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Short communication

Rare sesquiterpenes from South African *Pteronia* species

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Abstract

The genus *Pteronia* consists of approximately 80 species which are widely distributed in southern Africa. The essential oils isolated from the aerial parts of eleven species, analyzed by GC-MS varied both qualitatively and quantitatively. In *Pteronia pallens*, *Pteronia empetrifolia* and *Pteronia flexicaulis* uncommon sesquiterpenes such as presilphiperfolol-7-ene, 7-α-(H)-silphiperfol-5-ene, 7-β-(H)-silphiperfol-5-ene, α-campholene aldehyde, silphiperfol-5-ene, cameroonan-7-α-ol, silphiperfol-7-β-ol, presilphiperfolan-9-α-ol and presilphiperfolan-8-ol (a major compound in *P. pallens*) were identified. Cluster analysis based of the chemical composition of the oils revealed that individual plants of *Pteronia camphorata* collected in the same population had similar oil profiles with a high correlation coefficient ($S_{corr} \approx 0.98$). Similarly, the essential oil composition of *P. pallens* collected from two distinct localities also showed high levels of congruency ($S_{corr} \approx 0.99$).

Keywords: 7-α-(H)-silphiperfol-5-ene; 7-β-(H)-Silphiperfol-5-ene; Essential oil; Presilphiperfolan-8-ol; Presilphiperfolan-9-α-ol; Presilphiperfolol-7-ene; *Pteronia*; GC-MS; Silphiperfol-5-ene; Silphiperfol-7-β-ol; α-Campholene aldehyde

1. Introduction

Pteronia (L.) L. (Asteraceae) is represented by approximately 80 species, which are primarily found in southern Africa. The species are most abundant in the Karoo-Namib region although 12 species occur in the Fynbos biome of South Africa (Goldblatt and Manning, 2000). Members of this genus are often the dominant taxa in the plant communities in which they are found.

The morphology of the genus is diverse with regards to habit, flowers and leaf shape. They are mainly perennial and woody shrubs ranging from 0.3 to 1.5 m in height. The leaf texture varies from smooth, lacking hair and bristles (glabrous) to hairy, with leaves either opposite or alternate (Shearing, 1997).

Two species; *Pteronia pallens* and *Pteronia paniculata* are unpalatable species that dominate the veld in large parts of the Little Karoo. Another well documented characteristic of *Pteronia* (e.g. *P. pallens*) is that it is highly toxic to ungulate herbivores, and may be lethal to cattle.

Aromatherapy (uses of volatiles from plants for improvement of mood or health) is characteristic of the San culture of South Africa. The name San was used to refer to one of the most preferred species *Pteronia ombromoides* (Van Wyk, 2008). Since ancient times, the Khoi-San have been using *Pteronia* species which have been amongst the first plants to be used for their anti-infective properties and have also been used in the treatment of stomach ailments (cramps) and nausea (Shearing, 1997). Many species have also shown anti-insecticidal properties (Shearing, 1997). Considering the extensive historic use of *Pteronia* in traditional medicine, it is ironic that the volatiles of this genus remain poorly investigated.

2. Material and methods

2.1. Plant studied

Eleven species (14 samples) were collected from various areas in the eastern and southern Cape of South Africa (Table 1). All

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Table 1
List of species included in this study with voucher and locality information.

Species	Voucher number	Location
P. adenocarpa Harv.	Vlok 2822	Frinsgeword
P. camphorata (L.)L.	Vlok 2804	Montagu Pass near Herold
P. elongate Thunb.	Vlok 2819	Prins Albert on Klaarstroom road
P. empetrifolia Dc.	Vlok 2818	Prins Albert
P. fasciculata L.f.	Vlok 2814	Bakenshoogte
P. flexicaulis L.f.	Vlok 2805	Southern Hills near Daskop
P. glauca Thunb.	Vlok 2812	Lategansvlei
P. glomerata L.f.	Vlok 2824	West of Klaarstroom
P. pallens L.f.	Vlok 2815	Volmoed
P. pallens L.f.	Vlok 2816	Between Oudtshoorn and Volmoed
P. paniculata Thunb.	Vlok 2813	Bakenshoogte
P. viscosa Thunb.	Vlok 2817	Prins Albert

plants were identified by J Vlok, who also assisted in preparing the voucher specimens which are housed in the Department of Pharmaceutical Sciences (Tshwane University of Technology, Pretoria). Three individual samples were collected in the same population for *Pteronia camphorata* and two samples of *Pteronia pallens* were collected at different localities to assess possible within and between population variation.

2.2. Isolation of the essential oils

The aerial part of each plant was air-dried at room temperature and the oil was isolated by hydro-distillation using a Clevenger apparatus together with 500 ml of water over 4 h. The resultant oil was stored at 4 °C until analysis was carried out.

2.3. Gas chromatography coupled to mass spectroscopy (GC-MS)

The analysis of the essential oil was performed using a Hewlett-Packard GCD system equipped with a HP-Innowax column (60 m × 0.25 mm ∅, with 0.25 μm film thickness). Temperature at the injection port was 250 °C. Column temperature was initially 60 °C for 10 min then gradually increased to 220 °C at a 4 °C/min. Temperatures were held at 220 °C for 10 min until it was finally raised to 240 °C at a rate of 1 °C/min. Helium was used as carrier gas at a flow rate of 0.7 mL/min. An electron ionization system with an ionization energy of 70 eV was used with a split ratio of 50:1. The mass range was between 35 and 425 m/z. Percentage compounds were calculated by the computer from total ion chromatograms. Component identifications were performed by comparing their mass spectra and retention indices using the Başer Library of Essential Oil Constituents.

The percentage composition of the essential oil samples was used to determine the similarity between samples by cluster analysis using the NTSYS software (Rohlf, 1992). Correlation was selected as a measure of similarity and the unweighted pair-group method with arithmetic average (UPGMA) was used for cluster definition. The degree of correlation was evaluated according to Pestana and Gageiro (2000) where a very high correlation ranged between 0.90 and 1.00, high between 0.70 and 0.89, moderate between 0.40 and 0.69, low between 0.20 and 0.39 and very low if less than 0.20.

3. Results and discussion

The essential oil composition is given in Table 2. A total of 86 compounds representing 91.8% to 98.8% of the oil were identified. The following monoterpenes were present in all the oils; α-pinene (0.2–12.0%), β-pinene (0.7–32.1%), sabinene (2.0–33.6%), limonene (2.7–13.0%), p-cymene (0.5–21.1%), α-terpineol (0.1–4.8%) and terpinen-4-ol (0.8–9.2%). However, some major and minor compounds differ extensively within the genus *Pteronia* (Table 2). This confirms findings of Zedro et al. (1990) that the diterpenes and other compounds commonly present in *Pteronia* species are not uniform although there are some similarities.

In *P. pallens*, *Pteronia empetrifolia* and *Pteronia flexicaulis*, uncommon compounds characterise the essential oil; presilphiperfol-7-ene, 7-α-(H)-silphiperfol-5-ene, 7-β-(H)-silphiperfol-5-ene, α-campholene aldehyde, cameroonan-7-α-ol, silphiperfol-5-ene, silphiperfolan-7-β-ol, presilphiperfolan-9-α-ol and presilphiperfolan-8-ol (a major compound in *P. pallens*) (Table 2). Some of these compounds have previously only been recorded once in the essential oil literature, and have been isolated from *Eryophyllen staechadifolium* (König et al., 1997). Presilphiperfol-7-ene was first isolated from *Echinops giganteus* (Asteraceae) which is endemic to Cameroon and Nigeria (Weyerstahl et al., 1997a). It was also reported that cameroonan-7-α-ol also identified in *Pteronia* species contributed significantly to the strong woody amber-like odour of *E. giganteus*. α-Campholene aldehyde, another rare sesquiterpene (Fig. 1) was previously detected in the oil of *Juniperus oxycedrus* subsp. *macrocarpa* and *Salvia cyanescens* and represented 1.7–3.2% and 2.2% of the total oil composition, respectively (Sezik et al., 2005; Lucero et al., 2006). Presilphiperfolan-9-α-ol was reported in oils of *Artemisia chamaemelifolia* Vill. (Marco et al., 1996), *Artemisia laciniata* Willd. (Weyerstahl et al., 1997b) and *Haplopappus greenei* (Asteraceae) (Demirci et al., 2006), while presilphiperfolan-8-ol has been found in the hexane and ethanol rhizome extracts of *Silphium perfoliatum* and *Silphium integrifolium* in high levels (4.8–13.0%) (Kowalski, 2009).

In order to investigate variation between plants from the same population, three samples of *P. camphorata* were collected and the essential oil composition determined. The cluster analysis indicates that all three collections of *P. camphorata* are tightly grouped together (Fig. 2), with a very high correlation coefficient ($S_{corr} \approx 0.98$).

The variation between samples from different populations was also investigated for *P. pallens* which were collected at two different localities. The cluster analysis indicates that these samples all grouped together, with a high correlation ($S_{corr} \approx 1$) (Fig. 2), demonstrating that the oils are quantitatively and qualitatively similar in their composition for these species.

Table 2
Essential oil composition of the hydro-distilled essential oils as determined by GC-MS.

RRI	Compound	*P. adenocarpa*	*P. camphorata* A	*P. camphorata* B	*P. camphorata* C	*P. elongata*	*P. empetrifolia*	*P. fasciculata*	*P. flexicaulis*	*P. glauca*	*P. glomerata*	*P. pallens* (Volmoed)	*P. pallens* (between Oudtshoorn and Volmoed)	*P. paniculata*	*P. viscosa*
	Yield	0.43	0.29	0.19	0.71	0.15	0.48	0.14	0.11	0.51	0.2	0.28	0.55	0.34	0.28
952	1-Nonene	–	–	–	–	0.5	–	–	tr	–	–	–	–	–	–
1032	α-Pinene	4.8	0.5	0.2	0.7	4.5	10.3	8.7	5.1	4.7	10.2	4.2	5.0	12.0	2.4
1035	α-Thujene	–	0.3	–	0.1	–	–	–	–	–	2.1	–	–	–	–
1048	2-Methyl-3-buten-2-ol	–	–	–	–	2.2	–	–	–	–	–	–	–	–	–
1118	β-Pinene	25.4	1.1	0.7	0.9	28.6	14.5	15.8	32.1	26.9	14.3	21.6	22.5	19.0	14.9
1132	Sabinene	3.3	9.1	7.1	12.7	7.8	2.5	2	2.8	33.6	13.3	32.2	30.8	22.0	9.0
1142	Undecene	–	–	–	–	–	0.4	–	–	–	–	–	–	–	–
1174	Myrcene	2.9	–	–		12.4	4.7	13.8	17.6	1.1	2.8	1.3	0.9	2.1	10.4
1176	α–Phellandrene	–	1.6	1.7	5.5	–	–	–	–	–	–	–	–	–	–
1188	α–Terpinene	–	–	0.1	0.3	0.7	–	–	0.5	0.7	1.3	0.3	0.6	0.7	0.7
1203	Limonene	2.8	5.0	3.8	7.7	3.0	11.2	2.7	6.1	3.3	3.2	4.6	4.3	13	2.7
1213	1,8-Cineole	23.5	42.7	40.4	42.6	–	2.5	–	–	–	7.7	–	–	–	37
1218	β-Phellandrene	–	–	–	–	11.2	–	0.2	2.2	1.2		1.2	0.8	1.9	0.2
1246	Z-(β)-Ocimene	0.6	1.0	0.8	1.5	3.8	0.1	–	–	–	0.3	0.4	–	–	–
1255	δ-Terpinene	0.3	1.2	0.4	0.7	–	–	–	–		–	–	0.1	–	1.7
1255	γ-Terpinene	–	–	–	–	1.5	–	–	0.3		2.5	–	–	1.1	–
1266	(E)-β-ocimene	0.2	0.2	tr	0.1	1.4	0.3	0.9	–		1.1	1.0	1.4	0.5	0.9
1280	p-Cymene	14.3	17.1	21.1	10.0	3.6	9.8	9.2	1.3		9.9	0.5	1.1	2.9	1.7
1290	Terpinolene	0.2	0.3	–	0.1	0.5	0.1	–	0.3		0.6	0.1	tr	0.3	0.4
1327	3-Methyl-2-butenol	–	–	–	–	0.6	–	–	–		0.1	–	–	–	–
1399	Methyl-octanoate	–	–	–	–	0.1	0.1	–	–		0.2	–	–	–	0.7
1406	Presilphiperfol-7-ene	–	–	–	–	–	–	–	0.4		–	0.4	0.3	–	–
1424	7-α-(H)-Silphiperfol-5-ene	–	–	–	–		–	–	–		–	–	0.1	–	–
1435	β-bourbonene	–	–	–	–	–	–	–	0.5		–	–	–	–	–
1452	7 β-(H)-Silphiperfol-5-ene	–	–	–	–	–	–	–	0.8		–	–	0.1	–	–
1466	α-Cubebene	–	–	–	–	–	–	–	0.5		–	–	–	–	–
1474	*trans*-Sabinene hydrate	0.1	0.4	0.8	0.6	–	0.1	–	–		0.4	1.1	1.4	1.2	0.2
1479	Bicycloelemene	–	–	–	–	–	0.1	–	–		0.1	1.1	tr	0.1	–
1495	Silphiperfol-5-ene	–	–	–	–	–	–	–	1.7		–	0.9	0.9	–	–
1499	α-Campholene aldehyde	tr	–	–	–	–	0.4	–	–		–	–	tr	–	–
1553	Linalool	0.1	0.4	2.8	3.1	0.2	–	–	–		0.1	–	–	–	0.1
1556	*cis*-Sabinene hydrate	tr	–	–	–	–	–	–	–		0.2	0.8	0.8	1.0	
1571	*trans*-p-Menth-2-en-1-ol	0.3	0.3	0.1	0.2	0.3	0.1	–	tr		0.4	0.1	0.1	0.5	0.3
1583	Nopinone	0.5	–		–	0.1	0.3	–	–		0.1	0.1	0.2	–	–
1586	Pinocarvone	0.7	0.1	–	–	0.1	0.6	–	0.1		–	0.1	0.4	–	–
1600	Terpinen-4-ol	3.6	5.8	3.1	2.4	7.0	0.8	6.3	2.3		9.5	2.2	2.4	9.2	8.4

Table 2 (*continued*)

RRI	Compound	*P. adenocarpa*	*P. camphorata* A	*P. camphorata* B	*P. camphorata* C	*P. elongata*	*P. empetrifolia*	*P. fasciculata*	*P. flexicaulis*	*P. glauca*	*P. glomerata*	*P. pallens* (Volmoed)	*P. pallens* (between Oudtshoorn and Volmoed)	*P. paniculata*	*P. viscosa*
	Yield	0.43	0.29	0.19	0.71	0.15	0.48	0.14	0.11	0.51	0.2	0.28	0.55	0.34	0.28
1632	*cis*-p-Menth-2-en-1-ol	0.2	–	–	–	0.2	0.2	–	tr		0.3	–	–	0.3	0.2
1639	*trans*-p-Menth-2, 8-dien-1-ol	–	0.4	0.4	–	–	–	–	–		–	–	–	–	–
1648	Myrtenal	–	0.1	0.2	0.1	0.3	0.8	–	0.1		tr	0.3	0.6	–	–
1651	Sabinaketone	–	0.3	0.2	0.2	–	–	–	–		–	–	0.6	–	–
1661	*trans*-Pinocarveol	2.3	0.2	0.2	0.1	0.4	1.5				0.1	0.5	1		0.1
1671	Methyl chavicol (=estragol)	–	4.5	6.8	2.9	–	–	–	–		–	–	–	–	–
1682	α-Terpineol	1.7	2.3	4.8	3.9	1.9	0.8	1.3	1.1		1.4	0.1	0.2	1.6	1.5
1687	α-Humulene	–	–	–	–	–	–	–	–		–	0.3	0.5	–	–
1689	*trans*-Piperitol	–	–	–	–	–	–	–	–		0.2	–	–	–	0.1
1690	Cryptone	0.4	–	–	–	0.2	–	–	–		–	–	–	–	–
1726	Verbenone	0.3	–	–	–	–	0.7	–	–		–	–	–	–	–
1726	Germacrene D	–	–	–	–	–	–	–	0.1		–	0.8	0.9	1.1	–
1740	α-Muurolene	–	–	–	–	–	1.2	–	–		0.1	–	–	–	–
1743	α-Cadinene	0.3	–		–	–	–	–	0.4		–	–	–	–	–
1751	Carvone	0.5	1.2	0.1	0.7	–	–	–	–		–	–	–	–	–
1755	Bicyclogermacrene	–	–	–	–	0.8	1.5	–	0.2		2.8	1.3	0.7	2.4	
1773	δ-Cadinene	0.4	0.1		0.1		2.1	–	0.1		0.3	tr	0.1	0.8	0.3
1776	γ-Cadinene	–	–	0.1	–	–	–	–	–		0.1	0.1	0.2	–	–
1786	Kessane	0.1	–	–	–	0.1	1.5	–	0.1		0.5	0.6	0.7	–	–
1797	p-Methyl acetophenone	0.1	0.5	0.5	–	–	–	–	–		–	–	–	–	–
1802	Cumin aldehyde		0.4	0.4	0.2	–	–	–	–		–	–		–	–
1804	Myrtenol	1.4	–	0.2	0.1	0.3	0.8	–	0.2		0.2	0.4	0.7	–	–
1804	Liguloxide	0.3	–	–	–	0.1	1.4	–	–		0.2	0.5	0.6	–	–
1834	*trans*-Carveol	0.2	0.4	0.3	0.2	–	0.5	–	–		–	0.3	0.1	–	–

1864	p-cymen-8-ol	0.8	0.2	0.4	0.2	0.1	0.2	–	0.1	0.1		0.1	0.1	0.1
1867	Thymol acetate	–	–	–	–	–	11.0	–		–	–	–	–	–
1916	α-Agarofuran	–	–	–	–	–	0.2	–	0.3	–	0.3	0.6	–	–
1924	Silphiperfolan-7 β-ol	–	–	–	–	–	–	–	0.1	–	0.1	0.1	–	
1957	Cubebene	–	–	–	–	–	–	–	0.6	–	–	–	–	–
1976	Cameroonan-7-α-ol	–	–	–	–	–	–	–	0.6	–	0.4	–	–	–
2008	Caryophyllene oxide	tr	0.3	0.3	0.4	0.3	0.2	0.3	1.3	–	0.4	0.7	–	–
2030	Methyl eugenol	–	–	0.4	0.2	–	–	–	–	–	–	–	–	–
2030	Methyl eugenol	0.3	–	–	–	–	–	–	–	–	–	–	–	–
2024	Presilphiperfolan-9 α-ol	–	–	–	–	–	–	–	0.9	–	–	–	–	–
2037	(E)-Nerolidol	–	–	–	–	–	–	4	0.5	–	–	tr	0.2	–
2067	Presilphiperfolan-8-ol	–	–	–	–	–	0.9	–	0.9	–	7.5	7.9	–	–
2081	Humulene epoxide III	0.4	–	tr	–	–	–	–	–	–	–	–	–	–
2088	1-epi-Cubenol	–	–	–	–	0.1	0.1	–	0.2	–	–	–	–	–
2098	Globulol	–	–	–	–	0.1	–	–	–	0.4	–	tr	0.1	–
2104	Viridiflorol	0.1	–	–	–	0.1	1.9	31.1	2.3	0.3	–	–	0.2	–
2113	Cumin alcohol	0.3	0.2	0.1	0.1	0.1	–	–	–	–	0.1	0.3	–	–
2127	10-Epi-γ-Eudesmol	tr	–	–	–	–	0.7	–	5.4	–	8.8	6.4	–	–
2144	Spathulenol	2.4	0.1		0.1	3.6	4.7	2.1	1.3	8.6	–	–	2.3	2.2
2183	Thymol	–	–	–	–	–	1.7	–	–	–	–	–	–	–
2196	T-muurolol	0.3	–	–	–	–	0.9	–	0.4	0.2	–	–	0.2	0.1
2209	Torreyol	–	–	–	–	–	0.1	–	–	0.1	–	–		–
2232	α-Bisabolol	–	–	–	–	–	1.3	–	–	–	–	–	–	–
2247	*trans*-α-Bergamotol	–	–	–	–	–	0.1	–	–	0.6	–	0.1	–	–
2255	α-Cadinol	0.4	0.2	–	–	–	1.5	0.2	–	0.3	0.9	0.3	–	0.9
2264	Intermedeol	–	–	–	–	–	–	–		0.8	–	–	–	–
	Total	96.8	98.5	98.5	98.7	98.8	97.4	98.6	91.8	98.0	97.9	97.6	96.8	97.2

tr: trace amount (≤0.05%).
RRI: relative retention index.

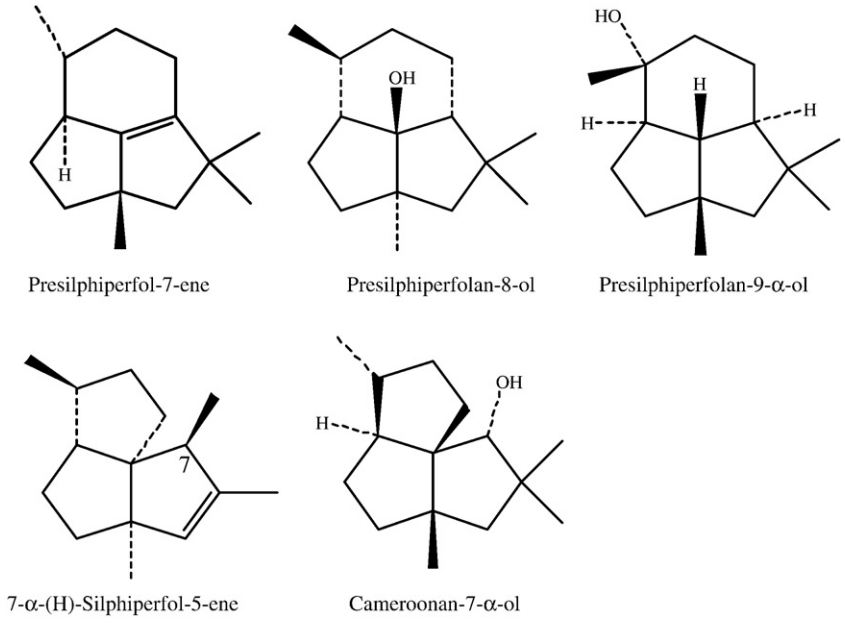

Fig. 1. Structure of rare sesquiterpenes identified in the oils of *Pteronia* species.

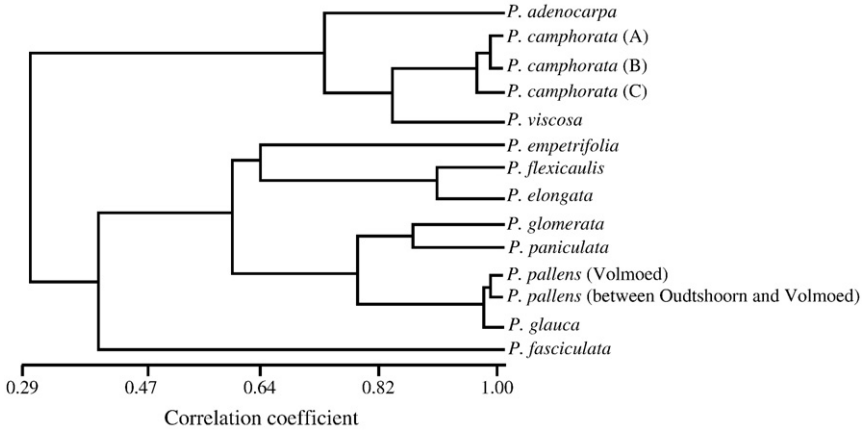

Fig. 2. Cluster analysis of essential oil composition.

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