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Chemistry

Volatile compound changes at *Brunfelsia uniflora* flower senescence

Mudanças nos compostos voláteis na senescência das flores de *Brunfelsia uniflora*

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ABSTRACT

The flower of *Brunfelsia uniflora* has few studies and no chemical characterization of volatiles by direct extraction via headspace and analysis by gas chromatography coupled to mass spectrometry (HS/GC-MS). This study provides background information on the flower senescence process. The objective of this study was to compare the chemical composition of volatiles from the purple and white flower stages of *B. uniflora* by HS/GC-MS. The volatile compounds from flowers incubated in headspace vials were analyzed by GC-MS. Oxygenated sesquiterpenes were the majority volatile class. The main volatile compounds for purple flower were *trans*-nerolidol (16.2%), *trans*-geranylgeraniol (5.8%), *cis*-linalool oxide (4.9%), and *cis-cis*-geranyl linalool (4.4%), and for white flower were *trans*-nerolidol (26.1%), *trans*-β-ocimene (9.5%), *trans*-geranylgeraniol (7.9%), and *trans*-γ-bisabolene (3.1%). Our results increased the knowledge of the types of volatile chemical compounds at different maturation stages of *B. uniflora* flower.

Keywords: Manacá; Volatiles; Headspace; Flower; Chemical composition

RESUMO

A flor de *Brunfelsia uniflora* tem poucos estudos e nenhuma caracterização química de voláteis por extração direta via headspace e análise por cromatografia gasosa acoplada à espectrometria de massa (HS/GC-MS). Este estudo fornece informações básicas sobre o processo de senescência das flores. O

objetivo deste estudo foi comparar a composição química dos voláteis da fase de flor roxa e branca de *B. uniflora* por HS/GC-MS. Os compostos voláteis das flores incubadas em frascos de headspace foram analisados por GC-MS. Os sesquiterpenos oxigenados foram a classe volátil majoritária. Os principais compostos voláteis para a flor roxa foram *trans*-nerolidol (16,2%), *trans*-geranilgeraniol (5,8%), óxido de *cis*-linalol (4,9%) e *cis*-*cis*-geranil linalol (4,4%), e para a flor branca foram *trans*-nerolidol (26,1%), *trans*-β-ocimeno (9,5%), *trans*-geranilgeraniol (7,9%) e *trans*-γ-bisaboleno (3,1%). Nosso estudo adiciona conhecimento aos tipos de compostos químicos voláteis em diferentes estágios de maturação da flor de *B. uniflora*.

Palavras-chave: Manacá; Voláteis; *Headspace*; Flor; Composição química

1 INTRODUCTION

Brunfelsia uniflora (Pohl.) D. Don. (Solanaceae) is native to Brazil and found in several regions of Brazil, Bolivia, Peru, Ecuador, Colombia, and Venezuela, popularly known as manacá, it has a bushy shape, simple leaves, and flowers in clusters or solitary. After the floral evocation, the open flower has a purple color and gradually fades to a white color in the senescence process (Althaus-Ottmann et al., 2006). The genus *Brunfelsia* is appreciated for the exuberant effect caused by the flower's chromatic variation and the fragrance (clove scent with musk hints in the evening) and *B. uniflora*, in particular, has several biological activities such as acaricidal (Sugauara et al., 2019), insecticidal (Sugauara et al., 2022), antioxidant, and antimicrobial (Thiesen et al., 2018) activities.

Few studies have evaluated the chemical composition of *B. uniflora* flowers. The oleoresin of *B. uniflora* flowers obtained by supercritical carbon dioxide had an antimicrobial activity with major compounds 21% (E,E)-geranyllinalool, 3.9% (E)-nerolidol, and 0.5% (2E,6Z)-farnesal (Thiesen et al., 2017) and antioxidant activity with major compounds 11-21% geranyl linalool, 9-24% tetracosane, and 10-22% alpha-amyrin (Jorge et al., 2017). The flower alcoholic extract of *B. uniflora* had insecticidal activity against *Aedes aegypti* larvae and the major compounds found were 36% alpha-amyrin, 16% beta-amyrin, and 10% (EE)-geranyl linalool (Sugauara et al., 2022).

However, those studies evaluated a mixture of purple and white flowers, and other studies of the chemical compounds of different floral stages have not been found.

Given the complexity of chemical compounds in *B. uniflora* flowers, evaluating their volatile compounds at different floral stages is crucial. Similarly, traditional Chinese medicine, which utilizes both female and male flowers of *Eucommia ulmoides* Oliv. (*duzhong* in Chinese) and *Trichosanthes* spp., has demonstrated significant variations in chemical composition and biological activity (Xu et al., 2019). Additionally, the characterization of underexplored plants is increasingly important in the search for novel bioactive compounds, as these species may harbor unique phytochemicals with significant therapeutic potential. For instance, studies on *Vassobia breviflora* (Sendtn.) Hunz, a South American native, have revealed novel phytochemicals and various biological activities (Viana et al., 2022; Viana et al., 2023). These findings underscore the importance of investigating lesser-known plants for their potential in developing new drugs and therapies.

The supercritical carbon dioxide technique has a high extraction capacity for a broad spectrum of chemical compounds, with high solvation selectivity and control of molecules, and is considered safe for the environment (Manjare & Dhingra, 2019). In addition, this technique leaves no solvent residues, the oil obtained has a high degree of purity, and promotes less degradation of volatile compounds (Kiran & Brenneecke, 1993; Mazutti et al., 2006). However, despite this extracting capacity for a large number of compounds, molecules of lower molar mass, abundant in flowers, are lost with the carbon dioxide evaporation at the final stage of the extraction process (Qamar et al., 2021). Therefore, the static headspace technique coupled with gas chromatography analysis is a useful, fast, economical, and efficient tool for characterizing volatile compounds from complex samples (Gałuszka et al., 2013; Tripler, 2013; Zhang et al., 2017) and could provide further elucidation of the volatile chemical compounds of *B. uniflora* flowers. This study aimed to compare the chemical compounds from the purple and white flower stage of *B. uniflora* by static headspace and gas chromatography coupled with mass spectroscopy (HS/GC-MS).

2 MATERIAL AND METHODS

2.1 Biological material

Brunfelsia uniflora (Pohl) D. Don (Solanaceae), current scientific name, and its synonyms and combinations such as *Martia opifera* Lacerda ex J. A. Schmidt, *Brunfelsia uniflora* var. *pubescens* (Benth.) Baker, *Franciscea uniflora* Pohl, *Brunfelsia hopeana* (Hook.) Benth., *Brunfelsia hopeana* var. *pubescens* Benth., *Franciscea hopeana* Hook., and *Franciscea mutabilis* H. Jacq. (Hassler, 2021) was taken from the Educational Herbarium of Paranaense University, number 2855, located at coordinates S23º 46.225' and WO 53º 16.730', 391 m altitude. The flowers of *B. uniflora* with intense purple coloring, the initial stage of flower development, and the white flowers with no remnants of purple coloring, the final stage of flower development, were harvested between 7 and 8 am. The fresh flowers (*in natura*) from each floral stage were stored in refrigerated containers before analysis on the same day. This plant is registered in the National System for Management of Genetic Heritage and Associated Traditional Knowledge (SisGen, in Portuguese) under registration number A0A10E1.

2.2 Flower extraction and chemical identification of volatile molecules

The *in natura* purple or white flowers (5 g) of *B. uniflora* were transferred to headspace flasks (20 mL clear borosilicate vial headspace) and the volatiles extracted by static headspace incubation. The headspace incubation parameters were: incubation temperature 130 °C, incubation time 60 min, agitation (on 60 s; off 10 s), syringe temperature 150 °C, agitation speed 750 rpm. The volatile compounds were analyzed by gas chromatography (Agilent 7890 B) coupled to mass spectrometry (Agilent 5977 A) (GC-MS). A 5% HP-5MS analytical column (30 m × 0.25 mm × 0.25 µm) was used with an initial temperature of 60 °C followed by a heating ramp of 3 °C/min to 170 °C, heating 2 °C/min to 250 °C, remaining for 10 min, and finally a heating ramp of 10 °C/min to 300 °C for 1 min. The carrier gas used was helium at a linear rate of 1 mL/min up

to 300 °C and a pressure of 56 kPa. The injector temperature was 260 °C; the injection volume was 2 mL; injection occurred in split mode (20:1). The transfer line was kept at 280 °C and the ionization source and quadrupole at 230 °C and 150 °C, respectively. The detection system was EM, in scan mode, in the mass-to-charge ratio range of 40 to 600 m/z. The volatile compounds were identified by comparing their mass spectra with the mass spectra from the NIST Library version 11.0 (Adams, 2007). The results of the volatile chemical composition of *B. uniflora* flowers at different development stages were compared and discussed.

3 RESULTS

In the fresh flowers of *B. uniflora*, 10 chemical compounds were identified for the purple flower and 16 out of 21 compounds for the white flower (Table 1). The major chemical compounds found in the purple flower were carbon dioxide (51.2%), *trans*-nerolidol (16.2%), *trans*-geranylgeraniol (5.8%), *cis*-linalool oxide (4.9%), and *cis*-*cis*-geranyl linalool (4.4%), and the major compounds in the white flower were carbon dioxide (36.4%), *trans*-nerolidol (26.1%), *trans*-β-ocimene (9.5%), *trans*-geranylgeraniol (7.9%), and *trans*-γ-bisabolene (3.1%) (Table 1 and Figure 1).

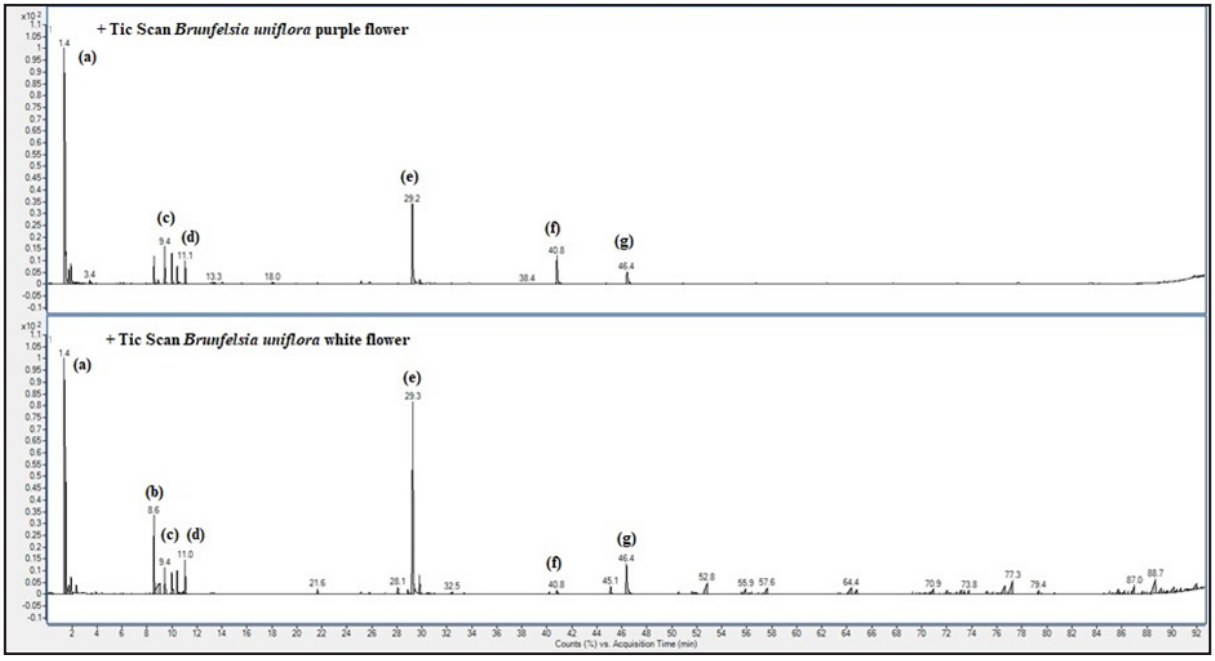
The purple flower had 29% carbon dioxide, 47% isovaleraldehyde, 47% *cis*-linalool oxide, 52% *trans*-linalool oxide, 20% linalool, and 36% *cis*-*cis*-geranyl linalool higher than the white flower. However, the purple flower had 13% *trans*-γ-bisabolene, 37% *trans*-nerolidol, and 37% *trans*-geranylgeraniol lower than the white flower. Furthermore, the compounds delta-3-carene, *trans*-β-ocimene, *trans*-β-farnesene, 2,3-dihydrofarnesol, *cis*-α-santalol, hexadecanoic acid ethyl ester, and 9,12-octadecadienoic acid ethyl ester were absent in the purple flower and present in the white flower, *trans*-β-ocimene was present in the purple flower but absent in the white flower, and five non-identified compounds were absent in the purple flower and present in the white flower (Table 1). This indicates that the flower senescence process promotes a change in the volatile chemical composition with decrease or increase and presence or absence of compounds.

Table 1 – Chemical composition of volatile molecules of *in natura* purple and white flowers of *Brunfelsia uniflora* obtained by headspace/GC-MS

Peak	RT (min)	Compound	RI	RA (%)		IM
				Purple flower	White flower	
1	1,377	Carbon dioxide	154	51.23	36.35	a,b,c
2	1,519	Isovaleraldehyde	658	2.97	1.57	a,b,c
3	1,928	Delta-3-carene	1011	–	1.80	a,b,c
4	2,344	n.i.	–	–	0.51	a,b,c
5	8,564	*cis*-β-Ocimene	1032	3.47	–	a,b,c
6	8,600	*trans*-β-Ocimene	1044	–	9.50	a,b,c
7	9,052	*cis*-Linalool oxide	1067	4.93	2.61	a,b,c
8	9,418	*trans*-Linalool oxide	1084	4.21	1.53	a,b,c
9	9,989	Linalool	1095	2.45	1.95	a,b,c
10	10,412	*trans*-β-Farnesene	1454	–	2.09	a,b,c
11	11,051	*trans*-γ-Bisabolene	1529	2.75	3.10	a,b,c
12	29,250	*trans*-Nerolidol	1561	16.17	22.13	a,b,c
13	29,279	2,3-Dihydrofarnesol	1688	–	0.53	a,b,c
14	29,814	*cis*-α-Santalol	1674	–	1.08	a,b,c
15	40,800	*cis*-*cis*-Geranyl linalool	1960	4.38	2.12	a,b,c
16	45,219	Hexadecanoic acid ethyl ester	1992	–	0.94	a,b,c
17	46,407	*trans*-Geranylgeraniol	2201	5.77	7.89	a,b,c
18	52,837	9,12-Octadecadienoic acid ethyl ester	2527	–	2.72	a,b,c
19	76,651	n.i.	–	–	1.84	a,b,c
20	77,270	n.i.	–	–	3.03	a,b,c
21	87,022	n.i.	–	–	1.41	a,b,c
22	88,713	n.i.	–	–	3.05	a,b,c
Total identified			**98.33**	**97.91**		
Hydrocarbon monoterpenes			3.47	11.30		
Oxygenated monoterpenes			11.59	6.09		
Hydrocarbon sesquiterpenes			2.75	5.19		
Oxygenated sesquiterpenes			16.17	23.74		
Oxygenated diterpenes			10.15	10.01		
Carbon dioxide			51.23	36.35		
Aldehydes			2.97	1.57		
Fatty acid esters			–	3.66		
n.i.			–	9.84		

Source: Authors (2024). RT = retention time (min); RI = retention index; IM = identification methods a: identification based on retention index from literature with Adams (2007) and NIST - Standard Reference Database 69 (NIST Chemistry WebBook); b: compounds listed in order of elution on HP-5MS UI column; c: identification based on comparison of mass spectra found in NIST 11.0 version 11.0 libraries; RA (%) = relative area (%): percentage of the area occupied by the compound in the chromatogram; n.i. = not identified; (–) = absent

Figure 1 – Chromatogram of volatile compounds of *Brunfelsia uniflora* purple and white flowers obtained by headspace/GC-MS



Source: Authors (2024). Major flower volatiles marked as (a) carbon dioxide, (b) *trans*-β-ocimene, (c) *cis*-linalool oxide, (d) *trans*-γ-bisabolene, (e) *trans*-nerolidol, (f) *cis*-*cis*-geranyl linalool, and (g) *trans*-geranylgeraniol

The predominant class of volatile compounds, without considering carbon dioxide, were oxygenated sesquiterpenes for the purple flower (16.2%) and the white flower (23.7%) (Table 1). The volatile compound classes changed from purple to white flower stage and were 47% oxygenated monoterpenes, 1% oxygenated diterpenes, 29% carbon dioxide, and 47% aldehydes higher in purple than white flowers, and 226% hydrocarbon monoterpenes, 89% hydrocarbon sesquiterpenes, and 47% oxygenated sesquiterpenes higher in white than purple flowers. Fatty acid esters (3.7%) occurred only in the white flower stage. This indicates that throughout the senescence process of the flowers, there is a change in the content of volatile compound classes.

4 DISCUSSION

Carbon dioxide is produced by cellular aerobic respiration (Nelson & Cox, 2012) and purple flowers showed higher carbon dioxide content, which indicates a higher

cellular respiration ratio than white flowers. This result is expected for purple (young) flowers and, therefore, have a higher metabolic ratio than white (mature) flowers (51.2% in purple flowers and 36.4% in white flowers in our study). However, carbon dioxide can be produced by endophytic microorganisms, where 46 endophytic fungi have been isolated from *B. uniflora* (Marsola et al. 2022), or microorganisms from the flower phyllosphere as plant surfaces can have great microbial diversity (Cilião Filho et al., 2017). Furthermore, these microorganisms may consume to exhaustion the carbohydrates present in the flower during the senescence process, which would explain the lower content of carbon dioxide in the white flowers. Therefore, because this compound is produced by most plants, it has not been listed as a major compound.

Nerolidol is sesquiterpene alcohol with *trans* and *cis* geometric isomers, and a common chemical component in several plants with floral odor (Chan et al., 2016; Cazella et al., 2019) such as *Arabidopsis lyrata* (Abel et al., 2009). It is volatile with production induced by herbivore attack (Chan et al., 2016) and in the *trans*-nerolidol form (16.2% in purple flower and 22.1% in white flower in our study) acts as a pheromone (PubChem, 2021). White flowers had probably a longer exposure time to herbivore attack and consequently produced a greater amount of nerolidol. Nerolidol is also broadly used in cosmetics, cleaning products, and as food flavorings. They have antimicrobial, antiparasitic, antibiofilm, antioxidant, antinociceptive, anti-inflammatory, anti-ulcerogenic, anticancer, and insect repellent activities (Chan et al., 2016). Due to its hydrophobic nature, it is easily permeable through the plasma membrane, can interact with intracellular proteins, and improves drug penetration by transdermal delivery, but has high cytotoxic potential with the ability to disrupt membranes (Chan et al., 2016).

β-Ocimene is a common volatile released by leaves and flowers of several plants and this acyclic monoterpene may play several biological functions having an attraction effect on floral pollinators and also defensive responses against herbivores (Farré-Armengol et al., 2017). In non-floral tissues, it functions as a chemical indicator to

attract natural enemies (phytophagous insects) as a plant systemic defensive response (Farré-Armengol et al., 2017). It has two stereoisomers, *cis*- and *trans*-β-ocimene, and it is synthesized by the enzyme (E)-β-ocimene synthase which mostly produces *trans*-β-ocimene. The *cis*-β-ocimene is less common than *trans*-β-ocimene in floral scents, being produced and emitted in small amounts, but detectable only in species where *trans*-β-ocimene is produced in moderately high amounts (Farré-Armengol et al., 2017). In our study, *cis*-β-ocimene (3.5% in purple flower and 0% in white flower) occurred in smaller amounts than *trans*-β-ocimene (0% in purple flower and 9.5% in white flower), which suggests that *B. uniflora* flowers might have a different compound synthesis due to the presence of *cis*-β-ocimene in the purple flower without *trans*-β-ocimene and the presence of *trans*-β-ocimene in the white flower without *cis*-β-ocimene.

Geranylgeraniol is an isoprenoid found in plants and an important metabolic derivative in the isoprenoid/cholesterol synthesis pathway. It plays very distinct roles in various physiological processes in plants and animals such as anti-inflammatory, antitumor, neuroprotective, and testosterone production enhancing activity in animals (Ho et al., 2018). *trans*-Geranylgeraniol (5.8% in purple flower and 7.9% in white flower in our study) is a plant metabolite precursor to phytol, the prenyl side chain of chlorophyll. The geranylgeraniol conversion to phytol is bound to chlorophyll synthesis and catalyzed by protein complexes associated with thylakoid membranes. Phytol is also used for the production of tocopherol (vitamin E), phylloquinone (vitamin K), and phytyl esters from fatty acids (Gutbrod et al., 2019). Therefore, as geranylgeraniol is related to different biochemical reactions, it is possible that the higher amount of this compound, in the white flowers, is related to the accumulation of this molecule throughout the flower senescence process.

Linalool is tertiary acyclic monoterpene alcohol, one of the most common floral fragrances in nature emitted mostly by petals and mostly converted into linalool oxides (Pichersky et al., 1994). It is usually added in perfumes, cosmetics, cleaning products, processed foods, beverages (fragrance and flavoring agents), and food

additives (PubChem, 2021), and also in the industry as an intermediate of vitamin E and A synthesis and as an insecticide. It has sedative, anxiolytic, anticonvulsant, analgesic, anti-inflammatory, antioxidant, antitumor, antimicrobial, cholesterol-reducing, and spasmodic activities (Aprotosoaie et al., 2014).

cis-*cis*-Geranyl linalool (4.4% in purple flower and 2.1% in white flower in our study) is produced by monoterpenes that capture the geranyl diphosphate and transform it into linalool (Landmann et al., 2007). The fragrance components of flowers are stored as non-volatile glycosides and degraded over flower senescence time (Pichersky et al., 1994), which may explain the higher linalool content in the purple flowers. Wild tobacco (*Nicotiana obtusifolia*) has geranyl linalool glycosides with antifeedant properties, substances that inhibit insect feeding but without toxic effects (Jassbi et al., 2010).

Bisabolenes are sesquiterpenes produced from nerolidol degradation and constitutively synthesized in plants (Tholl, 2015), which may explain the small variation of *trans*-γ-bisabolene (3.1% in purple flowers and of 2.8% in white flowers in our study) in *B. uniflora* flowers.

Isovaleraldehyde is a component of volatile oils in olives and has been used as a flavoring agent, food additive, and also to prevent, destroy or mitigate pests (PubChem, 2021). It is used as a pharmaceutical reagent and has antimicrobial and insecticidal activities (Kohlpaintner et al., 2013). Isovaleraldehyde (3.0% in purple flower and 1.6% in white flower in our study) is a plant metabolite produced by the catabolism of amino acids, mainly leucine (Palmer, 1984). The higher isovaleraldehyde content on the purple flowers could be related to their pest mitigating function.

The 9,12-octadecadienoic acid ethyl ester derived from linoleic acid can be found in grapes, coriander, sweet marjoram, and white mustard. It has a patent for treating diabetes, metabolic diseases, inflammation prevention, and is part of a pharmaceutical composition treatment containing ethyl linoleate as an active ingredient (PubChem, 2021). Moreover, it is abundant in volatile oil from flowers of *Coreopsis* spp. with antioxidant and antimicrobial properties (Kim et al., 2020). Our results indicate the

presence of this compound only in the white flowers (2.7%), which is expected since it is a derivative of fatty acid catabolism promoted by sugar depletion in the flower senescence.

Farnesene is the major compound of the death-alarm pheromone of aphids and is used as a natural repellent of these insects (Yu et al., 2012). *trans*-β-Farnesene (0% in purple flower and 2.1% in white flower in our study) is an herbivore-induced compound produced from farnesyl and is related to the senescence of plant tissues (Tholl, 2015).

Delta-3-carene is an anti-inflammatory (Martin et al., 1993) and one of the turpentine components that can irritate the skin and mucous membranes, and prolonged exposure can result in allergic contact dermatitis or impaired chronic lung function (Duisken et al., 2008). In our study, delta-3-carene was not found in the purple flower, but it was identified in the white flower (1.8%). This compound is associated with plant tissue senescence by cell division suppression, membrane rupture, and oxidative stress, and could affect the root apical meristem of other surrounding plants by inhibiting cell proliferation. It is still unclear whether this compound acts as a senescence initiation signaling molecule or whether it is a senescence direct effect (Korankye et al., 2017).

α-Santalol is a naturally occurring terpenoid with a chemopreventive effect on skin cancer (Zhang & Dwivedi, 2011). It is found in sandalwood (*Santalum album*) oil with anticancer, anti-inflammatory, antifungal, antihyperglycemic, and neuroleptic activities among others (Bommareddy et al., 2019). *cis*-α-Santalol (0% in purple flower and 1.1% in white flower in our study) is produced from a long metabolic route from nerolidyl pyrophosphate in the conversion route from farnesyl pyrophosphate to sesquiterpenoids. Santalol production is time-consuming and takes place after several metabolic steps (Croteau & Karp, 1994), which may explain its absence in purple (young) flowers and the presence in white (mature) flowers of *B. uniflora*, albeit in small quantities. Finally, 2,3-dihydrofarnesol is a fatty alcohol with patents applied for

pro-fragrance composition, umami flavor agent, high-intensity sweetener, dermatitis treatment among others (PubChem, 2021). Furthermore, it is a pheromone that attracts bees (buff-tailed bumblebee) such as *Bombus terrestris* (Coppée et al., 2011), an endangered pollinator species throughout the United Kingdom, protected by the Bumblebee Conservation Trust.

5 CONCLUSIONS

The major volatile chemical compounds of *B. uniflora* purple flowers are *trans*-nerolidol (16.2%), *trans*-geranylgeraniol (5.8%), *cis*-linalool oxide (4.9%), and *cis*-*cis*-geranyl linalool (4.4%); for white flowers are *trans*-nerolidol (26.1%), *trans*-β-ocimene (9.5%), *trans*-geranylgeraniol (7.9%), and *trans*-γ-bisabolene (3.1%). Young (purple) flowers have a higher concentration of secondary metabolism compounds, which have no direct action on the plant metabolism and act as an odor-attracting pollinator. Mature (white) flowers are related to the senescence process as from the catabolism of lipids and amino acids. The major class of volatile compounds was oxygenated sesquiterpenes in the purple flower (16.2%) and the white flower (23.7%). These results increase the knowledge of the types of volatile chemical compounds at different maturity stages of *B. uniflora* flowers. Further research may explore the understanding of these volatile compounds and their roles in the plant's life cycle. Potential areas of study include pollination and ecological interactions, molecular mechanisms, *Brunfelsia* spp. comparative analysis, environmental factors on volatile production, applications in horticulture and the fragrance industry, and strategies for prolonging the flowering period or improving the ornamental value of flowers.

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