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Composition of Essential Oils from the Leaf and Stem Bark of *Michelia foveolata*

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Authors' contributions

The work was carried out in collaboration with all authors. Authors DND and DBT collected the plant sample and performed the hydrodistillation of the oil samples while author TDT designed the study and performed the analysis of the oil samples. Authors IAO and TOO managed the literature searches. Author IAO wrote the first and final draft of the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

The chemical constituents of essential oils obtained from the hydrodistillation of the leaf and stem of *Michelia foveolata* Merr. et Dandy (Magnoliaceae) were being reported. The main compounds of the leaf essential oil are β-caryophyllene (37.1%) and bicyclogermacrene (23.3%). However, β-caryophyllene (26.4%), germacrene D (5.9%), *trans*-α-bergamotene (5.6%), δ-cadinene (5.5%), bicyclogermacrene (5.4%), bicycloelemene (5.3%), spathulenol (5.3%) and 3,7-guaiadiene (5.1%) were present in the stem.
Aims: The aim of this study was to isolate essential oils from the leaf and stem bark of *Michelia*

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foveolata Merr. et Dandy (Magnoliaceae) and investigate the volatile constituents present therein.
Study Design: The study involves the hydrodisitillation of essential oils from the air-dried plant materials and analysis of their chemical composition.
Place and Duration of Study: The leaf and stem bark of *M. foveolata* were collected from Vũ Quang National Park, Hà Tĩnh Province Vietnam, in July 2014.
Methodology: About 500 g of air-dried plant samples was shredded and their oils were obtained by separate hydrodistillation for 4 h at normal pressure, according to the Vietnamese Pharmacopoeia. The chemical constituents of the distilled oils were analyzed by means of gas chromatography-flame ionization detector (GC-FID) and gas chromatography coupled with mass spectrometry (GC-MS).
Results: The main compounds of the leaf essential oil are β-caryophyllene (37.1%) and bicyclogermacrene (23.3%) while the stem bark contained large amounts of β-caryophyllene (26.4%), germacrene D (5.9%), *trans*-α-bergamotene (5.6%), δ-cadinene (5.5%), bicyclogermacrene (5.4%), bicycloelemene (5.3%), spathulenol (5.3%) and 3,7-guaiadiene (5.1%).
Conclusion: The present oil compositions were found to be different from the results obtained previously from the essential oils of *Michelia* species grown in Vietnam and other parts of the world.

Keywords: Michelia foveolata; essential oil composition; sesquiterpenes.

1. INTRODUCTION

The aim of the present study was report the volatile compounds identified in the essential oils hydrodistilled from leaves and stems of *Michelia foveolata* Merr. et Dandy (Magnoliaceae) grown in Vietnam. This is in continuation of an extensive research aimed at the characterization of the volatile compounds of Vietnamese flora [1-3]. *Michelia foveolata* is a tree that grows up to 30 m tall with dark grey bark. The plant could be found growing wild wet forests of South-east Asia (altitude 500 to 1 800 m) and it is also found in the mountainous North-west of Vietnam. The leaves are densely reddish brown pale gray to dark gray. The fruits are about 7-20 cm long. Flowering takes place between March and May while fruiting occurs from September to October. Leaf extracts of *M. foveolata* inhibit the activity of cholinesterase [4].

Previous analysis revealed that the main compounds of the aril essential oil from China [5] were linalool (18.55%), 1a,2,3,4,4a,5,6,7b-octahydro-1,1,4,7-tetramethyl-1H-cyclopyl[e]azulene (17.70%) and eucalyptol (10.41%). The main constituents of essential oils isolated from various parts of the plant [6] were sabinene (32.4%) and terpinen-4-ol (13.7%). In addition, the oil exhibited significant antibacterial activity against *Salmonella enterica*, *Staphylococcus epidermidis*, *Staphylococcus aureus* and *Bacillus cereus* (MICs = 2–4 µl/ml). In another report, eucalyptol (>40%) was identified as the main component of the leaf oil [7]. Moreover, α-eudesmol (49.70%), eudesma-4(15)-7-dien-1β-ol (13.74%) and α-bisabolol (7.45%) were identified previously in *M. foveoleta* [8].

2. MATERIALS AND METHODS

2.1 Plant Material

The leaves and stems of *M. foveolata* were collected from Vũ Quang National Park, Hà Tĩnh Province Vietnam, in July 2014. The plant samples were authenticated by Dr. Dai, while voucher specimen DND 298 was deposited at the Botany Museum, Vinh University, Vietnam. Plant samples were air-dried prior to extraction.

2.2 Hydrodistillation of Essential Oils

Aliquots of 500 g each of pulverised plant samples was used for the experiment and their oils were obtained by hydrodistillation for 3 h, according to the Vietnamese Pharmacopoeia [9]. Briefly, 500 g of the pulverized sample were carefully introduced into a 5 L flask and distilled water was added until it covers the sample completely. Hydrodistillation was carried out in an all glass Clevenger distillation unit designed according to the specification. The volatile oils distilled over water and were collected separately in the receiver arm of the apparatus into a clean and previously weighed sample bottles. The oils were kept under refrigeration until the moment of analyses.

2.3 Analysis of Essential Oils

The GC analysis of essential oils was carried out using an Agilent Technologies HP 6890 Plus Gas

Chromatograph which was equipped with a flame ionization detector and HP-5MS column. The dimension of the column is 30 m x 0.25 mm (film thickness 0.25 μm). The GC operating parameters based on temperature programming were as follows column oven 40℃, injection port 250℃ while the detector temperature was 260℃. Time programming: 40℃ for 2 min, and then raise to 220℃ (and held isothermally for 10 min) at 4℃/min. The carrier gas used was H_2 at a flow rate of 1 mL/min. The split ratio was 10:1 while 1.0 μL of the essential oil was injected into the GC at inlet pressure was 6.1 kPa. Each analysis was performed in triplicate. Retention indices (RI) value of each component was determined relative to the retention times of a homologous *n*-alkane series with linear interpolation on the HP-5MS column. The relative amounts of individual components were calculated based on the GC peak area (FID response) without using correction factors.

An Agilent Technologies HP 6890 N Plus Chromatograph fitted with a fused silica capillary HP-5MS column (30 m x 0.25 mm, film thickness 0.25 μm) and interfaced with a mass spectrometer HP 5973 MSD was used for the GC/MS analysis, under the same conditions as those used for GC analysis. The conditions were the same as described above with He (1 mL/min) as carrier gas. The MS conditions were as follows: ionization voltage 70 eV; emission current 40 mA; acquisitions scan mass range of 35-350 amu at a sampling rate of 1.0 scan/s.

2.3.1 Identification of constituents of the oils

The identification of constituents was performed on the basis of retention indices (RI) determined by co-injection with reference to a homologous series of *n*-alkanes, under identical experimental conditions. Further identification was performed by comparison of their mass spectra with those from NIST [10] and the home-made MS library built up from pure substances and components of known essential oils, as well as by comparison of their retention indices with literature values [11].

3. RESULTS AND DISCUSSION

The yields of essential oils were 0.13% (v/w, leaf) and 0.15% (v/w, stem) calculated on a dry weight basis. Table 1 indicates the chemical constituents present in the oil, their percentages as well as retention indices on HP-5MS column. The main classes of compounds identified in the essential were the sesquiterpene hydrocarbons (71.8% and 70.8%) and oxygenated sesquiterpenes (17.5% and 16.6%). Monoterpene hydrocarbons (11.7%) were also present in the stem oil while the oxygen-containing monoterpene compounds were conspicuously absent in the leaf. The compounds occurring in higher amounts in the leaf oil are β-caryophyllene (37.1%) and bicyclogermacrene (23.3%). Two aromatic esters, benzyl salicylate (2.7%) and benzyl benzoate (1.9%) were also identified ion the leaf oil but absent in the stem. Also, β-caryophyllene (26.4%) and germacrene D (5.9%) occurred as the major constituents of the stem oil. In addition, *trans*-α-bergamotene (5.6%), δ-cadinene (5.5%), bicyclogermacrene (5.4%), bicycloelemene (5.3%), spathulenol (5.3%) and 3,7-guaiadiene (5.1%) were also present in sizeable amount. However, aromatic esters were not identified in the stem oil.

A comparative analysis of the present study with previous investigated oil samples elsewhere revealed some marked variations. The main compounds of previously study oil samples such as linalool, 1a,2,3,4,4a,5,6,7b-octahydro-1,1,4,7-tetramethyl-1H-cyclopyl[e]azulene and eucalyptol [5], sabinene and terpinen-4-ol [6], as well eucalyptol, α-eudesmol, eudesma-4(15)-7-dien-1β-ol and α-bisabolol [7,8] were conspicuously absent in the present oil composition. In addition, β-caryophyllene and bicyclogermacrene, the main constituents of the sample under investigation were not reported to be of quantitatively significant importance in the previous analysis.

The essential oils of *M. foveolata* may be taught to exists in different chemical forms mainly oils dominated by oxygenated monoterpenes [5,7], oils consisting mainly of oxygenated sesquiterpenes [8], oils whose major constituents were sesquiterpene hydrocarbons (this study) and oil with large amounts of monoterpene compounds [6].

It is well known that the essential oils of *Michelia* plants exhibited diversed chemical compositional patterns. For example, Linalool was the major component of the flower oil (72.8%) and the leaf oil (80.1%) of *M. alba* [12], while another report identified the abundance of phenyl ethyl alcohol (34.9%-39.0%) and indole (25.9%-67.9%) in the hot enfleurage, petroleum ether and hexane extracts of *M. alba* [13]. The major compounds of the essential oil at each vegetative stages of

M. alba [14] were dihydrocarveol (43.8-64.5%), linalool (59.1-79.4%) and butanoic acid-2-methyl, methyl ester (6.4-20.4%). The volatile components of the leaf oil of *M. balansae* [15]

Table 1. Chemical constituents of essential oils of *Michelia foveolata*

Compounds [a]	RI [b]	RI [c]	Percent composition	
			Leaf	Stem
α-Pinene	939	932	0.3	-
Sabinene	976	969	-	4.5
β-Myrcene	990	988	0.2	0.5
α-Phellandrene	1006	1004	1.9	0.4
δ-3-Carene	1011	1008	-	0.4
p-Cymene	1026	1020	0.3	-
β-Phellandrene	1028	1024	0.9	3.1
(*Z*)-β-Ocimene	1043	1032	-	0.5
(*E*)-β-Ocimene	1052	1044	-	0.6
γ-Terpinene	1061	1056	-	0.5
α-Terpinolene	1090	1087	-	0.9
allo-Ocimene	1128	1128	-	0.3
Citronella	1223	1223	-	0.6
Bicycloelemene	1327	1337	2.7	5.3
Cyclosativene	1371	1372	-	0.2
α-Copaene	1377	1373	0.2	0.2
β-Elemene	1391	1389	1.4	5.9
β-Caryophyllene	1419	1417	37.1	26.4
Thujopsene	1429	1430	-	0.8
β-Gurjunene	1434	1431	0.5	-
trans-α-Bergamotene	1435	1434	1.0	5.6
3,7-Guaiadiene	1444	1444	-	5.1
α-Humulene	1454	1452	2.5	3.5
Germacrene D	1485	1484	1.8	5.9
Zingiberene	1494	1493	0.4	-
Bicyclogermacrene	1500	1500	23.3	5.4
β-Bisabolene	1506	1505	0.3	1.0
cis-(*Z*)-α-bisabolene epoxide	1515	1518	1.1	0.4
δ-Cadinene	1525	1522	0.6	5.5
(*E*)-Nerolidol	1563	1561	0.5	2.4
Spathulenol	1578	1577	1.6	5.3
Valencene	1581	1581	-	-
Caryophyllene oxide	1583	1581	1.4	2.1
Ledol	1588	1589	0.4	3.9
Viridiflorol	1593	1594	0.6	-
Isospathulenol	1636	1636	1.4	-
τ-Muurolol	1646	1640	0.7	-
β-Eudesmol	1651	1646	2.8	-
α-Cadinol	1654	1652	1.0	2.5
Bulnesol	1672	1675	2.3	-
(*E*,*E*)-Farnesol	1718	1722	3.8	-
Benzyl benzoate	1760	1759	1.9	-
Benzyl salicylate	1866	1862	2.7	-
Total			97.5	99.7
Monoterpene hydrocarbons			3.6	11.7
Oxygenated monoterpenes			-	0.6
Sesquiterpene hydrocarbons			71.8	70.8
Oxygenated sesquiterpenes			17.5	16.6
Aromatic esters			4.6	-

[a] Elution order on HP-5MS column; [b] retention indices on HP-5MS column; [c] literature retention indices (see experimental)

from Vietnam were determined as α-pinene (18.4%), α-phellandrene (17.3%) and germacrene D (17.9%). The flower oil of *M. champaca* contained β-ionone (26.8%), linalool (11.0%) and dihydro-β-ionone (10.0%) as major constituents [16]. Another study identified β-caryophyllene (10.0-25.0%), α-humulene (9.4-14.0%) and β-elemene (7.0-16.0%) in higher proportions in *M. champaca* [17]. In addition, methyl linoleate (18.0-25.3%), methyl benzoate (8.6-11.5%) and phenyl acetonitrile (7.5-10.4%) were also reported as significant compounds of *M. champaca* [18]. The main constituents of *M. yunnanensis* [19] were bornyl acetate (12.4%), camphor (8.7%) and caryophyllene oxide (6.9%). The leaf oil of *M. compressa var. formosana* [20] consisted primarily of α-cadinol (18.9%) and germacrene D (18.5%) whereas α-cadinol was found in the twig (19.1%) and flower (11.4%). However, α-cadinol (25.7%) and β-eudesmol (20.2%) were present in the wood oil. Moreover, (*E*,*E*)-farnesol (10.82%) and germacrene A (16.05%) were the compounds occurring in higher quantity in *M. chapensis* [8].

4. CONCLUSION

In the present investigation of chemical constituents of essential oils from *M. foveolata,* β-caryophyllene and germacrene D were identified as the main constituents. The compositional pattern was found to differ from previously investigated oil samples of the same species as well as those from other *Michelia* plants.

ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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