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Volatile Components of *Oncoba spinosa* leaf and *Morus mesozygia* leaf and stem bark

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ABSTRACT

The essential oils of *Oncoba spinosa* leaf and *Morus mesozygia* leaf and stem bark were extracted using hydro distillation and analyzed by means of Gas chromatography (GC) and GC coupled with mass spectrometry (GC-MS). The yields of the essential oils were; 0.50 %, 0.165 % and 0.456 % respectively for *Oncoba spinosa* leaf, *Morus mesozygia* leaf and stem oil. A total of twenty eight, thirty four and twenty compounds representing 92.0%, 92.0% and 96.9% of the total oil contents were identified, respectively from the leaf of *O. spinosa*, leaf and stem oil of *M. mesozygia*. Leaf oil of *O. spinosa* contained linalool (22.1 %), β – caryophyllene (18.7 %), caryophyllene oxide (10.6 %) and pentadecanal (5.6 %) as the main constituents. *M. mesozygia* leaf oil was dominated with β – elemene (11.7 %), (*E*) –β- ionone (12.4 %), α- selinene (5.1 %), germacrene A (6.0 %), δ – cadinene (4.7 %) and spathulenol (7.4 %) while *M. mesozygia* stem oil had 2 –dodecanone (77%) and hexahydrofarnesylacetone (13 %) as its main constituents.

Key words: *Oncoba spinosa, Morus mesozygia,* linalool, (*E*) –β- ionone, 2 –dodecanone.

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INTRODUCTION

Plants essential oils and their components have been known to exhibit biological activities. Screening of plants extracts or their essential oil for these properties has become of great importance because of their growing interest in food and pharmaceutical industries (1). *Oncoba spinosa*, commonly known as the snuff-box tree is a plant species in the genus *Oncoba* and family *Flucourtiaceae*. It is a small to medium-sized deciduous tree of about 5 m high and has simple leaves. The tree is widely distributed along the eastern side of Africa as far as South Africa, mainly in dry woodland or open savanna in a wide range of sites from river valleys to rocky hills. In African medicine, the roots are used in the treatment of dysentery and bladder complaints and fruits in the treatment of leprosy. The anti-ulcerogenic potential (2), antihyperglycemic and antineoplastic activities (3), and anti-convulsant property (4) have been studied. The fruit was found to be effective in the treatment of urinary tract diseases of cows and camels (5). In addition, the antipasmodial (6) and antihelmintic properties (7) have been discussed in literature.

Morus mesozygia (Moraceae), commonly called black mulberry or African mulberry, is a small to medium sized forest tree. M*orus* comprises of ten to fifteen species with only one specie (*Morus mesozygia*) native to tropical Africa. It has a wide distribution in tropical Africa, from Senegal eastward to Ethiopia and southward to Zambia, Angola, Mozambique and South Africa (8). In African traditional medicine, parts of *M. mesozygia* are used in decoctions, baths, massages and enemas against rheumatism, lumbago, intercostal pain, neuralgia, colic, stiffness, debility, diarrhea and dysentery. It is evident from literature that extract of the plant is known to have antidepressant property (9). Arylbenzofurans and flavonoids were isolated from *M. mesozygia* (10) and the anti-plasmodial and cytotoxic properties of arylbenzofuran have been studied (11). The leaf essential oil of another species *Morus rotunbiloba* was characterized and dominated by benzyl alcohol (10.48%), dihydroactinolide (10.11%) and palmitic acid (7.01%); the oil also displayed good antibacterial and cytotoxic activities (12).

Therein, we describe the chemical composition of the essential oil from the leaves of *O. spinosa* and leaves and stem bark of *M. mesozygia* for the first time.

EXPERIMENTAL

Plant materials

Plant samples were collected from the Botanical garden, University of Ibadan, Ibadan, Nigeria in March 2014 by Mr. Owolabi. The plants were authenticated at the Herbarium of the Forestry Research Institute of Nigeria (FRIN) Oyo State, Nigeria where voucher specimens FHI 110037 and FHI 110038 for *Oncoba spinosa* and *Morus mesozygia* respectively were deposited. Plant samples were air-dried prior to extraction.

Isolation of essential oils

250 g of each of the dried plant samples were shredded and their oils were obtained by separate distillation for 3 hours in a Clevenger type apparatus. The essential oil fractions obtained were kept in a refrigerator at 4^{o}C prior to analysis.

Analysis of the Oils

Gas chromatography (GC) analysis was performed on a HP-5890 Gas chromatograph equipped with a Flame ionization detector and fitted with HP-5 capillary column (30 m x 0.25 mm, 0.25 μm film thickness). The GC oven temperature was programmed at 60°C (held for 10 min), heated to 220°C at 5°C/min. The injector and detector temperatures were maintained at 250°C. Helium was used as carrier gas at a flow rate of 2 mL/min with a split ratio of 1:30.

Gas chromatography / Mass spectrometry (GC-MS) analysis were carried out on a Varian CP-3800 gas chromatograph interfaced to a Varian Saturn 2000 ion trap Mass Detector operated at 70 eV. The injector and transfer line temperatures were 220°C and 240°C, respectively. The GC oven temperature was programmed from 60 to 240°C at 3°C/min. Helium was used as a carrier gas at a flow rate of 1 mL/min, injection of 0.2 μL aliquots as 10% Hexane solution, split ratio of 1:30.

Identification of the constituents on both columns was based on comparison of the retention times with those of authentic samples, comparing their retention indices relative to the series of n-hydrocarbons, and by comparison of their mass spectra with published spectra and those of reference compounds from NIST 98 and ADAMS (13, 14, 15, 16, 17). The relative concentration of each constituent was calculated by integration of GC peak areas.

RESULTS AND DISCUSSION

A total of 28 compounds amounting to 92% were identified in *O. spinosa* leaf essential oil. Among these are, 29.2% oxygenated monoterpenes, 22.3% sesquiterpenes and 9.2% oxygenated sesquiterpenes. It also contained non-terpene derivatives (14.7%) and apocarotenes

(16.6%). However, the major constituents identified are; linalool (22.1%), β- caryophyllene (18.7%), caryophyllene oxide (10.6%), pentadecanal (5.6%), methyl salicylate (4.2%) and *(E)* - β –ionone (3.8%). Linalool and β- caryophyllene which dominated the oil are noted for many commercial and medicinal applications. Linalool, a naturally occurring terpene alcohol has been utilized in perfumery (18) while β- caryophyllene is a dietary cannabinoid (19) which has been found to have clearly demonstrated its ability in the regulation of emotional beahviour, thereby suggesting that its receptor could be relevant therapeutic target for the treatment of anxiety and depressive disorders (20).

A total of 34 constituents were identified in the leaf of *M. mesozygia*, representing 92.0% of the total fraction. Monoterpenes (0.5%), sesquiterpenes (48.5%), oxygenated sesquiterpenes (22.1%), apocarotenes (18.4%) and non-terpene derivatives were identified in the essential oil. The main constituents are; (*E*)-β-ionone (12.4%), β-elemene (11.7%), Spathulenol (7.4%), α-selinene (5.1%), δ–cadinene (4.7%), humulene epoxide II (3.8%) *(E)* - α-ionone (3.4%), trans-α-bergamotene (3.5%). (*E*)-β-ionone is a flavouring agent and exhibits anti-proliferative function (21) while β-elemene has exhibited tremendous anti-tumor properties (22, 23).

The essential oil of the stem bark of *M. mesozygia* afforded identification of 20 constituents, corresponding to 96.9% of the oil constituents. Monoterpenes (0.1%), oxygenated monoterpenes (0.5%), sesquiterpene (2.5%), oxygenated sesquiterpenes (1.4%), apocarotenes (14.0%) and non-terpene derivative (78.4%) were identified in the essential oil. The main constituents in the oil are; 2-dodecanone (77.0%), hexahydrofarnesyl acetone (13.0%), ar-curcumene (1.0%). Moreover, it was observed that both *M. mesozygia* leaf and stem bark essential oils had *E*- β- ionone (12.4% and 0.4%) respectively as common constituents and also the most abundant in the leaf oil of *M. mesozygia.* Other common constituents are spathulenol

(7.4% and 0.3%), caryophyllene oxide (2.9% and 0.5%) alloaromadendrene (1.5% and 0.3%), *E*-geranylacetone (2.1% and 0.6%), α-humulene (1.1% and 0.2%), β- caryophyllene (1.2% and 0.6%), limonene (0.5% and 0.1%) and 2- pentylfuran (0.5% and 0.3%) respectively. The dominance of the stem oil of *M. mesozygia* by 2-dodecanone and hexahydrofarnesyl acetone has indicated that the bark oil may exhibit excellent insect repellant properties. In a study by Ndungu et al (24), it was revealed that 2-dodecanone was one of the constituents in the oil of *Cleome monophylla* that exhibited high repellent activity against tick and maize weevil. In another related work, it was deduced that 2-dodecanone exhibited good protection efficacy against *An. gambiae* (100% protection at 10% concentration) and also concluded that methyl ketone analogies are potential source of insect repellent including mosquitoes (25). Hexahydrofarnesylacetone is also a widespread ketone found in plant and insects which several insect uses as part of their pheromone bouquet (26).

The abundance of both compounds 2-dodecanone and hexahydrofarnesylacetone totaling 90% of the stem oil of *M. mesozygia* gives a strong lead that it possesses good insect repellent properties and has therefore opened an avenue to research into the repellent ability of the essential oil to different species of insects. On the contrast, these compounds were not found in the leaf oil yet confirming a known fact that plant tissues differ in their chemical constituents. The constituents of these oils were at variance to the compounds characterized in the oil of another species *Morus rotunbiloba*, fifty-one compounds were detected in the leaf essential oil with benzyl alcohol (10.48%), dihydroactinolide (10.11%) and palmitic acid (7.01%) being the most abundant (12). Out of the compounds characterized in *M. rotunbiloba*, only four were found in either of the two oils of *M. mesozygia*, these are E-α-ionone, hexahydrofarnesylacetone, geranylacetone and β-cyclocitral.

CONCLUSION

The different therapeutics and commercial importance of the characterized compounds in these essential oils justifies the use of different parts for the treatment of different ailments. This represents the first attempt to our knowledge at characterizing the essential oil constituents of the leaves of *O. spinosa* and leaves and stem bark of *M. mesozygia*.

Table 1: Essential Oil Constituents of the Leaves of *Oncoba spinosa* and Leaves and stem bark of *Morus mesozygia*.

S/N	Constituents	L.R.I[a]	L.R.I[b]	*O. spinosa* Leaves	*M. mesozygia* Leaves	*M. mesozygia* Stem bark
1	*(E)* -2-hexenal	865	846	0.8	-	-
2	*p*-xylene	867	883[a]	-	0.5	-
3	6-methyl-5-heptene-2-one	987	992	-	-	0.4
4	2-pentyl furan	993	984	0.7	0.5	0.3
5	*(E)* -2-pentenyl furan	1004	1000[b]	0.7	-	-
6	Limonene	1032	1024	-	0.5	0.1
7	1,8-cineole	1034	1026	-	-	0.3
8	Linalool	1101	1095	**22.1**	**-**	-
9	Nonanal	1103	1100	-	0.5	-

10	α –terpeneol	1191	1186	3.1	-	-
11	Methyl salicylate	1192	1190	4.2	0.5	-
12	β-cyclocitral	1222	1217	0.9	0.5	-
13	Nerol	1230	1227	1.2	-	-
14	Methyl carvacrol	1246	1298	-	-	0.2
15	Geraniol	1256	1249	2.8	-	-
16	α-copaene	1377	1374	0.7	2.9	-
17	β-elemene	1392	1389	0.9	**11.7**	-
18	2-dodecanone	1395	1394	-	-	**77.0`**
19	*cis*- α-bergamotene	1416	1411	-	0.5	-
20	β-caryophyllene	1419	1417	**18.7**	1.2	0.6
21	α- santalene	1420	1416	-	1.3	-
22	*(E)*- α-ionone	1428	1428	1.1	3.4	-
23	Trans-α-bergamotene	1438	1432	-	3.5	-
24	Precocene1	1448	1440[d]	-	-	0.3
25	epi-β-santalene	1450	1445	-	0.5	-
26	α-humulene	1455	1452	1.1	1.1	0.2
27	*(E)*-geranylacetone	1455	1453	1.5	2.1	0.6
28	Alloaromadendrene	1462	1461	-	1.5	0.3
29	Cabreuva oxide B	1463	1462	0.8	-	-
30	γ-muurolene	1478	1478	-	1.8	-

31	Germacrene D	1482	1484	-	1.3	-
32	ar-curcumene	1483	1479	-	-	1.0
33	*(E)*-β-ionone	1487	1487	3.8	**12.4**	0.4
34	10,11-epoxycalamenene	1492	1476[e]	-	-	0.4
35	α-selinene	1496	1498	-	5.1	-
36	Germacrene A	1504	1508	-	6.0	-
37	β-bisabolene	1509	1505	-	2.0	0.3
38	β-curcumene	1513	1514	0.9	2.3	
39	δ-cadinene	1524	1513	**-**	**4.7**	-
40	*(E)*- γ –bisabolene	1534	1529	-	0.5	-
41	Dihydroactinolide	1536	1531[f]	1.9	-	-
42	α- calacorene	1543	1544	-	0.6	-
43	*(E)*-nerolidol	1565	1561	1.8	-	-
44	Caryophyllene alcohol	1569	1568	-	-	0.6
45	*(Z)*-3-hexenyl benzoate	1570	1564[g]	0.5	-	-
46	Spathulenol	1577	1577	0.7	**7.4**	0.3
47	Caryophyllene oxide	1582	1582	**10.6**	2.9	0.5
48	Viridiflorol	1591	1592	1.1	-	-
49	Humlene epoxide II	1607	1608	0.8	3.8	-
50	Tetradecanal	1612	1611	1.2	-	-

51	Humulene-1,6-diene-3-ol	1618	1619[h]	-	1.9	-
52	Cedrenol	1630	I642[i]	-	1.7	
53	Caryophylla-4(14),8(15)-dien-5-ol	1637	1623[j]	1.1	-	-
54	Selin-11-en-4- α-ol	1655	1658	-	1.9	-
55	α-bisabolol	1684	1685	-	0.7	-
56	Pentadecanal	1716	1711 [k]	**5.6**	-	-
57	Hexahydrofarnesyl acetone	1845	1843[l]	**-**	**-**	**13.0**
58	Benzyl salicylate	1864	1864	-	-	0.4
1	Monoterpene hydrocarbons			0.0	0.5	0.1
2	Oxygenated monoterpenes			29.2	0.0	0.5
3	Sesquiterpenes hydrocarbons			22.3	48.5	2.5
4	Oxygenated sesquiterpenes			9.2	22.1	1.4
5	Apocarotenes			16.6	18.4	14.0
6	Non-terpene derivatives			14.7	2.5	78.4
	Total Identified			**92.0**	**92.0**	**96.9**

"Notes"
L.R.I[a] = Linear Retention Index From This Work
L.R.I[b] = Linear Retention Index From Literature (Adams)
^[d-l] = References 27-35.

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