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Extraction and Composition of Volatiles from Concrete of *Pandanus fascicularis* Lam

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Kewda (*Pandanus fascicularis* Lam) flowers are commercially hydro distilled to obtain essential oil and attars. The oil is obtained usually in 0.03 to 0.04 % depending on the season and time elapsed between harvests and processing. Extraction with hexane on the other hand provides concrete in 0.315 % yields. Hydro distillation of concrete is found to give a steam volatile oil in nearly double the yield. The oil obtained was analyzed and reported the comparison of the composition with the essential oil obtained in the conventional way.

Key Words: *Pandanus fascicularis* Lam, Concrete, Volatile oil, Essential oil, GC/MS, 2-Phenyl ethyl methyl ether, Terpinen-4-ol.

INTRODUCTION

Kewda or screw pine (*Pandanus fascicularis* Lam.) is a common species of the family pandanaceae found growing wild in India, all along the sea coast, on banks of the rivers, *etc*. However, gregarious growth is found along the coast of Ganjam district (19.18ºN, 84.51ºE), Orissa, India[1,2]. The plants are considered to be good soil binders. In the southern coastal district of Ganjam, it is an important economic plant and its male spadices are utilized in the production of essential oil and attars through a process of distillation with water[2]. Kewda flower distillation is a very old practice in Ganjam district and the industry has started in 1924[3]. Kewda products are used in flavouring tobacco and foods[3], particularly North-Indian sweets and in South East Asia, to flavour sweet rice dishes[1].

Kewda flower essential oil has been the subject of study by several investigators[3-9]. Despande[4] identified 2-phenyl ethyl methyl ether (PEME) as the major compound through isolation and structure elucidation by conventional methods. Dhingra *et al.*[5] examined the essential oil from Ganjam district, Orissa and reported identification of dipentene, linalool, phenyl ethyl acetate and citral besides PEME (65-95 %) as major components. Sadgopal[6] reported PEME, dipentene, linalool, 2-phenyl ethyl alcohol, citral and terpene esters. Chandra[7] has reported chemical composition as β-phenyl ethyl alcohol (67 %), dipentene (6 %), δ-linalool (19 %), caproic acid

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(0.5 %) and stearoptene (0.2 %). Nigam and Ahmed[8] have reported PEME (74.67 %), β-phenyl ethyl alcohol (16.23 %), phenyl ethyl acetate (2.76 %) and farnesol (0.55 %) as major components. Maheshwari[9] has identified for the first time the second major component terpinen-4-ol and reported 44 compounds by GC-FID and comparison of relative retention indices. Naqvi and Mandal[10] have also reported the composition of kewda oil. Misra *et al.*[11] have reported the composition of laboratory prepared as well as field collected samples of kewda essential oil and reported PEME and terpinen-4-ol as major compounds. They have also reported the chemical composition of Ketaki flower oil, a chemo variant of kewda. Rao[3] has reported variation in the composition of several samples of kewda oil prepared in the field as well as collected from Ganjam district. Raina *et al.*[12] have analyzed yet a different variant of Kewda essential oil and reported the oil to contain PEME (37.7 %), 2-phenyl ethyl alcohol (7.5 %), terpinen-4-ol (18.6 %), α-terpineol (8.3 %), geraniol (1.2 %), β-caryophyllene (1.8 %), β-gurjunene (1.8 %), γ-muurolene (2.0 %) and leden (1.2 %), a composition quite different from general samples. We have reported for the first time, the extraction of kewda flowers with hexane and reported the composition of concrete and absolute[13]. The hexane extract is obtained in much higher yields, but it contains large amounts of fatty acids and esters, fatty alcohols and aldehydes besides waxes co-extracted from the flowers. In order to separate these undesirable materials, the concrete was subjected to water distillation in a Clevenger apparatus. The volatile oil thus obtained is analyzed and its composition is compared with that of essential oil obtained in the conventional way.

EXPERIMENTAL

Concrete: Male flowers of *P. fascicularis* Lam were collected from the village Tulu, Ganjam district (19.18ºN, 84.51ºE), Orissa, India. The flowers were 25-35 cm long, 6-8 cm in diameter and on the average weighed *ca.* 100 g each. The extractions were carried out in three different batches, taking about 230-270 flowers (*ca.* 25 kg) per batch in a stainless steel conical extractor. The experimental procedure for extraction of concrete (yield 77-83 g) and its composition had appeared in previous publication[13].

Essential oil from flowers: The essential oil was prepared from 500 flowers (50 kg) collected from Tulu, Ganjam district, by distillation in a modified stainless steel field distillation unit wherein the yield of essential oil had improved to 0.04-0.045 %. Commercial samples were also collected and composition determined by GC and GC/MS analysis. The yield of the essential oil in the traditional commercial method ranged between 0.034-0.038 %. For comparative purposes, we have included in Table-1, the chemical composition of the essential oil published earlier[11].

Hydro distillation of concrete: 10 g of concrete is distilled with water for 2 h in a Clevenger type apparatus. The light oily portion is collected and dried over Na_2SO_4. The yield of the steam volatile oil is 2.2-2.4 g. The yields of oils directly distilled or from concrete reported here are results obtained from three identical experiments.

TABLE-1
COMPARATIVE COMPOSITION OF CONCRETE,
VOLATILE OIL AND ESSENTIAL OILS

RT	Compound	Concrete	Volatile oil	Essential oil	RRI lit
6.8	α-Thujene[a]	0.6 ± 0.2	0.4 ± 0.2	0.2 ± 0.1	931
7.4	α-Pinene[a]	0.1	0.1 ± 0.1	0.2 ± 0.1	939
8.4	Sabinene[a]	0.2	1.5 ± 0.7	-	976
8.5	β-Pinene[a]	0.4 ± 0.1	0.2 ± 0.1	t	980
9.3	Myrcene[a]	0.1	t	t	991
10.8	α-Terpinene[a]	0.1	0.2 ± 0.1	-	1018
11.1	P-Cymene[a]	0.2	0.5 ± 0.1	3.1 ± 0.3	1024
11.2	Limonene[a]	0.2	0.4 ± 0.1	0.5 ± 0.2	1031
11.3	1,8-Cineole[b]	0.1	0.3 ± 0.1	t	1033
12.9	γ-Terpinene[a]	0.2	0.8 ± 0.4	t	1062
13.9	Z-Sabinene hydrate[a]	0.1	t	-	1068
15.0	2-Phenylethyl methyl ether[d]	30.6 ± 1.5	87.3 ± 0.5	65.9 ± 1.2	1080
16.0	E-Sabinene hydrate[a]	0.1 ± 0.1	0.1	-	1097
17.9	2-Phenylethyl alcohol[d]	0.4 ± 0.1	0.2 ± 0.1	0.2 ± 0.1	1110
20.7	Terpinen-4-ol[b]	0.7 ± 0.1	1.8 ± 0.2	16.3 ± 0.9	1177
22.0	α-Terpineol[b]	0.1	0.2	2.9 ± 0.4	1189
28.0	Tridecane[f]	0.1	0.1	-	1289
30.3	α-Cubebene[c]	t	t	-	1351
41.5	α-Muurolene[c]	t	t	-	1499
43.5	δ-Cadinene[c]	0.1	t	-	1513
47.7	Hexadecene[f]	0.1	t	-	1593
48.7	Hexadecane[f]	0.2 ± 0.1	t	-	1600
54.1	Heptadecane[f]	0.1	t	t	1700
54.8	Methyl myristate[e]	t	t	-	1726
59.4	Ethyl myristate[e]	0.1	-	t	1777
65.9	2-Phenylethyl phenyl acetate[d]	0.3 ± 0.1	t	t	1909
66.7	Methyl palmitate[e]	0.2	-	t	1927
70.0	Ethyl palmitate[e]	0.3 ± 0.1	t	t	1976
72.2	Unidentified fatty acid[e]	9.0 ± 3.5	t	t	-
73.7	Methyl linoleate[e]	0.9 ± 0.2	t	0.2 ± 0.1	2092
74.8	Methyl linolenate[e]	1.4 ± 0.5	t	-	2096
76.0	Methyl oleate[e]	0.2	-	t	2105
76.5	Methyl stearate[e]	0.7 ± 0.1	-	-	-
81.8	9,12,15-Octadecatrienal[e]	1.3 ± 1.0	t	-	-
83.8	9,12-Octadecadienal[e]	3.3 ± 1.1	t	-	-
85.0	9,12-Octadecadienol[e]	7.4 ± 2.5	t	-	-
86.2	Oleic acid[e]	0.3 ± 0.2	t	-	-
92.2	Heneicosanol[e]	0.8 ± 0.1		-	-
98.2	4-Methyl tetracosane[f]	5.4 ± 0.4	0.1 ± 0.1	t	2438
108.6	Heptacosane[f]	0.7 ± 0.5	-	t	2700
114.5	Branched C28 hydrocarbon[f]	2.8 ± 1.1	-	-	-
126.8	Mixed hydrocarbon[f]	8.5 ± 0.5	-	-	-
140.8	n.i	8.4 ± 1.2	-	-	-
150.4	Mixed hydrocarbon[f]	7.4 ± 4.3	-	-	-

[a]monoterpene hydrocarbons, [b]oxygenated monoterpenes, [c]sesquiterpene hydrocarbons, [d]benzenoid components, [e]fatty acids/esters/alcohols/aldehydes, [f]hydrocarbons
RRI lit: Relative Retention Indices Literature reported, t < 0.1.

GC and GC/MS analysis: GC analysis is carried out on a Shimadzu GC 17A gas chromatograph equipped with a flame ionization detector and a 30 m × 0.25 mm WCOT column coated with 0.25 μm film thickness of 5 % diphenyl dimethyl siloxane supplied by J &W (DB-5). Helium is used as the carrier gas at a flow rate of 1.2 mL/min at a column pressure of 42 KPa. 0.2 μL of each sample is injected into the injection port of the GC using a split ratio of 50:1. Compound separation is achieved following a linear temperature program of 60-200 ºC (2 ºC/min), 200 ºC (90 min). Percentage composition is calculated using peak normalization method. The GC/MS analysis is carried out on a Shimadzu QP5000 GC/MS fitted with the same column and temperature programmed as above. MS parameters: ionization voltage (EI) 70 eV, peak width 2 s, mass range 40-500 amu and detector voltage 1.5 volts. Peak identification is carried out by comparison of the mass spectra with mass spectra available in NIST, Wiley and Adams libraries. The identification of compounds is confirmed by comparison of their relative retention indices with values reported in the literature[14].

RESULTS AND DISCUSSION

The kewda flowers on extraction with hexane afforded 0.315 ± 0.015 % of waxy extract (concrete)[13]. The yield of essential oil directly from flowers usually ranges between 0.034-0.038 %, while the steam volatile oil from the concrete is obtained in 0.072-0.075 % yields, which is almost double the amount of the essential oil. These oils contained benzenoids (65.1 ± 1.3, 87.5 ± 0.6 %), monoterpene hydrocarbons (3.6 ± 1.8, 4.2 ± 1.8 %) and oxygenated monoterpenes (18.2 ± 1.3 %, 2.3 ± 0.4) in essential oil and volatile oil, respectively.

The recoveries of major compound, 2-phenyl ethyl methyl ether was better by hexane extraction, the other semi polar major compounds terpenen-4-ol and α-terpineol, are obtained in much lower yield than usually obtained through hydro distillation. The non-polar compounds, α-thujene, β-pinene, sabinene and its hydrates have been obtained in appreciable quantities in the volatile oil obtained from the concrete. While sabinene and its hydrate have not been detected, α-thujene (0.1-1.0 %) and β-pinene (0.1-1.2 %) were reported in varying yields in the essential oil obtained directly from flowers through hydrodistillation[11]. The recovery of 2-phenyl ethyl methyl ether, terpinen-4-ol and α-terpineol are 63.7, 1.3 and 0.15 g, respectively from volatile oil obtained from concrete of 100 Kg of flowers. Similarly the recovery of 2-phenyl ethyl methyl ether, terpinen-4-ol and α-terpineol are 23.7, 5.8 and 1.0 g, respectively from essential oil obtained from 100 Kg of flowers. Isolation of the second major compound terpinen-4-ol is obtained in much lower yields, which may be due either to its presence in the flower in a bound form which gets released during hydro distillation or to its relative lower solubility in hexane. The presence of terpinen-4-ol in 3.3 ± 0.4 % in the floral volatiles as determined by headspace-solid phase microextraction (HS-SPME)[15] also has been much higher than observed in concrete and its steam volatile portion which shows that perhaps this compound is released continuously by the flower by a biochemical process (Table-2).

TABLE-2
AMOUNT OF PEME, TERPINEN-4-OL AND α-TERPINEOL
IN DIFFERENT EXTRACTS

Extract	Yield (%)	Volatile component (%)	PEME content (%)	PEME in g from 100 kg of flowers	Terpinen-4-ol content (%)	Terpinen-4-ol in g from 100 kg of flowers	α-Terpineol content (%)	α-Terpinenol in g from 100 kg of flowers
Concrete	0.315±0.015	77±1.5	30.6±1.5	74.2	0.7±0.1	1.7	0.1	0.24
Volatile oil (concrete)	0.073±0.005	~100	87.3±1.0	63.7	1.8±0.2	1.3	0.2	0.15
Essential oil	0.036±0.005	~100	65.9±1.9	23.7	16.3±0.5	5.8	2.9±0.4	1.0

Conclusion

Recovery of non-polar compounds and the semi-polar major compound *i.e.,* 2-phenyl ethyl methyl ether can be achieved through hexane extraction in much higher yields than the traditional water distillation process. The volatile oil is totally free of waxy materials. To achieve the complete extraction of all compounds, including minor and polar molecules from flowers, the solvent (hexane) may have to be modified suitably.

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