



Essential Oils of Aerial Parts of *Crassocephalum rubens* (Juss. ex Jacq.) S. Moore and *Cardiospermum grandiflorum* (Sweet) Stem

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Authors' contributions

This work was carried out in collaboration between all authors. Author OOO designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors IAO, OOA and GF managed the analyses of the study. Author OOO managed the literature searches. All authors read and approved the final manuscript.

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Short Communication

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ABSTRACT

Aim: This paper presents the essential oils constituents of aerial parts of *Crassocephalum rubens* and stem of *Cardiospermum grandiflorum*. *C. rubens* is a Nigerian vegetable used as a nutraceutical and traditionally has been used as antibiotic, anti-helminthic, anti-inflammatory, anti-diabetic, anti-malaria and blood regulating agents. *C. grandiflorum* (Sapindaceae) is considered a noxious weed in Australia and South Africa. The leaves are taken as vegetable in Ghana and have application for dermatological troubles, chest problems and fever. The plant has also been used in the treatment of jaundice, cough and kidney problems.

Methodology: The essential oils were obtained by hydro-distillation in a Clevenger-type apparatus

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designed to British Pharmacopeia specifications and analyzed using Gas-Chromatography/Mass-Spectrometry (GC-MS).

Results: Oil yields were 0.52% and 0.09% w/w for *C. rubens* and *C. grandiflorum* respectively. Fifty-six compounds representing 97.5% of *C. rubens* oil were identified. Main constituents in the leaf oil of *C. rubens* were monoterpenes; limonene (81.1%), (*Z*)- β -ocimene (4.6%), terpinolene (3.4%) and (*E*)- β -ocimene (2.2%). Oxygenated derivatives were detected at trace amount. Sixty-three compounds were identified that represented 97.8% of the essential oil of *C. grandiflorum* stems. The essential oil was characterized by high content of sesquiterpene hydrocarbons that accounted for 91.2% of the whole oil followed by 3.7% oxygenated sesquiterpene. The main compounds were germacrene D (26.2%), (*Z*)- γ -bisabolene (15.5%), β -caryophyllene (13.9%) and germacrene B (7.9%).

Conclusion: This paper presents essential oil components of *C. rubens* and *C. grandiflorum* that are scanty in the literature. Characterized compounds in each plant could be responsible for their ethno-medicinal activities.

Keywords: *Crassocephalum rubens*; *Cardiospermum grandiflorum*; hydro-distillation; essential oil; GC-MS; limonene; Germanene D.

1. INTRODUCTION

Crassocephalum rubens (Asteraceae) is an erect annual herb growing up to 80 cm tall. It is grown and consumed especially in Southwestern Nigeria and also as far away as Yemen, South Africa and Island of Indian Ocean. It is locally called Bologi and Ebolo among the Yorubas. Its mucilaginous leaves are used as dry or fresh vegetable in variety of dishes and as medicine for several different ailments [1]. Traditionally, *C. rubens* is used as a nutraceutical and believed to have antibiotic, anti-helminthic, anti-inflammatory, anti-diabetic, anti-malaria and blood regulation properties, and also treats indigestion, liver complaints, colds, intestinal worms and hepatic insufficiency [2,3]. Antimicrobial and hepatoprotective effects of the leaves of *Crassocephalum rubens* have been reported [4,5]. Also the antimicrobial, hepatoprotective, antioxidant, antinociceptive, anti-inflammatory, anthelmintic and cancer chemo-preventive actions of some species of the *Crassocephalum* genus are well established [6, 7,8,9]. Secondary metabolites detected in *C. rubens* were alkaloids, anthocyanins, quinone derivatives, saponins, triterpenoids, cyanogenic derivatives, cardiac glycosides and anthracene derivatives. The nutritional potential of *C. rubens* was evaluated through their proximate composition. The analysis revealed the contents of raw protein, total lipids, ash and carbohydrates to be 26.43 \pm 0.01%, 2.75 \pm 0.01%, 19.76 \pm 0.05% and 43.11 \pm 0.10 % respectively [2,3].

Literature on the essential oils composition of *C. rubens* are scarce. Nonetheless, Yehouenou et al. (2010) reported the presence of limonene

(48.8%), myrcene (30.7%), *E*-(β)-ocimene (7.4%) and α -thujene (4.6%) from fresh leaves of *C. rubens* at Ouèssè and Kpakpassa, in West-center of Republic of Benin [5]. It has been established that the essential oils composition pattern of medicinal plants could be affected by geographical and climatic conditions [8]. However, there is no previous report on the chemical compositions of essential oils of *C. rubens* found in Nigeria.

Cardiospermum grandiflorum (Sapindaceae) originated from America, Africa and West Indies. This plant is considered a noxious weed in Australia and South Africa [10]. It is commonly called balloon vine and locally known as 'Ako-ejirin' in Southwestern Nigeria [11].

The leaves are taken as vegetable in Ghana and have application for dermatological troubles, chest problems and fever while the fruits are used traditionally for abortion. The plant has also been used in the treatment of jaundice, cough and kidney problems [11,12]. Olaoluwa and Aiyelaagbe (2015) reported that *C. grandiflorum* extract displayed moderate antimicrobial activities against *Staphylococcus aureus*, *Escherichia coli* and *Candida albicans* [13].

C. halicacabum, another specie of *C. grandiflorum*, was stated to possess antimicrobial, antiparasitic, anxiolytic, antioxidant, antipyretic, antirheumatic, anticonvulsant, anti-inflammatory, neuroprotective, anticarcinogenic, anti-infertility, laxative and larvicidal activities [14-19].

Phytochemical investigations of *C. halicacabum* yielded some compounds that includes linoleic

acid, 1,2,4-trioxolane-2-octanic acid, ricinolenic acid, 2-hexyl-methyl ester, 7-methyl-7-tetradecan-1-ol acetate, 9-octadecenoic acid, 1,2,3-propanetriyl ester, (+)-pinitol, β sitosterol- β -o-glactoside, apigenin-7-o-glucuronide and luteolin-7-o-glucuronide [19].

Literature survey showed that there is no report on the essential oil constituents of *C. grandiflorum* found in Nigeria. Therefore, we have discussed the volatile constituents of the stems of *C. grandiflorum*.

2. MATERIALS AND METHODS

2.1 Plant Materials

Plants samples of *Crassocephalum rubens* and *Cardiospermum grandiflorum* were harvested, at full flowering stage, along Olorunda road, Akobo-Ojurin, Ibadan, Nigeria in September, 2010. They were identified, authenticated and deposited (FHI 109490 and 109489, respectively) at the herbarium unit at Forestry Research Institute of Nigeria (FRIN), Ibadan by Mr O. A. Micheal.

2.2 Extraction of Essential Oils

Fresh samples of *Crassocephalum rubens* aerial parts (775 g) and of *C. grandiflorum* stems (838 g) were subjected to hydro-distillation in a Clevenger-type apparatus designed to British Pharmacopeia specifications for 3 h [20]. The essential oil was collected over hexane and stored in a sealed vial under refrigeration prior to analysis.

2.3 Gas Chromatography- Mass Spectrometry (GC-MS)

Essential oil was subjected to GC analysis on Shimadzu model QP2010 system equipped with an AOCi-20i autosampler. This was coupled to Mass spectrometer, Shimadzu model QP2010 system, with split/split-less injector interfaced to mass selective detector operated at 70 eV, ion source temperature of 200°C and mass range of m/z 50 -700 at scan rate of 1428 amu/sec. The column used was DB5 (30 m \times 0.25 mm, 0.25 μ m film thickness). Helium was used as the carrier gas at a flow rate of 1 mL/min, linear velocity of 362 cm/sec and pressure 56.2 KPa. The oven temperature was set at 60°C, hold for 1 min to 180°C for 3 mins at 10°C/min, the final temperature was 280°C for 2 mins at 10°C/min. Both injector and detector temperatures were fixed at 250°C.

2.4 Identification of Compounds

Each constituent of the essential oil was identified based on their retention indices (determined with reference to a homologous series of normal alkane) and by comparison of their mass spectral fragmentation patterns (NIST data/base/chemstation data system) with data previously reported in the literature [21].

3. RESULTS AND DISCUSSION

Constituents of aerial parts essential oil of *C. rubens* and stems of *C. grandiflorum* are presented in Tables 1 and 2, respectively. The oils were colourless with 0.52% and 0.09% w/w yield, of fresh weight, respectively. Fifty-six compounds representing 97.5% of *C. rubens* oil were identified. Prominent compounds were monoterpenes (94.5%); limonene (81.1%), (Z)- β -ocimene (4.6%), terpinolene (3.4%) and (E)- β -ocimene (2.2%). Oxygenated derivatives were detected at trace amount. This result is partially in agreement with volatile constituents of *C. rubens* found in the Republic of Benin, which also had limonene (48.8%) as the most prominent compound. Variation in composition of limonene in *C. rubens* found in West-center of Republic of Benin and Nigeria could be due to different geographical and climatic conditions [5].

Limonene has been found to possess numerous biological activities that include insecticidal, antibacterial and anticancer [22-24]. Biological activities attributed to *C. rubens* in ethno-medicine may be due the copiousness of limonene in this plant. This plant could be a valuable alternative source of monoterpene especially limonene.

Sixty-three compounds were identified that represented 97.8% of the essential oil of *C. grandiflorum* stems. The essential oil was characterized by high content of sesquiterpene hydrocarbons. These hydrocarbons accounted for 91.2% of the whole oil. They were followed by oxygenated sesquiterpenes (3.7%). The other chemical classes were considerably less represented such as non-terpene hydrocarbons (1.1%), non-terpene aldehyde (0.4%), non-terpene ester (0.3%) and non-terpene ketone (tr). The main compound was germacrene D (26.2%), followed by (Z)- γ -bisabolene (15.5%), β -caryophyllene (13.9%) germacrene B (7.9%) and β -curcumene (4.6%). Also, essential oil consisted of *cis*- β -guaiene (1.3%), *trans*- β -guaiene (1.2%), gossonorol (0.5%), β -atlantol (0.4%) and methyl-2-hydroxybenzoate (tr).

Table 1. Essential oil composition of aerial parts of *Crassocephalum rubens*

S/N	Compounds	RI	(%) TIC
1.	n-nonane	899	Tr
2.	α -thujene	932	Tr
3.	α -pinene	940	1.6
4.	Camphene	953	Tr
5.	Sabinene	978	0.1
6.	β -pinene	982	Tr
7.	α -phellandrene	1007	0.5
8.	α -terpinene	1020	Tr
9.	Limonene	1034	81.1
10.	(Z)- β -ocimene	1041	4.6
11.	(E)- β -ocimene	1050	2.2
12.	γ -terpinene	1063	1.0
13.	Terpinolene	1090	3.4
14.	Linalool	1101	Tr
15.	1,3,8-p-mentha-triene	1113	Tr
16.	<i>trans</i> -p-mentha-2,8-dien-1-ol	1123	Tr
17.	<i>cis</i> -limonene oxide	1136	Tr
18.	<i>cis</i> -p-mentha-2,8-dien-1-ol	1141	Tr
19.	<i>trans</i> -limonene oxide	1142	Tr
20.	(E)-myroxide	1145	Tr
21.	4-terpineol	1179	Tr
22.	Naphthalene	1181	Tr
23.	p-cymen-8-ol	1185	Tr
24.	<i>trans</i> -carveol	1219	Tr
25.	<i>cis</i> -carveol	1231	Tr
26.	Carvone	1244	Tr
27.	<i>trans</i> -piperitone epoxide	1256	Tr
28.	Perilla aldehyde	1271	Tr
29.	isobornyl acetate	1287	Tr
30.	2-undecanone	1293	Tr
31.	Undecanal	1307	Tr
32.	Methyl geranate	1325	Tr
33.	Piperitenone	1343	Tr
34.	Neryl acetate	1367	Tr
35.	α -copaene	1376	Tr
36.	Geranyl acetate	1385	Tr
37.	β -elemene	1393	Tr
38.	n-tetradecane	1400	Tr
39.	β -caryophyllene	1419	0.1
40.	β -gurjunene	1432	Tr
41.	<i>trans</i> -bergamotene	1439	Tr
42.	α -humulene	1455	0.5
43.	6-demethoxy ageratochromene	1463	Tr
44.	γ -muurolene	1477	Tr
45.	Germacrene D	1482	0.6
46.	β -selinene	1487	Tr
47.	2-tridecanone	1496	1.7
48.	Germacrene A	1504	Tr
49.	(Z)- γ -bisabolene	1515	0.1
50.	δ -cadinene	1524	Tr
51.	Selina-3,7(11)-diene	1542	Tr
52.	Germacrene B	1556	Tr
53.	<i>trans</i> -nerolidol	1566	Tr
54.	Spathulenol	1577	Tr
55.	Caryophyllene oxide	1582	Tr
56.	Humulene epoxide II	1608	Tr
	Total identified		97.5%

RI-Retention Index, TIC- Total Ion Concentration

Table 2. Essential oil composition of stems of *Cardiospermum grandiflorum*

S/N	Compounds	RI	(%) TIC
1.	2-pentyl furan	976	0.1
2.	6-methyl-5-hepten-2-one	992	Tr
3.	Mesitylene	994	0.1
4.	<i>n</i> -decane	1000	0.2
5.	<i>p</i> -cymene	1027	Tr
6.	Limonene	1031	0.3
7.	1,8-cineole	1035	0.1
8.	(<i>E</i>)- β -ocimene	1051	Tr
9.	Linalool	1100	0.6
10.	(<i>E</i>)-2-nonenal	1164	Tr
11.	Methyl-2-hydroxybenzoate	1190	Tr
12.	<i>n</i> -dodecane	1200	Tr
13.	Decanal	1205	Tr
14.	(<i>E,E</i>)-2,4-nonadienal	1217	Tr
15.	3-methyl-3-hexen-1-yl-butanoate	1236	Tr
16.	(<i>E</i>)-2-decenal	1263	Tr
17.	(<i>E,Z</i>)-2,4-decadienal	1293	Tr
18.	Undecanal	1306	Tr
19.	(<i>E,E</i>)-2,4-decadienal	1317	0.2
20.	α -elemene	1340	0.4
21.	cyclosativene	1370	0.2
22.	α -copaene	1376	0.8
23.	β -patchoulene	1381	Tr
24.	β -bourbonene	1384	0.4
25.	β -cubebene	1390	Tr
26.	β -elemene	1392	0.5
27.	<i>iso</i> -italicene	1402	0.4
28.	(<i>Z</i>)-caryophyllene	1405	Tr
29.	α -gurjunene	1409	0.2
30.	β -caryophyllene	1418	13.9
31.	β -cedrene	1421	Tr
32.	β -copaene	1432	0.1
33.	γ -elemene	1433	Tr
34.	<i>trans</i> - γ -bergamotene	1439	1.0
35.	Aromadendrene	1441	4.4
36.	α -gurjunene	1444	1.2
37.	α -humulene	1455	2.2
38.	Alloaromadendrene	1461	2.1
39.	γ -muurolene	1477	0.7
40.	Germacrene D	1480	26.2
41.	<i>ar</i> -curcumene	1483	1.5
42.	<i>cis</i> - β -guaiene	1493	1.3
43.	α -muurolene	1500	0.2
44.	<i>trans</i> - β -guaiene	1503	1.2
45.	(<i>Z</i>)- α -bisabolene	1504	2.0
46.	(<i>Z</i>)- γ -bisabolene	1515	15.5
47.	β -curcumene	1516	4.6
48.	7- <i>epi</i> - α -selinene	1522	1.0
49.	δ -cadinene	1524	0.8
50.	Italicene ether	1538	0.5
51.	Germacrene B	1556	7.9
52.	(<i>Z</i>)-3-hexenyl benzoate	1570	0.3
53.	Caryophyllene alcohol	1572	0.3
54.	Caryophyllene oxide	1581	1.9
55.	<i>n</i> -hexadecane	1600	0.1
56.	Humulene epoxide II	1606	0.4
57.	β -atlantol	1608	0.4

S/N	Compounds	RI	(%) TIC
58.	α -acorenol	1632	0.2
59.	Gossonorol	1637	0.5
60.	Pentadecanal	1717	0.2
61.	n-docosane	2200	0.1
62.	n-tricosane	2300	0.2
63.	n-pentacosane	2500	0.4
Total identified			97.8%

RI-Retention Index, TIC- Total Ion Concentration

Germacrene D and germacrene B were reported to have exhibited anti-proliferative, antioxidant, and antimicrobial activities [25-27]. β -caryophyllene has also been reported to possess anticancer and analgesic properties [28]. These components may be responsible for aforementioned biological activities that are common in *Cardiospermum* genus. The minor constituents could also contribute to the bioactivity of the oils, as activities observed from essential oils may not be contributions from major constituents only [29].

4. CONCLUSION

Fifty-six and sixty-three compounds were identified in *Crassocephalum rubens* and stems of *Cardiospermum grandiflorum*; and oils were rich in monoterpenes and sesquiterpenes respectively. Dominant compounds in these essential oils have been reported to possess some biological activities which were observed in each plant genus.

CONSENT

It is not applicable.

ETHICAL APPROVAL

It is not applicable.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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