



Caryophyllene-Rich Essential Oils of Two Species from Southern Côte d'Ivoire: *Guibourtia ehie* (A. Chev.) J Léonard (Caesalpiniaceae) and *Oricia suaveolens* (Engl.) Verd. (Rutaceae)

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Toffé Alexis Kouao, Zana Adama Ouattara, Didjour Albert Kambiré, Bosson Antoine Kouamé, Janat Akhanovna Mamyrbékova-Békro, et al.. Caryophyllene-Rich Essential Oils of Two Species from Southern Côte d'Ivoire: *Guibourtia ehie* (A. Chev.) J Léonard (Caesalpiniaceae) and *Oricia suaveolens* (Engl.) Verd. (Rutaceae). *Compounds*, 2023, 3 (1), pp.73-82. <10.3390/compounds3010006>. <hal-05034487>

HAL Id: hal-05034487

<https://hal.science/hal-05034487v1>

Submitted on 15 Apr 2025

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Article

Caryophyllene-Rich Essential Oils of Two Species from Southern Côte d'Ivoire: *Guibourtia ehie* (A. Chev.) J Léonard (Caesalpiniaceae) and *Oricia suaveolens* (Engl.) Verd. (Rutaceae)

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Citation: Kouao, T.A.; Ouattara, Z.A.; Kambiré, D.A.; Kouamé, B.A.; Mamyrbékova-Békro, J.A.; Tomi, P.; Paoli, M.; Bighelli, A.; Békro, Y.-A.; Tomi, F. Caryophyllene-Rich Essential Oils of Two Species from Southern Côte d'Ivoire: *Guibourtia ehie* (A. Chev.) J Léonard (Caesalpiniaceae) and *Oricia suaveolens* (Engl.) Verd. (Rutaceae). *Compounds* **2023**, *3*, 73–82. <https://doi.org/10.3390/compounds3010006>

Academic Editors: Jorge Pereira and Cassamo U. Mussagy

Received: 16 December 2022

Revised: 6 January 2023

Accepted: 10 January 2023

Published: 12 January 2023



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Abstract: The essential oils of *Oricia suaveolens* and *Guibourtia ehie* from Southern Côte d'Ivoire were extracted by hydrodistillation then analyzed by gas chromatography in combination with retention indices [GC(RI)], gas chromatography coupled with mass spectrometry (GC-MS) and nuclear magnetic resonance of carbone-13 (¹³C NMR) And described here for the first time. A total of 42 compounds were identified in the essential oils of the leaves of *G. ehie* while 55, 40 and 23 components were identified in the leaves, stem bark and root bark oils of *O. suaveolens*, respectively. Essential oils samples were dominated by sesquiterpenes and (*E*)-β-caryophyllene was the major compound common to all samples: *G. ehie* leaf oil contained (*E*)-β-caryophyllene (26.9–40.8%), α-humulene (syn. α-caryophyllene) (6.7–9.7%), β-elemene (5.5–9.5%) and germacrene D (5.6–8.1%); *O. suaveolens*, leaf oil contained (*E*)-β-caryophyllene (33.5–39.3%), (*E*)-β-farnesene (5.9–9.3%), caryophyllene oxide (2.1–7.7%) and α-humulene (4.0–4.6%); stem bark oil contained α-humulene (38.3–45.8%) and (*E*)-β-caryophyllene (34.7–41.6%); root bark oil contained α-humulene (36.1–47.9%) and (*E*)-β-caryophyllene (34.3–43.3%). This study highlighted the abundant presence of (*E*)-β-caryophyllene, a phytocannabinoid sesquiterpene with countless biological properties, in two plant species: *Guibourtia ehie* and *Oricia suaveolens*.

Keywords: essential oil; *Guibourtia ehie*; *Oricia suaveolens*; hydrodistillation; chemical composition

1. Introduction

Guibourtia ehie (A. Chev.) J Léonard and *Oricia suaveolens* (Engl) are two plant-species growing wild in Côte d'Ivoire.

G. ehie is a tree of the Caesalpiniaceae (Fabaceae) family up to 45–50 m tall, with a straight, cylindrical bole, branchless for up to 25 m. The leaves are spirally arranged, paripinnate with a pair of leaflets; the stipules are leaf-like, up to 2 cm long, and often persistent; the petioles are 0.5–1 cm long. The distribution area of *G. ehie* extends from Guinea and Liberia to Cameroon and Gabon [1,2]. It is a medicinal plant present in Cameroon, Ghana, Liberia, Nigeria and Côte d'Ivoire [3]. It is used for the treatment of gonorrhea wounds [3], ulcers [4], high blood pressure and sexual infections [5]. Very few works in the literature deal with the phytochemistry of the *Guibourtia* genus. The phytochemical screening made from solvent extracts of *G. ehie* highlighted the presence of terpenes, sterols,

saponins, flavonoids and alkaloids [4]. A dihydrochalcone glucoside, 2',4-dihydroxy-4'-methoxy-6'-O- β -glucopyranoside dihydro-chalcone, a stilbene glycoside, 3,5-dimethoxy-4'-O-(β -rhamnopyranosyl-(1 $\rightarrow$ 6)- β -gluco-pyranoside) stilbene, and pterostilbene were isolated from *G. tessmanii* [6]. The leaves and trunk of *G. ehie* contained rhaponticine, 2,6-dimethoxybenzoquinone, lupeol, taraxerol, friedelan-3-one, lanosterol, scopoletine and pilloine [5]. Antioxidant and antibacterial activities are attributed to this plant [3–5]. However, no study concerning the chemical composition of the essential oils of *G. ehie* was mentioned in the literature.

Oricia suaveolens (Rutaceae) is a shrub or tree up to 10 m long. Its leaves are trifoliate, rarely simple, with a long petiole [7]. It is a species used in traditional medicine against toothache, fever and parasites [5]. The literature reports few studies on the phytochemicals identified in the *Oricia* genus. A furoquinoline alkaloid, 6,7-methylenedioxy-5-hydroxy-8-ethoxy-dictamine, and a flavanone glycoside, 5-hydroxy-4'-methoxy-7-O-[α -l-rhamnopyranosyl (1'' $\rightarrow$ 5'')- β -d-apiofuranosyl]-flavanoside, were isolated from *O. suaveolens* and *O. renieri* [8]. The root of *O. suaveolens* contained mainly alkaloids including skimmianine, kokusaginine and monrifoline and triterpenes such as limonine [9], while the leaves and stem contained β -indoloquinazoline alkaloids; orisuaveoline A, orisuaveoline B and furoquinoline alkaloids; quinosuaveoline A, and quinosuaveoline B [10]. No study reports the chemical composition of the essential oil of *O. suaveolens*.

In the course of our on-going work on the characterization of the aromatic plants of Côte d'Ivoire through the chemical composition of their essential oils [11–14], the aim of the present work was to i) determine, for the first time, the chemical composition of the essential oil from *G. ehie* and *O. suaveolens*, growing wild in Côte d'Ivoire, and ii) highlight the presence of (*E*)- β -caryophyllene as a major component.

In recent years, many studies have demonstrated the interesting biological properties of (*E*)- β -caryophyllene: antinociceptive and anti-inflammatory [15]; anticancer [16,17]; antioxidant [16,18]; antimicrobial [16]; analgesic [17]; and antiatherogenic [18]. Essential oils rich in caryophyllene therefore present a particular interest. Previous studies have shown the occurrence of (*E*)- β -caryophyllene in Ivorian plant species [14,19–22].

2. Materials and Methods

2.1. Plant Material

Four leaves samples of *G. ehie* were harvested in Boua M'po, Rubino, Côte d'Ivoire (G1, G2) (6°04'09'' N, 4°18'31'' W) and in Adiopodoumé, Abidjan, Côte d'Ivoire (G3, G4) (5°20'12'' N et 4°7'57'' W). The plant was authenticated (voucher n° IBAAN REG 00704) in "Centre National de Floristique", Université Félix Houphouët-Boigny, Abidjan.

Three samples of leaves, four samples of stem bark and five samples of root bark of *O. suaveolens* were harvested in Petit Yapo Forest (5°43'50.5'' N, 4°4'41.9'' W), Agboville, Côte d'Ivoire. The organs were conditioned under permanent air conditioning for three days at 18 °C. The identification of the plant has been confirmed (voucher n° LAA 10096) by "Centre National de Floristique", Université Félix Houphouët-Boigny, Abidjan.

2.2. Essential Oil Isolation

Vegetable material was submitted to hydrodistillation during 3.5 h with a Clevenger-type apparatus. The essential oil samples obtained were dried over anhydrous Na₂SO₄ and then conserved at 5 °C. The extraction yields were calculated from fresh material (*w/w*) (Table S1).

2.3. Gas Chromatography and Gas Chromatography–Mass Spectrometry in Electron Impact Mode

GC analyses were performed on a Clarus 500 PerkinElmer Chromatograph (PerkinElmer, Courtaboeuf, France), equipped with a flame ionization detector (FID) and two fused-silica capillary columns (50 m $\times$ 0.22 mm, film thickness 0.25 μ m), BP-1 (polydimethylsiloxane) and BP-20 (polyethylene glycol). The oven temperature was programmed from 60 °C to 220 °C at 2 °C/min and then held isothermal at 220 °C for 20 min; injector temperature:

250 °C; detector temperature: 250 °C; carrier gas: hydrogen (0.8 mL/min); split: 1/60; injected volume: 0.5 µL. Retention indices (RI) were calculated relative to the retention times of a series of *n*-alkanes (C8–C29) with linear interpolation (« Target Compounds » software from PerkinElmer).

GC/MS analyses were performed on a Clarus SQ8S PerkinElmer TurboMass detector (quadrupole), directly coupled with a Clarus 580 PerkinElmer Autosystem XL (PerkinElmer, Courtaboeuf, France), equipped with a BP-1 (polydimethylsiloxane) fused-silica capillary column (50 m × 0.22 mm i.d., film thickness 0.25 µm). The oven temperature was programmed from 60 to 220 °C at 2°/min and then held isothermal for 20 min; injector temperature, 250 °C; ion-source temperature, 250 °C; carrier gas, Helium (1 mL/min); split ratio, 1:80; injection volume, 0.5 µL; ionization energy, 70 eV. The electron ionization (EI) mass spectra were acquired over the mass range 35–350 Da.

2.4. Nuclear Magnetic Resonance

All nuclear magnetic resonance (NMR) spectra were recorded on a Bruker AVANCE 400 Fourier transform spectrometer (Bruker, Wissembourg, France) operating at 100.623 MHz for ¹³C, equipped with a 5 mm probe. Solvent used was CDCl₃, with all shifts referred to internal tetramethylsilane (TMS). ¹³C NMR spectra of the oil samples were recorded with the following parameters: pulse width, 4 µs (flip angle 45°); relaxation delay D1, 0.1 s; acquisition time, 2.7 s for a 128K data table with a spectral width of 25,000 Hz (250 ppm); CPD mode decoupling; digital resolution, 0.183 Hz/pt. The number of accumulated scans was 3000 for each sample or fraction (30 mg, in 0.5 mL of CDCl₃).

2.5. Identification of Individual Components

Identification of the individual components was carried out as follows: (i) by comparison of their GC retention indices on apolar and polar columns, with those of reference compounds [23,24]; (ii) on computer matching against commercial mass spectral libraries [24–26]; (iii) on comparison of the signals in the ¹³C NMR spectra of the samples with those of reference spectra compiled in the laboratory spectral library, with the help of laboratory-made software [27–29]. This method allowed the identification of individual components of the essential oil at content as low as 0.4–0.5%.

3. Results and Discussion

Four essential oils samples of the leaves of *G. ehie* harvested in two locations in the south of Côte d'Ivoire (Boua M'po and Adiopodoumé), were obtained with low yield (0.01–0.05 %: *w/w*; calculated on weight basis) (Table S1)

Essential oils of the leaves (three samples), stem bark (four samples) and root bark (five samples) of *O. suaveolens* harvested in Petit Yapo Forest were obtained with low yields (Table S1) (0.05–0.08%, 0.01–0.03% and 0.01–0.02%: *w/w*, calculated on weight basis, respectively).

A combination of techniques was applied to the essential oil analysis: GC (associated with retention indices on two columns with different polarity), GC-MS and ¹³C NMR following a computerized ¹³C NMR method developed at the University of Corsica [11,29]. Most compounds have been identified through these three techniques.

3.1. Chemical Composition of Leaf Oils of *G. ehie*

Forty-two compounds were identified in the leaf oil of *G. ehie* representing 89.9 to 93.8% of the overall composition (Table 1). The chemical compositions of the four oils were dominated by sesquiterpene hydrocarbons; among them, (*E*)- β -caryophyllene (26.9–40.8%) was the major compound. Other compounds were present in appreciable amounts: β -elemene (5.5–9.5%), α -humulene (6.7–9.7%), germacrene D (5.6–8.1%) and β -elemol (1.6–4.2%). No monoterpene was identified and the total amount of non-terpene linear compounds (0.9–2.9%) and diterpenes (0.5–3.3%) was limited.

Table 1. Chemical composition of leaf oil samples of *Guibourtia ehie*.

N°	Compounds	RIa	RIp	G1	G2	G3	G4	Identification
1	Undecan-2-one	1274	1591	-	-	0.2	0.2	RI, MS
2	δ -Elemene	1335	1472	0.9	0.3	1.7	1.7	RI, MS, ¹³ C NMR
3	α -Cubebene	1348	1459	0.1	0.2	0.2	0.1	RI, MS
4	α -Ylangene	1368	1580	0.1	0.4	0.1	0.1	RI, MS
5	α -Copaene	1375	1493	1.5	2.7	2.5	1.9	RI, MS, ¹³ C NMR
6	β -Elemene	1387	1593	9.0	5.5	9.5	7.7	RI, MS, ¹³ C NMR
7	(<i>E</i>)- β -Caryophyllene	1417	1600	40.4	40.4	40.8	26.9	RI, MS, ¹³ C NMR
8	β -Copaene	1426	1580	0.1	0.2	0.3	1.8	RI, MS, ¹³ C NMR
9	γ -Elemene	1429	1639	-	0.1	0.1	0.2	RI, MS
10	(<i>E</i>)- α -Bergamotene	1431	1587	1.2	1.7	1.3	0.9	RI, MS, ¹³ C NMR
11	α -Guaiene	1434	1591	1.0	1.0	0.8	0.6	RI, MS, ¹³ C NMR
12	(<i>E</i>)- β -Farnesene	1446	1669	0.8	1.1	1.0	0.7	RI, MS, ¹³ C NMR
13	α -Humulene	1448	1669	9.1	9.7	9.4	6.7	RI, MS, ¹³ C NMR
14	<i>allo</i> -Aromadendrene	1457	1645	0.5	0.9	0.8	0.9	RI, MS, ¹³ C NMR
15	γ -Muurolene	1469	1689	0.5	0.7	0.7	0.8	RI, MS, ¹³ C NMR
16	Germacrene D	1474	1709	7.7	5.6	7.1	8.1	RI, MS, ¹³ C NMR
17	Tridecan-2-one	1476	1809	0.4	0.7	0.6	0.5	RI, MS, ¹³ C NMR
18	β -Selinene	1482	1715	1.1	0.8	1.2	1.2	RI, MS, ¹³ C NMR
19	α -Selinene	1492	1715	0.1	1.0	0.1	1.2	RI, MS, ¹³ C NMR
20	α -Burnesene	1499	1718	1.4	1.4	1.1	1.0	RI, MS, ¹³ C NMR
21	β -Bisabolene	1505	2190	0.1	0.2	0.2	-	RI, MS
22	γ -Cadinene	1508	1750	0.1	0.3	0.1	0.2	RI, MS
23	δ -Cadinene	1515	1557	1.2	1.2	1.4	2.1	RI, MS, ¹³ C NMR
24	Selina-3.7(11)-diene	1529	1791	0.1	0.2	0.2	0.1	RI, MS
25	β -Elemol	1533	2077	2.6	1.6	1.6	4.2	RI, MS, ¹³ C NMR
26	(<i>E</i>)-Nerolidol	1547	2040	0.5	0.4	0.3	0.8	RI, MS, ¹³ C NMR
27	Germacrene B	1549	1826	0.4	0.2	0.3	0.5	RI, MS
28	Spathulenol	1566	2119	0.5	0.4	0.2	0.2	RI, MS
29	Caryophyllene oxide	1569	1778	3.3	3.1	1.9	1.4	RI, MS, ¹³ C NMR
30	Globulol	1571	2061	0.1	0.2	0.2	0.3	RI, MS
31	Viridiflorol	1580	2079	0.5	1.8	0.8	2.5	RI, MS, ¹³ C NMR
32	Guaiol	1583	2086	0.5	0.3	0.3	0.9	RI, MS, ¹³ C NMR
33	Cedrol	1592	2124	1.3	1.4	1.1	1.3	RI, MS, ¹³ C NMR
34	Humulene oxide II	1598	2034	1.3	1.4	1.1	1.3	RI, MS, ¹³ C NMR
35	τ -Muurolol	1625	2183	1.1	0.7	0.9	2.0	RI, MS, ¹³ C NMR
36	α -Muurolol	1627	-	0.4	0.2	0.3	0.8	RI, MS, ¹³ C NMR
37	Cubenol	1630	2071	0.1	-	0.1	0.2	RI, MS
38	α -Cadinol	1635	2227	2.0	1.2	1.6	2.7	RI, MS, ¹³ C NMR
39	Nonadec-1-ene	1675	-	0.5	0.1	0.6	1.3	RI, MS
40	Heptadecanal	1892	2247	-	0.1	0.2	0.9	RI, MS, ¹³ C NMR
41	(<i>E</i>)-Phytol	2097	2608	0.8	0.4	0.8	2.9	RI, MS, ¹³ C NMR
42	<i>neo</i> -Abietadiene	2136	2534	0.1	0.1	0.1	0.4	RI, MS
Sesquiterpene hydrocarbons				77.0	75.6	80.6	64.9	
Oxygenated sesquiterpenes				14.2	12.7	10.4	18.6	
Diterpenes				0.9	0.5	0.9	3.3	
Non-terpenic compounds				0.9	0.9	1.6	2.9	
Total				93.4	89.9	93.8	90.2	

Order of elution and percentages on apolar column (BP-1); RIa, RIp: retention indices measured on apolar and polar capillary columns, respectively.

The chemical composition of the four oil samples was homogenous and no chemical variability was observed in relation to the location. However, sample G4 was slightly different and exhibited a low amount of (*E*)- β -caryophyllene (26.9%) in comparison to the G1-G3 samples whose content is higher than 40%. (*E*)- β -Caryophyllene, the main compound of the essential oils of the leaves of *G. ehie*, is also found as the main component

in various species belonging to the same family (Caesalpiniaceae): *Copaifera multijuga*, 36% [30]; 57.5% [31]; *Indigofera microcarpa*, 56% [32].

3.2. Chemical Composition of Leaf, Stem and Root Bark Oils of *O. suaveolens*

Analysis of the essential oils of the leaves, stem and root barks of *O. suaveolens* allowed the identification of 55, 40 and 23 compounds, accounting for 88.1–93.7%, 94.0–96.0% and 94.3–98.8% of the global composition, respectively (Tables 2–4). These essential oils consisted almost exclusively of sesquiterpene hydrocarbons exhibiting (*E*)- β -caryophyllene and α -humulene as major components. (*E*)- β -caryophyllene is predominant in the leaf essential oil and associated with α -humulene (α -caryophyllene) in the trunk and root bark oil samples. The percentage of (*E*)- β -caryophyllene accounted for 33.5–39.3% in the leaf oils (Table 2). (*E*)- β -Farnesene (5.9–9.3%), caryophyllene oxide (2.1–7.7%) and α -humulene (4.0–4.6%) were present in appreciable content. Non-terpene linear compounds (0.6–6.1%) and diterpenes (1.5–3.9%) were represented in non-negligible amounts while monoterpenes (0.6–1.8%) were weakly represented. Stem bark oils were characterized by close amounts of α -humulene (38.3–45.8%) and (*E*)- β -caryophyllene (34.7–41.6%) (Table 3). Two oxygenated sesquiterpenes, caryophyllene oxide (2.3–4.1%) and humulene oxide II (2.1–3.7%), were present in non-negligible amounts. Amounts of monoterpenes (0.1–2.0%), diterpenes (0–0.4%), aromatic derivatives (0–1.2%) and non-terpene linear compounds (0.1–0.6%) were low. The same association of α -humulene (36.1–47.9%)/(*E*)- β -caryophyllene (34.3–43.3%) was also evidenced from root bark oil (Table 4). A sesquiterpene hydrocarbon, geijerene (1.4–5.9%), and three oxygenated sesquiterpenes, caryophyllene oxide (0.6–4%), humulene oxide II (1.1–4.5%) and humulene oxide III (2.4–7.7%), were present in appreciable content while monoterpenes and non-terpene linear compounds were represented in much lower content, 0–1.7% and 0–3.8%, respectively. Whatever the organ of the plant, the chemical variability was very low.

Table 2. Chemical composition of leaf oil samples of *Oricia suaveolens*.

N°	Compounds	RIa	RIp	F1	F2	F3	Identification
1	(<i>E</i>)-Hex-2-enal	823	1225	2.4	-	-	RI, MS, ¹³ C NMR
2	(<i>Z</i>)-Hex-3-en-1-ol	838	1387	-	1.3	-	RI, MS, ¹³ C NMR
3	(<i>Z</i>)-Hex-2-en-1-ol	842	1408	2.7	1.4	-	RI, MS, ¹³ C NMR
4	Hexan-1-ol	845	1352	0.9	0.6	-	RI, MS, ¹³ C NMR
5	α -Pinene	929	1020	0.4	0.3	-	RI, MS
6	Myrcene	981	1165	-	0.1	0.1	RI, MS
7	(<i>E</i>)- β -Ocimene	1035	1254	0.2	0.1	0.1	RI, MS
8	Linalool	1083	1549	1.1	0.8	0.1	RI, MS, ¹³ C NMR
9	Undecan-2-one	1270	1592	-	0.1	0.1	RI, MS
10	α -Copaene	1375	1493	0.1	0.1	0.1	RI, MS
11	β -Bourbonene	1383	1520	0.1	0.2	0.1	RI, MS
12	α -Funebrene	1386	1538	3.3	3.8	2.5	RI, MS, ¹³ C NMR
13	α -Ionone	1400	1849	0.1	0.1	0.1	RI, MS
14	(<i>E</i>)- β -Caryophyllene	1417	1600	39.3	33.5	38.7	RI, MS, ¹³ C NMR
15	β -Copaene	1427	1587	-	0.2	0.1	RI, MS
16	Geranyl acetone	1429	1855	0.1	0.1	0.3	RI, MS
17	<i>trans</i> - α -Bergamotene	1431	1587	1.4	2.3	2.3	RI, MS, ¹³ C NMR
18	α -Sesquisabinene	1434	1645	3.4	3.5	1.9	RI, MS, ¹³ C NMR
19	(<i>E</i>)- β -Farnesene	1446	1669	9.3	8.8	5.9	RI, MS, ¹³ C NMR
20	α -Humulene	1448	1669	4.0	4.3	4.6	RI, MS, ¹³ C NMR
21	(<i>Z,Z</i>)- α -Farnesene	1461	1691	0.1	0.2	0.1	RI, MS
22	β -Ionone	1464	1963	-	0.2	0.2	RI, MS
23	α -Curcumene	1469	1770	0.2	0.9	0.7	RI, MS, ¹³ C NMR

Table 2. Cont.

N°	Compounds	RIa	RIp	F1	F2	F3	Identification
24	γ -Curcumene	1471	1691	0.8	0.5	0.2	RI, MS, ¹³ C NMR
25	Germacrene D	1474	1709	1.3	1.7	1.1	RI, MS, ¹³ C NMR
26	Tridecan-2-one	1481	1807	0.1	0.7	0.5	RI, MS, ¹³ C NMR
27	β -Selinene	1485	1715	0.1	0.2	0.2	RI, MS
28	Bicyclogermacrene	1489	1733	0.2	-	-	RI, MS
29	(Z,E)- α -Farnesene	1490	1718	-	0.3	0.3	RI, MS
30	(Z)- α -Bisabolene	1491	1729	0.7	1.0	0.6	RI, MS, ¹³ C NMR
31	(E,E)- α -Farnesene	1495	1751	2.4	4.6	3.8	RI, MS, ¹³ C NMR
32	β -Bisabolene	1499	1727	-	1.1	1.3	RI, MS, ¹³ C NMR
33	β -Curcumene	1500	1741	1.8	0.9	0.2	RI, MS, ¹³ C NMR
34	β -Sesquiphellandrene	1513	1770	2.6	2.7	1.9	RI, MS, ¹³ C NMR
35	(E)- γ -Bisabolene	1521	1757	0.2	0.5	0.4	RI, MS
36	trans-Sesquisabinene hydrate	1529	1992	1.2	1.1	0.8	RI, MS, ¹³ C NMR
37	β -Elemol	1532	2080	2.4	1.8	0.8	RI, MS, ¹³ C NMR
38	(E)-Nerolidol	1547	2043	1.1	1.2	0.7	RI, MS, ¹³ C NMR
39	cis-Sesquisabinene hydrate	1563	2086	1.8	1.1	1.6	RI, MS, ¹³ C NMR
40	Caryophyllene oxide	1567	1981	2.1	2.9	7.7	RI, MS, ¹³ C NMR
41	cis-7-epi-Sesquisabinene	1574	2100	0.5	0.5	0.4	RI, MS
42	Guaiol	1583	2089	0.7	-	-	RI, MS, ¹³ C NMR
43	Globulol	1583	2077	-	0.7	0.1	RI, MS, ¹³ C NMR
44	Humulene oxide II	1592	2037	0.2	0.6	1.3	RI, MS, ¹³ C NMR
45	Zingiberenol I	1598	2110	0.2	0.6	0.4	RI, MS, ¹³ C NMR
46	Zingiberenol II	1617	2194	0.3	0.4	0.5	RI, MS
47	γ -Eudesmol	1619	2164	-	0.3	-	RI, MS
48	β -Eudesmol	1633	2119	0.3	0.2	0.3	RI, MS
49	α -Eudesmol	1637	2124	0.3	0.3	0.2	RI, MS
50	Bulnesol	1651	2206	0.2	0.1	-	RI, MS
51	β -Bisabolol	1652	2151	0.5	0.5	0.8	RI, MS, ¹³ C NMR
52	α -Bisabolol	1675	2212	0.1	0.1	0.1	RI, MS
53	iso-Phytol	1940	-	-	1.1	3.2	RI, MS
54	(Z)-Phytol	2095	2522	2.5	-	-	RI, MS, ¹³ C NMR
55	(E)-Phytol	2097	2608	-	0.4	0.7	RI, MS, ¹³ C NMR
Monoterpene hydrocarbons				0.6	0.5	0.2	
Oxygenated monoterpenes				1.2	0.9	0.4	
Sesquiterpene hydrocarbon				71.4	71.6	67.3	
Oxygenated sesquiterpenes				11.9	12.4	15.7	
Diterpenes				2.5	1.5	3.9	
Non-terpenic compounds				6.1	4.1	0.6	

Order of elution and percentages on apolar column (BP-1); RIa, RIp: retention indices measured on apolar and polar capillary columns, respectively.

(E)- β -Caryophyllene, a major component of *O. suaveolens* oils, was also found as a major component in the oils of species belonging to the same family, such as *O. suaveolens* (Rutaceae); *Murraya paniculata*, 57.57% [33]; *Acronychia pedunculata*, 57.63%; *Clausena excavata*, 55.41% [34]; *Bouchardatia neurococca*, 38.5% [35]; and *Melicope peninsularis*, 49% [36], as well as in the Ivorian species *Polyalthia oliveri* [19].

Table 3. Chemical composition of stem bark oil samples of *Oricia suaveolens*.

N°	Compounds	RIa	RIp	T1	T2	T3	T4	Identification
1	α -Pinene	931	1020	0.1	0.1	-	0.1	RI, MS
2	Sabinene	966	1127	0.1	0.3	0.2	0.3	RI, MS
3	Myrcene	981	1166	-	0.3	0.1	0.1	RI, MS
4	<i>p</i> -Cymene	1012	1276	-	0.1	-	0.1	RI, MS
5	Limonene	1022	1205	-	0.1	0.1	0.1	RI, MS
6	Nonanal	1083	1397	0.1	0.3	0.1	0.1	RI, MS
7	1,3-Dimethoxybenzene	1137	-	-	0.2	0.1	-	RI, MS
8	Decanal	1185	1501	-	0.1	-	0.1	RI, MS
9	Neral	1216	1682	-	0.4	0.2	0.3	RI, MS
10	Geraniol	1235	1848	-	0.1	-	0.1	RI, MS
11	Geranial	1244	1733	-	0.7	0.5	0.6	RI, MS, ¹³ C NMR
12	α -Copaene	1374	1492	0.2	0.2	0.1	0.8	RI, MS, ¹³ C NMR
13	β -Elemene	1386	1592	0.2	0.7	0.3	1.1	RI, MS, ¹³ C NMR
14	<i>cis</i> - α -Bergamotene	1410	1569	0.9	2.1	0.4	1.2	RI, MS, ¹³ C NMR
15	(<i>E</i>)- β -Caryophyllene	1417	1600	41.6	34.7	39.8	35.2	RI, MS, ¹³ C NMR
16	α -guaiaene	1435	1592	-	0.4	0.1	1.6	RI, MS, ¹³ C NMR
17	α -Humulene	1448	1669	45.8	38.5	44.1	38.3	RI, MS, ¹³ C NMR
18	γ -Muurolene	1471	1689	0.1	0.3	0.1	0.7	RI, MS, ¹³ C NMR
19	Germacrene D	1475	1708	0.1	0.1	0.1	0.3	RI, MS
20	β -Selinene	1490	1718	-	0.1	-	0.1	RI, MS
21	Valencene	1492	1724	0.2	0.2	0.1	0.7	RI, MS, ¹³ C NMR
22	(<i>E,E</i>)- α -Farnesene	1495	1750	0.6	0.1	-	1.9	RI, MS, ¹³ C NMR
23	α -Bulnesene	1498	1715	-	0.5	-	0.2	RI, MS
24	α -Muurolene	1501	1718	-	0.3	0.3	0.2	RI, MS
25	γ -Cadinene	1505	1752	-	0.1	-	0.3	RI, MS
26	δ -Cadinene	1513	1758	0.1	0.2	0.1	0.4	RI, MS
27	(<i>E</i>)- γ -Bisabolene	1522	1757	0.6	0.1	-	-	RI, MS, ¹³ C NMR
28	β -Elemol	1533	2077	-	1.4	-	1.6	RI, MS, ¹³ C NMR
29	(<i>E</i>)-Nerolidol	1547	2040	-	0.1	-	0.1	RI, MS
30	Germacrene B	1550	1827	0.1	0.2	-	0.1	RI, MS
31	Caryophyllene oxide	1567	1981	2.3	4.0	4.1	3.2	RI, MS, ¹³ C NMR
32	Humulene oxide I *	1583	2011	0.2	0.4	0.1	0.2	RI, MS
33	Guaiaol *	1583	2086	-	0.4	0.2	0.6	RI, MS, ¹³ C NMR
34	Humulene oxide II	1592	2037	2.1	3.7	3.5	3.2	RI, MS, ¹³ C NMR
35	Humulene oxide III	1615	2048	0.3	0.3	0.8	0.4	RI, MS
36	β -Eudesmol	1633	2228	0.1	0.2	0.1	0.2	RI, MS
37	α -Cadinol	1639	2224	0.1	0.3	-	0.1	RI, MS
38	α -Eudesmol	1652	2215	0.1	0.4	0.1	0.4	RI, MS
39	Benzyl benzoate	1722	2620	-	1.0	-	-	RI, MS, ¹³ C NMR
40	(<i>E</i>)-Phytol	2097	2608	0.1	0.4	-	0.3	RI, MS
Monoterpene hydrocarbons				0.1	0.8	0.4	0.6	
Oxygenated monoterpenes				0	1.2	0.7	1.0	
Sesquiterpene hydrocarbons				89.9	78.7	85.5	83.1	
Oxygenated sesquiterpenes				5.2	11.2	8.9	10.0	
Diterpenes				0.1	0.4	0	0.3	
Non-terpenic compounds				0.1	0.6	0.1	0.2	
Aromatic derivatives				0	1.2	0.1	0	
Total				96.0	94.0	95.7	95.2	

Order of elution and percentages on apolar column (BP-1), except components with an asterisk (*), where percentages are taken on a polar column (BP-20). RIa, RIp: retention indices measured on apolar and polar capillary columns, respectively.

Table 4. Chemical composition of root bark oil samples of *Oricia suaveolens*.

N°	Compounds	RIa	RIp	R1	R2	R3	R4	R5	Identification
1	α -Pinene	931	1020	-	0.7	0.1	0.1	-	RI, MS, ¹³ C NMR
2	Sabinene	966	1127	-	-	-	0.1	-	RI, MS
3	Geijerene	1135	1328	1.7	2.0	1.4	5.9	1.4	RI, MS
4	Geranial	1244	1733	-	0.1	0.2	0.4	0.1	RI, MS
5	Undecan-2-one	1274	1592	-	0.8	-	-	-	RI, MS, ¹³ C NMR
6	Pregeijerene	1277	1316	0.1	0.1	0.2	1.0	0.1	RI, MS
7	Myrtenyl acetate	1306	1689	-	0.9	-	-	0.9	RI, MS, ¹³ C NMR
8	α -Ylangene	1367	1482	0.2	-	-	-	-	RI, MS
9	Cyclosativene	1368	1483	-	0.4	0.3	1.0	0.2	RI, MS, ¹³ C NMR
10	α -Copaene	1375	1493	-	0.2	0.1	0.3	0.2	RI, MS
11	β -Elemene	1387	1593	-	0.1	0.1	0.4	0.1	RI, MS
12	(E)- β -Caryophyllene	1417	1600	43.3	35.3	40.5	34.3	34.8	RI, MS, ¹³ C NMR
13	α -Humulene	1448	1669	47.9	41.5	45.0	36.1	40.4	RI, MS, ¹³ C NMR
14	γ -Muuroolene	1469	1689	-	0.2	0.2	0.4	0.1	RI, MS
15	Tridecan-2-one	1476	1809	-	3.0	-	-	-	RI, MS, ¹³ C NMR
16	(E,E)- α -Farnesene	1500	1750	0.5	0.4	0.5	0.4	0.4	RI, MS
17	β -Elemol	1533	2077	-	-	0.3	-	0.7	RI, MS, ¹³ C NMR
18	Caryophyllene oxide	1567	1981	0.6	2.6	2.0	2.1	4.0	RI, MS, ¹³ C NMR
19	Humulene oxide I	1583	2013	0.3	0.6	0.6	0.7	0.9	RI, MS, ¹³ C NMR
20	Humulene oxide II	1592	2037	1.1	3.3	2.3	2.8	4.5	RI, MS, ¹³ C NMR
21	Humulene oxide III	1613	2051	2.9	4.6	2.4	7.7	6.8	RI, MS
22	τ -Muurolol	1625	2183	-	0.2	0.2	0.3	0.3	RI, MS
23	α -Cadinol	1635	2227	0.2	0.2	0.2	0.3	0.3	RI, MS
Monoterpene hydrocarbons				0	0.7	0.1	0.2	0	
Oxygenated monoterpenes				0	1.0	0.2	0.4	1.0	
Sesquiterpene hydrocarbons				93.7	80.2	88.3	79.8	77.7	
Oxygenated sesquiterpenes				5.1	11.5	8.0	13.9	17.5	
Non-terpenic compounds				0	3.8	0	0	0	
Total				98.8	97.2	96.6	94.3	96.2	

Order of elution and percentages on apolar column (BP-1); RIa, RIp: retention indices measured on apolar and polar capillary columns, respectively.

4. Conclusions

The leaf essential oil of *Guibourtia ehie* and leaf, stem bark and root bark essential oils of *Oricia suaveolens* were analyzed by GC(RI), GC-MS and ¹³C NMR. All of these essential oils were dominated by hydrocarbon sesquiterpenes. The stem and root bark oils of *Oricia suaveolens* were characterized by the couple α -humulene (α -caryophyllene)T(E)- β -caryophyllene, while the leaf oils of *Guibourtia ehie* and *Oricia suaveolens* were dominated by (E)- β -caryophyllene. The essential oils of these two plant species can therefore be considered as natural sources of caryophyllenes.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/compounds3010006/s1>, Table S1: Yields of hydrodistillation.

Author Contributions: Conceptualization, T.A.K., J.A.M.-B., Y.-A.B. and Z.A.O.; methodology, T.A.K., B.A.K. and Z.A.O.; software, Z.A.O., P.T., A.B. and M.P.; validation, A.B., J.A.M.-B., Y.-A.B. and F.T.; formal analysis, T.A.K., D.A.K. and Z.A.O.; essential oil investigation, T.A.K., D.A.K., B.A.K. and Z.A.O.; writing—original draft preparation, T.A.K., Z.A.O., A.B. and F.T.; writing—review and editing, T.A.K., Z.A.O., A.B. and F.T.; visualization, D.A.K., P.T. and M.P.; supervision, F.T. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Data Availability Statement: Data and samples of the essential oils are available from the authors.

Acknowledgments: The authors gratefully acknowledge the University of Corsica for accepting Z.A. Ouattara at “Laboratoire Sciences Pour l’Environnement” for an advanced training course. We acknowledge H. Téhé and Y. Konan for their valuable help in the plant identification.

Conflicts of Interest: The authors declare no conflict of interest.

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