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Profile of the volatile organic compounds of pink pepper and black pepper

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Abstract. Black pepper (Piper nigrum L.) and pink pepper (Schinus terebinthifolius Raddi) are two plant-based spices, which despite having a common popular name, have a botanical family and distinct centers of origin. Its fruits are known worldwide in cuisine as condiments; in addition, the extraction of essential oil from these species is interesting from a pharmacological and industrial perspective. In this sense, the present study aimed to analyze the chemical profile of volatile organic compounds (VOC's) present in black pepper and pink pepper. The solid phase microextraction method in headspace mode (HS-SPME) was used, using the fiber, polydimethylsiloxane-divinylbenzene (PDMS/DVB) for the extraction of VOCs. In the extraction of volatile compounds, 2g of the seeds of each sample were used, previously ground in an analytical mill, and placed in a 20 ml headspace flask. The adsorption of the compounds was carried out at a temperature of 60ºC, for 20 minutes, with the exposed PDMS/DVB fiber, after extraction, the desorption was carried

out in the gas chromatograph injector coupled to mass spectrometry (CG-MS), where the fiber was exposed for 5 minutes. The identification of VOCs was performed by comparing the mass spectra obtained with data from the NIST library. Thirty-six volatile organic compounds (VOCs) were identified and quantified among pink pepper and black pepper seed samples. Of which 16 were found in black pepper, and 20 in pink pepper. These compounds are divided into monoterpenes, sesquitepenes, and other classes such as alkaloids and sesquiterpenoids. The volatile organic compounds found in higher concentrations in black pepper were Carnegine with 36.32 %, beyerene (30.84%), alpha-gurjunene (6.10%) and 1R,4S,7S,11R-2, 2,4,8-Tetramethyltricyclo [5.3.1.0 (4.11)] undec-8-ene also with 6.10%. In pink pepper, the compounds with the highest concentrations were, phyllocladene (36.16%), 3-carene (12.49%), and 1R,4S,7S,11R-2,2,4,8-tetramethyltricyclo [5.3.1.0 (4.11)] undec-8-ene (12.43%).
Keywords: *Schinus terebinthifolius, Myracrodruon urundeuva*, Headspace Solid Phase Microextraction (HS-SPME)

Introduction

The spices, or condiments, are present in food has additives, enriching the food with colors, flavors and aroma since, at least, 5000 b.C. Besides providing a better sensorial experience to the consumers, it is common the spices present physiologically beneficial effects to the human health too. These products uses for pharmacological purposes are known since the ancestral and indigenous societies, in different places on the planet, and can be seen has one of the first registered functional foods (Biazotto, 2014; Srinivasan, 2008). Spices are pointed too has one of the main economic factors that impulsionated the development of the maritime expansion, pioneered by the kingdom of Portugal at the beginning of the XV century. From that historic moment, spices started to be widely traded, by boats, worldwidelily, while new lands were being discovered, mainly by europeans. The “discovery” and the american territory colonization was one of the in this maritime expansionist race in search for spices for trading (Ramos, 2013; Roggenbach, 2017). In this work, two spices of vegetal origin, the black pepper and pink pepper, respectively known has *Piper nigrum* L. and *Schinus terebinthifolius* Raddi, will be studied.

The *Piper nigrum* L. (Piperaceae), known as black pepper, is one of the most ancient and historically coveted spices in the world, being crucial to many maritime historic events. It is a fruit of a perennial, semi-woody species with a liana habit, originally from India, being, however, cultivated in tropical and subtropical areas around the world. The fruits, of the berry type, are produced from inflorescences formed on secondary branches and, when ripe, they present a color ranging from light green or yellowish to shades of red. For the spice exploration, the fruits are dried and crushed, and can be consumed has powder, as well as by extracting their essential oils and/or oleoresins, which amplifies the possibility of its industrial uses as a condiment, for example, in beverage production, canned food, cosmetics and even pharmaceltical drugs (Almeida, 2017; Alves, 2015; Andrade, 2015; Biazotto, 2014; Carnevalli & Araujo, 2015; Ferreira & Meirelles, 2002; Guimarães *et al*., 2015). Despite its high value and commercial importance to the kingdom of Portugal at the beginning of the colonization process of the Brazilian territory, the spice was only introduced in Brazil for cultivation in the 1930s, brought by Japanese immigrants. The national production established itself and maintained predominant in the Pará state, in which its production is correspondent to 80% of the country total production. It was only after 1950 that Brazil became self-sufficient in the production on the condiment, and, shortly thereafter, became one of the world’s largest exporters of the product, reaching 1st place in the world ranking, both in production and exportation, of the spice (Almeida, 2017; Bendaoud *et al*., 2010; Bortolucci *et al*., 2019; Cole *et al*., 2014; Lourinho *et al*., 2014; Meireles, 2014; Melo *et al*., 1997).

The *Schinus terebinthifolius* Raddi (Anacardiaceae) known as pink pepper, is a vegetal species with a generally shrubby or arboreal habit of small to medium size, native to South America, especially Brazil, where it is present from the Northeast to the South of the country (Clemente, 2006; Dourado, 2012; Figueiredo *et al*., 2021; Silva-Luz & Pirani, 2015). Similar to the black pepper, its fruits are rounded and appear in bunches, from inflorescences, however, they have smaller size and, when ripe, their color varies in shades of red or pink, only. Duo to its slightly sweet and spicy flavor, they are used in cooking and cocktail making, for example, despite being a species poorly explored in Brazil (Dourado, 2012). In addition, the astringent, antidiarrheal, depurative, diuretic and febrifugal properties of this species are known duo to the presence of various chemical compounds, such as alcohols, ketones, acids, monoterpenes, sesquiterpenes and triterpenes, present not only in fruits but also in the stem and in the leaves (Andrade, 2015; Macedo, 2018; Oliveira *et al*., 2014; Santana *et al*., 2012). It is a typical plant from the Brazilian Cerrado, a biome whose great animal and plant biodiversity suffers some prejudice because it is a dry climate biome most of the year and is characterized by its vegetation, largely creeping and/or shrubby and by a soil naturally poor in fertility (Figueiredo *et al*., 2021; Klink & Machado, 2005; Pereira *et al*., 2011). However, there are several species native to the Cerrado whose potential, both as a spice, for medicinal use or in the food industry is promising, but poorly explored in Brazil, such as pink pepper spices (*Schinus t.*) or even monkey pepper (*Xylopya aromatica* (Lam.) Mart.), whose flavor and aroma is similar to black pepper (*Piper n.*) (Costa *et al*., 2021; Pontes Pires, 2019).

A quick and efficient way to analyze the industrial, gastronomic and/or pharmacological potential of these spices is the extraction of their volatile organic compounds through the solid phase microextraction metho (SPME). This method, developed by Arthur and Pawliszyn (1990), uses the principle of “green chemistry”, since it requires a small amount of sample and does not use organic solvents, and can be used direct extraction mode or in headspace mode (HS-SPME). A great advantage of HS-SPME is its high capacity for adsorption of different classes of compounds, duo to the diversity of applicable coatings of SPME fibers, and it can be used to analyze complex matrices such as acerola, cagaita, callistemon, cambuí, beer, grumixama, pequi, corn, as well as spices such as black pepper and pink pepper, species which will be analyzed in this work (Franzin *et al*., 2020; Figueiredo *et al*., 2021; Srinivasan, 2008; García *et al*., 2019; Rodrigues *et al*., 2021; Silva *et al*., 2019; Silva *et al*., 2020; Silva *et al*, 2021; Oliveira Júnior, 2020; Silva *et al*., 2021; García *et al*., 2021; Pereira *et al*. 2021; Ramos *et al*., 2020; Assunção *et al*., 2020; Ramos, *et al*., 2021a; Ramos *et al*., 2021b; Botti *et al*., 2019; Nascimento *et al*., 2021a; Nascimento *et al*., 2021b; Santos *et al*., 2020; Mariano *et al*., 2020; Rocha *et al*., 2019; Bueno *et al*, 2021; Mazzinghy *et al*., 2021; Viana *et al*., 2018).

Material and Methods

Fruit collection

The *Schinus terebinthifolius* Raddi were collected from mature seeds of 1 adult progeny in Presidente Olegário – MG, Brazil, located at coordinates 18° 25' 04" S, 46° 25' 04" O, situated at an altitude of 947 meters. According to Köeppen, the climate is Cwa, that is, a typical savanna climate, with dry winter and wet and rainy summer (Garcia *et al*., 2016). Enough was collected for 2 grams samples.

As for black pepper, it was supplied by producers in the municipality of São Mateus - ES, located at coordinates 18°47'21.1"S 39°55'11.8"O.

Sample preparation

The seeds were transported to the chemistry laboratory of UFSJ – Campus Sete Lagoas, and manual process of removing dirt and unwanted plant parts was carried out.The next step consisted of crushing the seeds using a basic analytical mill A11 from the IKA brand and weighing each sample, which was performed with the aid of a digital scale from the Marbeg brand, and then storing them in headspace flasks.

Volatile compounds extraction

The solid phase microextraction method in headspace mode (HS-SPME) was used for the extraction of the volatile compounds, using the fiber polydimethylsiloxane-divinylbenzene (PDMS/DVB). In the extraction of the volatile compounds, 2g of previously crushed seeds were used, placed in 20mL headspace flasks, that were closed with an aluminum seal and a rubber septum. The 20mL headspace flask was attached to an aluminum block and heated to 60°C on a hot plate. After 20 minutes of heating, the SPME polymeric film (PDMS/DVB) mounted in a holder, the ground seed sample was exposed for 20 minutes, and then the holder containing the polymeric film was removed and manually inserted into the injector CGMS equipment, exposing the polymeric film for 5 minutes for the desorption of the extracted volatile organic compounds (Garcia *et al*., 2016; Silva *et al*., 2019; Garcia *et al*., 2021).

Volatile organic compounds identification

Black pepper and pink pepper seed samples were analysed by a gas chromatography system (Trace GC Ultra) coupled to a mass spectrometer detector (Polaris Q model, Thermo Scientific, San Jose, CA, USA), with a mass analyzer iontrap type, installed in the Mass Spectrometry Laboratory of the Department of Chemistry at UFMG. The samples were analyzed in the following attributions: injector temperature of 250°C; splitless mode injection, desorption time of 5 minutes; injector temperature of 200°C; interface temperature of 275°C. The column heating temperature was programmed: starting at 40°C and remaining for 1 minute and then with gradual heating from 10°C/minute to 100°C, maintaining the isotherm for 1 minute, from 12°C/minute to 150°C, maintaining the isotherm for 1 minute and then 15°C/minute up to 245/C, temperature at which the isotherm was maintained for 1 minute. The detector was kept in scan mode (fullscan, from 35 to 300 m/z), using the electron impact ionization (EI) technique, with energy of 70 electron-volt (eV). A capillary chromatographic column HP-5 MS (5% phenyl and 95% methylpolysiloxane) was used throughout the process, covering the following dimensions: 30m in length, 0,25mm in internal diameter and 0,25µm in film thickness (Agilent Techonolgies INC, Germany) (Garcia *et al*., 2016; Figueiredo *et al*., 2021).

To identify the volatile compounds found, it was based on the m/z ratio corresponding to each peak generated by the chromatogram, and compared with the mass spectra obtained by EI ionization, which used an energy of 70eV, with the range of scan from 35 to 300m/z (Garcia *et al*., 2019; Silva *et al*., 2019; Oliveira Júnior *et al*., 2020). Thus, the mass spectra of the analytes found were compared with the mass spectra data obtained from the NIST library (National Institute of Standards and Technology), using as an auxiliary tool to the data recorded in the literature for the confirmation of volatile compounds present in the samples of the seeds. The RSI index consists of a numerical comparison factor where, the higher its value, the closer the compound is to the finding in the NIST library literature. However, only peaks with a value above 500, relative standard intensity (RSI), and a signal-to-noise ratio (S/N) above 50 decibels were selected. The peak intensity values obtained and the S/N ratio were obtained from the Thermo Electron Corporation Xcalibur 1.4 program and transferred to Microsoft Office Excel 2013, through this programs peak selections were made according to the S/N ratio at the UFSJ/CSL Chemistry Laboratory.

Results and discussion

36 volatile organic compounds (VOCs) were identified and quantified among the pink pepper and black pepper seed samples using the DVB-PDMS fiber, of which 16 were found in black pepper, and 20 in pink pepper (Table 1).

Table 1. Volatile organic compounds found in the pink pepper and black pepper samples.

N°	Volatile Organic Compounds	CAS	Formula	Sample %	
				Piper nigrum	***Schinus***
		MONOTERPENES			
1	3-Carene[a, b, c, d, e, g, h, l]	13466-78-9	$C_{10}H_{16}$	3,04	12,49
2	Terpinolene[a, c, d, e, g, l]	586-62-9	$C_{10}H_{16}$	1,10	0,90
3	p-Mentha-1(7),8-diene[a, b,]	499-97-8	$C_{10}H_{16}$	1,00	-
4	Trans-carene	18968-23-5	$C_{10}H_{18}$	-	0,45
5	(+) -cis-Sabinol	471-16-9	$C_{10}H_{16}O$	-	0,38
6	Bornilacetate	76-49-3	$C_{12}H_{20}O_2$	-	0,66
7	Beyerene	3564-54-3	$C_{20}H_{32}$	30,84	-
8	Linalyl butyrate	78-36-4	$C_{14}H_{24}O_2$	0,08	-
9	(+)-Terpinen-4-ol[a, b, h, l]	562-74-3	$C_{10}H_{18}O$	0,16	-
10	alpha-Terpineol[a, b, c, k]	98-55-5	$C_{10}H_{18}O$	0,07	-
		SESQUITERPENES			
10	4,4-Dimethyl-3- (3-methylbut-3-enylidene) -2-methylenebicyclo[4.1.0]heptane	79718-83-5	$C_{15}H_{24}$	-	3,08
11	10s,11s-Himachala-3(12),4-diene	NA	$C_{15}H_{24}$	-	0,76
12	Himachalene[d, e,]	1461-03-6	$C_{15}H_{24}$	-	1,13
	α – Guaiene	3691-12-1	$C_{15}H_{24}$	-	1,50
13	*β*-Guaiene[d]	88-84-6	$C_{15}H_{24}$	5,29	7,11
14	(-) -alpha-Gurjunene[a, d, j, k]	489-40-7	$C_{15}H_{24}$	6,10	3,11
	(+) -gamma-Gurjunene	22567-17-5	$C_{15}H_{24}$	-	1,66
15	Cariofileno[a, b, c, d, e, f, g, h, k, l]	87-44-5	$C_{15}H_{24}$	-	2,29
16	(-) -Cyperene	2387-78-2	$C_{15}H_{24}$	5,60	-
17	Elemene[a, b,]	20307 – 84-0	$C_{15}H_{24}$	1,26	-
	δ-Elemene[a, l]	20307-84-0	$C_{15}H_{24}$		1,78
18	*α* – Muuroleno[a, b,]	12306047		1,36	-
19	*β*-Cadinene[j, k]	523-47-7	$C_{15}H_{24}$	-	5,39
20	Epizonarene	41702-63-0	$C_{15}H_{24}$	0,53	-
21	1,1,4a-Trimethyl-5,6-dimethylenedecahydronaphthalene	NA	$C_{15}H_{24}$	1,15	-
22	1R,4S,7S,11R-2,2,4,8-Tetramethyltricyclo[5.3.1.0(4,11)]undec-8-ene	NA	$C_{15}H_{24}$	6,10	12,43
		OTHER CLASSES			
23	Carnegine	490-53-9	$C_{13}H_{19}NO_2$	36,32	-
24	Isopseudocumenol[d,]	697-82-5	$C_9H_{12}O$	-	7,26
25	*γ*-Eudesmol	1209-71-8	$C_{15}H_{26}O$	-	1,02
26	(-) -Phyllocladene	20070-61-5	$C_{20}H_{32}$	-	36,16
27	β-Eudesmol[d, e, k]	473-15-4	$C_{15}H_{26}O$	-	0,44

Letters inficate compounds that haven been identified by other authors, (a)Singh et al. 2004; (b)Kapoor *et al*. 2009; (c)Melo *et al*. 2021; (d)Figueiredo *et al*. 2021; (e)Silva 2017; (f)Santos *et al*. 2014; (g)Cole 2008; (h)Clemente 2006; (i)Costa *et al*. 2010; (j)Dourado 2012; (k)Oliveira *et al*. 2014; (l)Barbosa *et al*. 2007

Among the 16 VOCs found in black pepper, 7 were classified as monoterpenes, 8 as sesquiterpenes, and carnegine which is na alkaloid. As in the work by Costa *et al*. (2010), a higher number of sesquiterpenes in black pepper seed was identified. Among the monoterpenes, 4 oxygenated: bornylacetate, linalyl butyrate, terpinen-4-ol, and alpha-terpineol, results that resemble those of Costa *et al*. (2010). However, these results differ from those found by Figueiredo *et al*. (2021) where the proportion of monoterpenes were higher (80%).

The VOCs found in higher concentrations in black pepper were carnegine with 36,32%, beyerene (30,84%), alpha-gurjunene (6,10%) and 1R,4S,7S,11R-2,2,4,8-Tetramethyltricyclo [5.3.1.0 (4,11)]undec-8-ene also with 6,10%. The compound carnegine is a simple tetrahydroisoquinoline, isolated from different plants around the world, such as Cactaceae, Chenopodiaceae and Boraginaceae

(Bracca & Kaufman, 2004). Beyerene configures as a diterpene, reported by Guerreiro *et al*. (1984) when it was extracted from aerial parts of *Petunia patagonica* (Speg.) Millán, (Solanaceae). *α*-Gurjene is a sesquiterpene, and was also found in the work made by Singh *et al*. (2004). However the compound with higher concentrations found in the present study differ from those found in Melo *et al*. (2021), as well as in Costa *et al*. (2010), where they found E-Caryophyillene, Limonene, e Sabinene with the highest concentrations.

In pink pepper, of the 20 compounds found, 5 are classified as monoterpenes, 11 as sesquiterpenes, 4 belonging to diferente classes (phyllocladene, isopseudocumenol, γ-eudesmol and β-eudesmol). The compounds with the highest concentrations were phyllocladene (36,16%), 3-carene (12,49%), and 1R,4S,7S,11R-2,2,4,8-Tetramethyltricyclo [5.3.1.0 (4,11)]undec-8-ene (12,43%). Of those compounds, the monoterpene 3-carene is often found in the literature regarding the chemical profile of *Schinus terebinthifolius* (Figueiredo *et al*. 2021; Silva, 2017; Cole, 2008, Clemente, 2006), however, analyzing its concentration, in the present study, 3-carene had a lower concentration (12,49%) compared to the works of Figueiredo *et al*. (2021) (26,81%), Silva (2017) analysingg 11 seed samples, presenting a minimum concentration of 33,78%, and a maximum of 36,73%; Cole (2008) (30,37%), and Clemente (2006) with 29,22%, where all evaluated the pink pepper fruit. In the work of Santos *et al*. (2014), evaluating samples of pink, green and ripe pepper fruits, respectively, 3-carene was not even identified, the compound with highest concentration being limonene (70,49%) (67,15%), followed by α-phellandrene (19,24%) (18,94%). Volatile organic compounds found in pink pepper in higher concentrations, in this study, also differ in terms of the composition foun in studies related to *Schinus terebinthifolius*, as in Figueiredo *et al*. (2021), tracing the comparative profile of pink pepper and aroeira, pink peper had the highest concentrations (in addition to the 3-carene already discussed), β-guaiene (14,93%), and isopseudocumenol (14,62%) in this study was found with a concentration of 7,26%, as well as in Clemente (2006) that in addition to 3-carene, the others with higher concentrations were β-phellandrene (18,08%), and α- phellandrene (13,04%).

Among the oxygenated compounds in the pink pepper, two were found, isopseudocumenol and β-eudesmol, the same ones observed in Figueiredo *et al*. (2021).

In both samples, pink pepper and black pepper, 3 compounds were found in common, 3-carene, terpinolene, and β-guaiene.

The different concentrations and qualities of volatile organic compounds found in this study and compared with the literature on the subject are inherent, as is known, to the extraction method, the parts of the plants used, the place of harvest of the species, as well as the local edaphoclimatic conditions at a given time, and that exert different influences on the production and composition of secondary metabolites.

Conclusion

The headspace solid phase microextraction method was eficiente in the extraction of volatile compounds, with 36 compounds being identified and quantified among the black pepper and pink pepper seed samples, using DVB-PDMS fiber, being identified 16 compounds in black pepper and 20 in pink pepper samples. There was a greater number of sesquiterpenes between both samples.

The compounds found in higher concentrations in black pepper were carnegine with 36,32%, beyerene (30,84%), alpha-gurjunene (6,10%), as well as 1R,4S,7S,11R-2,2,4,8-Tetramethyltricyclo[5.3.1.0(4,11)]undec-8-ene also with 6,10%. In pink pepper, the compounds with the highest concentrations were phyllocladene (36,16%), 3-carene (12,49%), and 1R,4S,7S,11R-2,2,4,8-Tetramethyltricyclo[5.3.1.0(4,11)]undec-8-ene (12,43%). In both samples, 3 compounds were found in common, being 3-carene, terpinolene and β-guaiene.

The extraction of volatile compounds by the HS-SPME method used in the two species in this study helps in greater knowledge of the chemical profile of these two spices, and in the prospection of pharmacological, gastronomic and industrial potentialities.

The gas chromatography technique coupled with mass spectrometry has been shown to be very efficient and has been highlighted due to its practicality in execution, in addition to providing representative results.

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