Chemical Science International Journal
17(3): 1-7, 2016; Article no.CSIJ.29572
Previously known as American Chemical Science Journal
ISSN: 2249-0205

SCIENCEDOMAIN *international*
www.sciencedomain.org

Essential Oil from the Fruits of *Fissistigma bracteolatum* and *Fissistigma maclurei*

Nguyen V. Hung[1], Do N. Dai[2*], Tran H. Thai[3], Tran D. Thang[1] and Isiaka A. Ogunwande[4]

[1]Faculty of Chemistry, Vinh University, 182-Le Duan, Vinh City, Nghe An Province, Vietnam.
[2]Faculty of Agriculture, Forestry and Fishery, Nghe An College of Economics, 51-Ly Tu Trong, Vinh City, Nghe An Province, Vietnam.
[3]Institute of Ecology and Biological Resources, Vietnam Academy of Science and Technology, Cau Giay, Hanoi, Vietnam.
[4]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.

Authors' contributions

This work was carried out in collaboration between all authors. Authors NVH, THT and DND collected the plant samples and performed the hydrodistillation of the oil, while authors DND and TDT designed the study and performed the instrumental analysis on the oil samples. Author IAO managed the literature searches, wrote the first and final draft of the manuscript. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CSIJ/2016/29572
Editor(s):
(1) T. P. West, Department of Chemistry, Texas A&M University-Commerce, USA.
Reviewers:
(1) Monthon Lertcanawanichakul, Walailak University, Thailand.
(2) Charu Gupta, Amity University UP, India.
Complete Peer review History: http://www.sciencedomain.org/review-history/16879

Original Research Article

Received 18th September 2016
Accepted 11th October 2016
Published 11th November 2016

ABSTRACT

Aims: The aim of this study was to isolate essential oils from the fruits of *Fissistigma bracteolatum* and *Fissistigma maclurei* (Annonaceae) and investigate the volatile constituents present they contained.
Study Design: The study involves the hydrodisitillation of essential oils from the air-dried fruit samples of *F. bracteolatum* and *F. maclurei* and analysis of their chemical compositions by GC and GC-MS.

**Corresponding author: E-mail: daidn23@gmail.com;*
Co-author: E-mail: isiaka.ogunwande@lasu.edu.ng;

Place and Duration of Study: Fruits of *F. bracteolatum* and *F. maclurei* were collected from Pù Mát National Park, Nghệ An Province, Vietnam, in May 2014. Analysis of the oil samples was performed between June and August, 2014.
Methodology: About 500 g of air-dried fruit samples was shredded and their oils were obtained by separate hydrodistillation for 4 h at normal pressure, according to the Vietnamese Pharmacopoeia specifications. 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 constituents in the fruit of *F. bracteolatum* are α-pinene (15.5%) and δ-cadinene (11.0%), with significant amounts of β-caryophyllene (8.0%) and germacrene D (7.0%) while spathulenol (21.2%) and β-cubebene (10.6%) are the quantitatively significant compounds of *F. maclurei* fruit.
Conclusion: The present oil compositions of fruit of *F. bracteolatum* and *F. maclurei* were reported for the first for these species and the results were found to differ from previous studies on other parts of the plants.

Keywords: Fissistigma bracteolatum; Fissistigma maclurei; monoterpenes; sesquiterpenes.

1. INTRODUCTION

In recent times, the analyses of chemical constituent of essential oils from Vietnamese plants have received attention [1-3]. This paper therefore present the chemical compounds identified in the fruit essential oils of two *Fissistigma* species. *Fissistigma bracteolatum* Chatt. is a scandent evergreen shrub which grows up to 10m long. The leaves are ovate-oblong in shape. Flowering takes place from March to June while fruiting occurs between August and November. This herb is used for the treatment of contusions and strains [4]. Phytochemical studies have revealed that *F. bracteolatum* contains chalcones and dihydrochalcones with potent inhibitory effects against superoxide anion production [5-8], phenanthrene [7], flavonoids [9] and alkaloids [10]. The essential oil from the leaf was reported to contained *epi*-α-muurolol (17.8%), caryophyllene oxide (13.1%), bicycloelemene (10.5%), while β-bourbonene (13.6%), (*E*)-β-ocimene (12.6%) and (*E,E*)-α-farnesene (11.2%) were the main volatiles of the stem bark [11]. The monoterpene myrcene (83%) occurred as the most abundant compound in another result [12]. However, β-myrcene (17.0%), β-caryophyllene (7.4%), δ-cadinene (6.9%), and bicyclogermacrene (6.2%) in the leaf, as well as β-caryophyllene (20.0%), δ-cadinene (9.9%), α-humulene (6.4%), bicyclogermacrene (6.3%) and linalool (6.2%) in the stem were the principal components of other investigated sample [2].

Fissistigma maclurei Merr. (syn. *Meiogyne maclurei* Sinclair) are climbers that grow up to 6 m tall. Little is known about the pharmacological potential and chemistry of this plant. The major compounds of the leaf oil of *F. maclurei* [13] were germacrene D (26.1%), spathulenol (10.0%) and α-terpinene (8.2%).

Till moment there are no reports on the chemical constituents of essential oils from the fruits of the studied plant species. This paper reports for the first time the components of fruit volatile of both *F. bracteolatum* and *F. maclurei* grown in Vietnam.

2. MATERIALS AND METHODS

2.1 Plant Material

Fruits of *F. bracteolatum* and *F. maclurei* were collected from Pù Mát National Park, Nghệ An Province, Vietnam, in May 2014. Botanical identification was carried out by Dr. Dai. Voucher specimens LVH 425, LVH 426 respectively have been deposited at the Botany Museum, Vinh University, Vietnam. Plant samples were air-dried prior to extraction.

2.2 Hydrodistillation of Essential Oil

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-type distillation unit designed according to the specification [14]. 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 Oil

Gas chromatography (GC) analysis was performed on an Agilent Technologies HP 6890 Plus Gas chromatograph equipped with a FID and fitted with HP-5MS column (30 m x 0.25 mm, film thickness 0.25 μm, Agilent Technology). The analytical conditions were: carrier gas H_2 (1 mL/min), injector temperature (PTV) 250ºC, detector temperature 260ºC, column temperature programmed from 60ºC (2 min hold) to 220ºC (10 min hold) at 4ºC/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.

An Agilent Technologies HP 6890N Plus Chromatograph fitted with a fused silica capillary HP-5 MS 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 70eV; emission current 40 mA; acquisitions scan mass range of 35-350 amu at a sampling rate of 1.0 scan/s.

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 [15] 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 [16].

3. RESULTS AND DISCUSSION

The yield of essential oils were 0.18% (v/w, *F. bracteolatum*) and 0.20% (v/w, *F. maclurei*), calculated on a dry weight basis. Oil samples were leaf light yellow in colouration. Table 1 indicates the chemical constituents present in the oils, their percentages as well as retention indices on HP-5 column.

Table 1. Percentage composition of essential oils of *F. bracteolatum* and *F. maclurei*

Compounds [a]	RI (Cal.)	RI (Lit.)	Percent composition [b]	
			F. br	***F. ma***
α-Pinene	939	932	15.5	0.7
Camphene	953	946	-	0.1
β-Pinene	980	976	-	0.2
β-Myrcene	990	988	1.4	0.1
α-Phellandrene	1006	1004	-	0.2
p-Cymene	1026	1024	-	0.5
Limonene	1032	1030	2.0	0.5
(*E*)-β-Ocimene	1052	1044	0.7	0.1
γ-Terpinene	1061	1056	2.3	0.1
α-Terpinolene	1090	1089	2.0	0.1
n-Nonanal	1106	1100	-	0.2
β-Citronellol	1228	1232	-	0.1
(*Z*)-Citral	1250	1249	-	0.4
Bornyl acetate	1289	1287	-	0.1
2-Undecanone	1291	1294	-	0.1
Bicycloelemene	1327	1337	-	2.9
α-Cubebene	1351	1345	-	0.9
α-Copaene	1377	1374	1.1	1.5
β-Bourbonene	1385	1381	-	0.2
β-Cubebene	1388	1387	5.4	10.6
α-Gurjunene	1412	1409	-	0.3
β-Caryophyllene	1419	1417	8.0	6.9
α-Guaiene	1440	1433	3.7	-
Aromadendrene	1441	1439	1.2	1.0

Compounds [a]	RI (Cal.)	RI (Lit.)	Percent composition [b]	
			F. br	*F. ma*
α-Humulene	1454	1452	4.2	2.0
Germacrene D	1485	1485	7.0	-
α-Amorphene	1485	1485	-	1.8
β-Selinene	1486	1488	4.9	-
Cadina-1,4-diene	1496	1496	-	0.4
Bicyclogermacrene	1500	1500	-	7.0
α-Muurolene	1500	1502	2.3	-
(*E*,*E*)-α-Farnesene	1506	1505	-	1.1
β-Bisabolene	1506	15	-	0.8
cis-(Z)-α-Bisabolene epoxide[c]	1515	1515	-	1.4
δ-Cadinene	1525	1522	11.0	6.9
α-Cadinene	1539	1539	1.3	-
Calacorene	1546	1541	2.6	0.6
Elemol	1550	1548	2.0	-
Germacrene B	1561	1561	2.4	-
(*E*)-Nerolidol	1563	1561	1.0	1.3
1,5-Epoxysalvial-4(14)-ene	1564	1564	-	1.0
Spathulenol	1578	1577	2.4	21.2
Caryophyllene oxide	1583	1581	4.0	6.8
Globulol	1585	1583	2.1	-
Viridiflorol	1593	1591	0.6	1.1
Longiborneol	1599	1592	-	1.1
Guaiol	1601	1600	1.2	-
α-Cedrol	1601	1601	-	1.5
β-Oplopenone	1608	1607	1.2	-
Aromadendrene epoxide	1623	1623	1.5	1.4
τ-Muurolol	1646	1640	1.1	2.3
α-Cadinol	1654	1652	-	3.8
Vulgarol B	1688	1688	-	1.2
Benzyl benzoate	1760	1759	0.5	3.1
Phytol	2125	2119	0.9	0.2
Total			97.5	95.8
Monoterpene hydrocarbons			23.9	2.6
Oxygenated monoterpenes			-	0.7
Sesquiterpene hydrocarbons			44.1	44.9
Oxygenated sesquiterpenes			28.6	47.2
Diterpenes			0.9	0.2
Non-terpenes			-	0.2

[a] Elution order on HP-5MS column; [b] SD Standard deviation, values were insignificant and were omitted from the Table to avoid congestion; RI (Cal.) Retention indices on HP-5MS column; RI (Lit.) Literature retention indices (see Experimental); [c] Tentative identification; - Not identified; F. br Fissistigma bracteolatum; F. ma Fissistigma maclurei

Thirty compounds representing 97.5% of the total oil contents were identified in the fruit of *F. bracteolatum*. The main classes of compounds present in the oil were monoterpene hydrocarbons (23.9%), sesquiterpene hydrocarbons (44.1%) and oxygenated sesquiterpenes (28.6%). Oxygen-containing monoterpene compounds are conspicuously absent in the oil. The main constituents of the oil were α-pinene (15.5%), δ-cadinene (11.0%), β-caryophyllene (8.0%), germacrene D (7.0%) and β-cubebene (5.4%). It was well noted that some compounds such as *epi*-α-muurolol, bicycloelemene, β-bourbonene, (*E*)-β-ocimene, (*E*,*E*)-α-farneseneα-humulene, bicyclogermacrene and linalool [2,11] that are characteristics of previous reports were not identified in the present oil sample. In addition, the contents of myrcene (1.4 vs.83%) and caryophyllene oxide (4.0 vs.13.1%) are low when compared with previous analysis [12].

Table 2. Main chemical constituents of previously studied *Fissistigma* oil samples

Plant	Part	Main constituents	References
F. chloroneurum	leaf	(*E*)-α-bergamotene (52.0%) and (*E*)-β-caryophyllene (9.0%)	[17]
F. cupreonitens	leaf	(*E*)-β-caryophyllene (27.7%) and bicyclogermacrene (14.7%)	[17]
F. pallens	leaf	germacrene D (30.2%), bicyclogermacrene (26.4%) and bicycloelemene (18.3%)	[17]
F. bicolor	leaf	(*E*)-β-caryophyllene (13.3%), (E)-α-bergamotene (13.1%), (*Z*)-β-farnesene (12.3%) and *p*-cymene (8.9%)	[17]
F. shangtzeense	leaf	(*E*)-β-caryophyllene (28.1%), bicyclogermacrene (10.3%) and α-muurolene (8.8%)	[17]
F. petelotii	leaf	α-pinene (15.4%), bicyclogermacrene (13.9%), bicycloelemene (10.8%) and (*E*)-β-caryophyllene (9.9%)	[17]
F. maclurei	leaf	germacrene D (26.1%), spathulenol (10.0%), α-terpinene (8.2%) and bicyclogermacrene (6.6%),	[13]
F. rufinerve	leaf	α-santalene (14.3%), beta-caryophyllene (6.3%) and terpinen-4-ol (6.3%)	[13]
F. villosissimum	leaf	bicycloelemene (13.0%), bicyclogermacrene (11.5%), spathulenol (11.4%) and germacrene D (10.6%)	[11]
F. villosissimum	stem	elema-1,3,11(13)-triene-12-ol (17.0%), germacrene D (10.3%),globulol (8.2%) and heneicosane (8.2%),	[11]
F. latifolium	leaf	γ-elemene (14.9%), dihydro carveol (14.0%), eugenol (9.2%) and viridiflorol (9.1%)	[11]
F. latifolium	stem	γ-elemene (19.0%), valerenol (16.9%), eugenol (11.6%) and viridiflorol (8.5%)	[11]
F. glaucesens	leaf	β-bourbonene (16.4%), eicosane (9.1%) and and β-caryophyllene (6.1%) docosane (5.8%) and heptadecane (5.1%)	[11]
F. glaucesens	stem	β-caryophyllene (9.7%), α-bisabolol (9.5%), γ-terpinene (7.6%), bicycloelemene (5.8%), bicyclogermacrene (5.1%) and longifolene (5.0%).	[11]
F. scandens	leaf	(*E*)-β-ocimene (14.8%) and limonene (11.0%)	[1]
F. scandens	stem	campherenone (13.0%) and (*E*)-β-ocimene (8.8%)	[1]
F. poilanei	leaf	β-elemene (33.2%), germacrene D (10.9%), bicyclogermacrene (8.7%) and bicycloelemene (8.5%)	[1]
F. poilanei	stem	β-elemene (36.9%), germacrene D (12.9%) and benzyl benzoate (10.0%)	[1]
F. acuminatissimum	leaf	β-bisabolene (14.5%), β-elemene (12.2%) and caryophyllene oxide (11.0%)	[1]
F. acuminatissimum	stem	caryophyllene oxide (15.6%), β-bisabolene (12.1%), and spathulenol (9.8%)	[1]
F. thorelii	leaf	γ-terpinene (22.0%), β-phellandrene (7.3%), and bicyclogermacrene (7.2%)	[12]
F. oldhamii	leaf	(*E*)-β-ocimene (10.2%), β-caryophyllene (23.7%) and γ-cadinene (27.2%)	[12]
F. polyanthoides	leaf	β -phellandrene (14.3%), (*E*)-β ocimene(43.4%) and α-phellandrene (8.3%)	[12]
F. rubigirosa	leaf	(*E*)-β-ocimene (21.4%) and δ-cadinene (13.3%)	[18]

On the other hand, forty-five compounds amounting to 95.8% of the total oil contents were identified in *F. maclurei* fruit oil. The classes of compounds found in the oil were mainly sesquiterpenes with 44.9% of the hydrocarbon contents and 47.2% of the oxygenated

derivatives. Monoterpenes are less common (3.3%). The constituents occurring in higher proportions were spathulenol (21.2%) and β-cubebene (10.6%), along with bicyclogermacrene (7.0%), β-caryophyllene (6.9%) and δ-cadinene (6.9%). A previous report on the leaf oil of *F. maclurei* [13] identified germacrene D (26.1%), spathulenol (10.0%) and α-terpinene (8.2%). It could be seen that spathulenol content of the fruit oil competes favourably with that of the leaf. However, germacrene D and α-terpinene were not identified in the fruit oil.

There is dirt of literature on the volatile compositions of both fruit oils and as a result the present study may represent the first of its kind. The observed compositional variations between the present oil samples and previous studies may be attributed to the different plant parts being analysed. It is well known that different parts of the same plant accumulate different phytochemicals. For example *F. scandens* leaf oil comprised mainly of (*E*)-β-ocimene and limonene while campherenone and (*E*)-β-ocimene were identified in higher proportions in the stem oil.

Literature information revealed that the chemical constituents of some Vietnamese grown *Fissistigma* have been studied and reported (Table 2). Although ubiquitous monoterpene and sesquiterpenes were the main compounds identified in this study, it is evident that each sample has its own compositional pattern different from each other.

4. CONCLUSION

In the present investigation of chemical constituents of essential oils from the fruits of *F. bracteolatum* and *F. maclurei* were found to be different from previous investigated oil samples from other parts of the plant.

CONSENT

It is not applicable.

ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Thang TD, Luu HV, Hung NH, Dai DN, Ogunwande IA. Constituents of essential oils from three Vietnamese species of *Fissistigma* (Annonaceae). Chem Nat Comp. 2016;52(1):155-8.
2. Thang TD, Dai DN, Ogunwande IA. Essential oil constituents of Vietnamese species of *Fissistigma bracteolatum* Chatt. and *Fissistigma chloroneurum* (Hand-Mazz) Tsiang. In: Utilization and Management of Medicinal Plants (Gupta, V.K., Verma, A.K. and Koul, S. ed.), Daya Publishing House, New Delhi. 2016;3:85-94.
3. Dai DN, Thang TD, Ogunwande IA. Essential oil composition of four Magnoliaceae species cultivated in Vietnam. J Herbs, Spices Med Plants. 2016;22(3):279-83.
4. Li B, Gilbert MG. Annonaceae. In: Flora of China. Wu, Z., Raven, P.H. and Hong, D. (eds.) Science Press, Beijing & Missouri Botanical Garden Press, St Louis, 2001;19:672-675,708.
5. Lien TP, Porzel A, Schmidt J, Sung TV, Adam G. Chalconoids from *Fissistigma bracteolatum*. Phytochem. 2000;53(7): 991-5.
6. Yu HL, Chen CY, Rong CF, Long HT, Chaung LC, et al. Potential anti-inflammatory activities of bractelactone and other compounds isolated from *Fissistigma bracteolatum*. Helv Chim Acta. 2005;88(4):905-9.
7. Zhu HP, Lu XL, Sun XH, Xu QZ, Jiao BH. Dihydrochalcones and phenanthrene derivatives from *Fissistigma bracteolatum.* J Med Coll PLA. 2010;25(4):226-34.
8. Wu YC, Sureshbabu M, Fang YC, Wu YH, Lan YH, Chang FR, et al. Potent inhibition of human neutrophil activations by bractelactone, a novel chalcone from *Fissistigma bracteolatum*. Appl Toxicol Pharmacol. 2013;266(3):399-407.
9. Porzel A, Phuong LT, Schmidt J, Drosihn S, Wagner C, Merzweiler K, et al. Fissistigmatins A-D. Novel type natural products with flavonoid-sesquiterpene hybrid structure from *Fissistigma bracteolatum*. Tetrahed. 2000;56(6):865-72.
10. Deng Y, Chen J, Wu FE. Two new aporphine alkaliods from *Fissistigma bracteolatum*. Chin Chem Lett. 2002;13(9): 862-4.

11. Thang TD, Luu HV, Dung VC, Tuan NN, Hung NH, Dai DN, Ogunwande IA. Chemical constituents of essential oils from the leaves and stem barks of four Vietnamese species of *Fissistigma* (Annonaceae). Nat Prod Res. 2014;28(3): 174-84.
12. Dai DN, Thang TD, Hoi TM, Nguyen XD. Chemical composition of the stems essential oil of *Fissistigma thorelii* (Pierre ex Fin. & Gagnep.) Merr. from Hatinh. Proceedings of the 3rd National Scientific Conference on Ecology and Biological Resources, Agricultural Publishing House, Hanoi, Vietnam. 2009;933-7.
13. Thang TD, Dai DN, Hoi TM, Ogunwande IA. Essential oils from five species of Annonaceae from Vietnam. Nat Prod Comm. 2013;8(2):239-42.
14. Vietnamese Pharmacopoeia. Medical Publishing House, Hanoi, Vietnam. 1997;1-134.
15. NIST. Chemistry Web Book. Data from NIST Standard Reference Database. 2011;69.

    Available:http://www.nist.gov/
16. Joulain D, Koenig WA. The atlas of spectral data of sesquiterpene hydrocarbons. E. B. Verlag, Hamburg, Germany. 1989;1-234.
17. Hoeferl M, Dai DN, Thang TD, Jirovetz L, Schmidt E. Leaf essential oils of six Vietnamese species of *Fissistigma* (Annoanceae). Nat Prod Comm. 2013;8(5): 663-5.
18. Thang TD, Dung NX. Progress in the study of some *Fissistigma* species from Vietnam. In aromatic plants from asia their chemistry and application in food and therapy. Jirovetz L, Nguyen XD, Varshney VK. (Eds). Har Krishan Bhalla & Sons, Dehradun, India. 2007;119-26.

Peer-review history:
The peer review history for this paper can be accessed here:
http://sciencedomain.org/review-history/16879