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# Chemical composition of Essential Oils from Nigerian Plants

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**ABSTRACT**

*Essential oils obtained by separate hydrodistillation of leaves of three plants collected in Nigeria were analysed comprehensively for their constituents by means of gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). Germacrene D (28.4%), α-pinene (17.1%), β-caryophyllene (7.5%) and caryophyllene oxide (6.0%) were the major compounds of Aspilia africana (Pers.) C. D. Adams (Asteraceae). The oil of Lippia multiflora Moldenke (Verbenaceae) comprised mainly of sabinene (13.0%), β-caryophyllene (21.8%) and rimuene (14.6%) as dominant compounds, with significant quantity of abietatriene (7.1%), β-pinene (4.0%) and caryophyllene oxide (4.3%). The species is a source of new chemotype. β-Caryophyllene (19.0%) and geranial (10.6%) were identified in higher proportions from Spondias lutea L. (Anacardiaceae), with significant amounts of neral (5.1%), caryophyllene oxide (5.6%) and δ-cadinene (4.6%). The paper will discuss further the chemotaxonomic importance of the results.*

**Keywords:** *Aspilia africana, Lippia multiflora, Spondias lutea*, essential oils, chemical composition, terpenes

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## INTRODUCTION

*Aspilia africana* (Pers) C. D. Adams, is an important member of Asteraceae family of plant. It is a tropical weed with deep yellow flowers and reaching about 1.5m long. It is used in traditional medicine for the teatment of varoius disorders such as gonorrhea, cough wouds and insect bites [1]. Variuos biological activities such as anticoagulant, anti-inflammatory and anti-malarial have been attibuted to the plant and its extracts [2, 3]. To date only three reports have appeared in literature of the composition of its essential oils [2-4].

*Lippia multiflora* Moldenke, Verbenaceae, is a shrubby aromatic plant, growing up to 1.2 m whitish flowers on cone-like heads in a terminal panicle. The volatile oils of *L. multiflora* have been extensively investigated with variations in the chemical compounds depending on chemotype and location [5, 6]. Oxygenated monoterpenoids such as thymol, 1, 8-cineole, eugenol, geranial, carvacrol etc were the predominat class of compounds identified in the oil samples.

*Spondias lutea* L., or *Spondias mombin* is a tree, a species of flowering plant in the family Anacardiaceae. It is native to the tropical Americas, including the West Indies. The tree has been naturalized in parts of Africa, India, Sri

Lanka and Indonesia. It is rarely cultivated. The pulp is either eaten fresh, or made into juice, concentrate, jellies , and sherbets. The mature fruit has a leathery skin and a thin layer of pulp. The seed has oil content [7]. In Nigeria, the fruit is called '*Iyeye*' in Yoruba, '*ngulungwu'* in Igbo and '*isada'* in Hausa. They are pulped, boiled in water, and drunk, or used as a lotion or for baths. The bark is used as a purgative and in local applications for leprosy. The fruit-juice is used as a febrifuge and diuretic. The leaf extract of *S. mombin* displayed an anti-inflammatory effect and suppresses inducible formation of tumor necrosis factor-α and nitric oxide [8]. Over 200 different volatile compounds have been isolated from the fruit pulps [9-11].

## MATERIALS AND METHODS

***Plant materials***

Fresh leaves of *Aspilia africana* were collected from Lagos State University, Ojo, Nigeria, in June 2010, while the leaves of *Lippia multiflora* and *Spondia lutea* were harvested from Ijede Area, Ikorodu, Lagos, Nigeria, in January 2011. The plants were authenticated by Curators at the Herbarium of Botany Department, University of Lagos, Nigeria. Voucher specimens LUH 3224 (*A. africana*), LUH 3320 (*L. multiflora*) and LUH 3324 (*S. lutea*) were deposited at the Herbarium for future references.

***Extraction of the essential oils***

Air-dried leaves (300g) were subjected to separate hydrodistillation in all-glass Clevenger type apparatus for 4 h in accordance with British Pharmacopoeia method [12]. The plant samples yielded a low content of essential oils: 0.52% (v/w; *A. africana*; yellow); 0.45% (v/w; *L. multiflora*; colourless) and 0.50% (v/w; *S. lutea*; yellow), calculated on a dry weight basis.

***Gas chromatography (GC) –and gas chromatography-mass spectrometry (GC-MS).***

GC analysis was accomplished with 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 $^{0}$C for 10 min, rising from 5 $^{o}$C/min to 220 $^{o}$C. Both injector and detector temperatures were maintained at 250 $^{o}$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 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 temperature were 220 $^{o}$C and 240 $^{o}$C respectively. Oven temperature programmed from 60 $^{o}$C- 244 $^{0}$C at 3 $^{o}$C/min; carrier gas was helium at a flow rate of 1 mL/min; injection of 0.2 µL (10% 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 the constituents***

Identification of the constituents was based on comparison of the retention times with those of authentic samples, comparing their linear indices relative to the series of n-hydrocarbons, and on computer matching against commercially spectral [13]. Further identification were also made possible by the use of self constructed spectral library built up from pure substances and components of known oils and MS literature data [14, 15]. Moreover, the molecular weights of all the identified substances were confirmed by gas chromatography-chemical ionisation mass spectrometry, using methanol as CI ionising gas.

## RESULTS AND DISCUSSION

Eighty-three constituents categorised as monoterpene hydrocarbons (26.8%), oxygenated monoterpenes (3.9%), sesquiterpene hydrocarbons (53.6%) and the oxygenated derivatives (11.9%) were identified in *A. africana*. The major compounds of the oil were germacrene D (28.4%), β-caryophyllene (7.5%), caryophyllene oxide (6.0%) and α-humulene (4.3%). α-Pinene (17.1%), β-pinene (4.1%) and methyl carvacrol (2.7%) were the prominent monoterpenes present in the oil (Table1). The composition of the major compounds of this oil was qualitatively similar to previous studies from Nigeria [2] and Cameroon [4], but differs from other reports in the fact that α-cubebene [3] and limonene [4] were identified in insignificant quantities. Therefore the oils of the genus *Aspilia* reported so far in the literature could be classified into different chemical forms as shown in Table 2.

**Table 1: Essential oil composition of *A. Africana***

| Constituents | LRI [a] | Percentage (%) |
|---|---|---|
| α-thujene | 931 | tr |
| α-pinene | 939 | 17.1 |
| Camphene | 953 | tr |
| thuja-2,4(10)-diene | 957 | tr |
| Benzaldehyde | 961 | tr |
| Sabinene | 976 | 1.1 |
| β-pinene | 980 | 4.1 |
| 6-methyl-5-hepten-2-one | 985 | tr |
| Myrcene | 991 | 0.8 |
| α- phellandrene | 1005 | 1.6 |
| α-terpinene | 1018 | tr |
| ρ-cymene | 1027 | 0.8 |
| Limonene | 1031 | 1.1 |
| 1,8-cineole | 1034 | tr |
| *(Z)*-β-ocimene | 1041 | tr |
| Phenylacetaldehyde | 1045 | tr |
| *(E)*-β-ocimene | 1051 | 0.2 |
| γ-terpinene | 1062 | tr |
| *cis*-sabinene hydrate | 1070 | tr |
| terpinolene | 1089 | tr |
| linalool | 1099 | 0.1 |
| nonanal | 1103 | tr |
| α-campholenal | 1127 | tr |
| *trans*-pinocarveol | 1140 | tr |
| geijerene | 1143 | tr |
| *cis*-verbenol | 1144 | 0.3 |
| pinocarvone | 1164 | tr |
| borneol | 1167 | tr |
| 4-terpineol | 1179 | 0.2 |
| *p*-cymen-8-ol | 1185 | tr |
| α -terpineol | 1190 | 0.2 |
| myrtenal | 1194 | 0.2 |
| safranal | 1200 | tr |
| decanal | 1205 | tr |
| verbenone | 1207 | tr |
| β-cyclocitral | 1219 | 0.2 |
| methyl thymol | 1235 | tr |
| cumin aldehyde | 1239 | tr |
| methyl carvacrol | 1244 | 2.7 |
| pregeijerene | 1288 | 0.3 |
| δ-elemene | 1340 | tr |
| α-cubebene | 1351 | 0.6 |
| cyclosativene | 1370 | tr |
| α-ylangene | 1372 | tr |
| α-copaene | 1376 | 1.0 |
| β-bourbonene | 1384 | 0.5 |
| β-cubebene | 1390 | 0.6 |
| β-elemene | 1392 | 1.1 |
| β-caryophyllene | 1418 | 7.5 |
| β-gurjunene | 1432 | 0.3 |
| γ-elemene | 1433 | 0.2 |
| α-guaiene | 1439 | tr |
| aromadendrene | 1441 | tr |

| | | |
|---|---|---|
| *trans*-muurola-3,5-diene | 1454 | tr |
| α-humulene | 1455 | 4.3 |
| *(E)*-β-farnesene | 1459 | tr |
| *cis*-muurola-4(14),5-diene | 1462 | 0.2 |
| γ-muurolene | 1477 | 0.5 |
| germacrene D | 1480 | 28.4 |
| β-selinene | 1485 | 0.2 |
| *trans*-muurola-4(14),5-diene | 1492 | 0.4 |
| bicyclogermacrene | 1494 | 1.6 |
| α-muurolene | 1500 | 0.4 |
| α-bulnesene | 1505 | 0.9 |
| β-bisabolene | 1509 | 0.2 |
| δ-amorphene | 1512 | tr |
| *trans*-γ-cadinene | 1513 | 0.9 |
| δ-cadinene | 1524 | 2.3 |
| *trans*-cadina-1,4-diene | 1532 | 0.2 |
| α-cadinene | 1538 | tr |
| α-calacorene | 1542 | 0.2 |
| germacrene B | 1556 | 0.8 |
| *trans*-nerolidol | 1565 | 0.3 |
| spathulenol | 1576 | 1.2 |
| caryophyllene oxide | 1581 | 6.0 |
| guaiol | 1595 | 0.3 |
| humulene oxide II | 1606 | 2.0 |
| γ-eudesmol | 1632 | tr |
| τ-cadinol | 1641 | 0.4 |
| τ-muurolol | 1643 | 0.2 |
| β-eudesmol | 1649 | 0.2 |
| selin-11-en-4-α-ol | 1653 | 1.1 |
| hexahydrofarnesylacetone | 1845 | 0.2 |
| **Total** | | **96.2** |
| Monoterpene hydrocarbons | | 26.8 |
| Oxygenated monoterpenes | | 3.9 |
| Sesquiterpene hydrocarbons | | 53.6 |
| Oxygenated sesquiterpenes | | 11.9 |

[a] *Retention indices on HP-5MS capillary column; tr, trace amount < 0.1%*

**Table 2: Chemical forms of essential oil of *A. Africana***

| Chemotype | Species | Major constituents | References |
|---|---|---|---|
| Oil with abundance of germacrene D, α-pinene and β-caryophyllene | *A. africana* | germacrene D (28.4%), α-pinene (17.1%), β-caryophyllene (7.5%) | This study |
| | *A. africana* | germacrene D (15.6%), α-pinene (13.6%), β-caryophyllene (10.8%) | 2 |
| Oil rich in α-cubebene and α-pinene [*] | *A. africana* | α-cubebene (13.1%), α-pinene (6.7%), α-thujene/car-3-ene (5.0%) | 3 |
| Oil with high proportions of α-pinene, germacrene D and limonene | *A. africana* var. *africana* | α-pinene (38.7%), germacrene D (13.8%), limonene (7.5%) | 4 |
| | *A. africana* var. *africana* | α-pinene (26.5%), germacrene D (24.4%), limonene (9.4%) | 4 |
| Oil with high amounts of germacrene D, β-caryophyllene and limonene | *A. africana* var. *africana* | germacrene D (45.0%), β-caryophyllene (8.7%), limonene (7.1%) | 4 |
| | *A. africana* var. *africana* | germacrene D (54.2%), β-caryophyllene (13.2%), δ-cadinene (8.2%) | 4 |
| Oil rich in limonene, α-pinene and germacrene D | *A. africana* var. *ambigua* | limonene (23.2%), α-pinene (21.8%), germacrene D (6.7%) | 4 |

[*] *Very unusual constituents*

**Table 3: Compounds identified in the essential oil of *L. multiflora***

| Constituents | LRI [a] | Percentage (%) |
|---|---|---|
| α-thujene | 931 | 0.4 |
| α-pinene | 939 | 1.1 |
| camphene | 953 | tr |
| sabinene | 976 | 13.0 |
| β-pinene | 980 | 4.0 |
| myrcene | 991 | tr |
| octanol | 993 | 0.2 |
| α-phellandrene | 1005 | tr |
| δ-3-carene | 1011 | tr |
| α-terpinene | 1018 | 0.7 |
| *p*-cymene | 1027 | 0.6 |
| limonene | 1031 | 1.0 |
| 1,8-cineole | 1034 | 0.2 |
| α-terpinene | 1062 | 1.1 |
| *cis*-saninene hydrate | 1070 | 1.4 |
| terpinolene | 1089 | 3.1 |
| *trans*-sabinene hydrate | 1098 | 1.0 |
| *exo*-fenchol | 1117 | 0.3 |
| *cis*-*p*-menth-2-en-1-ol | 1122 | 0.2 |
| *trans*-*p*-menth-2-en-1-ol | 1142 | tr |
| isoborneol | 1156 | tr |
| 4-terpineol | 1178 | 3.5 |
| *p*-cymen-8-ol | 1185 | 0.2 |
| α-terpineol | 1190 | 0.2 |
| *trans*-piperitol | 1207 | tr |
| geranial | 1272 | tr |
| α-copaene | 1376 | tr |
| β-elemene | 1391 | 0.2 |
| *(Z)*-caryophyllene | 1405 | tr |
| β-caryophyllene | 1418 | 21.8 |
| *(E)*-α-ionone | 1428 | tr |
| *trans*- α-bergamotene | 1439 | 2.3 |
| α-humulene | 1455 | 1.6 |
| *allo*-aromadendrene | 1461 | tr |
| 6-demethoxy-ageratochromene | 1463 | 0.3 |
| β-chamigrene | 1475 | tr |
| germacrene D | 1480 | tr |
| β-selinene | 1485 | 1.6 |
| bicyclogermacrene | 1494 | 3.1 |
| α-bulnesene | 1505 | 0.3 |
| β-sesquiphellandrene | 1524 | tr |
| spathulenol | 1576 | 2.6 |
| caryophyllene oxide | 1581 | 4.3 |
| viridiflorol | 1590 | tr |
| humulene epoxide II | 1606 | 0.2 |
| humulane-1,6-dien-3-ol | 1611 | 0.8 |
| γ-eudesmol | 1632 | tr |
| τ-cadinol | 1641 | tr |
| selin-11-en-4-α-ol | 1653 | 1.8 |
| 8-cedren-13-ol | 1689 | 1.4 |
| *trans*- α-bergamotol | 1691 | 0.5 |
| isopimara-9(11),15-diene | 1899 | 0.2 |
| rimuene | 2018 | 14.6 |

| | | |
|---|---|---|
| abietatriene | 2054 | 7.1 |
| abietadiene | 2080 | 1.8 |
| **Total** | | **98.7** |
| Monoterpene hydrocarbons | | **25.0** |
| Oxygenated monoterpenes | | **7.0** |
| Sesquiterpene hydrocarbons | | **30.9** |
| Oxygenated sesquiterpenes | | **11.8** |
| Diterpenes | | **23.5** |
| Others | | **0.5** |

[a]*Retention indices on HP-5MS capillary column; tr, trace amount < 0.1%*

**Table 4: Oil composition of *S. Lutea***

| Constituents | LRI [a] | Percentage ( %) |
|---|---|---|
| 3-methyl-2-hexanone | 844 | 0.6 |
| *(E)*-2-hexenal | 854 | 1.0 |
| α-pinene | 939 | 0.4 |
| benzaldehyde | 961 | 0.9 |
| β-pinene | 980 | 0.3 |
| 6-methyl-5-hepten-2-one | 985 | 0.5 |
| 2-pentyl furan | 995 | 1.2 |
| *n*-decane | 1000 | 0.6 |
| α-terpinene | 1018 | 0.5 |
| *o*-cymene | 1022 | 1.2 |
| *p*-cymene | 1027 | 2.2 |
| limonene | 1031 | 0.5 |
| 1,8-cineole | 1034 | 0.3 |
| *(Z)*-β-ocimene | 1041 | tr |
| *(E)*-β-ocimene | 1051 | tr |
| γ-terpinene | 1062 | 0.5 |
| *cis*-linalool oxide (furanoid) | 1075 | tr |
| dehydro-*p*-cymene | 1088 | 1.4 |
| linalool | 1099 | 0.5 |
| nonanal | 1103 | 0.5 |
| 1,3,8-*p*-menthatriene | 1112 | tr |
| *p*-mentha-1,5-dien-8-ol | 1166 | tr |
| 4-terpineol | 1178 | 0.4 |
| naphthalene | 1180 | tr |
| *p*-cymen-8-ol | 1185 | tr |
| α-terpineol | 1190 | tr |
| methyl salicylate | 1192 | tr |
| *cis*-piperitol | 1195 | 0.6 |
| β-cyclocitral | 1219 | 0.8 |
| neral | 1240 | 5.1 |
| geranial | 1272 | 10.6 |
| 1,2-dihydro-1,1,6-trimethyl naphthalene | 1354 | 0.4 |
| α-ylangene | 1372 | 0.3 |
| α-copaene | 1376 | 0.7 |
| geranyl acetate | 1385 | 0.6 |
| *(Z)*-caryophyllene | 1405 | 0.3 |
| β-caryophyllene | 1418 | 19.0 |
| γ-elemene | 1433 | 1.7 |
| *trans*- α-bergamotene | 1439 | 0.7 |
| aromadendrene | 1441 | 0.6 |
| *(E)*-geranyl acetone | 1453 | 1.2 |
| α -humulene | 1455 | 3.8 |

| | | |
|---|---|---|
| 6-demethoxyageratochromene | 1463 | 0.4 |
| γ-muurolene | 1477 | 3.7 |
| germacrene D | 1480 | 1.0 |
| β-selinene | 1485 | 1.9 |
| *(E)*-β-ionone | 1488 | 1.2 |
| valencene | 1491 | 2.7 |
| *cis*-β-guaiene | 1493 | 0.4 |
| α-muurolene | 1499 | 2.3 |
| *trans*-γ-cadinene | 1513 | 2.3 |
| δ-cadinene | 1524 | 4.6 |
| α-cadinene | 1538 | 0.5 |
| α-calacorene | 1542 | 1.2 |
| spathulenol | 1576 | 0.3 |
| caryophyllene oxide | 1581 | 5.6 |
| humulene oxide II | 1606 | 1.0 |
| τ-cadinol | 1641 | 0.5 |
| α-cadinol | 1653 | 0.8 |
| pentadecanal | 1717 | 0.8 |
| abietatriene | 2054 | 1.2 |
| **Total** | | **94.9%** |
| Monoterpene hydrocarbons | | **7.0** |
| Oxygenated monoterpenes | | **22.7** |
| Sesquiterpene hydrocarbons | | **48.7** |
| Oxygenated sesquiterpenes | | **8.2** |
| Diterpenes | | **1.2** |
| Aliphatic | | **4.0** |
| Aromatic | | **2,5** |
| Others | | **0.4** |

[a] *Retention indices on HP-5MS capillary column; tr, trace amount < 0.1%*

Table 3 revealed the identities of the fifty-six compounds identified in the oil of *L. multiflora*. The major components were rimuene (14.6%), a diterpene; β-caryophyllene (21.8%), a sesquiterpene and the monoterpene, sabinene (13.0%). There were significant quantities of abietatriene (7.1%), caryophyllene oxide (4.3%) and β-pinene (4.0%). Considering the major constituents, literature information revealed that *L. multiflora* exhibited intraspecific variation in its oil compoisition. Though sabinene and β-caryophyllene as found in this oil sample have been reported as major compounds of *L. multiflora*, the occurrenece of rimuene or any diterpenes is uncommon. Hence, the rimuene/ β-caryophyllene/sabinene chemotype described in this report has is uncommon [5, 6]. The commonly reported compounds such as thymol, 1, 8-cineole, tagetone, geranial, *p*-cymene, nerolidol etc were either conspicuosly absent or occurred in low amounts in the present investigation.

From Table 4, it could be seen that terpenes were the major constituents of *S. lutea*. The classes of compounds occurring in higher amounts were oxygenated monoterpene (22.7%) and sesquiterpene hydrocarbons (48.7%). The main compounds were β-caryophyllene (19.0%) and geranial (10.6%). Other significant constituents of the oil were caryophyllene oxide (5.6%), neral (5.1%), δ-cadinene (4.6%), α-humulene (3.8%) and γ-muurolene (3.7%). Over 200 different volatile compounds have been isolated and characterized from the fruit pulps [9-11]. Previously (*E*)-caryophyllene (18.7%), ethyl butyrate (10.0%); myrcene (41.1%), β-phellandrene (8.5%) and ethyl hexanoate (4.9%); (*Z*)-caryophyllene (13.2%), limonene (9.5%) and ethyl hexanoate (6.2%); myrcene (38.0%), ρ-cymene (6.2%) and α-terpineol (5.1%) were the main compounds of *S. lutea* [10]. In another report, butanoic acid, ethyl 3-hydroxyhexanoate, butanol, ethyl 3-hydroxybutanoate, butyl butanoate and butyl 3-hydroxybutanoate were the compounds identified in higher proportions [11]. However, terpenic esters and acids could not be identified in the present oil sample.

## CONCLUSION

The volatile oil of *L. multiflora* afforded a new chemotype which has not been described previously. Each volatile oil exhibited differing chemical constituents which may be attributed to several factors such as ecological and climatic conditions, age of the plant, variety etc.

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