



Original article

Volatile profile and sensory descriptors of *Melaleuca* quinquenervia (Cav.) S.T. Blake essential oil from Madagascar as a promising food flavouring

Maria Merlino, 1* Fabrizio Cincotta, 1 Antonella Verzera, 1 Anthea Miller, 1 Marco Torre 1,2 & Concetta Condurso 1

- 1 Department of Veterinary Sciences, University of Messina, Viale G. Palatucci, 98168 Messina, Italy
- 2 Department of Agricultural, Forestry and Food Sciences DISAFA, University of Turin, Via Verdi 8, 10124 Torino, Italy

(Received 13 May 2024; Accepted in revised form 15 July 2024)

Summary

The present study investigated the volatile and sensory profiles and antioxidant activity of 1,8-cineole type Niaouli essential oil from Madagascar for which limited information is reported in literature. The volatile compounds were identified by Gas chromatography–mass spectrometry and semi-quantitative results were obtained by Gas chromatography with a flame-ionisation detector; the odour descriptors were determined by a trained panel using a Quantitative Descriptive Analysis. The antioxidant activity was determined, too. Several volatiles, most of these here firstly, have been identified; 1,8-cineole (eucalyptus-like) prevailed, followed by viridiflorol and (*E*)-nerolidol. 'Eucalyptus-like, fresh-mint, woody, sweet-floral and spicy' odour descriptors have been defined. Volatiles resulted in agreement with the odour descriptors. The data allowed to characterise the Niaouli essential oil; considering its volatile composition, pleasant sensory notes and antioxidant activity the 1,8-cineole type Niaouli essential oil from Madagascar could be considered a promising flavouring agent and natural preservative in the food industry.

Keywords

1,8-cineole chemotype, antioxidant activity, *Melaleuca quinquenervia* (Cav.) S.T. Blake, Niaouli essential oil, sensory descriptors, volatile composition.

Introduction

The Myrtaceae family, mainly spread in Australia, Southeast Asia, tropical to southern temperate America and Africa, is a rich source of essential oils, with a hundred genera and approximately 3800 species (Wilson et al., 2001). Belonging to the Myrtaceae family, Melaleucas include a diverse range of forms, from woody shrubs to tall timber-producing trees. Leaves vary in size and shape, from short to long, elliptic, cordate and lanceolate; venation is pinnate, longitudinal or longitudinal-pinnate (Brophy et al., 2013). The leaf essential oils of various Melaleuca species differ in composition, also due to pedoclimatic conditions; methyl eugenol, (E)-methyl isoeugenol, 1,8-cineole, terpinen-4-ol, terpinolene, (E)-nerolidol, viridiflorol, ledol, β-caryophyllene have been reported as the main volatile compounds (Wheeler et al., 2007).

Melaleuca quinquenervia (Cav.) S.T. Blake is native and mainly spread in Australia, New Caledonia and Madagascar; it is highly valued for its leaf essential oil

*Correspondent: E-mail: maria.merlino@unime.it

named Niaouli oil or 'broad-leaf paperbarks'. Several chemotypes have been described in Madagascar: a chemotype rich in 1,8-cineole; a chemotype rich in (E)-nerolidol; a chemotype rich in 1,8-cineole, viridiflorol and terpinolene and a chemotype rich in viridiflorol. Niaouli oil with a high content of 1,8-cineole is the most widespread and appreciated chemotype (Ramanoelina $et\ al.$, 2008).

The oil has been used in traditional medicine against many pathological conditions and recent studies have demonstrated different pharmacological effects (Nam et al., 2008; Acha et al., 2019). The global market for Niaouli oil has experienced significant growth in recent years mainly due to the increasing consumer interest in natural and holistic health products.

Recently the high antimicrobial and antioxidant activities of niaouli oil as an alternative to chemical preservatives in the food industry have been demonstrated; it has been indicated as an innovative agent for shelf-life prolonging of different types of food products, such as bread, carrot and potato (Valková et al., 2022) due to its antioxidant properties, pleasant flavour and sensory notes.

To be used as food flavouring, the knowledge of the oil volatile profile is required; this can be obtained using human senses and at the same time, high-resolution advanced analytical techniques. The application of advanced analytical techniques in the volatile studies of essential oils is of great importance since they allow the determination of volatiles even present in trace amounts in a complex mixture.

As regards the Niaouli essential oil, 1,8-cineole chemotype produced in Madagascar different studies have been carried out on its volatile composition even if this dates back to more than 10 years ago (Ramanoelina et al., 1992, 1994, 2005, 2008); moreover, no information is present in the literature on its sensory features, neither on their correlation with the volatile compounds. The composite characteristics of the Niaouli oil are not defined by the International Standardization Organization such happens for other *Melaleuca* varieties, such as *Melaleuca alternifolia* (tea tree oil) terpinen-4-ol type (ISO 4730: 2017).

Considering what was above reported, this research aims to characterise the volatile composition and sensory descriptors of 1,8-cineole chemotype Niaouli essential oil from Madagascar, which represents the most commercially widespread, providing useful elements for its wide application as a flavour ingredient in food products. Advanced analytical techniques such as GC-MS and sensory qualitative descriptive analysis were applied for the essential oil characterisation.

Materials and methods

Materials

Eight samples of *Melaleuca quinquenervia* (Cav.) S.T. Blake essential oil 1,8-cineole type produced in Madagascar, supplied by commercial retailers with attestation of genuineness, have been analysed. According to the producers' information, the essential oils were obtained by hydrodistillation at atmospheric pressure for 3 h, using a Clevenger-type apparatus, and the distilled oils were collected and dried over anhydrous sodium sulphate, filtered snf stored in amber flash. The samples have been analysed in triplicate by Gas chromatography–mass spectrometry (GC-MS) for volatile identification and by Gas chromatography with a flame-ionisation detector (GC-FID) for the quantitative results.

Gas chromatography-mass spectrometry (GC-MS) analysis

The GC-MS analysis was performed using a GC2010 Plus (Shimadzu) gas chromatograph equipped with an SLB-5 ms capillary column (30 m length, 0.25 mm ID, 0.25 µm film thickness), interfaced with a TQMS 8040

triple quadrupole mass spectrometer (Shimadzu). The oven temperature was programmed from 60 °C to 110 °C at a rate of 2 °C/min, from 110 °C to 160 °C at a rate of 3 °C/min and from 160 °C to 260 °C at a rate of 10 °C/min. Helium was used as a carrier gas at a constant flow rate of 0.8 mL/min. The split ratio was 1:200, the injection temperature was set to 260 °C and the transfer line temperature was 250 °C. Mass selective detector operated in electron impact ionisation (EI) mode at 70 eV electron energy, the ion source temperature was set to 230 °C, and the quadrupole temperature was 150 °C. The mass scan range was 40-360 m/z, scan speed of 1250. One microliter of the essential oil, dissolved in hexane (1:100 v/v), was injected. The peak identification has been carried out using mass spectral data, NIST '23' (NIST/EPA/NIH Mass Spectra Library, USA), FFNSC 3.0 database and linear retention indices (LRI) by comparing with those reported in the literature. Linear retention indices were calculated according to the Van den Dool and Kratz equation based on homologue n-alkane hydrocarbons C7–C30 injected under the same operating conditions.

Gas chromatography with flame-ionisation detector (GC-FID) analysis

The GC-FID analysis was performed using a GC2010 Plus (Shimadzu) gas chromatograph equipped with a flame-ionisation detector (FID) set at 290 °C under the same analytical conditions as above reported. The quantification of the compounds was obtained by the internal normalisation method with a response factor equal to unity for all of the sample constituents.

Sensory analysis

A Quantitative Descriptive Analysis (QDA) has been used to identify and quantify the sensory odour descriptors of the Niaouli essential oils. A sensory panel of 12 judges (6 males and 6 females; age range 24–62 years old) has been trained according to the ISO 8586: 2012 at the Aroma and Sensory Laboratory of the Department of Veterinary Sciences of the University of Messina. The members of the panel were selected concerning their experiences in the sensory evaluation of essential oils. The panel participated in eight training preliminary sessions (2-h each, over 2 weeks), the panellists were asked to list the perceived odour descriptors by sniffing a test strip (dipped in the essential oil around 1 cm). During the training sessions, all the samples were evaluated at least twice to generate appropriate descriptors. The final scorecard for odour analysis contained the descriptors selected by at least one-third of the panel. According to this, five attributes were selected and defined by consensus:

6456

'eucalyptus-like, fresh-minty, woody, sweet-floral and spicy' as reported in Table S1. The samples were evaluated in a sensory booth room at a temperature of 20 °C under fluorescent lighting equivalent to the day-light. Each judge was located in an individual booth during each sensory evaluation session. Each of the eight samples of Niaouli essential oils has been evaluated randomly three times over six sessions over 3 weeks. For each sample oil, three sniffs from the test strip were performed and fresh air was breathed between each sample. The panellists were asked to mark the intensity of the descriptors on a 9 cm line scale length. For each sample, the scorecards were decoded, and the mean values of the descriptors were calculated.

DPPH radical scavenging assay

The antioxidant activity of the Niaouli essential oil, was assessed by measuring its ability to scavenge the DPPH stable radical. Different concentrations (20–100 $\mu L/mL)$ of each essential oil were prepared in methanol. Three mL of methanolic solution of DPPH (0.004%) to 0.1 mL of each test concentration was added. The resulting mixtures were incubated in the dark for 30 min at room temperature. The decrease in absorbance was measured at 517 nm using a UV-2250 (Shimadzu) spectrophotometer against a black. Scavenging (%) of DPPH free radical by essential oil was calculated as follows:

Scavenging (%) =
$$(A_{blank} - A_{sample}/A_{blank}) \times 100$$

where A_{blank} is the absorbance of the control reaction (containing all reagents except the test compound) and A_{sample} is the absorbance of the test oil.

All determinations were performed in triplicate. The DPPH scavenging activity of butylated hydroxytoluene (BHT), a widely used antioxidant recognised as safe for use in foods, was evaluated as a comparison.

Statistical analysis

The means values and standard deviations of volatile compound data were calculated using Excel 2019 software (Microsoft, Milan, Italy).

For the QDA analysis, the confidence interval method was used to eliminate the variance associated with the panellists. The odour descriptors were mean-centred per panellist by scaling with unit variance at a 95% confidence level. Then, the mean values and percentiles of the 8 samples were derived from the values assigned by the 12 panellists, and differences between them were analysed by one-way Analysis of Variance ANOVA and Duncan's multiple range test at a confidence level of 95% using XLstat software 2014 (Addinsoft, Paris, France). Frequency histograms of

all odour scores were generated by using Excel 2019 software (Microsoft, Milan, Italy). A spider plot of odours was constructed by using Microsoft Excel 2019 (MS Office 2010, Microsoft Corporation, Seattle, WA, USA) based on the mean scores for the different odours (mean values for 8 samples assessed by 12 panellists).

Results and discussion

Volatile aroma compounds

Table 1 reports the average volatile composition as single compounds and classes of substances of the Niaouli essential oils analysed and a comparison with literature data (Ramanoelina et al., 1992, 1994, 2005, 2008). Figure 1 shows a GC-MS chromatogram of a Niaouli essential oil. Globally, about ninety volatile compounds have been identified by GC-MS analysis, most of these, here for the first time reported. The identified volatiles belonged to different classes of compounds with monoterpenes and sesquiterpenes, both hydrocarbon and oxygenated, being the most represented. Looking at the table the percentage of each volatile agrees with Ramanoelina et al. (2008), at least for the common ones. Among monoterpenes, 1,8-cineole (eucalyptus-like) prevailed, as expected; numerous studies have shown that 1,8-cineole has anti-inflammatory, antioxidant, analgesic and potent antimicrobial properties against a wide range of microorganisms, thus has the potential to be an antibacterial agent for meat preservation et al., 2023).

Limonene (citrus, lemon), α -pinene (pine, hearty, woody) and β -pinene (woody, pine) followed in agreement with Ramanoelina *et al.* (2008). Several monoterpenes were, here, for the first time identified, especially monoterpene alcohols and esters such as α -terpenyl acetate at a level of 2.10% which has been found in Niaouli essential oil 1,8-cineole chemotype from New Caledonia (Trilles *et al.*, 2006).

Sesquiterpenes represented 20.72% of the total volatile fraction; viridiflorol and (E)-nerolidol were the most abundant, responsible for green-sweet and woody-flower notes, respectively; β -caryophyllene (2.06%) and viridiflorene (1.32%) followed. These are typical sesquiterpenes of *Melaleuca* sp. leaf essential oils with a different amount in the four chemotypes of *Melaleuca quinquenervia*. Viridiflorol and viridiflorene are rare aromadendrane tricyclic sesquiterpenoids with a characteristic skeleton, a dimethyl cyclopropane ring fused to a hydroazulene ring system; they have been detected in the extracts of many Myrtaceae and Lamiaceae plants but until now only two plant viridiflorol synthases have been identified (Ntana *et al.*, 2021). Viridiflorol has recently demonstrated

aded from https://ifst.onlinelibrary.wiley.com/doi/10.1111/ijfs.17390 by University Degli Studi Di Messina, Wiley Online Library on [02/09/2024]. See the Terms nditions) on Wiley Online Library for rules of use; OA articles are governed by the applicable Creative Commons License

Table 1 Average composition as single compound and classes of substances of the 1,8 cineole type Niaouli essential oils from Madagascar

Table 1 (Continued)

from	Madagascar					Area			Compounds	LRI	LRI lit.	Area %	SD	Area % Ref. 4	Re
(Compounds	LRI	LRI lit.	Area %	SD	% Ref. 4	Ref.	26	Fenchyl alcohol	1120	1120	0.06	0.01	nd	2
						-		27	trans-Pinocarveol	1141	1141	0.04	0.01	nd	-
ı	Monoterpene hydrod	arbons						28	Isopulegol	1149	1145	0.11	0.02	nd	-
	x-Thujene	924	924	0.20	0.03	nd	1	29	Camphene hydrate	1156	1150	0.02	0.01	nd	-
	x-Pinene	932	932	9.38	0.52	10.92	1,2,3,4	30	<i>iso</i> -Isopulegol	1159	1160	0.05	0.01	nd	-
	x-Fenchene	947	944	0.03	0.01	nd	-	31	δ-Terpineol	1171	1165	0.20	0.01	nd	-
. (Camphene	948	948	0.11	0.01	nd	2	32	Borneol	1172	1173	0.06	0.01	nd	-
: :	Sabinene	971	969	0.01	0.01	nd	2	34	Terpinen-4-ol	1181	1179	1.05	0.26	0.71	1
ı f	3-Pinene	977	977	2.73	0.14	2.84	2,3,4	35	<i>p</i> -Cymen-8-ol	1188	1183	0.05	0.01	nd	-
1 [Myrcene	988	987	0.89	0.05	0.89	1,2,3,4	36	α-Terpineol	1196	1199	7.10	0.84	6.35	1
2 /	<i>p</i> -Menta-1(7),8-	1006	1008	0.07	0.01	nd	_	37	Citronellol	1229	1228	0.07	0.01	nd	-
	diene							38	Geraniol	1253	1253	0.02	0.01	nd	_
3 (x-Phellandrene	1007	1003	0.07	0.01	nd	1,2	43	Eugenol	1353	1356	0.05	0.01	nd	_
	5-3-Carene	1009	1010	0.01	0.01	nd	2		Total			9.12	1.25	7.30	
	x-Terpinene	1016	1016	0.27	0.02	nd	2		Epoxides						
	p-Cymene	1010	1015	1.82	0.02	1.06	2,3,4	25	cis-Rose oxide	1112	1112	0.02	0.01	nd	_
	Limonene	1023	1025	9.18	0.84	8.12	1,2,3,4	-	Esters				-		
		1030	1030	0.04	0.84		1,2,3,4	40	δ-Terpineol acetate	1314	1316	0.04	0.01	nd	_
	(E)-β-Ocimene					nd 171		41	Myrtenyl acetate	1324	1328	0.02	0.01	nd	_
	y-Terpinene	1056	1056	1.17	0.07	1.71	2,3,4	42	α-Terpinyl acetate	1348	1350	2.10	0.34	nd	_
	Terpinolene	1084	1086	0.59	0.03	0.68	1,2,3,4	42	Total	1040	1000	2.16	0.34	iiu	_
•	p-Cymenene	1090	1090	0.05	0.01	nd	_		Eters			2.10	0.30		
	Total			26.62	1.86	26.22		18	1,8-Cineole	1033	1033	44.06	1.19	55.38	1
	Sesquiterpene hydro							18	•		1033	44.06	1.19	55.38	1,
	soledene	1371	1371	0.01	0.01	nd	-		Oxygenated sesquite	rpenes					
5 c	x-Copaene	1376	1374	0.13	0.01	nd	2		Alcohols						
6 (x-Gurjunene	1406	1406	0.17	0.02	0.04	1,2,3,4	68	<i>epi</i> -Longipinalol	1557	1557	0.04	0.01	nd	-
7 f	3-Caryophyllene	1418	1417	2.06	0.14	1.37	1,2,3,4	69	(E)-Nerolidol	1562	1563	3.18	0.29	2.62	1,
8 /	Aromadendrene	1438	1438	0.06	0.01	nd	2	70	Palustrol	1570	1570	0.25	0.03	nd	-
9 (x-Guajene	1439	1432	0.17	0.01	nd	_	71	Thujopsan-2-α-ol	1582	1579	0.38	0.05	nd	-
0 /	Amorpha-4,11-	1441	_	0.03	0.01	nd	_	73	Viridiflorol	1594	1591	2.59	0.57	4.98	1,
	diene							74	Cubeban-11-ol	1597	1592	0.26	0.03	nd	_
1 9	Selina-5,11-diene	1445	1441	0.02	0.01	nd	_	75	Copaborneol	1606	1592	0.11	0.07	nd	_
	x-Humulene	1454	1452	0.37	0.04	nd	1,2	77	Eremoligenol	1630	1631	0.04	0.01	nd	_
	Alloaromadendrene	1459	1460	0.59	0.05	nd	2	78	epi-Cubenol	1635	1631	0.02	0.01	nd	_
	Cadina-1(6),4-diene	1472	1475	0.03	0.01	nd	_	79	, γ-Eudesmol	1639	1637	0.06	0.01	nd	_
	Selina-4,11-diene	1473	-	0.03	0.01	nd	_	80	α-Caryophylladienol	1645	1645	0.03	0.01	nd	_
	y-Muurolene	1475	- 1477	0.04	0.01	nd	_	81	epi-α-Cadinol	1650	1656	0.08	0.01	nd	3
	•							82	Cadin-4-en-10-ol	1652	1658	0.02	0.01	nd	-
	3-Selinene	1488	1490	0.46	0.06	nd 0.70	1	83	α-Muurolol	1655	1643	0.02	0.01	0.06	4
	Viridiflorene	1491	1493	1.32	0.17	0.73	1,2,3,4	84	β-Eudesmol	1664	1664	0.01	0.01	nd	4
	x-Selinene	1495	1494	0.30	0.04	nd	_		•		1672	0.16			_
	x-Muurolene	1498	1499	0.07	0.01	nd	-	85	Bulnesol	1673	10/2		0.01	nd 7.66	_
	3-Bisabolene	1508	1509	0.01	0.01	nd	-		Total			9.97	1.15	7.66	
	γ-Cadinene	1513	1513	0.25	0.03	0.23	1,2,3,4		Epoxides	4500	450.	0.00	0.01	0.00	_
3 8	6-Cadinene	1519	1522	0.30	0.01	0.23	1,2,3,4	72	Caryophyllene	1586	1584	0.09	0.01	0.29	1,
4 i	<i>trans</i> -Calamenene	1521	1520	0.03	0.01	nd	2		oxide						
5 t	<i>trans</i> -Cadina-1,4- diene	1532	1530	0.02	0.01	nd	2	76	Humulene epoxide II	1612	1603	0.05	0.01	nd	-
6 (x-Cadinene	1537	1541	0.03	0.01	nd	2		Total			0.14	0.02	0.29	
	(E)-α-Bisabolene	1541	1547	0.01	0.01	nd	_		Aliphatic compounds	•					
	Total			6.53	0.72	2.60			Alcohols						
	Oxygenated monote	rpenes		0.00	J., Z			1	(Z)-4-Hexen-1-ol	873	879	0.02	0.01	nd	_
	Carbonyl compounds	•							Ketones						
	<i>cis</i> -Pinocamphone	1179	_	0.06	0.01	nd	_	10	6-Methyl-5-hepten-	986	986	0.03	0.01	nd	_
	Alcohols	11/3	_	0.00	0.01	IIu	_		2-one						
		1100	1101	0.24	0.02	0.24	1224		Hydrocarbons						
4 I	Linalool	1102	1101	0.24	0.03	0.24	1,2,3,4		,						

^{© 2024} The Author(s). International Journal of Food Science & Technology published by John Wiley & Sons Ltd International Journal of Food Science and Technology 2024 on behalf of Institute of Food Science & Technology (IFST).

1-Nitro-2-Phenyl

Hydrocarbons

ethane

Styrene

6458

	•		LRI	Area	0.0	Area % Ref.	5.
	Compounds	LRI	lit.	%	SD	4	Ref.
86	Eicosane Aromatic compoun	1997 ds	2000	0.53	0.06	nd	-
7	Aldehydes Benzaldehyde Esters	962	962	0.29	0.05	0.19	1,2,3,4
23	Methyl benzoate Nitrogen compound	1096 ds	1094	0.06	0.01	nd	-

1300

0.02

0.01 nd

 $LRI = Linear \ Retention \ Index \ calculated \ on \ SLB-5 \ capillary \ column; \ LRI \ lit. = Linear \ Retention \ Index \ from \ Literature; \ Area \ \% = average \ of \ all \ the \ essential \ oil \ samples \ analysed \ in \ triplicate; \ Area \ \% \ Ref.$

1293

897 897

nd, not determined; SD, Standard Deviation.

anti-neoplastic properties inducing apoptosis of breast, lung and brain cancer cells (Akiel et al., 2022). (E)-Nerolidol is an acyclic sesquiterpene widely spread in the essential oil of various medicinal plants which are well known for their pharmacological and biological activities; for this reason, the U.S. Food and Drug Administration (FDA) allows its use as a food flavouring agent (Chan et al., 2016). β-caryophyllene is a bicyclic sesquiterpene widely spread in nature, too; it is responsible for woody, sweet, spicy notes and it has wide applications as food flavouring. Moreover, sesauiterpenes are described as possessing inflammatory and antibacterial properties including enhancement of bacterial susceptibility to antibiotics, and to exhibit anticarcinogenic and antioxidant effects (Cincotta et al., 2015). Among the sesquiterpenes identified for the first time, α -selinene and α -bisabolene isomers were present. α-Selinene is one of the major sesquiterpenes in the celery volatile profile (Naghneh $et\ al.,\ 2023$) while α -bisabolene is a primary component in black pepper (Eddin $et\ al.,\ 2022$).

Sensory analysis

The QDA revealed different perceived odours with different intensities (Fig. 2a). According to the judges' evaluation, the oils have been characterised by the following descriptors: 'woody, fresh-mint, eucalyptus-like, sweet-floral and spicy'. The descriptors 'eucalyptus-like and woody' odour showed the highest mean scores followed by 'fresh-minty' odour.

Further, 'eucalyptus-like' showed the highest median score, with the narrowest lowest-to-highest range and interquartile range; the descriptor of 'fresh-minty' odour was second to this (Fig. 2b); their intensity was the most stable and the highest.

'Eucalyptus-like' odour is mainly related to the high content of 1,8-cineole or eucalyptol while 'fresh-minty' note could be related to monoterpene alcohols such as α -terpineol. 'Woody' odour could be attributable to the presence of monoterpenes and sesquiterpene hydrocarbons including α and β -pinene, p-cymene, γ -terpinene, β -caryophyllene and (E)-nerolidol; the 'sweet-floral' odour could be related to oxygenated terpenes such as (E)-nerolidol whereas 'spicy' to sesquiterpenes such as β -caryophyllene and germacrene D which are responsible for 'woody and spicy' notes. Hence, the typical aromatic characteristics of Niaouli oil were mostly attributed to the dominant 'eucalyptus-like' and 'fresh-minty' odour.

Antioxidant activity

The antioxidant activity of the Niaouli essential oils was assessed using the DPPH assay. The % inhibition was found to increase from 4.66% to 20.02% with the increase in the concentration of the essential oil from 10 to 100 μ L/mL. The oils were found to exhibit similar antioxidant activity if compared to BHT (Table 2). This could be due to other constituents such as terpenoids with reactive sites such as hydroxyl

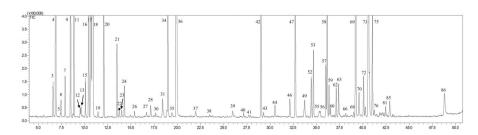


Figure 1 HS-SPME-GC-MS chromatogram of 1,8-cineole type Niaouli essential oils from Madagascar. The identification of peaks is reported in Table 1.

^{4 =} average composition of 1,8-cineole type Niaouli essential oil from madagascal reported by Ramanoelina *et al.*, 2008; Ref. = (1) Ramanoelina *et al.*, 1992; (2) Ramanoelina *et al.*, 1994; (3) Ramanoelina *et al.*, 2005; (4) Ramanoelina *et al.*, 2008.

13652621

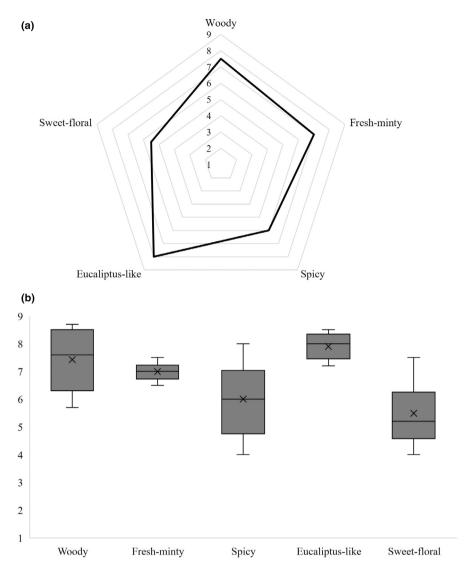


Figure 2 Graphical representation of the average data of QDA analysis (a) and score percentiles box for the odours (b) assigned by 12 panel-lists of all the 1,8-cineole type Niaouli essential oils from Madagascar analysed.

Table 2 Antioxidant activity of Niaouli essential oils measured in terms of DPPH radical scavenging

	Concentration (μL/mL)	Niaouli oils	SD	ВНТ
DPPH (%)	10	4.66	0.23	6.18
	20	6.76	0.59	9.22
	50	11.06	0.97	17.42
	100	20.02	1.34	31.98

BHT, antioxidant activity of butylated hydroxytoluene; SD, Standard Deviation.

groups useful for electron transfer which can contribute to the antioxidant potential of Niaouli essential oil (Bakkali *et al.*, 2008).

Conclusions

The consumer request for natural ingredients in food and beverages is increasing and the market of natural flavourings is expected to witness significant growth in the next years. In this context, the characterisation of the 1,8-cineole type Niaouli essential oil from Madagascar using the volatile and odour profile results in considerable interest. Using advanced analytical techniques, many volatiles, especially sesquiterpenes, have been identified. The Quantitative Descriptive Sensory Analysis has been able to identify the descriptors that characterised the oil; volatile compounds and odour descriptors resulted in agreement. Considering its volatile composition, the pleasant sensory notes of

^{© 2024} The Author(s). International Journal of Food Science & Technology published by John Wiley & Sons Ltd International Journal of Food Science and Technology 2024 on behalf of Institute of Food Science & Technology (IFST).

'eucalyptus-like' and 'fresh-minty' and, the antioxidant activity, the 1,8-cineole type Niaouli essential oil from Madagascar could be considered as a flavouring agent and natural preservative in the food industry mainly in the field of candy, baked products, meat and beverages.

Author contributions

Maria Merlino: Methodology; investigation; writing – original draft. Fabrizio Cincotta: Investigation; data curation; visualization; supervision. Antonella Verzera: Conceptualization; writing – review and editing; methodology; data curation; resources. Anthea Miller: Formal analysis. Marco Torre: Formal analysis. Concetta Condurso: Conceptualization; writing – review and editing; supervision.

Conflict of interest

There is no conflict of interest.

Ethical approval

Ethics approval was not required for this research. All participants involved in the sensory analysis signed an informed consent according to the principles of the Declaration of Helsinki before the beginning of the study.

Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

References

- Acha, E., Ahounou Aikpe, J.F., Adovelande, J., Assogba, M.F., Agossou, G. & Sezan, A. (2019). Anti-inflammatory properties of Melaleuca quinquenervia (Cav.) ST Blake Myrtaceae (Niaouli) leaves' essential oil. International Journal of Current Research in Chemistry and Pharmaceutical Sciences, 6, 30–40.
- Niaouli essential oil has been used in traditional medicine against many pathological conditions and recent studies have demonstrated different pharmacological effects (Acha *et al.*, 2019).
- Akiel, M.A., Alshehri, O.Y., Aljihani, S.A. *et al.* (2022). Viridiflorol induces anti-neoplastic effects on breast, lung, and brain cancer cells through apoptosis. *Saudi Journal of Biological Sciences*, **29**, 816–821.
- Bakkali, F., Averbeck, S., Averbeck, D. & Idaomar, M. (2008). Biological effects of essential oils—a review. *Food and Chemical Toxicology*, **46**, 446–475.
- Brophy, J.J., Craven, L.A. & Doran, J.C. (2013). *Melaleucas: their Botany, Essential Oils and Uses. Monograph No. 156.* Canberra, Australia: ACIAR Australian Centre for International Agricultural Research.
- Chan, W.K., Tan, L.T.H., Chan, K.G., Lee, L.H. & Goh, B.H. (2016). Nerolidol: a sesquiterpene alcohol with multi-faceted pharmacological and biological activities. *Molecules*, **21**, 529.

- Cincotta, F., Verzera, A., Tripodi, G. & Condurso, C. (2015). Determination of sesquiterpenes in wines by HS-SPME coupled with GC-MS. *Chromatography*, **2**, 410–421.
- Eddin, L.B., Jha, N.K., Goyal, S.N. *et al.* (2022). Health benefits, pharmacological effects, molecular mechanisms, and therapeutic potential of α-bisabolol. *Nutrients*, **14**, 1370.
- ISO (2012). Sensory Analysis. General Guidance for the Selection, Training, and Monitoring of Assessors. Geneva (ISO 8586): International Organization for Standardization.
- ISO (2017). Essential Oil of Melaleuca, Terpinen-4-ol Type (Tea Tree Oil). Geneva (ISO 4730): International Organization for Standardization.
- Naghneh, N.S., Rafiei, F., Shahbazi, E. & Gheisari, M.M. (2023).Variation of volatile compounds in celery through different vegetative stages. *Plant Physiology Reports*, 28, 476–480.
- Nam, S.Y., Chang, M.H., Do, J.S., Seo, H.J. & Oh, H.K. (2008). Essential oil of niaouli preferentially potentiates antigen-specific cellular immunity and cytokine production by macrophages. *Immu-nopharmacology and Immunotoxicology*, 30, 459–474.
- Niaouli essential oil has been used in traditional medicine against many pathological conditions and recent studies have demonstrated different pharmacological effects (Nam *et al.*, 2008).
- Ntana, F., Bhat, W.W., Johnson, S.R. *et al.* (2021). A sesquiterpene synthase from the endophytic fungus Serendipita indica catalyzes formation of viridiflorol. *Biomolecules*, **11**, 898.
- Ramanoelina, P.A., Bianchini, J.P., Andriantsiferana, M., Viano, J. & Gaydou, E.M. (1992). Chemical composition of niaouli essential oils from Madagascar. *Journal of Essential Oil Research*, **4**, 657–658.
- Ramanoelina, P.A., Viano, J., Bianchini, J.P. & Gaydou, E.M. (1994). Occurrence of various chemotypes in niaouli (*Melaleuca quinquenervia*) essential oils from Madagascar using multivariate statistical analysis. *Journal of Agricultural and Food Chemistry*, **42**, 1177–1182.
- Niaouli essential oil with a high content of 1,8-cineole is the most widespread and appreciated chemotype. Different studies have been carried out on Niaouli essential oil with a high content of 1,8-cineole volatile composition even if this dates back to more than ten years ago (Ramanoelina *et al.*, 1994).
- Ramanoelina, P.A., Gaydou, E.M. & Bianchini, J.P. (2005). Caractérisation des huiles essentielles industrielles de niaouli (*Melaleuca quinquenervia*) de Madagascar-Propositions d'Avant-projet de Normes. *Terre Malgache*, **24**, 59–91.
- Ramanoelina, P.A., Bianchini, J.P. & Gaydou, E.M. (2008). Main industrial niaouli (*Melaleuca quinquenervia*) oil chemotype productions from Madagascar. *Journal of Essential Oil Research*, 20, 261– 266
- Niaouli essential oil with a high content of 1,8-cineole is the most widespread and appreciated chemotype. Different studies have been carried out on Niaouli essential oil with a high content of 1,8-cineole volatile composition even if this dates back to more than ten years ago (Ramanoelina *et al.*, 2008).
- Sobhy, M., Ali, S.S., Cui, H., Lin, L. & El-Sapagh, S. (2023). Exploring the potential of 1,8-cineole from cardamom oil against food-borne pathogens: antibacterial mechanisms and its application in meat preservation. *Microbial Pathogenesis*, **184**, 106375.
- Trilles, B.L., Bombarda, I., Bouraïma-Madjebi, S., Raharivelomanana, P., Bianchini, J.P. & Gaydou, E.M. (2006). Occurrence of various chemotypes in niaouli [Melaleuca quinquenervia (Cav.) ST Blake] essential oil from New Caledonia. Flavour and Fragrance Journal, 21, 677–682.
- Valková, V., Ďúranová, H., Vukovic, N.L., Vukic, M., Kluz, M. & Kačániová, M. (2022). Assessment of chemical composition and anti-penicillium activity of vapours of essential oils from Abies Alba and two melaleuca species in food model systems. *Molecules*, 27, 3101
- Wheeler, G.S., Pratt, P.D., Giblin-Davis, R.M. & Ordung, K.M. (2007). Intraspecific variation of *Melaleuca quinquenervia* leaf oils

in its naturalized range in Florida, the Caribbean, and Hawaii. *Biochemical Systematics and Ecology*, **35**, 489–500.

The leaf essential oils of various *Melaleuca* species differ in composition, also due to pedoclimatic conditions; methyl eugenol, (E)-methyl isoeugenol, 1,8-cineole, terpinen-4-ol, terpinolene, (E)-nerolidol, viridiflorol, ledol, β -caryophyllene have been reported as the main volatile compounds (Wheeler *et al.*, 2007).

Wilson, P.G., O'Brien, M.M., Gadek, P.A. & Quinn, C.J. (2001).
Myrtaceae revisited: a reassessment of infrafamilial groups. American Journal of Botany, 88, 2013–2025.

Supporting Information

Additional Supporting Information may be found in the online version of this article:

Table S1. Lexicon used to describe the sensory characteristics of the 1,8-cineole type Niaouli essential oils from Madagascar.