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Constituents of Some Essential Oil Bearing Plants from Vietnam

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Abstract

Essential oils obtained from hydrodistillation of three medicinal plants grown in Vietnam were analyzed by gas chromatography (GC) and gas chromatography/mass spectrometry (GC-MS). The monoterpene hydrocarbons, *α*-pinene (50.2%), *β*-pinene (23.6%) and limonene (5.3%) were the most abundant constituents of the rhizome oil of *Zingiber collinsii* Mood & Theilade (Zingiberaceae). The main compounds of the stem oil of *Croton kongensis* Gagnep., (Euphorbiaceae) were benzyl benzoate (12.7%), *β*-selinene (9.8%), bulnesol (8.0%) and 5,6,7,8-tetrahydroquinoxaline (7.4%). The leaf oil of *Goniothalamus albiflous* Ban., consisted mainly of *α*-pinene (26.2%), caryophyllene oxide (10.6%) and 1,8-cineole (9.7%). The composition of the oils of *Zingiber collinsii* and *Croton kongensis* was being reported for the first time.

Keywords

***Zingiber collinsii*; *Croton kongensis*; *Goniothalamus albiflorus*; Essential Oil; Terpenes**

1. Introduction

In this paper, we report on the volatile constituents identified from the three medicinal plants, as part of our continued interest on the chemical analysis of the flora of Vietnam [1] [2]. *Zingiber collinsii* Mood & Theilade (Family Zingiberacea), a deciduous species, was recently collected in Vietnam and introduced by Mark Collins [3]. Naturally dormant in winter, leaves have silver streaks on green above and maroon beneath. Cones are dull

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orange and form at the base of the plant. It is the most beautiful ginger in cultivation. The foliage is spectacular with silver bands on the leaves and basal flowers in clusters of orange to red that look like brightly colored hot pokers [4]. Literature information is scanty on its volatile and non-volatile constituents as well as the biological potential of this plant.

Croton kongensis Gagnep., (family Euphorbiaceae), is known in Vietnam as Cù đèn cửu long. The leaves are used for medicinal purposes. It is frequently used in folk medicine for dysmenorrhoea. The leaves are used in Indo-China for various stomach disorders including ulcers, and a decoction is externally applied for furuncles and impetigo [5]. Crude extract of the plant was previously reported to have exhibited antimalarial and antimycobacterial activities [5]. Several metabolites including antimycobacterial and antimalarial diterpenes [6]-[9] and phenolic compounds [9] have been characterized from this plant. The volatile constituent has not been any subject of literature discussion.

Goniothalamus albiflorus Ban., is a species of plant in the Annonaceae family. This plant is endemic to Vietnam [10]. A previous analysis of the volatile constituents identified higher amounts of benzoic acid (18.4%), *β*-caryophyllene (12.4%) and *α*-pinene (10.3%) leaf oil, while limonene (21.2%), *β*-caryophyllene (12.8%) and *α*-phellandrene (9.3%) were present in stem oil [11].

2. Materials and Methods

2.1. Plants Collection

Rhizomes of *Z. collinsii* and the stem of *C. kongensis* were collected from Pu Mat National Park, Nghe An Province in July 2012 and September 2011 respectively. The leaves of *G. albiflorus* were collected from Bạch Mã National Park, Thừa Thiên—Huế Province, Vietnam, in August 2011. Voucher specimens were coded DND 278, HDT 288 and DND 807 respectively. All specimens were deposited at the Botany Museum Vinh University, Vietnam.

2.2. Isolation of Essential Oils

0.5 Kg of air-dried of each plant sample was shredded and their oils were obtained by hydrodistillation for 3h at normal pressure, according to the Vietnamese Pharmacopoeia [12].

2.3. Gas Chromatography (GC) Analysis of the Oils

Gas chromatography (GC) analysis was performed on Agilent Technologies HP 6890 Plus Gas chromatograph equipped with an FID and fitted with HP-5MS column (30 m × 0.25 mm, film thickness 0.25 μm, Agilent Technology). The analytical conditions were: carrier gas H_2 (2 mL/min), injector temperature (PTV) 250˚C, detector temperature 260˚C, column temperature programmed 60˚C (2 min hold) to 220˚C (10 min hold) at 4 ˚C/min. Samples were injected by splitting and the split ratio was 10:1. The volume injected was 1.0 μL. Inlet pressure was 6.1 kPa. The relative amounts of individual components were calculated based on the GC peak area (FID response) without using correction factors.

2.4. Gas Chromatography-Mass Spectrometry (GC-MS) Analysis of the Oils

An Agilent Technologies HP 6890N Plus Chromatograph fitted with a fused silica capillary column HP-5 MS column (30 m × 0.25 mm, film thickness 0.25 μm) and interface with a mass spectrometer HP 5973 MSD was used for the GC/MS analysis, under the same condition 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 70eV; emission current 40 mA; acquisitions scan mass range of 35 - 350 amu at a sampling rate of 1.0 scan/s. The MS fragmentation patterns was checked with those of other essential oils of known composition patterns with Wiley (Wiley 9th Version), NIST 08 Libraries (on ChemStation HP), with those in the literature and also with standard substances.

2.5. Identification of Constituents

The identification of constituents was performed on the basis of retention indices (RI) determined with reference to the homologous series of *n*-alkanes, under identical experimental conditions, co-injection with standards

(Sigma-Aldrich, St. Louis, MO, USA) or known essential oil constituents, MS library search (NIST 08 and Wiley 9th Version), and by comparing with MS literature data [13] [14].

3. Results and Discussion

The yields obtained from the hydrodistillation procedures were 0.20% (v/w; *Z. collinsii*; light yellow), 0.12% (v/w; *C. kongensii* (colourless) and 0.25% (v/w; *G. albiflorus*, light yellow). All calculations were done on a dry weight basis. **Table 1** displays the identities of compounds identified from the studied oil samples. 35 compounds representing 96.9% of the total contents were identified from the oil of *Z. collinsii*. Monoterpene hydrocarbons (89.9%) were the exclusive class of compound identified in *Z. collinsii*. The main compounds in this class were *α*-pinene (50.2%), *β*-pinene (23.6%) and limonene (5.3%). The oxygenated monoterpenes and the sesquiterpene compounds, though present in the oil were identified in lesser quantities. This report was the first attempt on the analysis of the volatile constituents of this plant. Recently, the major constituents of rhizome oil of Vietnamese *Zingiber rubens* were identified as (Z)-citral (30.1%), camphene (9.7%), *β*-phellandrene (7.5%) and 1,8-cineole (7.0%) and zingiberene (5.3%) while (Z)-citral (26.1%), camphene (16.3%), sabinene (14.6%), zingiberene (7.2%) and lavandulyl acetate (6.7%) were the principal compounds of *Zingiber zerumbet* [1]. It was noted that all the main constituents of *Z. rubens* and *Z. erumbet* except camphene were conspicuously absent in *Z. collinsii*.

Table 1. Compounds identified from the studied samples.

Compounds	RI	RI	Percent composition (%)		
			Z.c	***C.k***	***G.a***
α-Pinene	939	932	50.2	5.4	26.2
Camphene	953	946	2.3	2.3	1.7
Verbenene	968	961	0.2	-	1.0
β-Pinene	980	974	23.6	2.1	2.2
β-Myrcene	990	988	2.9	3.5	-
α-Phellandrene	1006	1002	1.3	-	-
α-Terpinene	1017	1014	0.3	-	-
p-Cymene	1024	1020	-	-	0.3
Limonene	1032	1024	5.3	0.7	0.4
1,8-Cineole	1034	1026	-	-	9.7
(*Z*)-*β*-Ocimene	1043	1032	0.5	-	0.1
(*E*)-*β*-Ocimene	1052	1044	0.2	-	2.2
γ-Terpinene	1061	0154	1.0	-	-
α-Terpinolene	1090	1086	0.5	-	-
Linalool	1100	1095	0.2	0.4	-
1,3,8-*p*-Menthatriene	1110	1108	-	-	0.1
α-Campholenal	1126	1122	-	-	0.4
allo-ocimene	1128	1128	0.1	-	-
trans-Verbenol	1145	1140	-	-	1.8
Isoborneol	1162	1155	-	-	0.6
Pinocarvone	1165	1160	-	-	1.1
Borneol	1167	1165	-	0.6	-
Terpinen-4-ol	1177	1174	0.1	-	-
α-Terpineol	1189	1187	-	-	0.7
Myrtenal	1190	1195	-	-	1.3
Mesitol	1204	1202	-	1.1	-
Verbenone	1205	1204	-	-	0.5
trans-Carveol	1217	1215	-	-	0.2
5,6,7,8-Tetrahydroquinoxaline	1226	1226	-	7.4	-
Fenchyl acetate	1228	1228	0.6	-	-
Z-Citral (=Neral)	1238	1235	-	-	2.3
Carvone	1243	1239	-	-	0.1
Bornyl acetate	1289	1287	0.4	1.2	0.5

Continued

trans-Pinocarvyl acetate	1297	1298	0.2	-	1.7
Bicycloelemene	1337	1338	0.4	-	-
δ-Elemene	1340	1335	-	2.1	-
α-Cubebene	1351	1345	-	-	2.5
α-Ylangene	1375	1373	-	-	4.0
α-Copaene	1377	1374	0.1	-	0.6
β-Elemene	1391	1389	0.1	1.9	-
β-Caryophyllene	1419	1417	-	3.5	0.1
Aromadendrene	1441	1439	0.1	-	2.7
α-Humulene	1454	1452	-	2.0	-
Dehydroaromadendrene	1463	1460	-	-	3.3
γ-Gurjunene	1479	1475	0.1	-	-
γ-Curcumene	1480	1481	-	1.0	-
Germacrene D	1484	1484	-	2.8	-
α-Amorphene	1485	1483	-	-	0.1
β-Selinene	1484	1489	0.1	9.8	-
cis-Cadina-1,4-diene	1496	1495	-	-	2.3
α-Selinene	1498	1498	0.8	-	-
α-Chamigrene	1505	1503	-	2.0	-
(*E*,*E*)-*α*-Farnesene	1513	1505	0.2	-	-
cis-Calamenene	1514	1513	-	-	2.4
γ-Cadinene	1514	1513	-	1.8	0.6
7-*epi*-*α*-Selinene	1515	1520	0.1	1.2	-
α-Calacorene	1544	1544	-	-	4.7
Cadina-4,9-diene	1546	1546	-	-	1.4
Elemol	1550	1548	-	0.4	-
(*E*)-Nerolidol	1563	1561	0.1	1.0	-
Palustrol	1567	1567	-	-	0.3
Spathulenol	1578	1577	0.2	2.3	-
Caryophyllene oxide	1583	1582	-	2.2	10.6
α-Guaiol	1601	1600	-	1.2	-
Ledol	1604	1602	-	-	3.2
epi-Cedrol	1617	1618	-	1.6	-
Alloaromadendrene epoxide	1641	1639	-	-	0.4
α-Cadinol	1652	1652	0.1	1.1	-
neo-Intermedeol	1658	1658	-	2.8	-
Bulnesol					
3-Ethyl-6-methoxy-2-Naphthol	1674	1676	-	4.7	-
Farnesol[d]	1718	-	0.1	-	-
Benzyl benzoate	1760	1759	-	12.7	-
1,2-Benzenedicarboxylic acid	19171	1917	1.5	0.3	-
Hexadecanoic acid	1959	1959	0.1	-	-
(*Z*)-9-Octadecamide	2398	2398	0.2	5.4	-
(*Z*)-13-Docosenamide	2499	2499	2.7	-	-
Total			**96.9**	**96.5**	**94.3**
Monoterpene hydrocarbons			**88.4**	**14.0**	**34.2**
Oxygenated monoterpenes			**1.5**	**3.3**	**20.9**
Sesquiterpene hydrocarbons			**2.0**	**28.1**	**24.7**
Oxygenated sesquiterpenes			**0.5**	**20.6**	**14.5**
Aromatic compounds			**-**	**12.1**	**-**
Aromatic esters			**-**	**12.7**	**-**
Others			**4.5**	**5.7**	**-**

[a]Compounds identified by RI from column, co-injection, literature MS pattern and literature retention indices, except where stated; [b]Retention indices on HP-5 MS capillary column; [c]Literature retention indices; - not identified and not found in Literature; [d]correct isomer not identified; *Z.c* = *Zingiber collinsii*; *C.k* = *Croton kongensii*; *G.a* = *Goniothalamus albiflorus*.

The classes of compounds present in *C. kongensis* were monoterpenes (16.2%), sesquiterpenes (48.7%), aromatic ester (12.7%), aromatic compounds (12.1%) among others. The main compounds of *C. kongensis* were benzyl benzoate (12.7%), *β*-selinene (9.8%), bulnesol (8.0%) and *α*-selinene (5.4%). *α*-Pinene (5.4%), *α*-ter- pinene (3.5%) and camphene (2.3%) were the representatives of the monoterpene compounds. Two unusual constituents of essential oils, namely 5,6,7,8-tetrahydroquinoxaline (7.4%) and 3-ethyl-6-methoxy-2-naphthol (4.7%) were also present in significant amounts (**Table 1**). This report was the first of its kind aimed at characterizing the volatile compound present in *C. kongensis.* However, the compositions of essential of some *Croton* species from Vietnam had been reported in literature. The oil of *Croton cascarilloides* consisted mainly of *α*-pinene (10.5%), *β*-caryophyllene (13.5%), *α*-humulene (5.9%), germacrene D (6.0%) and *α*-selinene (6.7%) while cyclohexanone (6.8%), *cis*-carane (6.5%), 1-menthol (30.4%) and benzyl benzoate (18.8%) were the main constituents of *Croton chevalieri*. Linalool (7.8%), bicycloelemene (8.0%), *β*-caryophyllene (10.1%), *α*-humulene (7.1%), *β*-bisabolene (9.6%) and *β*-sesquiphellandrene (6.9%) could be identified in higher amounts in *Croton tonkinensis* [15]. Some major compounds of previously studied Croton oils from Vietnam were not detected in the oil of *C. kongensis*. These were *cis*-carane, 1-menthol, *β*-bisabolene, *β*-sesquiphellandrene and cyclohexanone.

As could be seen in **Table 1**, monoterpenes (53.4%) and sesquiterpenes (39.2%) were the main class of compounds present in *G. albiflous*. The major constituents were identified to be *α*-pinene (26.2%), 1, 8-cineole (9.7%) and caryophyllene oxide (10.6%). Quantitative amounts of *α*-calacorene (4.7%), *α*-ylangene (4.0%), dehydroaromadendrene (3.3%) and ledol (3.2%) were also present in the oil. We reported earlier the composition of the leaf oil from Nghệan Province, to be benzoic acid (18.4%), *β*-caryophyllene (12.4%) and *α*-pinene (10.3%) while limonene (21.2%), *β*-caryophyllene (12.8%) and *α*-phellandrene (9.3%) were identified in stem [11]. Benzoic acid, a major compound in previous study was not identified in the present sample, which also had low content of *β*-caryophyllene (2.7 vs. 12.4%). Our previous studies on other *Goniothalamus* revealed that the leaf oil of *Goniothalamus macrocalyx* was rich in *α*-pinene (50.0%) *β*-caryophyllene (9.9%), α-cadinol (5.3%) and *β*-selinene (5.2%) while *α*-pinene (33.4%), viridiflorol (18.5%) and *β*-caryophyllene (12.4%) were the abundant compounds of *Goniothalamus tamirensis* [11]. Literature information indicated that *α*-pinene (44.5%), *β*-pinene (22.1%) and *β*-phellandrene (12.0%) were the quantitative compounds of *Goniothalamus glabraciamus* [16]. Both qualitative and quantitative variations in chemical compositions could be observed between the various investigated samples.

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