD (× 10^6)				Odor description
		LRI (lit)	LRI (cal)	MOSCATEL	BRUT ROSÉ	BRUT	DEMI-SEC	
Ke 102	2-Hydroxycyclo-pentadecanone	1846	1840	0.08 ± 0.05	nd	nd	nd	
	Total ketones			4.87^{a}	0.09^{b}	$0.18^{a,b}$	0.06^{b}	
	Phenol (2)							
Ph 59	*p*-Vinylguaiacol	1307	1312	$0.05^{a,b} \pm 0.03$	nd	nd	$0.49^{a} \pm 0.20$	Dry, woody, fresh, amber, cedar, peanut, smoky, spicy, smoked meat [8,16,17]
Ph 80	2,4-Di-tert-butylphenol	1515	1515	nd	0.31 ± 0.20	nd	nd	Phenolic [16,17]
	Total phenol			$0.05^{a,b}$	0.31^{a}	nd	0.49^{a}	
	Pyran (2)							
Pr12	2,2,6-Trimethyl-6-vinyltetrahydropyran	972	972	0.44 ± 0.42	nd	nd	nd	Fresh, camphor, herbal, rosemary [16,17]
Pr32	Hydroxydihydromaltol	1151	1149	$0.66^{b} \pm 0.28$	$3.58^{a,b} \pm 2.32$	$2.05^{a,b} \pm 0.16$	$6.33^{a} \pm 0.26$	
	Total pyran			1.10^{b}	$3.58^{a,b}$	$2.05^{a,b}$	6.33^{a}	
	Pyrazine (1)							
Pz 28	6-Methyl-2-pyrazinylmethanol	1084	1084	nd	$0.74^{a} \pm 0.22$	$0.18^{a,b} \pm 0.04$	$0.24^{a,b} \pm 0.04$	
	Total pyrazine			nd	0.74^{a}	$0.18^{a,b}$	$0.24^{a,b}$	
	Terpenes (16)							
Te 19	*p*-Cymene	1025	1022	0.07 ± 0.02	nd	nd	nd	Fresh, citrus, terpene, woody, spice [16,17]
Te 20	Limonene	1030	1026	$0.28^{a} \pm 0.23$	$0.18^{a} \pm 0.01$	nd	nd	Orange, fresh, sweet, flowery, green, citrus [8]
Te 21	1,8-Cineole	1033	1030	nd	0.03 ± 0.01	nd	nd	Eucalyptus, herbal, camphor medicinal [16]
Te 23	(*Z*)-β-Ocimene	1049	1050	0.14 ± 0.00	nd	nd	nd	Sweet, herbal [16,17]
Te 25	γ-Terpinene	1060	1059	$0.08^{a,b} \pm 0.02$	$0.37^{a} \pm 0.12$	nd	$0.08^{a,b} \pm 0.02$	Oily, woody, terpene, lemon/lime tropical herbal [16,17]
Te 26	(*E*)-2-(Tetrahydro-5-methyl-5-vinylfuran-2-yl)-propan-2-o((*E*)-Linalool oxide))	1074	1073	0.03 ± 0.00	nd	nd	nd	Floral, herbal, earthy, green [16,17]
Te 33	Nerol oxide	1153	1154	$0.43^{a} \pm 0.33$	$2.33^{a} \pm 1.81$	nd	nd	Green, weedy, cortex, herbal, diphenyl oxide, narcissus, celery, flower, fragrant [16,17,18]
Te 34	(*E*)-Ocimenol	1169	1167	0.05 ± 0.04	nd	nd	nd	Fresh, citrus, lemon lime, cologne, sweet mace [16,17]
Te 39	*p*-Cymen-8-ol	1186	1185	$0.71^{a,b} \pm 0.63$	nd	nd	$5.02^{a} \pm 2.88$	Sweet, fruity, cherry, coumarin, floral balsamic, camphor [16,17,19]
Te 40	α-Terpineol	1192	1192	$2.78^{a,b} \pm 2.32$	$1.05^{a,b} \pm 0.42$	nd	$5.02^{a} \pm 2.14$	Oil, anise floral, pine like, lilac, citrus, woody, floral [2,13,15]
Te 44	Nerol	1228	1228	0.06 ± 0.06	nd	nd	nd	Sweet, natural neroli, citrus, magnolia, rose, lime, flower [1,2, 16,17]
Te 48	Carvone	1255	1255	0.47 ± 0.04	nd	nd	nd	Minty, spearmint, herbal [16,17]
Te 50	Geranial	1268	1267	$0.32^{a} \pm 0.25$	$0.55^{a} \pm 0.05$	nd	nd	Citrus, lemon [16,17]
Te 74	α-Humulene	1453	1454	$0.10^{a,b} \pm 0.06$	$0.17^{a} \pm 0.01$	$0.07^{a,b} \pm 0.01$	nd	Woody [16,17]

Table 2 (continued)

Code	Volatile compound*	LRI (lit)	LRI (cal)	"Peak area count" ± SD ($\times 10^6$)				Odor description
				MOSCATEL	BRUT ROSÉ	BRUT	DEMI-SEC	
Te 86	(*Z*)-α-Bisabolene epoxide	1586	1585	0.52 ± 0.35	nd	nd	nd	
Te 88	Germacrene B	1591	1588	0.03 ± 0.02	nd	nd	nd	Woody, earthy, spicy [(17)]
	Total terpenes			$6.07^{a,b}$	$4.68^{a,b}$	0.07^{b}	10.12^{a}	

*The compounds are listed by chemical class; LRI lit.: linear retention index of literature; LRI cal.: linear retention index calculated; SD: standard deviation; nd: not identified. E: identification by the mass spectrum, the others were identified by the mass spectrum and the literature retention index. Results with different letters for the same class of compounds differ significantly by Kruskall-Wallis test and Dunn's post hoc multiple comparison test ($p < 0.05$)

[1]Welke, Zanus, Lazarotto, and Zini (2014)

[2]Caliari et al. (2015)

[3]Ubeda et al. (2019)

[4]Ferreira et al. (2002)

[5]Torrens et al. (2010)

[6]Zhao; Gao; Qian; Li (2017)

[7]Korenika et al. (2020)

[8]Komes (2006)

[9]Tao, Y.-S., & Li, H. (2009)

[10]Shinohara (1985)

[11]Sacks et al. (2012)

[12]Martínez-García et al. (2017)

[13]Fan, Xu, Jiang, and Li (2010)

[14]Fan and Qian (2005)

[15]Vararu; Moreno-García; Zamfir; Cotea; Moreno (2016)

[16]Pherobase (2020)

[17]*The good scents company* (2020)

[18]Wang et al. (2017)

[19]Bellincontro et al. (2016)

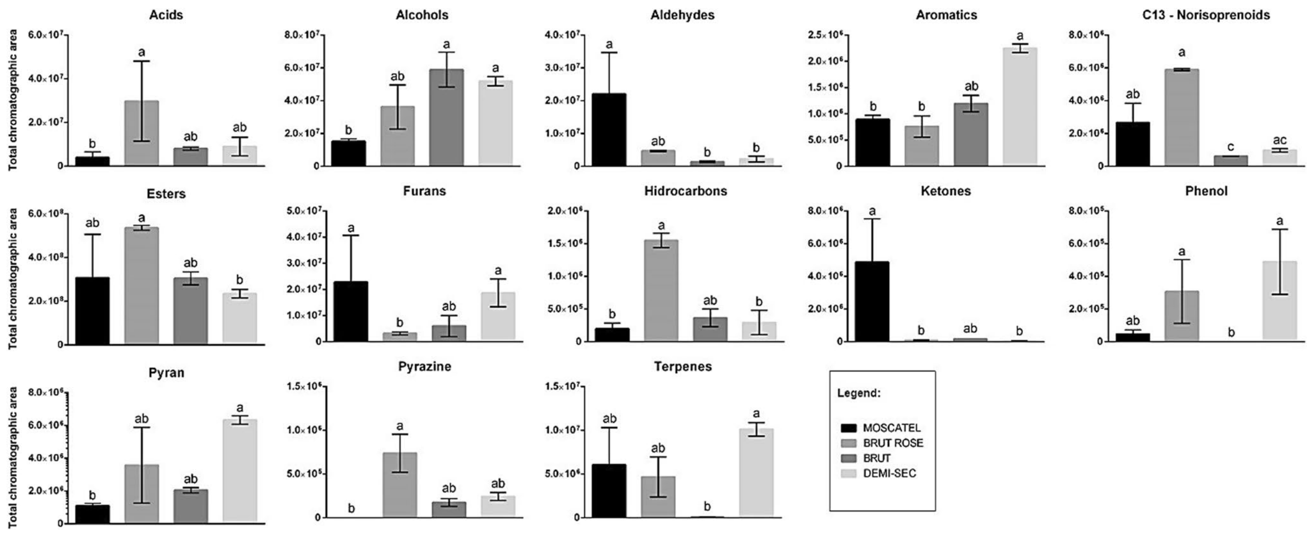

Fig. 1 Graph of the total area values of each chemical group according to the four commercial sparkling wine groups. Results with different letters for the same class of compounds differ significantly by the Kruskal–Wallis test and Dunn's post hoc multiple comparison test ($p < 0.05$)

67 for brut rosé, 61 for brut, and 52 for demi-sec. Some of these were the major in the four sparkling wines analyzed: ethyl octanoate, ethyl decanoate, ethyl hexanoate, ethyl 9-decenoate, 3-methyl-1-butanol. These five compounds together represented 72.69%, 82.2%, 79.07%, and 71.13% of the total chromatography area of the identified compounds in moscatel, brut rosé, brut and demi-sec, respectively.

The main compounds among the chemical groups were as follows: esters (37), followed by terpenes (16), alcohols (14), aromatics (8), acids (8), and furans (6). The comparative total area values of each volatile compound chemical group of the sparkling wines are shown in the Fig. 1. It can be seen that significant differences were presented within a class between at least two categories of sparkling wines according to the Kruskal–Wallis test and the Dunn's post hoc multiple comparison test ($p < 0.05$).

The esters class was the most abundant among the 13 groups of compounds, representing larger areas in the brut rosé category (85.43%), followed by brut (79.39%), moscatel (79.34%), and demi-sec (69.56%), and presenting significant differences ($p < 0.05$) between them (as can be seen in the Fig. 1). Ethyl octanoate with 44%, 43%, 38%, and 34% and ethyl decanoate with 18%, 25%, 24%, and 21% of the total area of the moscatel, brut rosé, brut, and demi-sec categories, respectively, are highlighted in this group. These compounds were also identified in other sparkling wine categories (Pérez-Magariño et al. 2015; Ubeda et al. 2019; Voce et al. 2019), however, in different proportions. It is also worth mentioning the ethyl hexanoate as a fruity (pineapple and pear) and floral aromatic descriptor, and ethyl decanoate with a sweet, fruity, fatty, and pleasant aroma (Jiang et al. 2013).

Other esters detected with larger areas in the brut, brut rosé and demi-sec samples were ethyl hexanoate, diethyl succinate, and ethyl 9-decenoate (Table 2). When analyzing experimental sparkling wines from the Chenin Blanc cultivar, Nascimento et al. (2018) suggested that diethyl succinate is one of the most relevant esters for the volatile profile of sparkling wines produced in the São Francisco Valley. These compounds were also identified in moscatel sparkling wines produced in southern Brazil (Nicolli et al. 2015). The presence of Diethyl succinate in sparkling wines from the Ribolla Gialla variety of Northeast Italy, suggests that the sparkling wine was produced with a more or less prolonged period of aging, and/or a refermentation inside the bottle, as diethyl succinate is considered an aging ester (Voce et al. 2019).

When studying Italian sparkling wines of the Maresco cultivar, Tufariello et al. (2019) detected ethyl octanoate, isoamyl acetate, ethyl hexanoate, and ethyl decanoate esters. All of these compounds were identified in the four groups of sparkling wines in this study. Esters are mainly derived from alcoholic fermentation (Étievant, 1991), and contribute to the sensory attributes of wines, especially concerning their fruity aroma. Their production depends on factors such as the yeast used, temperature, and aeration during fermentation and sugar content in the must (Jiang et al. 2013).

A total of 16 terpenes were identified, which fifteen of them were present in the moscatel sparkling wine and only one (α-humulene) was present in the brut sparkling wine. Interestingly, the demi-sec sparkling wine contained only three terpenes (γ-terpinene, p-cymen-8-ol, and α-terpineol) and represented the largest area (3% of the total area of compounds identified), significantly differing ($p < 0.05$) from the brut sparkling wine (Fig. 1). All of these number particularities and terpene area, mainly in moscatel and demi-sec sparkling wines, may be related to the different cultivars and winemaking processes used in each category of sparkling wine. The terpenes belong to the secondary constituents of the plant whose biosynthesis starts with acetylcoA (Jiang et al. 2013); consequently, the concentration of sugars and the technological process used in the production of sparkling wines, one conducted by the *asti* method (moscatel—Italia Muscat) with only one fermentation, and the other by the *charmat* method (brut-Sauvignon Blanc, Chenin Blanc, and Verdejo) with two fermentations, justify this variation between the terpenes. A study performed by Ubeda et al. (2019) verifying the evolution of different chemical families of volatile compounds during the production of sparkling wines from the País cultivar showed that the second fermentation slightly reduced the terpene concentration in sparkling wines of Chile, elaborated by the traditional method (champegnoise). Furthermore, according to a study by Nascimento et al. (2018), terpenes are not frequently identified in wines from the Chenin Blanc and Syrah cultivars, only finding the Carvone terpene in sparkling wines produced from Chenin Blanc. However, p-cymen-8-ol, α-terpineol, and nerol oxide showed the largest relative areas in this study that can provide aromatic notes of balsamic, anise floral, pine like, lilac, citrus, woody, floral, fragrant, etc. (Wang et al. 2017; Bellincontro et al. 2016; Caliari et al. 2015; Ubeda et al. 2019; Vararu et al. 2016).

A total of 14 compounds were identified in the alcohol class, representing a larger percentage area for the demi-sec sparkling wine category (15.40%), followed by brut (15.37%), brut rosé (5.77%), and moscatel (3.94%), and differing significantly (as shown in Fig. 1). Superior alcohols are composed of volatile molecules with more than two carbon atoms, considered to have a strong aromatic effect on wines, and whose final concentration in this beverage mainly depends on the yeast metabolism, among other factors such as the type of wine and chemical composition (Ruiz et al. 2019). Most alcohols can attribute strong aromas to wines (like the herbaceous), which in high concentration can mask the fragrance of the drink and in low concentrations (up to 0.3 g/L) help in aromatic complexity (Jackson, 2020). 3-Methyl-1-butanol alcohols and phenylethyl alcohol were the major compounds in sparkling wines in this study. Their contributions to the wine aroma range from floral, rose, honey, flower, and woody (Phenylethyl alcohol) (Caliari et al, 2015; Torrens et al 2010) to fruity, banana, alcohol, whiskey, fusel, and oil solvent (3-Methyl-1-butanol) (Welke et al. 2014). Tyrosol was only identified in brut rosé and demi-sec sparkling wines, being associated with the honey aroma in wines (Lambrechts and Pretorius, 2000). Among the identified alcohols (14), five were detected in only one type of sparkling wine (2-Methyl-1-butanol, 3-methyl-2-hexanol, 3-methyl-4-heptanol, 1-nonanol and 2-heptadecanol), thus suggesting the influence of the varietal and the winemaking process on the aromatic quality of the final product.

Of the seven compounds identified in the aromatic class (aromatic hydrocarbons), all were detected for the first time in sparkling wines from VSF and do not have flavor descriptors in the literature (Jiang et al. 2013; Pherobase, 2020). This is because normally these do not directly influence the sensory characteristics of the wine, due to their solubility characteristics (Jackson, 2008). From these, only 5-phenylundecane was identified in a survey of wine produced with the Cabernet Sauvignon grape and parts of the stalk of this grape (Nan et al. 2019). The particularities of these sparkling wines may be due to the combination of varietal characteristics and peculiarities of the *terroir* (Marcon et al. 2021). As shown in Fig. 1, the total area of the aromatic class in demi-sec sparkling wine differed from moscatel and brut rosé sparkling wines, and the class of C13-norisoprenoid compounds in brut rosé sparkling wine differed from brut sparkling wine, with both classes representing $< 1\%$ of the total area. The distinction of compounds is possibly associated with the different cultivars used for winemaking these sparkling wines, for which the brut rosé has a fermentative maceration protocol (contact with the Grenache grape skins). Moreover, 1,2-dihydro-1,5,8-trimethyl-naphthalene showed the largest chromatographic areas among the class C13-norisoprenoid compounds identified, but it is worth noting the presence of TDN (1,1,6-trimethyl-1,2-dihydronaphthalene) which was only detected in brut sparkling wine, and the presence of which has also been identified in sparkling wines from Chile (Ubeda et al. 2019) and Spain (Muñoz-Redondo et al. 2020). TDN has been classified as an important aging marker with aromatic notes of burned, tabac, and herb (Ubeda et al. 2019).

Many of the volatile acids in wines are generally saturated linear chain lengths ranging from 2 to 18 carbon atoms; another small group of branched-chain organic acids includes 3-methyl butanoic acid, 2-methyl butanoic acid, and 2-methyl propanoic acid (Ruiz et al. 2019). The total acid area differed significantly between brut rosé and moscatel sparkling wine, which represented 4.74% and 1.02%, respectively. Decanoic acid and hexadecanoic acid were identified in the four types of sparkling wines, and presented the largest areas among the eight acids found in this study. These two acids were also identified in commercial sparkling wines of the Ribolla Gialla variety produced in Northeastern Italy (Voce et al. 2019). Organic acids have been described with aromatic notes of fruit, cheese, fat and rancidity, while long-chain acids have a reduced effect on the aroma of wines, and C6-C10 chain acids have a positive impact on the quality of the overall aroma of wines (Fernandes et al. 2018).

The total area of the furan class in the moscatel (5.90%) and demi-sec (5.55%) sparkling wines differed significantly from brut rosé (0.51%) (Fig. 1). The 5-hydroxymethylfurfural compound is an intermediate product of the Maillard reaction and caramelization process (Gong et al. 2020), and was identified in all sparkling wines in this study, especially in demi-sec (representing 5.15% of the total area of the identified compounds), with caramel being one of its aromatic descriptors (Table 2). Since the winemaking process of these sparkling wines does not apply heating, the formation of this compound may have been influenced by the climatic conditions with high temperatures in the São Francisco Valley region. According to Lampír and Pavlousek (2013), each cultivated grape in a specific *terroir* reflects the location in its chemical composition. Storage time and temperature are also possible markers of the formation of this compound as described in a study by Serra-Cayuela et al. (2014) in commercial sparkling wines.

A total of 6 aldehydes were identified, most of which are produced during fermentation, processing or extracted from oak during the aging stage. When compared to ketones (which had four identified compounds in this study), the aldehydes are carbonyl compounds, which are differentiated by the terminal location of the functional carbonyl group (–C = O), while ketones are compounds related to the carbonyl group located in an internal carbon (Jackson, 2008). The total area of both classes (Fig. 1) in the moscatel sparkling wine differed significantly from demi-sec. The following aldehydes may be highlighted: decanal, found in moscatel and brut rosé sparkling wines, with a grassy, orange skin-like aroma (Jiang et al. 2013); and dodecanal, found in the four sparkling wines, which can confer a soapy, waxy, aldehydic, citrus, green, and floral aroma (Welke et al. 2014). Three of the identified ketones were only found in moscatel sparkling wine, with the 3-Dodecanone compound with the largest chromatographic area (representing 1% of the total area), and whose aromatic descriptors can be fatty, soapy, waxy, and fruity (The good scents company 2020).

Only four high molecular weight hydrocarbons were identified in present study, observing variation among the sparkling wines studied, as shown in Table 2 and Fig. 1. The hydrocarbons are generally associated to grape cell debris and lost before or during clarification or maceration (Jackson, 2008). Thus, they do not directly influence the sensory characteristics of the wine; however, the hydrocarbon degradation products may produce important volatile compounds, such as β-damascenone, and 1,1,6-trimethyl-1,2-dihydronaphthalene (TDN) (Jackson, 2008).

Other compound classes (phenol, pyran, pyrazine) were also identified in smaller numbers and with less representation in the total area. Phenol, 2,4-di-tert-butylphenol was detected in brut rosé sparkling wine, compound also identified in other studies with sparkling wines (Nicolli et al. 2015; Soares et al. 2015) and p-vinylguaiacol in moscatel and demi-sec sparkling wine. In addition, pyrazine 6-methyl-2-pyrazinylmethanol was observed in all sparkling wines, except for moscatel. However, the compound pyrane 2,2,6-trimethyl-6-vinyltetrahydropyran was only found in this wine and in the study of Nicolli et al. (2015). This volatile variability among the analyzed samples was also observed by Arcanjo et al. (2018), which suggests the influence of factors such as the grape harvest time and the technological processes used in the final aromatic quality of the wine.

Throughout the wine-making process, saccharomycetes may grow and produce a diverse range of metabolic end-products, which may have desirable or undesirable effects (Wu et al. 2021). The compounds best known for imparting undesirable aromas to wines during the winemaking process were not identified in the studied sparkling wines. Some off-flavors occasionally present during aging in oak wood were also not detected.

Principal Component Analysis of Volatile Compounds in Sparkling Wines from the São Francisco Valley

The volatile profile of sparkling wines (Table 2) was analyzed by principal component analysis (PCA) (Fig. 2). The first principal component (PC1) explained 33.34% of the total variation among the samples, which PC2 explained 29.25% of total variability. PC1*x*PC2 explained 62.59% of the variability among the volatile profiles. Sparkling wines with similar volatile profiles were positioned in the quadrants in nearby regions. PC1 separated the brut rosé (in the positive side of PC1) from the brut and demi-sec (in the negative side of PC1) sparkling wines. The identified volatiles which best characterized each group of wines are represented by vectors, and those with significant factor loads and ≥ 0.8 are considered for discussion. The vectors which are very close are also indicative of highly correlated variables and the compounds with higher loading values contribute most significantly to the explanatory meaning of the factors. Table 3 shows the loadings of each compound in each one of the selected factors, as well as the eigenvalue and the cumulative variance of each factor.

There are 40 associated volatile compounds in the positive side of PC1 and negative PC2 that were associated to the brut rosé sparkling wine. This indicates that these sparkling wines had higher concentrations of volatile compounds associated with PC1 (Factor 1) such as hexadecanoic acid (Ac 108), (E)-2-dodecenal (Ald 75), 1-octanol (Al 27), 1,2-dihydro-1,5,8-trimethyl-naphthalene (No 63), ethyl hexanoate (Es 35), ethyl octanoate (Es 41), ethyl 2-phenylacetate (Es 46), isobornyl acetate (Es 54), propyl octanoate (Es 55), isomenthol acetate (Es 57), methyl decanoate (Es 60), ethyl 9-decenoate (Es 67), 2-methylbutyl octanoate (Es 73), 1-methoxy-2-ethylbutane (Hy 1), 3-butyl-1,2,4-cyclopentanetrione (Hy 78), γ-terpinene (Te 25), geranial (Te 50), and

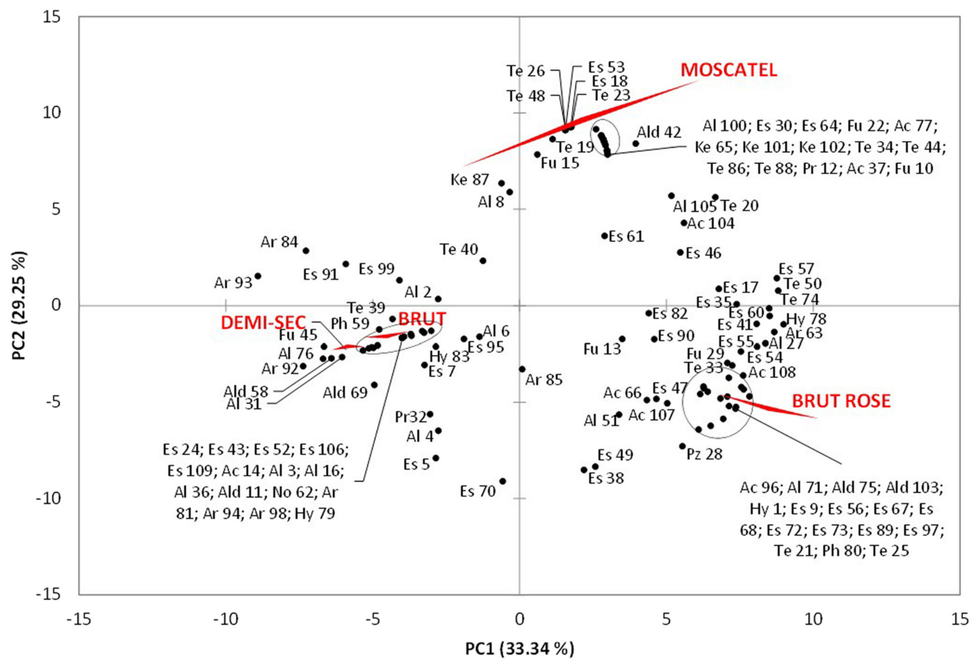

Fig. 2 Principal components analysis (PCA) of the volatile compounds* of sparkling wines from the São Francisco Valley.*Legend of the compound codes is in Table 2

α-humulene (Te 74). And also, to factor 2: diethyl succinate (Es 38), β-phenethyl acetate (Es 49).

Demi-sec and brut sparkling wines, both produced with the same cultivar, were the richest in volatile compounds are located in the negative side of PC1 and positive PC2, associated to 33 vectors. These sparkling wines were richer in volatile compounds associated with factor 1, characterized by the volatiles 6-phenyl-undecane (Ar 92), 5-phenyl-undecane (Ar 93), associated with factor 2: ethyl butanoate (Es 5), ethyl isopentyl succinate (Es 70), and associated with factor 3: 2,3-butanediol (Al 2), 1-hexanol (Al 6), 3-methyl-4-heptanol (Al 16), 1-nonanol (Al 36), benzaldehyde (Ald 11), ethyl glutarate (Es 52), 2,6,10-trimethyltetradecane (Hy 83), 1,1,6-trimethyl-1,2-dihydronaphthalene (No 62).

PC2 separated the moscatel sparkling wine (in the positive side) from the others, located in the negative side of PC2. The volatile compounds with the greatest contribution in the discrimination of the moscatel sparkling wine were octanoic acid (Ac 37), undecanoic acid (Ac 77), 2-heptadecanol (Al 100), decanal (Ald 42), hexyl acetate (Es 18), ethyl 2,4-hexadienoate (Es 30), bornyl acetate (Es 53), propyl 2,4-hexadienecarboxylate (Es 64), methoxy phenyl oxime (Fu 10), 2-pentyl furan (Fu 15), tetrahydro-2,2-dimethyl-5-(1-methyl-1-propenyl)-furan (Fu 22), 3-dodecanone (Ke 65), 2-hexadecan-2-one (Ke 101), 2-hydroxycyclopentadecanone (Ke 102), 2,2,6-trimethyl-6-vinyltetrahydropyran (Pr 12), p-cymene (TE 19), (*Z*)-β-ocimene (Te 23), (*E*)-linalool oxide (Te 26), (*E*)-ocimenol (Te 34), nerol (Te 44), carvone (Te 48), (*Z*)-α-bisabolene epoxide (Te 86), and germacrene B (Te 88). Additionally, one compound was associated with Factor 4, Isobutyl octanoate (Es 61). All of these compounds are negatively correlated to the compounds associated with demi-sec and brut sparkling wines. Previous studies highlight that Brazilian muscatel wines have a high concentration of the compounds like isoamyl acetate, hexyl acetate, limonene, rose oxide, linalool, and citronellol (Marcon et al. 2021).

Thus, PCA discriminated sparkling wines in 3 groups, and the cultivar used (Itália Muscat in moscatel sparkling wines; Grenache in brut rosé sparkling wines and blend of Chenin Blanc, Sauvignon Blanc, and Verdejo, around 33% of each one in demi-sec and brut sparkling wines) was a marker in the volatile composition of the beverage, influencing the quality and differentiating the wines produced in the same winery, from different technological processes. Moscatel was elaborated by the *asti* method with just one fermentation and the 3 other sparkling wines were elaborated by the *charmat* method with two fermentations. In this context, only brut and demi-sec sparkling wines, made with the same cultivars and with the same vinification method, were allocated in the same quadrant.

Hierarchical Cluster Analysis and Heatmap Applied to the Profile of Volatile Sparkling Wines in the São Francisco Valley

A hierarchical cluster and heatmap analysis (Fig. 3) were performed considering all the identified volatile compounds (Table 2) in order to analyze the expressive volatile composition of each sparkling wine. The color of the obtained boxes and its intensity is used to represent changes on each compound concentration. The hierarchical cluster analysis

Table 3 Factor loadings between volatile compounds and first four principal components of the sparkling wines

Compounds*	Factor 1	Factor 2	Factor 3	Factor 4	Compounds*	Factor 1	Factor 2	Factor 3	Factor 4
Ac 37	0.3198	0.8650		−0.3753	Es 54	0.8288			
Ac 77	0.3064	0.9067		−0.2789	Es 55	0.8893			
Ac 108	0.8372	−0.3729		−0.2639	Es 57	0.9638			
Al 2	−0.3040		−0.8657	−0.3687	Es 60	0.9369			
Al 6			−0.8400	−0.4286	Es 61	0.3185	0.3708	0.0872	0.8416
Al 16	−0.4040		0.8300		Es 64	0.3140	0.8857		−0.3308
Al 27	0.9207		−0.2967		Es 67	0.8611	−0.4839		
Al 36	−0.4397		0.8500		Es 70		−0.9385	−0.2485	
Al 100	0.2862	0.9419			Es 73	0.8393	−0.4465	0.2657	
Ald 11	−0.4397		0.8500		Fu 10	0.3303	0.8069		−0.4771
Ald 42	0.4349	0.8645			Fu 15		0.8066		0.5703
Ald 75	0.8106	−0.5414			Fu 22	0.3220	0.8556		−0.3937
No 62	−0.4399		0.8496		Hy 1	0.8089	−0.5495		
No 63	0.9542				Hy 78	0.9886			
Ar 92	−0.8103	−0.3231	−0.3604	−0.2885	Hy 83	−0.3125		0.8822	−0.2554
Ar 93	−0.9793				Te 19		0.8889		0.4257
Ke 65	0.3145	0.8842		−0.3342	Te 23		0.9537		
Ke 101	0.3059	0.9078		−0.2758	Te 25	0.8300	−0.4345		
Ke 102	0.3095	0.8988		−0.2995	Te 26		0.9378		0.2869
Es 5	−0.3125	−0.8143	−0.2863	−0.2603	Te 34	0.3270	0.8297		−0.4404
Es 18		0.9514		0.2267	Te 44	0.3276	0.8256		−0.4472
Es 30	0.3172	0.8747		−0.3552	Te 48		0.9383		0.2852
Es 35	0.8134			−0.3204	Te 50	0.9682	0.0792		−0.1145
Es 38	0.2414	−0.8771	−0.2592	−0.1205	Te 74	0.9347		0.3175	
Es 41	0.8881			−0.4067	Te 86	0.3118	0.8923		−0.3154
Es 49	0.2837	−0.8593	0.3627		Te 88	0.3171	0.8751		−0.3543
Es 52	−0.4382		0.8515		Pr 12	0.3292	0.8151		−0.4643
Es 53		0.9373		0.2889					

*Legend of the compound codes is in Table 2. Loadings lower than absolute values of 0.250 are not shown. Values in bold indicate the highest weight (≥ 0.8) of each compound in each factor

based on the volatile markers grouped the sparkling wines into two clusters with weak association between them. The first one is formed by the blends of sparkling wines: brut and demi-sec; and the second one is formed by the monovarietal sparkling wines: moscatel and brut rosé. These results suggest a strong association with the amount of sugars (brut and demi-sec), type and quantity of grape varieties/blend used during the technological winemaking practices.

The heat map (Fig. 3) shows the volatile compounds for brut sparkling wines of which 50% belong to the ester class and 22.22% to the alcohols class. Among these are the volatile compounds: 2,6,10-trimethyltetradecane, ethyl tetradecanoate, and phenylethyl alcohol represented superior chromatographic areas in relation to the other sparkling wines (Table 2). It is worth mentioning that the compounds which were exclusively identified in the brut sparkling wine: hexanoic acid, 1-nonanol, benzaldehyde, 1,1,6-trimethyl-1,2-dihydronaphthalene, ethyl glutarate, ethyl pentadecanoate, and isopropyl octanoate, including 2-phenyl-undecane and 3-methyl-4-heptanol, which stand out for being identified for the first time in sparkling wines. Of the most relevant compounds identified in demi-sec sparkling wine (Fig. 3) 25% belong to the aromatics class and 20.83% to the esters class. Among these, 2.3-butanediol and 5-hydroxymethylfurfural obtained superior chromatographic areas in relation to other sparkling wines (Table 2). The pentadecane, 6-phenyl-undecane, and undecanal compounds showed greater area in relation to brut and p-vinylguaiacol, p-cymen-8-ol, in relation to moscatel. in addition, the 4-phenyl-undecane, 5-phenyl-dodecane, 2-methyl-1-butanol, and ethyl furoate compounds were only identified in demi-sec sparkling wine, and 4-Phenyl-undecane was identified for the first time in sparkling wines. This composition is possibly associated to the *terroir* of the São Francisco Valley, considering that the synthesis and concentration of volatile compounds in the grape berry are influenced by factors such as temperatures

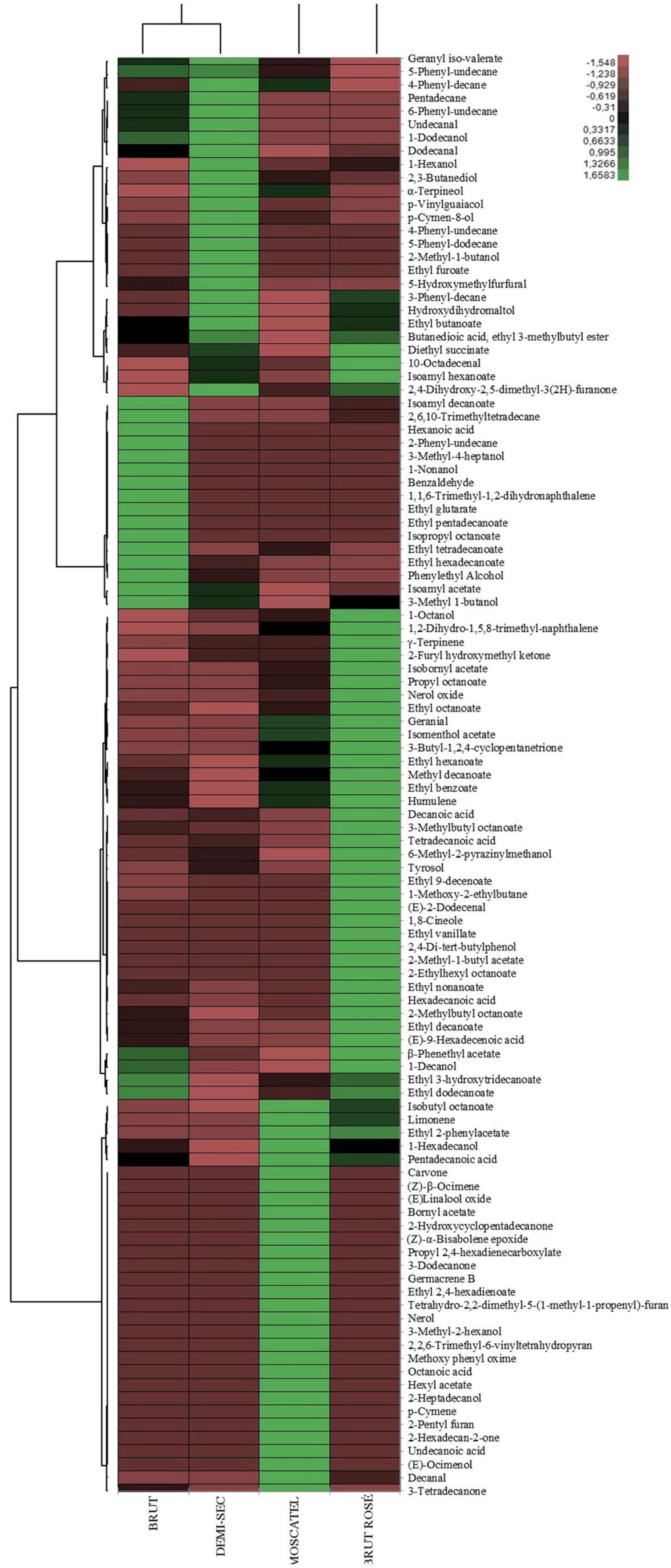

Fig. 3 Hierarchical cluster analysis and heatmap for each sparkling wine from the São Francisco Valley performed by Pearson's correlation coefficient

during grape maturation, light intensity/solar radiation, rainfall index, thermal amplitude and soil conditions. These variables possibly participated in regulating the biosynthesis of volatile compounds of the grape berry, thus determining the geographical characteristics of the wines (Jiang et al. 2013).

The volatile compounds identified as most relevant in moscatel sparkling wine (Fig. 3), 30% belong to the terpene class and 20% to the ester class. Of these compounds, the decanal obtained the highest chromatographic area among these compounds in relation to the brut rosé (Table 2). Exclusive compounds were also identified in moscatel sparkling wine, which were as follows: carvone, (*Z*)-β-ocimene, (*e*)-linalool oxide, bornyl acetate, 2-Hydroxycyclopentadecanone, (*Z*)-α-bisabolene epoxide, propyl 2,4-hexadienecarboxylate, 3-dodecanone, germacrene B, ethyl 2,4-hexadienoate, tetrahydro-2,2-dimethyl-5-(1-methyl-1-propenyl) furan, nerol, 3-methyl-2-hexanol, 2,2,6-trimethyl-6-vinyltetrahydropyran, methoxy phenyl oxime, octanoic acid, hexyl acetate, 2-heptadecanol, *p*-Cymene, 2-pentyl furan, 2-hexadecan-2-one, undecanoic acid, and (*E*)-ocimenol, of which they account for 50% of the total terpenes in this study. Terpenes in muscat wines attribute floral aromas and the esters attribute fruity and floral notes, constituting important characteristics and possibly being responsible for the varietal aroma of moscatel sparkling wine (Bordiga et al. 2013; Soares et al. 2015). Of the terpenes, Germacrene B has woody, earthy, and spicy notes as its aromatic descriptors ("The good scents company," 2020), being identified in Baga grapes (Coelho et al. 2006) and in Nero d'Avola, Frappato, Nerello Mascalese, and Cabernet Sauvignon grape stalk (Ruberto et al. 2008).

The most relevant volatile compounds to distinguish brut rosé 53.5% belong to the ester class. Among these compounds are the γ-terpinene, 3-methyl octanoate, tetradecanoic acid, 6-Methyl-2-pyrazinylmethanol, ethyl 9-decenoate, 1-methoxy-2-ethylbutane, hexadecanoic acid, 2-methylbutyl octanoate, ethyl decanoate, and β-phenethyl acetate obtained superior chromatographic areas in relation to other sparkling wines (Table 2). The following compounds also obtained superior areas: 3-butyl-1,2,4-cyclopentanetrione in relation to moscatel and tyrosol in relation to demi-sec. the compounds: (E)-2-dodecenal, 1,8-cineole, ethyl vanillate, 2,4-di-tert-butylphenol, and 2-methyl-1-butyl acetate were only identified in brut rosé. In a study of wines made with the same varietal used in brut rosé (Grenache), Arias et al. (2019) obtained high concentration (280.11 μg/L) of ethyl vanillate compound associated to the origin of the cultivar. The 1,8-cineole (eucalyptol) and 2,4-di-tert-butylphenol compounds were also identified in moscatel (Moscato Bianco and Moscato R2) sparkling wines produced in Rio Grande do Sul, Brazil (Soares et al. 2015). According to Capone et al. (2012), the proximity of vines to Eucalyptus trees may influence the concentration of 1.8-Cineole in wines, which could attribute mint aromatic notes (Pherobase, 2020).

Furthermore, 51.78% of the most expressive compounds indicated by heatmap were also important markers in the principal component analysis, contributing with factor loads in each quadrant.

All the obtained results generally address the importance of knowing the volatile chemical composition of wine varieties. Measuring these characteristics can help winemakers in the technological adjustments of winemaking, making it possible to obtain wines with the typicality of the most pronounced cultivar and improving their quality. Moreover, the characterization and differentiation of the varietal wines obtained in this study may increase their commercialization value, as well as with helpful information for consumers (Lukic and Horvat, 2017).

Conclusions

A strong association with the type and/or quantity of grape cultivars and the technological processes (*asti* and *charmat* methods) used in winemaking was detected. Several volatile compounds of brut, brut rosé, demi-sec and moscatel commercial sparkling wines were identified for the first time in sparkling wines from VSF to the best of our knowledge. In comparison, significant compounds were exclusively identified in each type of sparkling wine, such as moscatel (23), brut (9), brut rosé (5), and demi-sec (4), with an emphasis on the compounds 2-Phenyl-undecane, 3-Methyl-4-heptanol, 4-Phenyl-undecane and Germacrene B. This indicates a specific aromatic profile for each sparkling wine, in addition to different overall aromas which establish themselves as possible authenticity markers for commercial sparkling wines from the São Francisco Valley. In this context, future studies should be developed to identify the impact of these compounds on the overall aroma of wines, especially those reported for the first time in sparkling wines.

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Author Contribution Samara de Macêdo Morais: methodology, validation, formal analysis, investigation, data curation, writing—original draft, writing—review & editing, visualization.

Mércia de Sousa Galvão: validation, formal analysis, data curation.

Leila Moreira de Carvalho: validation, formal analysis, data curation.

Lary Souza Olegario: data curation, writing—original draft, writing—review & editing, visualization.

Giuliano Elias Pereira: supervision, writing—review & editing, funding acquisition.

Luciana Leite de Andrade Lima: term, resources, supervision, writing—review & editing.

Flávio Luiz Honorato da Silva: term, resources, supervision, writing—review & editing.

Marta Suely Madruga: term, conceptualization, resources, writing—review & editing, supervision, project administration, funding acquisition.

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Data Availability The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests The authors declare no competing interests.

Informed consent Informed consent not applicable.

Conflict of interest Samara de Macêdo MORAIS declares she has no conflict of interest. Mércia de Sousa GALVÃO declares she has no conflict of interest. Leila Moreira de CARVALHO declares she has no conflict of interest. Lary Souza OLEGARIO declares he has no conflict of interest. Giuliano Elias PEREIRA declares he has no conflict of interest. Luciana Leite de Andrade LIMA declares she has no conflict of interest. Flávio Luiz Honorato da SILVA declares he has no conflict of interest. Marta Suely MADRUGA declares she has no conflict of interest.

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