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Essential Oils from the Leaves of Six Medicinal Plants of Nigeria[#]

Isiaka A. Ogunwande[a,*], Nudewhenu O. Avoseh[a], Guido Flamini[b], Alimot-Sadiat O. Hassan[a], AbdulRazaq O. Ogunmoye[c], Akindele O. Ogunsanwo[a], Kamorudeen O. Yusuf[a], Atuonwu O. Kelechi[a], Zainab A. Tiamiyu[a] and Godgift O. Tabowei[a]

[a]*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*

[b]*Dipartimento di Scienze Farmaceutiche, sede Chimica Bioorganica e Biofarmacia, Universita di Pisa, Via Bonanno 33, 56126 Pisa, Italy*

[c]*Department of Chemistry, Crescent University, Abeokuta, Ogun State, Nigeria*

isiaka.ogunwande@lasu.edu.ng

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The chemistry of *Cassia siamea* L., *C. occidentalis* L. (Fabaceae), *Cnestis ferruginea* Vahl ex DC (Connaraceae), *Anthocleista djalonensis* A. Chev (Loganiaceae), *Solanum torvum* Swartz and *S. erianthum* G. Don (Solanaceae) volatiles grown in Nigeria have been studied. The essential oils were obtained by hydrodistillation and analyzed by GC and GC-MS. The main compounds of *C. siamea* were (*E*)-geranyl acetone (5.8%), 1-octen-3-ol (5.8%), linalool (7.8%), *iso*-italicene (15.4%) and (*E*)-β-damascenone (11.0%). On the other hand, *C. occidentalis* consisted mainly of (*E*)-geranyl acetone (8.0%), hexahydrofarnesylacetone (24.0%) and (*E*)-phytol acetate (40.7%). The oil of *C. ferruginea* was comprised mainly of (*E*)-geranyl acetone (13.7%), (*E*)-α-ionone (9.5%), phytol (5.8%), pentadecanal (6.1%) and 1-octen-3-ol (5.5%). The main compounds of *A. djalonensis* were α-humulene (31.9%), β-caryophyllene (17.8%), humulene epoxide II (12.7%) and caryophyllene oxide (5.9%). The main volatiles of *S. torvum* were *(E)*-phytol acetate (38.7%), pentadecanal (25.3%) and *(E)*-geranyl acetone (5.0%). Apart from methyl salicylate (4.5%), tetradecanal (2.2%), 2-pentyl furan (1.8%), hexahydrofarnesylacetone (1.6%) and hexadecanal (1.1%), all other compounds were either present in trace quantity or in amounts less than 1%. On the other hand, α-humulene (46.6%) and β-caryophyllene (20.6%) were the compounds occurring in higher quantities in *S. erianthum*. The volatile oil contents of *Cassia siamea, Cnestis ferruginea, Anthocleista djalonensis* and *Solanum torvum* are being reported for the first time.

Keywords: *Cassia siamea*, *Cassia occidentalis*, *Cnestis ferruginea*, *Anthocleista djalonensis*, *Solanum torvum, Solanum erianthum*, Essential oil composition.

Cassia or *Senna* is a large genus of flowering plants in the family Fabaceae, subfamily Caesalpinioideae. This diverse genus is native throughout the tropics, with a small number of species reaching into temperate regions. The number of species is usually estimated to be about 260 [1], but some authors believe that there are as many as 350. The *Cassias* are typically shrubs or subshrubs, some becoming scandent when growing into other vegetation. *Cassia* species make good ornamental plants and are used for landscape gardening. Some are herbs or small trees. The fruit is a legume, indehiscent or tardily dehiscent. The present investigation reports on the volatile compounds identified in the leaf oils of *Cassia siamea* L. and *C. occidentalis* L. These plants have been studied extensively for their pharmacological activities and phytochemistry, and a large number of active compounds have been isolated and characterized [2-7]. However, there is little literature information on the volatile contents of these and several other *Cassia* species. The authors are aware of three reports on the oil contents of *C. alata* [8-10], and one report each on *C. occidentalis* [9], *C. hirsuta* [9], *C. fistula* [11], and *C. grandis* [12]. *C. alata* and *C. occidentalis* essential oils were found to be cytotoxic, inhibiting the growth of Hs 578T human tumor breast cell lines. Moreover, *C. alata, C. hirsuta* and *C. occidentalis* oils displayed only moderate antimicrobial activities to the assayed standard strains of *Bacillus cereus, Staphylococcus aureus, Pseudomonas aeroginosa, Escherichia coli, Candida albicans* and *Aspergillus niger* [9]. The antioxidant activity of the oil of *C. alata* was reported to be low compared with that of butylated hydroxytoluene (BHT) [10].

The shrub, *Cnestis ferruginea* Vahl ex DC (Connaraceae) is native to Africa, with local names such as 'Oko-Aja' or 'Gboyín-Gboyín'. It is the commonest of the 13 species of forest shrubs and climbers of the genus *Cnestis*. It bears orange-red fruits with velveting hairs on the follicle. Extracts or compounds of *C. ferruginea* have been shown to possess analgesic, anti-inflammatory [13, 14], antidepressant, anxiolytic [15], and anticonvulsant activities [16]; inhibit haemoglobin glycosylation *in vitro* [17]; possess hypoglycemia activity [18]; hepatoprotective potential [19] and improve sexual dysfunction [20]. The light petroleum fraction of *C. ferruginea* fruit has been shown to contain octacosanyl stearate and 1-myristo-2-stearo-3-palmitin [21]. A novel isoflavone glycoside, afrormosin-7-*O*-β-D-galactoside with antimicrobial activity was isolated from the fruit testa [22]. Squalene, myricyl alcohol, β-sitosterol, cyanidin, delphinidin and apigenidin have also been isolated from the plant [23]. No report on its volatile contents could be found in the literature.

Anthocleista djalonensis A. Chev (Loganiaceae) is a small tree up to 15 m tall, with a bole up to 40 cm in diameter; twigs sometimes with 2 erect spines or small cushions above the leaf axils. It is widely used throughout its distribution area as a strong purgative and diuretic [24]. A root decoction is commonly taken to treat constipation, to regulate menstruation and as an abortifacient. It is used as a wash, bath or as a vapour bath to treat leprosy, venereal diseases, oedema and scrotal elephantiasis. A root infusion is taken to treat intestinal problems, acute inflammations, and boils on the

skin [25,26]. The plant is known to possess anti-inflammatory [27] and free radical scavenging activities [28]. The plant is a source of secondary metabolites which are cytotoxic [29, 30] and antifungal [31].

The genus *Solanum* (Solanaceae) is comprised of 1700 species commonly found in the temperate and tropical regions of the world [32]. The genus is represented by some 25 species in Nigeria, including *S. wrightii* Benth, *S. torvum* Swartz, *S. melongena* L., *S. tuberosum* L., *S. mammorum* L. and *S. seaforthianum* Andr. (var. *disjunctum*). *S. erianthum* D. Don is a shrub or small tree about 6 m high with dense soft stellate hairs. The leaves act as an abortifacient and are considered a potent medicine for expelling all impurities through the urine and in particular to treat leucorrhoea [32]. The plant is also used to treat stomach ache, sores in the mouth and applied externally to skin irritations and rashes. *S. torvum* Swartz., is a bushy, erect and spiny perennial plant used horticulturally as a rootstock for eggplant. The plant is usually 2 or 3 m in height and 2 cm in basal diameter, but may reach 5 m in height and 8 cm in basal diameter. The shrub usually has a single stem at ground level, but it may branch on the lower stem. The fruits are berries that grow in clusters of tiny green spheres (ca. 1 cm in diameter) that look like green peas. They become yellow when fully ripe. They are thin-fleshed and contain numerous flat, round, brown seeds. These plants have been studied extensively for their pharmacological activities and phytochemistry, where a large number of active compounds have been isolated and characterized [33-38]. *S. erianthum* leaf volatile oil was reported to have potent inhibitory activity against Hs 578T and PC-3 human breast and prostate tumor cells, respectively. In addition, *S. erianthum* and *S. macranthum* essential oils exhibited significant antimicrobial activity (19.5–625 μg/mL) on pathogens employed in the assay [39].

In continuation of our studies on the volatile constituents of Nigerian medicinal plants and herbs [8, 9, 39], we report herein compounds identified from the above mentioned plant samples. The essential oil yields were: 3.4%, v/w, *C. siamea*, pale yellow; 0.15%, v/w, *C. occidentalis*, light yellow; 0.14%, v/w, *C. ferruginea*; brownish; 0.06%, v/w, *A. djalonensis*, greenish yellow; 0.51%, v/w, *S. torvum*, pale yellow; and 0.14%, v/w, *S. erianthum*, pale yellow, calculated on a dry weight basis.

The compounds identified in *C. siamea* and *C. occidentalis* can be seen in Table 1. The ubiquitous terpenoid compounds were mostly represented among the volatile contents. Oxygenated monoterpenes and sesquiterpene hydrocarbons were the main classes of compound in *C. siamea*, while the oil of *C. occidentalis* consisted largely of diterpenoids and oxygenated sesquiterpenoids.

The main compounds of *C. siamea* were *iso*-italicene (15.4%), (*E*)-β-damascenone (11.0%), linalool (7.8%), 1-octen-3-ol (5.8%) and (*E*)-geranyl acetone (5.8%). However, (*E*)-phytol acetate (40.7%) and hexahydrofarnesylacetone (24.0%) occurred in higher amounts in *C. occidentalis*. The oil also features significiant amounts of *(E)*-geranyl acetone (8.0%), β-caryophyllene (4.1%), (*E*)-β-ionone and farnesylacetone (ca. 3.7%). The oil of *C. occidentalis* previously analysed from Nigeria contained (*E*)-phytol (26.0%), hexadecanoic acid (17.3%) and 6,10,14-trimethyl-2-pentadecanone (9.9%) as major compounds [9]. However, hexadecanoic acid and 6,10,14-trimethyl-2-pentadecanone could not be identified in the present investigation, while the phytol content was low. It may be postulated that the oil of *C. occidentalis* from Nigeria could exist in two chemical forms, one with an abundance of diterpenoid and oxygenated sesquiterpenoids (this study), and another whose major compounds were fatty acids and aliphatic compounds [9].

Table 1: Volatile compounds identified from *C. siamea* and *C. occidentalis*.

Constituents	LRI [a]	LRI[b]	*C.s*	*C. o*	MI
(*E*)-2-Hexenal	855	846	0.5	0.4	Cmrl
2-Heptanone	890	889	-	Tr	Cmrl
Heptanal	900	901	-	Tr	Cmrl
Benzaldehyde	962	952	Tr	0.4	Cmrl
1-Octenol	978	974	5.8	-	Cmrl
6-Methyl-5-hepten-2-one	986	981	1.1	0.8	Cmrl
2-Pentyl furan	986	984	0.8	1.2	Cmrl
trans-2-(2-pentenyl) Furan	1000	-	0.6	-	Cmr
δ-2-Carene	1001	1001	-	0.9	Cmrl
cis-2-(2-pentenyl) Furan	1004	-	-	0.2	Cmr
ρ-Methyl anisole	1019	1015	1.3	-	Cmrl
(*E*)-β-Ocimene	1051	1044	Tr	-	Cmrl
trans-Linalool oxide (furanoid)	1084	1084	Tr	-	Cmrl
cis-Linalool oxide (furanoid)	1088	1087	0.7	-	Cmrl
Linalool	1099	1095	7.8	-	Cmrl
Nonanal	1103	1100	0.6	0.4	Cmrl
2,3-Dimethyl anisole	1105	-	0.9	-	Cmr
neo-allo-Ocimene	1144	1140	0.5	-	Cmrl
Veratrole (=*o*-Methyl anisole)	1146	1141	0.7	-	Cmrl
1,4-Dimethoxy benzene	1164	1163	Tr		Cmrl
1,3-Dimethoxy benzene	1169	1165	-	0.3	Cmrl
2,4-Dimethyl benzaldehyde	1180	-	0.7	-	Cmr
Naphthalene	1182	1178	0.6	Tr	Cmrl
α-Terpineol	1190	1186	1.4	-	Cmrl
Methyl salicylate	1192	1190	0.5	-	Cmrl
β-Cyclocitral	1217	1217	1.6	0.6	Cmrl
Geraniol	1256	1249	1.3	-	Cmrl
neo-3-Thujanol acetate	1276	1273	0.6	-	Cmrl
7-*epi*-Silphiperfol-5-ene	1348	1345	1.0	-	Cmrl
α-Terpinyl acetate	1350	1346	1.1	-	Cmrl
(*E*)- β-Damascenone	1381	1383	11.0	0.3	Cmrl
2,7-Dimethyl naphthalene	1392	-	-	0.6	Cmr
iso-Italicene	1400	1401	15.4	-	Cmrl
Methyl eugenol	1404	1401	0.6	-	Cmrl
Longifolene	1407	1407	-	Tr	Cmrl
2,6-Dimethyl naphthalene	1409	-	-	0.5	Cmr
1,7-Dimethyl naphthalene	1416	-	0.8	-	Cmr
β-Caryophyllene	1418	1418	2.2	4.1	Cmrl
β-Ylangene	1421	1419	0.4	-	Cmrl
trans-Dictamnol	1428	1428	0.8	-	Cmrl
β-Gurjunene	1432	1431	-	0.6	Cmrl
γ-Elemene	1437	1434	-	tr	Cmrl
trans-α-Bergamotene	1439	1432	-	0.5	Cmrl
1-Methoxy naphthalene	1446	1444	1.0		Cmrl
(*E*)-Geranyl acetone	1454	1453	5.8	8.0	Cmrl
γ-Muurolene	1477	1478	-	0.3	Cmrl
(*E*)-β-Ionone	1485	1487	1.9	3.7	Cmrl
cis-Eudesma-6,11-diene	1489	1489	3.1	-	Cmrl
β-Selinene	1490	1489	0.4	-	Cmrl
Viridiflorene	1493	1496	0.4	-	Cmrl
(*E*)-Methyl eugenol	1495	1494	0.6	-	Cmrl
α-Selinene	1498	1498	Tr	0.4	Cmrl
(*Z*)-α-Bisabolene	1504	1506	2.2	Tr	Cmrl
β-Curcumene	1512	1514	0.6	-	Cmrl
(*E*)-Dihydroapofarnesal	1520	1520	0.5	-	Cmrl
δ-Cadinene	1524	1522	1.9	0.9	Cmrl
β-Thujaplicinol	1536	1529	1.4	1.0	Cmrl
α-Calacorene	1542	1544	1.1	-	Cmrl
Elemicin	1554	1555	0.5	-	Cmrl
(*E*)-Nerolidol	1564	1561	1.0	0.5	Cmrl
Caryophyllene oxide	1581	1582	-	0.6	Cmrl
Gleenol	1585	1586	-	0.7	Cmrl
Longiborneol	1594	1599	-	0.4	Cmrl
β-Oplopenone	1604	1607	-	0.1	Cmrl
Selin-11-en-4α-ol	1652	1656	0.5	-	Cmrl
α-Cadinol	1653	1652	-	0.3	Cmrl
Cadalene	1674	1675	-	0.6	Cmrl
Pentadecanal	1717	1717	-	0.5	Cmrl
Hexahydrofarnesylacetone	1845	1843	1.2	24.0	Cmrl
Farnesylacetone [c]	1927	1913	1.5	3.7	Cmrl
Phytol	1950	1942	-	0.4	Cmrl
Hexadecanoic acid	1959	1959	2.8	-	Cmrl
Abietatriene	2054	2055	1.0	-	Cmrl
(*E*)-Phytol acetate	2218	2218	-	40.7	Cmrl
Total			**91.0**	**98.6**	
Monoterpene hydrocarbons			**1.5**	**0.9**	
Oxygenated monoterpenes			**24.8**	**12.3**	
Sesquiterpene hydrocarbons			**39.2**	**7.8**	
Oxygenated sesquiterpenes			**6.1**	**31.2**	
Diterpenoids			**1.0**	**41.1**	
Aliphatic/fatty acids			**10.2**	**2.1**	
Aromatic compounds			**7.4**	**3.2**	

[a]Retention indices on HP-5MS capillary column; [b]Literature retention indices ([42] and lower version); M.I = Modes of identification: cmrl, Co-injection, mass fragmentation pattern, Retention indices from column and Literature Retention indices; cmr, Co-injection, Mass fragmentation pattern, Retention indices from column, literature other than [42] and its lower versions; [c] correct isomer not identified; - not identified and not present in literature; Tr < 0.1%; *C.s* = *C. siamea*; 2, *C.o* = *C. occidentalis*.

Table 2: Compounds identified from *C. ferruginea*.

Constituents	LRI [a]	LRI [b]	Percent	M.I
(*E*)-2-Hexenal	855	846	1.2	Cmrl
1-Hexanol	868	863	0.3	Cmrl
2-Heptanone	890	889	Tr	Cmrl
Heptanal	900	901	1.1	Cmrl
Benzaldehyde	962	952	0.9	Cmrl
1-Heptanol	970	959	0.4	Cmrl
1-Octen-3-ol	978	974	5.5	Cmrl
6-Methyl-5-hepten-2-one	986	981	3.8	Cmrl
2-Pentyl furan	986	984	2.3	Cmrl
δ-2-Carene	1001	1001	0.7	Cmrl
trans-2(2-pentenyl) Furan	1004	-	1.4	Cmr
Octanal	1005	998	Tr	Cmrl
Dihydro-tagetone	1050	1046	0.4	Cmrl
m-Tolualdehyde	1068	1064	0.7	Cmrl
1-Octanol	1071	1063	Tr	Cmrl
Terpinolene	1088	1086	0.6	Cmrl
Linalool	1099	1095	1.9	Cmrl
Nonanal	1103	1100	3.5	Cmrl
Isophorone	1120	1118	0.6	Cmrl
4-keto-Isophorone	1143	1140	0.6	Cmrl
iso-Isopulegol	1155	1145	Tr	Cmrl
(*E*)-2-Nonenal	1162	1157	Tr	Cmrl
2,4-Dimethyl Benzaldehyde	1171	-	1.6	Cmr
Menthol	1174	1167	1.0	Cmrl
4-Terpineol	1178	1174	0.5	Cmrl
p-Methyl acetophenone	1179	1179	0.7	Cmrl
Naphthalene	1180	1178	0.7	Cmrl
α-Terpineol	1190	1186	Tr	Cmrl
Methyl salicylate	1192	1190	3.5	Cmrl
Safranal	1200	1196	2.0	Cmrl
Decanal	1204	1201	0.8	Cmrl
β-Cyclocitral	1217	1217	3.7	Cmrl
Pulegone	1237	1233	1.4	Cmrl
(*E*)-β-Damascenone	1381	1383	1.3	Cmrl
β-Elemene	1392	1389	0.7	Cmrl
(*E*)-α-Ionone	1426	1428	9.5	Cmrl
trans-α-Bergamotene	1439	1432	0.4	Cmrl
(*E*)-Geranyl acetone	1454	1453	13.7	Cmrl
(*E*)-β-Ionone	1485	1487	2.5	Cmrl
(*E*)-Nerolidol	1564	1561	0.8	Cmrl
Caryophyllene oxide	1581	1582	0.6	Cmrl
Tetradecanal	1611	1611	0.6	Cmrl
7-*epi*- α-Eudesmol	1665	1662	0.7	Cmrl
Elemol acetate	1680	1680	0.9	Cmrl
Pentadecanal	1717	1717	6.1	Cmrl
(*E*,*E*)- α-farnesyl acetate	1843	1843	1.0	Cmrl
Hexahydrofarnesylacetone	1845	1843	2.4	Cmrl
Farnesylacetone [c]	1927	1913	4.3	Cmrl
Phytol	1949	1945	5.8	Cmrl
Total			**93.1%**	
Monoterpene hydrocarbons			**1.3**	
Oxygenated monoterpenes			**41.3**	
Sesquiterpene hydrocarbons			**2.4**	
Oxygenated sesquiterpenes			**10.7**	
Diterpenoids			**5.8**	
Aliphatic compounds			**16.6**	
Aromatic compounds			**8.3**	
Fatty acids			**6.7**	

[a] Retention indices on HP-5MS capillary column; [b] Literature retention indices (Adams, [42] and its lower versions); Tr, trace amount < 0.1%; - not present in literature; [c] Correct isomer not identified; M.I = Modes of identification which are: cmrl, Co-injection, Mass fragmentation pattern, Retention indices from column and Literature Retention indices; cmr, Co-injection, Mass fragmentation pattern, Retention indices from column, literature other than Adams [42] and its lower version.

Previous results for *Cassia* species revealed that the major compounds of *C. alata* were *ar*-turmerone (13.5%), β-caryophyllene (7.3%), (*E*)-phytol (7.0%) and 6,10,14-trimethyl-2-pentadecanone (6.8%) [9]; 1,8-cineole (39.8%), β-caryophyllene (19.1%) and caryophyllene oxide (12.7%) [8], as well as linalool (23.0%), borneol (8.6%) and pentadecanal (9.3%) [9]. Also, (*E*)-phytol (30.8%) and pentadecanal (21.7%) were identified as the main components of *C. hirsuta* [9]. On the other hand, linalool (31.5%) was the main constituent of *C. grandis* from Cuba [11], while *C. fistula* from Egypt [12] had (*E*)-nerolidol (38.0%), 2-hexadecanone (17.0%) and heptacosane (12.8%) in the flower oil, while the leaf oil was characterized by an abundance of phytol (16.1%), tetradecane (10.5%) and hexadecane (8.7%).

Therefore, from various reports, the following delineation of the chemical forms of the oils of *Cassia* species analyzed from all regions is being proposed: (i) oils whose major compounds consist of diterpenoid and fatty acids, e.g. leaves of *C. occidentalis* [9], *C. fistula* [11] and *C. hirsuta* [9]; (ii) oils whose major compounds are composed of oxygenated sesquiterpenoids and fatty acids, as seen in *C. fistula* flower [11]; (iii) oils whose major compounds were dominated by oxygenated monoterpenoid and fatty acids, e.g *C. grandis* flower [12] and *C. alata* leaf [10]; (iv) oils with significant proportions of mono- and sesquiterpenes, as seen in leaves of *C. alata* [8,9], and *C. siamea* (this study); (v) oils in which oxygenated sesquiterpenoid and diterpenoid compounds predominate, as seen in *C. occidentalis* (this study).

Table 2 shows the compounds identified from *C. ferruginea*. Oxygenated monoterpenoids (41.3%), aliphatic compounds (16.6%) and oxygenated sesquiterpenoids (10.7%) represent the major classes of compounds identified in the oil. The main oil constituents were (*E*)-geranyl acetone (13.7%), (*E*)-α-ionone (9.5%), phytol (5.8%), pentadecanal (6.1%) and 1-octen-3-ol (5.5%). Farnesylacetone (4.3%), 6-ethyl-5-hepten-2-one (3.8%), β-cyclocitral (3.7%), nonanal (3.5%) and methyl salicylate (3.5%) also occurred above 2%. The authors are unaware of any literature information on the volatile contents of *C. ferruginea* either from Nigeria or elsewhere, and as such the present report may represent the first of its kind.

Sesquiterpene compounds (82.5%) are the dominant class of compounds in *A. djalonensis* (Table 3). The main ones were α-humulene (31.9%) β-caryophyllene (17.8%), humulene epoxide II (12.7%) and caryophyllene oxide (5.9%). Apart from phytol acetate (2.2%), (*Z*)-caryophyllene (1.7%), δ-cadinene (1.7%), hexahydrofarnesyl acetone (1.3%), α-copaene 1.0%) and cubebol (1.0%), all other compounds were identified in insignificant amount. The authors are unaware of any literature information on the volatile contents of *A. djalonensi* or any other member of the family, either from Nigeria or elsewhere, and as such the present report may represent the first of its kind.

In *S. torvum,* diterpenoids (38.7%), fatty acids (30.5%), and oxygenated monoterpenoids were the most abundant classes of compounds, while sesquiterpenoids (77.6% vs. 15.2%; hydrocarbon vs. oxygen derivatives) predominated in *S. erianthum*. The main volatiles of *S. torvum* were (*E*)-phytol acetate (38.7%), pentadecanal (25.3%) and (*E*)-geranyl acetone (5.0%). Apart from methyl salicylate (4.5%), tetradecanal (2.2%), 2-pentyl furan (1.8%), hexahydrofarnesylacetone (1.6%) and hexadecanal (1.1%), all other compounds were either present in trace quantity or amounts less than 1%. On the other hand, α-humulene (46.6%) and β-caryophyllene (20.6%) were the compounds ocuurring in higher quantities in *S. erianthum*. Other compounds in significant quantities were germacrene D (4.8%), humulene epoxide II (4.4%) and caryophyllene oxide (4.0%). Monoterpenoids (1.4%) were rare among the identified compounds (Table 4)

There are literature reports on the oil constituents of some *Solanum* species grown in Nigeria (Table 5), but not for *S. torvum*. The volatile oil of *S. erianthum* was characterized by the abundance of α-terpinolene (17.8%), α-phellandrene (17.5%), *p*-cymeme (15.7%) and β-pinene (11.7%) in the leaves; and α-humulene (23.1%), humulene epoxide II (20. 0%), caryophyllene oxide (16.5%), methyl salicylate (11.8%) and β-caryophyllene (10.9%) in the fruits [39]. The leaf oil of *S. macranthum* consisted of (*E*)-phytol (29.0%), pentadecanal (28.1%), pentadecane (7.7%) and ethyl palmitate (5.7%), while the fruit oil had α-humulene (36.5%), (*E*)- caryophyllene (17.8%), ethyl palmitate (9.4%), and methyl salicylate (8.2%) as major compounds [39]. Germacrene D (14.8%),

Table 3: Leaf oil constituents of *A. djalonensis*.

Constituents	LRI [a]	LRI [b]	Percent	M.I
(*E*)-3-Hexen-1-ol	856	-	Tr	Cmr
2-Heptanone	890	889	Tr	Cmrl
Heptanal	900	901	Tr	Cmrl
Benzaldehyde	962	952	Tr	Cmrl
6-Methyl-5-hepten-2-one	986	981	Tr	Cmrl
2-Pentyl furan	986	984	0.1	Cmrl
Limonene	1031	1024	Tr	Cmrl
2-Nonanone	1092	1087	Tr	Cmrl
Nonanal	1102	1100	0.2	Cmrl
(*E,Z*)-2,6-Nonadienal	1156	1150	0.2	Cmrl
(*E*)-2-Nonenal	1162	1157	0.1	Cmrl
1-Nonanol	1173	1165	Tr	Cmrl
Naphthalene	1180	1178	Tr	Cmrl
Decanal	1205	1201	Tr	Cmrl
2-Undecanone	1292	1293	Tr	Cmrl
Undecanal [c]	1306	1300	0.1	Cmrl
α-Cubebene	1351	1345	Tr	Cmrl
α -Copaene	1376	1374	1.0	Cmrl
β-Bourbonene	1384	1387	0.4	Cmrl
β-Elemene	1391	1389	0.4	Cmrl
n-Tetradecane	1399	1400	Tr	Cmrl
(*Z*)-Caryophyllene	1404	1408	1.7	Cmrl
β-Caryophyllene	1418	1418	17.8	Cmrl
β-Gurjunene	1432	1431	0.2	Cmrl
α-Guaiene	1439	1437	0.3	Cmrl
α-Humulene	1454	1454	31.9	Cmrl
cis-Muurola-4(14),5-diene	1461	1465	Tr	Cmrl
γ-Muurolene	1477	1478	0.3	Cmrl
α-Amorphene	1480	1483	Tr	Cmrl
(*E*)-β-Ionone	1485	1487	1.6	Cmrl
epi-Cubebol	1494	1493	0.3	Cmrl
trans- β-Guaiene	1500	1502	0.4	Cmrl
n-Pentadecane	1500	1500	0.4	Cmrl
Germacrene A	1505	1508	0.8	Cmrl
Cubebol	1515	1514	1.0	Cmrl
δ-Cadinene	1524	1522	1.7	Cmrl
α-Calacorene	1542	1544	Tr	Cmrl
Elemol	1549	1548	0.2	Cmrl
(*E*)-Nerolidol	1565	1561	0.2	Cmrl
Germacrene D-4-ol	1574	1574	0.8	Cmrl
Caryophyllene oxide	1581	1582	5.9	Cmrl
cis-β-Elemenone	1590	1589	0.2	Cmrl
Cedrol	1596	1600	0.1	Cmrl
Humulene epoxide II	1606	1608	12.5	Cmrl
Caryophylla-4(14),8(15)-dien-5-ol	1636	-	0.2	Cmr
Epoxy-allo-aromadendrene	1639	1639	0.9	Cmrl
τ-Cadinol	1641	-	0.5	Cmr
α-Cadinol	1654	1652	0.8	Cmrl
Acorenone	1696	1692	0.3	Cmrl
n-Heptadecane	1700	1700	0.1	Cmrl
Pentadecanal	1717	1717	0.5	Cmrl
Mint sulfide	1741	1740	0.4	Cmrl
Hexahydrofarnesylacetone	1845	1843	1.3	Cmrl
Methyl hexadecanoate	1927	1921	0.2	Cmrl
Phytol	1949	1942	0.5	Cmrl
n-Heneicosane	2100	2100	Tr	Cmrl
(*E*)-Phytol acetate	2218	2218	2.2	Cmrl
Total			**88.2%**	
Monoterpene hydrocarbons			**-**	
Oxygenated monoterpenes			**1.6**	
Sesquiterpene hydrocarbons			**57.3**	
Oxygenated sesquiterpenes			**25.2**	
Diterpenoids			**2.7**	
Aliphatic compounds			**0.6**	
Aromatic compounds			**0.1**	
Fatty acids			**0.7**	

[a] Retention indices on HP-5MS capillary column; [b] Literature retention indices ([42] and its lower version); Tr, trace amount < 0.1%; - not identified and not present in literature; - not present in literature; [c] Correct isomer not identified; M.I = Mode of identification: cmrl, Co-injection, Mass fragmentation pattern, Retention indices from column and Literature Retention indices; cmr, Co-injection, Mass fragmentation pattern, Retention indices from column, Literature other than [42] and its lower version.

Table 4: Constituents of *S. torvum* and *S. erianthum*.

Constituents	LRI [a]	LRI [b]	*S. t*	*S. e*	M.I
(*E*)-2-Hexenal	854	846	0.4	Tr	Cmrl
2-Heptanone	890	889	-	Tr	Cmrl
Heptanal	900	901	Tr	Tr	Cmrl
α-Pinene	939	932	-	0.3	Cmrl
Benzaldehyde	962	952	-	Tr	Cmrl
Sabinene	977	969	-	Tr	Cmrl
6-Methyl-5-hepten-2-one	986	981	0.2	Tr	Cmrl
2-Pentyl furan	986	984	1.8	0.3	Cmrl
(*Z*)-3-Hexenyl acetate	1007	1004	Tr	-	Cmrl
p-Cymene	1026	1020	-	Tr	Cmrl
Limonene	1031	1024	1.9	0.2	Cmrl
(*E*)-β-Ocimene	1051	1044	Tr	Tr	Cmrl
(*E*)-2-Octenol	1063	1060	-	Tr	Cmrl
1-Octanol	1071	1063	-	Tr	Cmrl
Terpinolene	1088	1086	-	Tr	Cmrl
Linalool	1099	1095	0.6	-	Cmrl
Nonanal	1103	1100	0.5	0.3	Cmrl
Geijerene	1143	1138	-	Tr	Cmrl
(*E*)-2-Nonenal	1152	1157	0.5	Tr	Cmrl
(*E,Z*)-2,6-Nonadienal	1156	1150	0.3	Tr	Cmrl
Naphthalene	1180	1178	0.4	-	Cmrl
(*Z*)-3-Hexenyl butanoate	1186	1184	0.2	-	Cmrl
Methyl salicyalte	1191	1190	4.5	-	Cmrl
n-Dodecane	1199	1200	0.2	-	Cmrl
Decanal	1204	1201	0.3	Tr	Cmrl
β-Cyclocitral	1217	1217	0.2	Tr	Cmrl
Pregeijerene	1288	1285	1.0	Tr	Cmrl
n-Tridecane	1299	1300	0.2	Tr	Cmrl
Undecanal [c]	1306	1300	-	Tr	Cmrl
(*E, E*)-2,4-Decadienal	1314	1315	Tr	-	Cmrl
Hexenyl tiglate	1332	1319	-	Tr	Cmrl
α-Cubebene	1351	1345	-	Tr	Cmrl
α-Copaene	1376	1374	Tr	0.9	Cmrl
β-Bourbonene	1384	1387	-	0.5	Cmrl
β-Elemene	1391	1389	-	0.6	Cmrl
n-Tetradecane	1400	1400	-	Tr	Cmrl
(*Z*)-Caryophyllene	1405	1408	-	0.3	Cmrl
β-Caryophyllene	1418	1418	0.2	20.6	Cmrl
β-Gurjunene	1432	1431	-	0.2	Cmrl
α-Guaiene	1439	1437	-	Tr	Cmrl
α-Humulene	1454	1454	-	46.6	Cmrl
(*E*)-Geranyl acetone	1456	1453	5.0	-	Cmrl
cis-Muurola-4(14),5-diene	1461	1465	-	Tr	Cmrl
γ-Muurolene	1477	1478	-	Tr	Cmrl
Germacrene D	1480	1484	0.3	4.8	Cmrl
(*E*)-β-Ionone	1485	1487	0.9	0.9	Cmrl
Bicyclogermacrene	1494	1500	-	0.2	Cmrl
epi-Cubebol	1496	1493	-	0.2	Cmrl
α-Muurolene	1499	1500	-	Tr	Cmrl
n-Pentadecane	1500	1500	0.4	-	Cmrl
Germacrene A	1503	1508	Tr	1.5	Cmrl
Tridecanal	1509	1509	0.3	Tr	Cmrl
Cubebol	1515	1514	-	0.7	Cmrl
(*E*)-Dihydroapofernesal	1522	1520	Tr	-	Cmrl
δ-Cadinene	1524	1522	-	1.4	Cmrl
(*E*)-Nerolidol	1565	1561	0.3	-	Cmrl
(*Z*)-3-Hexenyl benzoate	1570	1565	0.3	-	Cmrl
Caryophyllene oxide	1581	1582	-	4.0	Cmrl
n-Hexadecane	1600	1600	0.6	-	Cmrl
Humulene epoxide II	1606	1608	-	4.4	Cmrl
1-*epi*-Cubenol	1628	1627	-	0.3	Cmrl
Caryophylla-4(14),8(15)-dien-5-ol	1636	-	-	0.6	Cmr
epi-α-Cadinol	1640	1638	-	0.7	Cmrl
α-Muurolol	1645	1644	-	0.2	Cmrl
α-Cadinol	1653	1652	-	1.3	Cmrl
1-Tetradecanol	1674		0.3	-	Cmrl
Eudesma-4(15),7-dien-1-β-ol	1688	1687	-	0.2	Cmrl
n-Heptadecane	1700	1700	-	0.2	Cmrl
Pentadecanal	1717	1717	25.3	0.8	Cmrl
n-Octadecane	1800	1800	0.5	-	Cmrl
Hexadecanal	1830	-	1.1	-	Cmr
Hexahydrofarnesylacetone	1845	1843	1.6	0.8	Cmrl
Benzyl salicylate	1866	1864	0.2	-	Cmrl
n-Nonadecane	1900	1900	0.6	-	Cmrl
Farnesylacetone [c]	1927	1913	-	1.0	Cmrl
Hexadecanoic acid	1959	1958	-	0.8	Cmrl
n-Eicosane	2000	2000	0.5	-	Cmrl
n-Heneicosane	2100	2100	0.5	-	Cmrl
(*E*)-Phytol acetate	2218	2218	38.7	-	Cmrl
Total			**90.8%**	**95.8%**	
Monoterpene hydrocarbons			**2.9**	**0.5**	
Oxygenated monoterpenes			**11.2**	**0.9**	
Sesquiterpene hydrocarbons			**0.5**	**77.6**	
Oxygenated sesquiterpenes			**1.9**	**15.2**	
Diterpenoids			**38.7**	**-**	
Aliphatic compounds			**2.7**	**0.3**	
Aromatic compounds			**2.4**	**0.3**	
Fatty acids			**30.5**	**1.0**	

[a] Retention indices on HP-5MS capillary column; [b] Literature retention indices ([42] and its lower version); Tr, trace amount < 0.1%; - not identified and not present in literature; [c] Correct isomer not identified; M.I = Mode of identification: cmrl, Co-injection, Mass fragmentation pattern, Retention indices from column and Literature Retention indices; cmr, Co-injection, Mass fragmentation pattern, Retention indices from column, Literature other than [42] and its lower version.
S. t = Solanum torvum; *S. e = Solanum erianthum*

pentadecanal (11.4%), β-elemene (10.1%), α-bulnesene (7.9%), δ-cadinene (6.0%), β-caryophyllene (6.5%) and α-copaene (5.5%) were the major components of the oil of *S. nigrum* var. *virginicum* [40].

Table 5: Major components of *Solanum* oils from Nigeria.

Species/Plant Parts	Major constituents	Ref
S. erianthum (leaves)	α-terpinolene, α-phellandrene, *p*-cymene, β-pinene	[39]
S. erianthum (fruits)	α-humulene, humelene epoxide, caryophyllene oxide, methyl salicylate, β-caryophyllene	[39]
S. macranthum (leaves)	(*E*)-phytol, pentadecanal, pentadecane, ethyl palmitate	[39]
S. macranthum (fruits)	α-humelene, (*E*)-caryophyllene, ethyl palmitate, methyl salicylate	[39]
S. nigrum var. *nigrum* (leaves)	Germacrene D, pentadecanal, β-elemene α-bulnesene, δ-cadinene, β-caryophyllene, α-copaene	[40]
S. torvum (leaves)	*(E)*-phytol acetate, pentadecanal *(E)*-geranyl acetone	This study
S. erianthum (leaves)	α-humulene, β-caryophyllene	This study

It is evident that the leaf oils of *S. erianthum* analysed from Nigeria exist in two chemical forms, namely one with an abundance of monoterpene hydrocarbons [39] and one whose major compounds are sesquiterpene hydrocarbons, as seen in the present study (Table 5).

Moreover, the oils of *Solanum* species so far analysed from Nigeria could be thought to exist in three chemical forms (Table 5) namely; (i) oil dominated by sesquiterpenoid compounds, such as *S. nigrum* var. *nigrum, S. erianthum* (leaf and fruit) and *S. macranthum* (fruits); (ii) oil consisting of monoterpene hydrocarbons, as seen in *S. erianthum* (leaf); and (iii) oil with abundant diterpenoids and fatty acids, typified by *S. torvum* and *S. macranthum* (leaves). Further studies are on-going to ascertain the biological activities of these essential oils and the compounds responsible for such activities.

Experimental

Plants collection: Mature leaves of *C. siamea, C. occidentalis* and *A. djalonensis* were collected at Abule-Eera, a suburb along the Badagry Expressway Lagos, in May 2011. Leaves of *C. ferruginea* were harvested at Igbesa Waterside, Agbara, also along Badagry Expressway, Lagos, in June 2011. Botanical identifications were performed at the Herbarium, Forestry Research Institute of Nigeria (FRIN), Ibadan, Nigeria, where voucher specimens FHI 109435, FHI 109436, FHI 109434 and FHI 109438, respectively have been deposited for future reference. The leaves of *S. torvum* and *S. erianthum* were obtained respectively from Ijede Town and Egan Town, Lagos. Identification was accomplished at the Herbarium, Department of Botany, University of Lagos, Nigeria, where voucher specimens LUH 5227 and LUH 5226, respectively were deposited. Leaves were air-dried for 2 weeks under laboratory shade prior to extraction.

Extraction of the oils: Aliquots of air-dried and pulverized leaves (30 g each) were subjected to separate hydrodistillation in an all-glass Clevenger type apparatus for 3 h in accordance with the British Pharmacopoeia method [41]. The oils obtained were collected and stored under refrigeration until analyses were performed.

***Gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS)*:** GC analysis was accomplished with a HP-5890 series II instrument equipped with HP-wax and HP-5 capillary columns (both 30 m x 0.25 mm, 0.25 µm film thickness) with the following temperature programme; 60°C for 10 min, rising from 5°C/min to 220°C. Both injector and detector temperatures were maintained at 250°C; carrier gas, nitrogen (2 mL/ min); detector, FID; ratio, 1:30. The volume injected was 0.5 µL. The relative proportions of the oil constituents were the percentages obtained (% area) by FID peak-area normalisation without the use of response factor.

Gas chromatography-electron ionization mass spectroscopy (GC-EIMS) analysis was performed with a Varian CP-3800 gas chromatography equipped with a HP-5 capillary column (30 m x 0.25 mm; film thickness 0.25 µm) and a Varian Saturn 2000 ion trap mass detector. Analytical conditions: injector and transfer line temperatures were 220°C and 240°C respectively. Oven temperature programmed from 60°C to 244°C at 3°C/min; carrier gas was helium at a flow rate of 1 mL/min; injection of 0.2 µL (10% *n*-hexane solution); split ratio 1:30. Mass spectra were recorded at 70 eV. The acquisition mass range was 30-300 m/z at a scan rate of 1 scan/s.

***Identification of constituents*:** Identification of the constituents was based on comparison of the retention times with those of authentic samples, comparing their linear indices relative to a series of *n*-hydrocarbons, and by computer matching against commercial spectra. Further identification was also made possible by the use of a self constructed spectral library built up from pure substances and components of known oils and MS literature data [42, 43]. Moreover, the molecular weights of all the identified substances were confirmed by gas chromatography-chemical ionisation mass spectrometry, using methanol as CI ionizing gas.

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