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Chemical Constituents of the Leaf, Stem-bark and Root Essential oils of *Antiaris africana*

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ABSTRACT

Leaf, stem bark and root volatile oils were obtained from *Antiaris africana* Engl. a large attractive and medicinal deciduous timber tree (Moraceae) in 0.24%; 0.67% and 0.76% yields respectively. GC and GC-MS analyses revealed 10 compounds are responsible for leaf oil; 22 compounds make up 98.9% of the stem bark oil, while 6 compounds make-up 99.6% of root oil. 9 compounds were identified in leaf, which make-up 92.2% of it and 5 identified compounds in root represent 96.6% of it. Most abundant compounds in leaf oil are camphor (20.7%), 5-(1methylethylidene)-1,3-cyclopentadiene (16.8%), isomenthol (12.6%) and 1,3,5-trimethylbenzene (12.6%). Prominent compounds in stem bark oil are eucalyptol (19.3%), 4-methyl-1-(1methylethyl)-R-3cyclohexen-1-ol (17.4%), n-hexadecanoic acid (14.2%) and p-menth-1-en-8-ol (8.5%). Root oil is dominated by methylhexadecanoate (41.3%), 10-methyleicosane (30.4%) and methyl-13-octadecenoate (17.3%). Six other compounds in leaf oil, 18 others in stembark oil and 2 others in root oil along with above as reported are composition of *Antiaris africana* Engl. Leaf and stem bark oils are highly terpenic, having many interesting C_{10} and C_{15} terpenoids; Eucalyptus the most abundant compound in stem bark oil, is known to be medicinal; Presence of isomers of linalooloxide in stem bark oil is unique. Compositions of these three volatile oils are very interesting, which is scarce in literature.

INTRODUCTION

The highly medicinal *Antiaris africana* Engl. is a large attractive deciduous timber tree (family Moraceae), which grows up to 130 ft. (30-40m) high; with grey bark, on slashing exudes a watery latex which soon darkens to the colour of milky tea. Its ripe fruits are red or orange; synonyms are *Antiaris usambarensis*, *Antiaris welwitschii*. (Olofinboba, 2002; Gotz,1983; Lavers, 1983; HMSO, 1981).

They are found in the drier forest of tropical Africa, Oceania and S.E. Asia, and are widely distributed in many African countries. They are referred to by many common names such as Akede, Ako, Andoum, Antiaris, Bark cloth tree, Bofu-kenge, Bofue, Bofui, Bofuni, Bonkonko, Bovili, Chenchen, Diolosso, Egyany, Elwa, False iroko, False mvule, Fou, Hachu, Handame, Hanton, Jafo, Ju-u, Kakulu kodzo, Kan, Kesuba, Kirundu, Kudzo, Kyenkyen, Logotsi, Lulundu, Man, Mbondo, Mkunde, Mkunzu, Mkuzu, Mlulu, Mnguonguo, Muherere, Mulundo, Mulundulundu, Mumaka, Munsende, Mutie, Ofu, Ogiovu, Olwaa, Oro, Pau bicho branco, Pou, Ripi, Sansama, Sili, Terap, Tide, Tomboiro blanc, Toumboturo blanc, Tsangu, Upas tree, Zaadi (JSTOR Home, 2000-2011; WCMC, 1992; Keay, 1989; Lincoln, 1986; Murira, 1984).

It also have important decorative applications such as in constructions of agricultural implement, bedroom suite, general boat building, boxes and crates, building materials, furniture and interior construction, charcoal, chemical derivatives, chests, concealed parts, cooperages, core stock, decorative plywood, veneer, door, dowell pins, dowells, drawer sides, excelsior, floor lamps, flooring, matches, millwork, moldings, packing cases, plywood pulpwood, shipbuilding, sporting goods, tool handles, vehicle parts, decorative (Patterson, 1988; Malaysian Timber Industry Board,1986). *Antiaris africana* is utilized traditionally in ethno-medicine for epilepsy, lumbargo, skin irritant, purgative, nervous disorders (Odugbemi, 2008). It is used ethnobotanically in Nigeria to relieve rheumatic, respiratory and stomachic pains. Literature reports that *A. africana* contains flavonoids and it has antibacterial activity (Banso and Mann, 2008). Okogun *et al.* (1976) reported they isolated triterpenoids and betaines like Phenylalaninebetaine from *Antiaris africana* (Moraceae).

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Ten compounds have been isolated and reported in methanol extract of the stem bark of *A. africana*. They are betulinic acid; 3β-acetoxy-1β,11α-dihydroxy-olean-12-ene; ursolic acid; oleanolic acid; strophanthidol; periplogenin; convallatoxin; strophanthidinic acid; methyl strophanthinate and 3,3′-dimethoxy-4′-*O*-β-d-xylopyronosylellagic acid.

They have been tested and confirmed to have antioxidant and anticancer activities, results of the study provide supportive data for the traditional anticancer use of *A. africana* and indicate that the methanol extract as well as the ten compounds are potential source of medicine for the treatment of cancer, having also interesting antioxidant properties. (Kuete *et al*., 2009; Okogun *et al*., 1976).

Four compounds were isolated from the stem bark of *Antiaris africana*. They are lichenxanthone, *β*-sitosterol, betulinic acid and a γ-lactone named antialactone (Bertina *et al.*, 2008).

We report the essential oil compositions of leaf, stem bark and root of *Antiaris africana* which is scarce in literature and may be responsible for its odour, vast applications in decorations and high ethno-medicinal values.

MATERIALS AND METHODS

Plant Materials

Experienced herb sellers in Ago-Iwoye assisted in getting right locations of *Antiaris africana* in Ago-Iwoye, Ogun- State, Nigeria, because it is an endangered species. Leaf; stem bark and root samples were collected in Ago-Iwoye, Ogun- State, Nigeria, for extraction of their volatile oils.

Plant was authenticated at the Herbarium, Department of Botany, University of Ibadan, Ibadan, where voucher samples were deposited and signed certificate of identification was obtained (Dr Ayodele and Mr Donatus O. Esimekhuai). Dr Soladoye M.O. (Plant taxonomists, OOU, Ago-Iwoye) also confirmed the plant.

Isolation of Essential oils

The plant was separated into leaf, stem bark and root parts. Weighed amounts of each (500 g of leaf, 350 g of stem bark and 450 g of root) were hydro-distilled in an all glass Clevenger-type apparatus, over very little distilled hexane (0.3 ml), which was removed afterwards. The distillation time was 3 h in each case. 1.18 g of leaf, 2.36 g of stem bark and 3.41 g of root essential oils were procured from the samples, each with characteristic odour. The yields were respectively 0.24%; 0.67% and 0.76%.

Gas Chromatography and Gas Chromatography-Mass Spectrometry

The three essential oils were subjected to GC analyses on a QP2010 PLUS Shimadzu gas chromatograph, AOC-20i gas chromatography- mass spectrometry, with following programming: 250 0C injection temperature with split mode; column flow 0.97 mL/min; total flow 52.5 mL/min; oven temperature programming from 75 0C (5 min hold) to 250 0C (rate is 4 0/min). GC-MS (AOC-20i QP2010 Plus) ion-source temperature 200 0C; interface temperature 250 0C; threshold 3000; start time 3 mins, end time 56 mins; ACQ mode scan; scan speed 1428; used MS program, mass selective detector operated at 70 eV with a mass range of m/z 40-700.

Identification of components

Identification of the essential oil components were based on their retention indices (determined with reference to a homologous series of *n*-alkanes), and by comparison of their mass spectral fragmentation patterns with in-built computer data and commercial systems, such as the NIST database/ Chemstation data system, Wiley GC/MS Library (Massada, 1976), Adams Library (Adams, 1995), Mass Finder 3.1 Library (Joulain & Koenig, 1998), and in-house "Başer Library of Essential Oil Constituents" built up from genuine compounds and components of known oils.

RESULTS AND DISCUSSION

Antiaris africana an endangered species was collected in Ago-Iwoye, Ogun- State, Nigeria. It was separated into leaf, stem bark and root parts. Their volatile oils were obtained by hydro-distillation in 0.24%; 0.67% and 0.76% yields respectively. GC and GC-MS analyses revealed 10 compounds are responsible for leaf oil; 22 compounds make up 98.9% of the stembark oil, while 6 compounds make-up 99.6% of root oil [see figures 1,2 and 3 for the chromatograms]. 9 compounds were identified in leaf, which make-up 92.2% of it and 5 identified compounds in root represent 6.6% of it.

Table. 1: Chemical Composition of the Essential oil of *Antiaris africana* Leaf.

Peak No[a]	MS (Base peak + most abundant peaks) [b]	Identified compound [c]	%TIC [d]	Retention time (mins) [e]	Calculated RI[f]
1	83,41,55,69,111,44,43,57,67	Ethylcyclohexane;	7.1	1.67	166
2	91,106,105,77,43,65,51,	5-(1methylethylidene)-1,3-cyclopentadiene	16.8	1.75	172
3	91,106,55,69,111,77,83,44,	p-xylene	9.3	1.85	180
4	105,120,44,77,91,103,106,	1-ethyl-2methylbenzene	4.5	2.27	214
5	105,120,55,97,77,119,44,81,91,	1,3,5-trimethylbenzene	12.6	2.54	236
6	57,43,71,41,44,85	u.i[g]	7.8	2.72	250
7	67,84,43,69,44,57,71,81,93,111,	isopulegol	4.4	2.94	291
8	57,43,44,41,55,71,85,81,56,95,109,119,	z,z-2,5-pentadecaden-1-ol	4.2	4.16	369
9	95,81,105,152,106,69,41,55,83,67,	Camphor	20.7	4.41	385
10	71,81,95,123,41,55,138,43,69,82,	isomenthol	12.6	5.23	475

[a]According to the rention time from GC; [b](m/e) values of base peak 1st stated, and other most prominent ions; [c]see identification of components; [d]Total ion concentration in %; [e]Retention time in minutes; [f]Retention Index determined with reference to homologous series of n-alkanes; [g]unidentified component).

Table. 2: Chemical Composition of the Essential oil of *Antiaris africana* Stembark .

Peak No[a]	MS (Base peak + most abundant peaks) [b]	Identified compound [c]	%TIC [d]	Retention time (mins) [e]	Calculated RI[f]
1	67,41,17,55,44,57,69,82,111,42,43,83	3-methyl,4-penten-1-ol	1.2	1.67	166
2	55,54,43,41,42,69,91	1-methylbutyl-oxirane	2.7	1.72	170
3	91,105, 44,104,77,51, 43	5-(1-methylethylidene)-1,3-cyclopentadiene	1.5	1.75	172
4	,91, 44,106,69,77,111	Ethylbenzene	1.2	1.85	180
5	,93,77,91,92,105,44,67	α-pinene	1.3	2.14	204
6	105,81,120,44,138,77,79,53	2,4-dimethyl-2,3-heptadien-5yne	1.7	2.53	235
7	119, 134,91, 117,120,77,65,115	1-methyl-4(1-methylethyl)benzene	4.1	2.85	260
8	43,81,108,154, 71,111,139,84,93, 69	Eucalyptol	19.5	2.94	291
9	93,91,136,17,77, 121,79,43,41,	1-methyl-4(1methylethyl)-1,4-cyclohexadiene	2.6	3.31	315
10	59,94,55,93,43,111,68,67,41,81,	Cis-linlooloxide	3.5	3.44	323
11	59,94,43,55,68,93,111,67,81,	Trans-linalooloxide	1.5	3.65	336
12	71,93, 41,55,43,69,80,67,121,83,81,136,	β-myrcene	3.5	3.87	350
13	71,111,93, 43,85,41,55,69,154,67,91,136,	4-methyl-1-(1methylethyl)-R-3cyclohexen-1-ol	17.4	5.31	478
14	59,93,121,136,81, 43,67,92,41,55,	p-menth-1-en-8-ol	8.5	5.57	488
15	152,123,91,109,44,66,77,40,107,	3,5-dimethoxytoluene	1.5	7.32	714
16	74,87,43,143,149,55,57,41,83,227,270,44,	12-methyl,-methyl(S)tetradecanoate	1.1	27.21	2425
17	73,60,43,55, 129, 41,57,69,71,	n-hexadecanoic acid	14.2	31.74	3150
18	152, 71,43,96,44,55,97,115,185,248,125	3methyl-N-dimethylaminomethylene-pyrazol-5amine	1.9	33.72	3203
19	113,43,55,67,41,81,95,135,181,57,69,83,85	1,5-dihydro-4methoxy-2H-pyrrol-2-one	1.2	36.58	3560
20	57,71,43,85,41,55,69,99,44,56,83,97	2,6-dimethylheptadecane	1.9	41.54	3963
21	57,71,43,85,17,97,113,127,	2,6,10,15-tetramethylheptadecane	2.7	44.17	4284
22	57,71,43,85,55,41,99,113,97,127,	10-methyl-eicosane	4.2	47.26	4617

[a]According to the rention time from GC; [b](m/e) values of base peak 1st stated, and other most prominent ions; [c]see identification of components; [d]Total ion concentration in %; [e]Retention time in minutes; [f]Retention Index determined with reference to homologous series of n-alkanes; [g]unidentified component).

Table. 3: Chemical Composition of the Essential oil of *Antiaris africana* Root .

Peak No[a]	MS (Base peak+most abundant peaks) [b]	Identified compound [c]	%TIC [d]	Retention time (mins) [e]	Calculated RI[f]
1	74,87,43,55,143,227,270,41,75,	Methylhexadecanoate	41.3	29.94	2806
2		u.i[g]	3.0	33.19	3189
3	55,44,69,83,84,41,96,74,97,87,67,111,	Methyl-13-octadecenoate	17.3	35.20	3242
4	57,71,43,85,44,40,55,69,97,41,113,	2,6-dimethylheptadecane	4.0	44.10	4285
5	57,71,85,43,55,69,44,83,99,97,	10-methyleicosane	30.4	44.23	4289
6	,57,71,43,85,44,55,56,69,40,	2,6,10-trimethylpentadecane	3.6	52.29	5205

[a]According to the rention time from GC; [b](m/e) values of base peak 1st stated, and other most prominent ions; [c]see identification of components; [d]Total ion concentration in %; [e]Retention time in minutes; [f]Retention Index determined with reference to homologous series of n-alkanes; [g]unidentified component).

Most abundant compounds in leaf oil are camphor (20.7%), 5-(1methylethylidene)-1,3-cyclopentadiene (16.8%), isomenthol (12.6%) and 1,3,5-trimethylbenzene (12.6%). Prominent compounds in stem bark oil are eucalyptol (19.3%), 4-methyl-1-(1methylethyl)-R-3cyclohexen-1-ol (17.4%), n-hexadecanoic acid (14.2%) and p-menth-1-en-8-ol (8.5%). Root oil is dominated by methylhexadecanoate (41.3%), 10-methyleicosane (30.4%) and methyl-13-octadecenoate (17.3%). This paper report 6 other compounds in leaf oil, 18 other compounds in stem bark oil and 2 others in root oil along with above as composition of *Antiaris africana* Engl. [tables 1,2 and 3]. The leaf and stembark oils are highly terpenic, having many interesting C_{10} and C_{15} terpenoids; Eucalyptus is the most abundant compound in stembark oil, and is known to be highly medicinal. There is presence of isomers of linalooloxide in stem bark oil. Generally, compositions of the three volatile oils are very interesting, report of which is scarce in literature.

CONCLUSION

Leaf, stem bark and root essential oils from *Antiaris africana* Engl., were isolated and analyzed using GC and GC-MS analyses. Nine compounds were identified in leaf, which make-up 92.2% of it and 5 identified compounds in root represent 96.6% of it. Most abundant compounds in leaf oil are camphor (20.7%), 5-(1methylethylidene)-1,3-cyclopentadiene (16.8%), isomenthol (12.6%) and 1,3,5-trimethylbenzene (12.6%). Prominent compounds in stembark oil are eucalyptol (19.3%), 4-methyl-1-(1methylethyl)-R-3cyclohexen-1-ol (17.4%), n-hexadecanoic acid (14.2%) and p-menth-1-en-8-ol (8.5%). Root oil is dominated by methylhexadecanoate (41.3%), 10-methyleicosane (30.4%) and methyl-13-octadecenoate (17.3%). *Antiaris africana* Engl. essential oils contain high amount of terpenoids. This paper report 6 other compounds in leaf oil, 18 other compounds in stem bark oil and 2 others in root oil along with above as composition of Antiaris africana Engl., which is new in literature.

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