DOI 10.1007/s10600-019-02691-x
Chemistry of Natural Compounds, Vol. 55, No. 2, March, 2019

CONSTITUENTS OF ESSENTIAL OIL OF *Zingiber nudicarpum* FROM VIETNAM

Nguyen D. Hung,[1] Le T. Huong,[2*] Ly N. Sam,[3] Tran M. Hoi,[1,4] and Isiaka A. Ogunwande[5*]

Zingiber Mill. is a genus of gingers found in pantropical Asia. The genus comprises about 144 species, ranging from India to Japan and Southeast Asia, with the center of biodiversity of the genus residing in the Indochinese Peninsula and southern China [1]. Currently 32 *Zingiber* species are known from Vietnam [2]. *Zingiber nudicarpum* D. Fang was recently discovered and collected in the forest protected areas in Pu Huong and Pu Hoat Nature Reserves [3]. It is a rhizomatous herb up to 2.8 m tall, forming small clumps, with up to six leaf shoots per clump [3].

To date, the authors are not aware of any literature citation on the volatile composition of *Z. nudicarpum*. The aim of the present study was to report the chemical compounds identified in the essential oils obtained from the leaf, root and fruit of *Z. nudicarpum* grown in Vietnam. In recent times, the volatile constituents of some Vietnamese plants have been published [4, 5].

The leaves, roots, and fruits of *Z. nudicarpum* were collected from Pu Hoat Natural Reserve, Nghe An Province, Vietnam, in August 2014. A voucher specimen, NDH 474, was deposited at the Botany Museum, Vinh University, Vietnam. Plant samples were air-dried prior to extraction. Hydrodistillation was carried out separately in an all-glass Clevenger-type distillation apparatus according to established procedure [6]. The volatile oils distilled over water and were collected in the receiver arm of the apparatus into a clean and previously weighed sample bottle. The oils were kept under refrigeration until the moment of analysis.

Gas chromatography (GC) analysis was performed on an Agilent Technologies HP 6890 Plus Gas chromatograph (HP-5MS column, 30 m × 0.25 mm, film thickness 0.25 μm). Temperature parameters: column oven 40°C, injection pot – 250°C, detector – 260°C. Time programming: 40°C for 2 min, temperature rose to 220°C (10 min hold) at 4°C·min^{-1}, carrier gas H_2 (1 mL·min^{-1}), split ratio 10:1, volume injected 1.0 μL. Inlet pressure, 6.1 kPa. Each analysis was performed in triplicate. Retention indices (RI) of each component was determined relative to the retention times of a homologous *n*-alkane series with linear interpolation on the HP-5MS column. Gas chromatography-mass spectrometry (GC/MS) was performed on an HP 5973 MSD mass spectrometer with HP 6890N Plus GC (HP-5 MS, 30 m × 0.25 mm, film thickness 0.25 μm). MS conditions: ionization voltage 70 eV; emission current 40 mA; acquisitions scan mass range 35–350 amu, sampling rate 1.0 scan·s^{-1}.

Most constituents were identified by gas chromatography by comparison of their retention indices with those in the literature or with those of available authentic compounds. The retention indices were determined in relation to a homologous series of *n*-alkanes (C_8–C_{24}) obtained under the same operating conditions. Further identification was made by comparison of their mass spectra with those stored in the library [7], peak enrichment on co-injection with authentic standard where possible are as described previously [4, 5].

The yields of the hydrodistilled essential oils of *Z. nuricarpum* were 0.20% (v/w, leaves), 0.35% (v/w, roots), and 0.51% (v/w, fruits), calculated on a dry weight basis. Table 1 indicates the percentages and identities of compounds present in the oils.

1) Graduate University of Science and Technology, Vietnam Academy of Science and Technology, 18-Hoang Quoc Viet, Cau Giay, Hanoi, Vietnam; 2) School of Natural Science Education, Vinh University, 182 Le Duan, Vinh City, Nghe An Province, Vietnam, e-mail: lehuong233@gmail.com; 3) Institute of Tropical Biology, Vietnam Academy of Science and Technology, 85 Tran Quoc Toan road, District 3, Hochiminh City; 4) Institute of Ecology and Biological Resources, Vietnam Academy of Science and Technology, 18-Hoang Quoc Viet, Cau Giay, Hanoi, Vietnam; 5) Natural Products Research Unit, Department of Chemistry, Faculty of Science, Lagos State University, Badagry Expressway Ojo, P. M. B. 0001, LASU Post Office, Ojo, Lagos, Nigeria, e-mail: isiaka.ogunwande@lasu.edu.ng. Published in *Khimiya Prirodnykh Soedinenii*, No. 2, March–April, 2019, pp. 305–307. Original article submitted November 19, 2017.

TABLE 1. Essential Oil Constituents of *Z. nudicarpum*

Compound	RI[a]	Leaves	Roots	Fruits	Compound	RI[a]	Leaves	Roots	Fruits
Tricyclene	926	–	0.5	–	Germacrene D	1485	–	1.1	–
α-Pinene	939	2.4	8.5	4.4	α-Amorphene	1485	–	0.3	–
Camphene	953	–	4.0	–	Bicyclogermacrene	1500	3.1	2.9	0.6
Sabinene	976	–	–	8.5	(*E*,*E*)-α-Farnesene	1508	–	–	1.9
β-Pinene	980	11.7	27.6	5.9	γ-Cadinene	1514	0.5	0.2	–
β-Myrcene	990	0.3	2.3	0.6	δ-Cadinene	1525	1.2	2.8	0.6
α-Phellandrene	1006	–	2.4	1.0	Cyclohexasiloxane dodecamethyl	1527	–	–	1.2
δ-3-Carene	1011	0.3	0.6	2.1					
α-Terpinene	1017	0.3	0.6	–	*cis*-Calamene	1529	1.4	–	–
p-Cymene	1026	–	5.2	0.3	Calacorene[b]	1546	1.0	0.4	–
Limonene	1032	1.0	5.0	–	Elemol	1550	1.0	–	–
(*Z*)-β-Ocimene	1043	–	0.1	–	Germacrene B	1561	0.5	–	–
(*E*)-β-Ocimene	1052	0.1	0.3	4.7	(*E*)-Nerolidol	1563	1.3	0.9	30.0
γ-Terpinene	1061	0.1	1.3	0.5	Ledol	1573	–	0.7	–
α-Terpinolene	1090	–	0.9	–	Spathulenol	1578	3.7	0.9	–
(*E*)-4,8-Dimethyl-1,3,7-nonatriene	1098	–	–	3.3	Caryophyllene oxide	1583	3.0	1.9	0.7
Linalool	1100	–	0.3	–	α-Cedrol	1601	14.8	–	–
Fenchyl alcohol	1110	–	0.2	–	γ-Eudesmol	1622	5.5	–	–
allo-neo-Ocimene	1135	0.7	–	–	β-Eudesmol	1651	13.8	–	–
Borneol	1167	–	0.5	–	α-Cadinol	1654	–	0.5	0.8
Terpinen-4-ol	1177	–	0.3	0.5	Zerumbone	1732	0.7	–	1.1
α-Terpineol	1189	–	0.3	0.6	Mint sulfide	1741	–	0.2	–
Bornyl formate	1205	–	0.2	–	Guaiazulene	1772	0.5	–	–
Fenchyl acetate	1206	0.4	0.3	–	Octadecane[b]	1800	–	–	0.7
Bornyl acetate	1289	–	1.2	–	Vulgarol B	1869	1.4	–	–
Bicycloelemene	1327	1.9	2.5	–	1,2-Benzenedicarboxylic acid	1917	7.3	3.4	4.4
1,5,5-Trimethyl-6-methylene-cyclohexene	1338	0.8	0.6	–	Vulgarol A	1950	1.8	–	–
					Total		95.7	95.2	91.7
Isoledene	1375	–	0.5	–	Monoterpene hydrocarbons		16.9	59.3	28.0
α-Copaene	1377	–	0.1	–	Oxygenated monoterpenes		0.4	3.3	1.1
β-Bourbonene	1385	0.7	0.5	–	Sesquiterpene hydrocarbons		22.8	23.7	20.3
β-Cubebene	1388	0.5	–	–	Oxygenated sesquiterpenes		45.7	4.9	32.7
α-Humulene	1454	1.4	–	–	Diterpenes		1.8	–	–
(*E*)-β-Farnesene	1458	–	2.1	2.3	Non-terpenes		8.1	4.0	9.6

[a]Elution order on HP-5 MS column; [b]correct isomer not identified; –: not identified.

A total of 37, 46, and 27 components, representing 95.7%, 95.2%, and 91.7% of the total oil contents, was identified from the leaves, roots, and fruits oils, respectively. In the leaf oil, sesquiterpene compounds (68.5%) predominate over the monoterpenes (17.3%). The main constituents of the leaves oil were α-cedrol (14.8%), β-eudesmol (13.8%), β-pinene (11.7%), β-caryophyllene (7.4%), and 1,2-benzenedicarboxylic acid (7.3%).

On the other hand, monoterpene compounds (62.6%) occurred in higher quantities than the sesquiterpenes (28.6%) in the root oil. The volatile compounds occurring in the root oil were β-pinene (27.6%), α-pinene (8.5%), β-caryophyllene (6.3%), *p*-cymene (5.2%), and limonene (5.0%). Also, the sesquiterpene compounds (53.0%) were prominent in the fruit oil compared to the monoterpene compounds (29.1%). However, the significant constituents of the fruit oil were identified as (*E*)-nerolidol (30.0%), β-caryophyllene (9.4%), sabinene (8.5%), and β-pinene (5.9%).

Regardless of *Zingiber* being a large genus, with mostly aromatic plants, the essential oils of *Z. nuricarpum* have not been previously investigated. Therefore, the present results may represent the first of its kind aimed at the comprehensive characterization of the volatile constituents of *Z. nuricarpum*. The chemistry of volatile compounds of *Zingiber* species grown in Vietnam have been reported [8–17]. Several terpenoid and non-terpenoid compounds have been described from the essential oils of several species in the genus. The chemical variability in the constituents of *Zingiber* oils could be described as oils with abundant phenylpropanoids such as *Z. niveum* (roots and rhizome) [8]; oils with a large amount of zerumbone such as

Z. zerumbet (roots) [9] and *Z. ottensii* (rhizome) [10]; oils with a large content of phenylbutanoids such as *Z. cassumunar* (rhizome) [11] and *Z. neesanum* (rhizome) [12]; and oils with abundant diversed monoterpene and sesquiterpene compounds that are commonly found in some other *Zingiber* species such as *Z. nitens* (all parts) [13], *Z. rufopilosum* (leaves), *Z. gramineum* (leaves) [14], *Z. rubens* (rhizome) and *Z. collinsii* (rhizome), [15], *Z. pellitum* (rhizome) [16], and *Z. purpureum* (leaves) [17].

It is well known that the essential oils and compounds of *Zingiber* have exhibited notable biological activities. The anti-inflammatory action of *Z. neesanum* [9] and cytotoxicty potential of *Z. ottensii* [17] have been reported.

ACKNOWLEDGMENT

This research is funded by the Vietnam National Foundation for Science and Technology Development (NAFOSTED) under Grant No. 106-NN.03-2017.328.

REFERENCES

1. T. L. Wu and K. Larsen, *Zingiberaceae*, in: *Flora of China*, Vol. 24, Z. Y. Wu, P. H. Raven (eds.), Science Press, Beijing & Missouri Botanical Garden Press, St. Louis, 2000, pp. 322–377.
2. N. S. Ly, *Phytotaxa*, **265**, 139 (2016).
3. N. S. Ly, V. S. Dang, D. G. Do, T. T. Tran, N. D. Dai, and D. H. Nguyen, *Biosci. Disc.,* **8**, 1 (2017).
4. T. D. Thang, D. N. Dai, and I. A. Ogunwande, *Chem. Nat. Compd*, **52**, 152 (2016).
5. D. N. Dai, L. T. Huong, and I. A. Ogunwande, *Chem. Nat. Compd*, **53**, 570 (2017).
6. *Vietnamese Pharmacopoeia*, Medical Publishing House, Hanoi, 1997, 106 pp.
7. *NIST*, Chemistry Web Book, Data from NIST Standard Reference Database, 2011, p. 69.
8. T. Orawan, J. Thaya, and M. Withawat, *Biol. Chem. Res.*, **15**, 157 (2015).
9. S. Thubthimthed, P. Limsiriwog, U. Rerkam, and T. Suntotntanasat, *ISHS Acta Hort*, **675**, 107 (2015).
10. C. B. Singh, S. B. Chanu, K. Lenin, N. Swapana, C. Cantrell, and S. A. Ross, *J. Pharmacogn. Phytochem.*, **3**, 130 (2014).
11. M. N. I. Bhuiyan, J. U. Chowdhury, and L. Begum, *Bangladesh J. Pharmacol*, **3**, 69 (2008).
12. B. Sabulal, M. Dan, J. J. Anil, R. Kurup, S. P. Chandrika, and V. George, *Flav. Fragr. J.*, **22**, 521 (2007).
13. N. V. Hung, D. N. Dai, T. H. Thai, N. D. San, and I. A. Ogunwande, *J. Essent. Oil-Bear. Pl.,* **20**, 69 (2017).
14. L. T. M. Chau, T. D. Thang, N. T. M. Tu, D. N. Dai, and I. A. Ogunwande, *Bol. Latinoam. Caribe Plantas Med.*, **14**, 449 (2015).
15. D. N. Dai, T. D. Thang, L. T. M. Chau, and I. A. Ogunwande, *Am. J. Plant Sci*, **4**, 7 (2013).
16. P. M. Giang, P. T. Son, and W. A. Konig, *J. Essent. Oil-Bear. Pl*, **14**, 494 (2011).
17. Y. Wang, C. X. You, K. Yang, Y. Wu, R. Chen, W. J. Zhang, Z. L. Liu, S. S. Du, Z. W. Deng, Z. F. Geng, and J. Han, *J. Econ. Entomol*, **108**, 925 (2015).