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Chemical Constituents and Cytotoxic Activities of Essential Oils from the Flowers, Leaves and Stems of *Zingiber striolatum* Diels Minyi Tian^{® 1}, Yi Hong^{® 1}, Xianghuan Wu^{® 1}, Min Zhang^{® 1}, Bing Lin^{® 1,2*} and Ying Zho^{® 1,2*}

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Abstract: The purpose of this study was to investigate chemical composition and cytotoxic activities of essential oils from *Zingiber striolatum* Diels flowers, leaves and stems. 73, 68 and 66 compounds representing 97.0%, 94.8% and 93.7% of flowers, leaves and stems essential oils were identified using GC-FID and GC-MS, respectively. The main constituents of the flowers oil were β -phellandrene (28.5%), α -humulene (14.7%), β -pinene (8.2%), β -elemene (5.5%), humulene oxide II (3.5%), cryptone (3.3%) and tricosane (3.2%). The predominant components in the leaves oil were hexahydrofarnesyl acetone (24.7%), α -humulene (12.2%), phytol (11.9%), humulene oxide II (6.3%), β -pinene (4.3%), sandaracopimaradiene (3.1%) and β -elemene (3.0%). The stems oil contained mainly α -humulene (15.6%), humulene oxide II (7.9%), hexahydrofarnesyl acetone (7.4%), phytol (7.2%), humulene oxide I (4.1%), β -elemene (3.8%) and 4-terpineol (3.2%). The cytotoxic activity against human leukemic (K562), prostatic carcinoma (PC-3) and lung cancer (A549) cell lines of essential oils was assessed using MTT assay. The essential oils of flowers, leaves and stems exhibited significant cytotoxicity against K562 (IC₅₀: 12.94–37.89 µg/mL), PC-3 (IC₅₀: 69.06–82.56 µg/mL) and A549 (IC₅₀: 45.73–66.12 µg/mL) cell lines. This is the first report on the chemical constituents and cytotoxic activities of *Z. striolatum* flowers, leaves and stems essential oils.

Keywords: Zingiber striolatum Diels; essential oils; GC-FID/MS; cytotoxic activity. © 2019 ACG Publications. All rights reserved.

1. Plant Source

The flowers, leaves and stems of *Z. striolatum* were collected from Guizhou Province of China in September 2018. Identity of the species was confirmed by Prof. Shenghua Wei of Guizhou University of Chinese Medicine. The voucher specimen (NO.1936) was deposited at Guizhou Engineering Center for Innovative Traditional Chinese Medicine and Ethnic Medicine, Guizhou University.

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2. Previous Studies

The genus *Zingiber*, as an important source of essential oil, is widely used as food and traditional medicinal plant [1,2]. *Zingiber striolatum* Diels, a perennial plant of this genus widely cultivated in China, is a unique healthy vegetable and used as a traditional Chinese medicine for treatment of abdominal pain and diarrhea [3,4]. In previous studies, the ethanol extract of *Z. striolatum* was found to possess nematicidal activity [5] and hypoglycemic activity [6]. It was reported that the major constituents of *Z. striolatum* rhizome essential oil were β -phellandrene (23.96%), sabinene (17.34%), β -pinene (11.36%) and the essential oil demonstrated significant antimicrobial and anticancer properties [7]. To the best of our knowledge, the chemical composition and cytotoxic activities of *Z. striolatum* flowers, leaves and stems essential oils have not been reported.

3. Present Study

The dry Z. striolatum flowers, leaves and stems (800 g) were separately subjected to hydrodistillation for 5 h using a Clevenger-type apparatus to obtain the essential oils. The flowers, leaves and stems essential oils were separately dried over anhydrous Na_2SO_4 and stored in amber bottle at 4°C until further analysis.

Compounds ^a	RI ^b	RI ^c		% Area	Idontificationd	
Compounds			flowers	leaves	stems	- Identification ^d
α-Pinene	933	931-939 ^{a,b,c}	1.7	0.2	-	MS, RI
Sabinene	973	954-976 ^{a,b,c,}	0.6	0.2	-	MS, RI
β -Pinene	977	973-980 ^{a,b,c}	8.2	4.3	0.2	MS, RI
β -Myrcene	992	988-991 ^{a,b,c}	1.1	0.1	-	MS, RI
α -Phellandrene	1006	1005 ^{b,c}	1.0	0.1	0.1	MS, RI
α-Terpipene	1017	1017-1018 ^{a,c}	0.1	0.1	-	MS, RI
<i>p</i> -Cymene	1025	1023-1026 ^{a,b,c}	1.4	0.1	0.1	MS, RI
β -Phellandrene	1032	1031°	28.5	1.2	0.5	MS, RI
cis-Ocimene	1038	1038-1040 ^{b,c}	0.2	-	-	MS, RI
trans-Ocimene	1048	1048-1050 ^{b,c,d}	0.9	0.1	-	MS, RI
γ-Terpinene	1059	1051-1062 ^{a,b,c}	1.1	0.1	-	MS, RI
Tetramethylpyrazine	1086	1089°	0.3	-	-	MS, RI
α-Terpinolene	1088	1082-1088 ^{a,b,c}	0.2	-	-	MS, RI
Linalool	1102	1093-1098 ^{a,b,c,d}	1.7	0.1	0.2	MS, RI
Nonanal	1104	1100-1102 ^{c,d}	-	0.1	-	MS, RI
Fenchol	1115	1113 ^c	0.2	0.1	0.2	MS, RI
<i>p</i> -menth-2-en-1-ol	1123	1126 ^c	0.5	0.1	0.1	MS, RI
Nopinone	1136	1137°	0.1	0.2	0.2	MS, RI
trans-Pinocarveol	1139	1139°	0.2	0.8	0.4	MS, RI
trans-Verbenol	1146	1144 ^c	0.1	0.2	0.1	MS, RI
Pinocarvone	1162	1162-1164 ^{b,c}	0.2	0.2	0.2	MS, RI
Borneol	1167	1165-1167 ^{b,c}	0.4	0.1	0.3	MS, RI
Nonanol	1172	1165-1173 ^{c,d}	0.2	-	0.2	MS, RI
4-Terpineol	1179	1169-1177 ^{a,b,c,}	1.3	0.7	3.2	MS, RI
Cryptone	1187	1184°	3.3	0.4	2.7	MS, RI
α -Terpineol	1193	1177-1189 ^{a,b,c}	0.7	0.5	1.1	MS, RI
Myrtenal	1196	1193 ^{b,c}	0.1	0.3	0.4	MS, RI
Myrtenol	1198	1994-1213 ^{b,c}	0.5	1.7	1.3	MS, RI
Sabinol	1204	1143°	0.2	-	-	MS, RI
Decanal	1206	1206 ^c	-	-	0.3	MS, RI
trans-Piperitol	1209	1208 ^c	0.3	0.1	0.1	MS, RI
trans-Carveol	1221	1217°	0.1	0.2	0.3	MS, RI
Cuminic aldehyde	1241	1239°	0.2	-	0.1	MS, RI
Carvone	1244	1243°	-	-	0.1	MS, RI
Piperitone	1254	1253°	0.1	0.1	0.2	MS, RI
2-Phenylcrotonaldehyde	1273	1274 ^c	0.1	0.1	-	MS, RI
Phellandral	1275	1273°	1.4	0.1	0.6	MS, RI
Borneol acetate	1286	1284-1285 ^{b,c}	0.1	-	0.1	MS, RI

Table 1. Chemical composition of essential oils from the flowers, leaves and stems of Z. striolatum

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$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Cuminic alcohol	1293	1289°	0.2	-	0.2	MS, RI
Myrtenyl acetate 1326 1327° 0.1 0.4 0.2 MS, R a-Cobebene 1351 1351' 0.1 0.1 MS, R a-Copaene 1377 1376 ^{b,c} 0.3 0.1 0.6 MS, R β -Bourhonene 1386 1384 ^{b,c} 0.2 0.4 0.5 MS, R β -Caryophyllene 1407 1404-1415 ^{b,c} - - 1.8 MS, R α -Gurjunen 1411 1406 ^c 0.1 0.1 0.1 MS, R α -Conone 1429 1420 ^c -1427 ^{cd} - 0.5 - MS, R α -Ionone 1435 1435 ^{b,d} 0.2 0.1 2.3 MS, R α -Innene 1435 1435 ^{c,d} - 0.5 - MS, R α -Innene 1435 1435 ^{c,d} - 0.2 0.1 2.3 MS, R α -Innene 1448 1445 ^{c,d} 0.1 - - MS, R α -Innene 1448 1448 ^{c,d} 0.3 0.1 - MS, R				0.3		0.2	MS, RI
a^{-} Cubene 1351 1351 ^s 0.1 0.1 0.1 0.1 0.1 0.6 MS, R a^{-} Copane 137 1376 ^{b,c} 0.3 0.1 0.6 MS, R a^{-} Caryophyllen 1397 1391 ^{b,c} 5.5 3.0 3.8 MS, R a^{-} Caryophyllen 1407 1404-1415 ^{b,c} - - 1.8 MS, R a^{-} Caryophyllene 1411 1400 ^s 0.1 0.1 0.1 MS, R a^{-} Caryophyllene 1421 1424-1427 ^{s,d} - 0.5 . MS, R a^{-} Caryophyllene 1435 1432 ^{h,d} - 2.5 . MS, R a^{-} Chunne 1435 1442 ^{h,d} - 2.5 . MS, R a^{-} Itumulene 1453 1456-1481 ^{h,b,c} 0.1 - . MS, R a^{-} Itumulene 1487 1448 ^{h,c,d} 0.4 0.3 1.0 MS, R a^{-} Itumilene 1487 1448 ^{h,c,d} 0.4 0.3 1.0 MS, R a^{-} Itake 1497							MS, RI
$a-Copane$ 137 1376 ^{he} 0.3 0.1 0.6 MS, R β -Bourbonene 1386 1384 ^s 0.2 0.4 0.5 MS, R α -Caryophyllene 1407 1404-1415 ^{he} - - 1.8 MS, R α -Caryophyllene 1421 1418-1425 ^{hbb} 2.5 2.5 1.0 MS, R α -Conone 1429 1426-1427 ^{red} - 0.5 - MS, R α -Ionone 1435 1433 ^{he} 0.2 0.1 2.3 MS, R α -Immulene 1455 1452-1454 ^{e-4} - 2.5 - MS, R α -Humulene 1455 1452-148 ^{heb} 0.1 - - MS, R α -Humulene 1485 1456-148 ^{heb} 0.1 - - MS, R α -Humulene 1487 1485-148 ^{heb} 0.1 - - MS, R β -Bonone 1487 1495 ^{cd} - - 1.1 MS, R β -Solinene 1488 148 ^{heb} 0.4 0.3 0.1 - <	5 5						MS, RI
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$a-Garjophyllene 1411 1400° 0.1 0.1 0.1 N. N. R. \beta-Caryophyllene 1421 1418-1425nbd 2.5 2.5 1.0 MS. R. q-Inome 1423 1433hb 0.2 0.1 2.3 MS. R. q-Humulene 1435 1433hb 0.2 0.1 2.3 MS. R. a-Humulene 1455 1452-1454id - 0.5 - MS. R. a-Humulene 1453 1452-1454id - 2.5 - MS. R. A-tomadendrene 1463 1469° - 0.1 - - MS. R. A-tomadendrene 1483 1455-1481hbc 0.1 - - MS. R. \beta-Selinene 1485 1485hd 0.4 0.3 1.0 MS. R. \alpha_1-conne 1497 1494-1497hd - - MS. R. \alpha_1-conne 1497 1494-1497hd - - MS. R. $							MS, RI
$\begin{array}{cccccccccccccccccccccccccccccccccccc$							MS, RI
1 -Lonon 1439 1433 ^{hc} 0.2 0.1 2.3 MS, R γ -Elemene 1435 1433 ^{hc} 0.2 0.1 2.3 MS, R Geranylacetone 1455 1452-1454 ^{hcd} - 2.5 - MS, R α -Humulene 1458 1454 ^{hcf} 1.4 12.2 15.6 MS, R Aromadendrene 1483 1456-1481 ^{hbf} 0.1 - - MS, R β -Bonne 1487 1485 ^{idh} 0.1 - - MS, R β -Selinene 1488 1485 ^{idh} 0.4 0.3 1.0 MS, R β -Selinene 1497 1494-1497 ^{r,d} - - 1.1 MS, R Pentadecane 1501 1500 ^{f,d} 1.8 0.1 0.5 MS, R Germacrene A 1507 1504 ^s 0.3 0.6 1.3 MS, R β -Branesene 1510 1509 ^c 0.5 0.2 - MS, R β -Branesene 1510 1509 ^c 0.5 0.2 - MS, R							MS, RI
y-Elemene 1435 1433^{hc} 0.2 0.1 2.3 MS, R Geranylacetone 1455 $1452-1454^{hcd}$ $ 2.5$ $-$ MS, R MS, R Aromadendrene 1463 1469 ^c $ 0.1$ 0.3 MS, R Germacrene D 1483 1456-1481 ^{abac} 0.1 $ -$ MS, R β -Seniene 1488 1485-1486 ^{bac} $ 0.8$ 0.4 MS, R β -Seniene 1488 1485-1486 ^{bac} $ 0.8$ 0.4 MS, R β -Tridecanone 1497 1495-1500 ^{bac} 0.7 0.8 $-$ MS, R β -Tridecanone 1501 1500 ^{abd} 0.3 0.6 1.3 MS, R β -Farnesene 1510 1508 ^{abde} 0.3 0.6 1.3 MS, R β -Branesene 1510 1509 ^{bb} 0.5 0.2 $-$ MS, R β -Sequiphellandrene 1520 1524 ^s 0.4 $-$ -							MS, RI
Geranylacetone 1455 1452-1454 ^{c,4} - 2.5 - MS, R a -Hamulene 1458 1454 ^{b,c} 14.7 12.2 15.6 MS, R Aromadendrene 1463 1469 ^c - 0.1 0.3 MS, R Germacrene D 1483 1455-1481 ^{h,b,c} 0.1 - - MS, R β -Sclinene 1488 1485-1486 ^{h,c,d} 0.4 0.3 1.0 MS, R 2 -Tridecanone 1497 1495-1500 ^{h,c,d} - - 1.1 MS, R Bicyclogermacrene 1501 1500 ^{h,c,d} 0.3 0.6 1.3 MS, R β -Farnesene 1510 1509 ^{h,b,c} 0.3 0.6 1.3 MS, R β -Bisabolene 1510 1509 ^{h,b,c} 0.5 0.2 - MS, R β -Bisabolene 1510 1509 ^{h,b,c} 0.5 0.2 - MS, R β -Bisabolene 1510 1509 ^{c,6} 0.3 0.2 - MS, R β -Bisabolene 1510 1509 ^{c,6} 0.3 0.2 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td></td></t<>							
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2-Tridecanone 1497 1494-1497-d - - 1.1 MS, R Bicyclogermacrene 1497 1495-1500 ^{a,c} 0.7 0.8 - MS, R Perntadecane 1501 1500 ^{c,d} 1.8 0.1 0.5 MS, R β -Franesene 1510 1500 ^{c,d} 0.3 0.6 1.3 MS, R β -Bisabolene 1510 1500 ^{c,d} 0.5 0.2 - MS, R β -Branesene 1559 1557 ^c 0.5 0.2 - MS, R Germacrene B 1559 1557 ^c 0.5 0.2 - MS, R Nerolidol 1581 1572-1576 ^{c,d} 0.8 1.9 2.8 MS, R Caryophyllene oxide 1585 1581 ^{b,c} 0.7 2.2 2.3 MS, R Humulene oxide II 1615 1606 ^{b,c} 3.5 6.3 7.9 MS, R a -Bisabolol 1690 1683-1685 ^{b,c} - 0.1 0.4 MS, R Pentadecanal 1715 1715 ^c 0.2 - 0.8 <td< td=""><td></td><td></td><td></td><td></td><td></td><td></td><td>MS, RI</td></td<>							MS, RI
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Others 11.1 6.1 15.2		S					
	Others Total			11.1 97.0	6.1 94.8	15.2 93.7	

^aCompounds are listed in order of their elution from a FB-5MSi column; ^bRI: Retention index on FB-5MSi column, calculated using homologous series of C₉–C₃₀ alkanes; ^cRI: Retention index of literature; a) [8], b) [9], c) NIST 14 and Wiley 275 databases, d) [10]; ^dIdentification: MS, based on comparison with Wiley 275 and NIST 14 MS databases; RI, based on comparison of calculated RI with those reported in the literature, Wiley 275 and NIST 14 databases; - : not detected.

The yields of the hydrodistilled flowers, leaves and stems essential oils were 0.39% (w/w), 0.27% (w/w) and 0.18% (w/w) of dry weight, respectively. The essential oils were identified and quantified by GC-FID and GC-MS and the results were presented in Table 1. A total of 73, 68 and 66 compounds representing 97.0%, 94.8% and 93.7% of the total oil were identified in essential oils of flowers, leaves and stems, respectively. The chemical constituents of the essential oils were classified as monoterpene hydrocarbons (0.9-45.0%), oxygenated monoterpenes (5.3-8.9%), sesquiterpene hydrocarbons (21.1-30.0%), oxygenated sesquiterpenes (5.6-20.6%), diterpenes (0.3-40.6%) and others (6.1-15.2%). The main constituents of the flowers oil were β -phellandrene (28.5%), α -humulene (14.7%), β -pinene (8.2%), β -elemene (5.5%), humulene oxide II (3.5%), cryptone (3.3%) and tricosane (3.2%). The major components of the leaves oil were hexahydrofarnesyl acetone (24.7%), α -humulene (12.2%), phytol (11.9%), humulene oxide II (6.3%), β -pinene (4.3%), sandaracopimaradiene (3.1%) and β -elemene (3.0%). The stems oil contained mainly α -humulene (15.6%), humulene oxide II (7.9%), hexahydrofarnesyl acetone (7.4%), phytol (7.2%), humulene oxide I (4.1%), β -elemene (3.8%) and 4terpineol (3.2%). In our previous study, the main components of the Z. striolatum rhizomes oil were β phellandrene (23.96%), sabinene (17.34%), β -pinene (11.36%), geranyl linalool (8.56%), 4-terpineol (8.27%), α -pipene (5.56%) and crypton (4.49%) [7]. These compounds are present in different ratios in other Zingiber species. For example, β -phellandrene was the main compound of Z. spectabilis inflorescence oil, which was a key ingredient of cleaning, cosmetic and medical products [11,12]. α -Humulene was found to be the main constituent in Z. zerumbet rhizome essential oil, which was known for its anti-inflammatory property [13,14]. Hexahydrofarnesyl acetone and phytol have been reported to be the major constituents in Z. chrysanthum leaf hexane extract, and hexahydrofarnesyl acetone was the oxidation product of phytol [15,16]

In the previous studies, essential oils from genus *Zingiber* have been characterized. The major constituents in the essential oil from *Z. nimmonii* rhizome were β -caryophyllene (42.2%) and α -humulene (27.7%) [1]. The most abundant constituents in the essential oil from *Z. officinale* rhizome were reported as zingiberene and *ar*-curcumene [1]. Zerumbone was the predominant component in the rhizome oil of *Z. zerumbet*, but the major components of its leaves and flowers oil were (*E*)-nerolidol, β -caryophyllene and linalool [9,13]. The main components of *Z. cassumunar* rhizome essential oil were terpinen-4-ol (40.5%) and sabinene (17.4%) [17]. The major components of the essential oil from *Z. spectabile* leaf were β -caryophyllene (21.3%) and β -elemene (12.5%), the main component in its rhizome oil was zerumbone (59.1%) [18]. The main constituents of the rhizome oil from *Z. anamalayanum* were δ -2-carene (52.83%), camphene (9.83%), endo-fenchol (9.42%) [19]. In the present study, the most abundant constituents in *Z. striolatum* flowers, leaves and stems essential oils were β -phellandrene (0.5-28.5%), α -humulene (12.2-15.6%), hexahydrofarnesyl acetone (0.2-24.7%) and phytol (0.1-11.9%). The chemical constituents of *Z. striolatum* essential oils varied according to different parts.

The cytotoxic activities of *Z. striolatum* flowers, leaves and stems essential oils were evaluated against human leukemic (K562), prostatic carcinoma (PC-3) and lung cancer (A549) cell lines using MTT assay. The results were presented in Table 2. The cytotoxic activities of the essential oils were compared with cisplatin as positive control. The essential oils of flowers, leaves and stems exhibited significant cytotoxicity against A549 (IC₅₀: 45.73–66.12 µg/mL), PC-3 (IC₅₀: 69.06–82.56 µg/mL) and K562 (IC₅₀: 12.94–37.89 µg/mL) cell lines in a concentration-dependent manner. The cytotoxic activities of the flowers, leaves and stems essential oils can be attributed to the high content of terpenoids, such as α -humulene, β -pinene, β -elemene and phytol, which have been reported to have cytotoxic activity [20-23]. In addition, β -caryophyllene, as a chemical component with less content in *Z. striolatum* essential oils, significantly potentiated the cytotoxic activity of α -humulene against human breast adenocarcinoma cell line (MCF-7) [24]. Therefore, the cytotoxic activities of *Z. striolatum* flowers, leaves and stems essential oils may be attributed to the specific chemical constituents, and/or synergies between various components.

Z. zerumbet rhizome oil exhibited significant cytotoxicity towards human cancer cell lines (A549, MDAMB-231, A431, K562, WRL-68, and HaCaT) with IC₅₀ values ($33.37-46.02 \mu g/mL$) [13]. *Z. officinale* essential oil was found strong cytotoxic activity against human cancer cell lines (HO-8910)

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and Bel-7402) and IC₅₀ values were 0.00643 and 0.00256% (v/v), respectively [25]. In the present study, the essential oils of *Z. striolatum* flowers, leaves and stems exhibited significant cytotoxicity against A549 (IC₅₀: 45.73–66.12 µg/mL), PC-3 (IC₅₀: 69.06–82.56 µg/mL) and K562 (IC₅₀: 12.94–37.89 µg/mL) cell lines. The cytotoxic activity of the essential oils against K562 cell line was found significantly higher than that against PC-3 and A549 cell lines (p < 0.05).

Table 2. Cytotoxic activity of essential oils from the flowers, leaves and stems of Z. striolatum

Treatment	Cel	a	
Treatment	K562 ^b	PC-3 ^c	A549 ^d
Flowers	26.75 ± 1.28	82.56 ± 2.83	52.65 ± 1.82
Leaves	37.89 ± 1.67	77.20 ± 1.87	45.73 ± 1.56
Stems	12.94 ± 1.38	69.06 ± 2.31	66.12 ± 2.34
Cisplatin	3.42 ± 0.28	10.26 ± 0.64	7.98 ± 0.23

^aIC₅₀: The concentration of compound that affords a 50% reduction in cell growth (after 48 h of incubation); ^bHuman leukemic cell line; ^cHuman prostatic carcinoma cell line; ^dHuman lung cancer cell line.

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Supporting Information

Supporting Information accompanies this paper on <u>http://www.acgpubs.org/journal/records-of-natural-products</u>

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