Revista Brasileira de Farmacognosia 27 (2017) 414–418

Brazilian Journal of Pharmacognosy
REVISTA BRASILEIRA DE FARMACOGNOSIA
www.elsevier.com/locate/bjp

Original Article

# Chemical composition of the leaf oil of *Artabotrys jollyanus* from Côte d'Ivoire

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ARTICLE INFO

*Article history:*
Received 29 September 2016
Accepted 3 April 2017
Available online 2 May 2017

*Keywords:*
7-Hydroxycalamenene
Essential oil
Côte d'Ivoire
$^{13}$C NMR
Sesquiterpenes

ABSTRACT

One oil sample isolated from leaves of *Artabotrys jollyanus* Pierre, Annonaceae, from Côte d'Ivoire has been analyzed by GC(RI), GC-MS and $^{13}$C NMR. In total, thirty-seven compounds accounting for 96.9% of the relative composition have been identified. The composition of the essential oil was dominated by *trans*-calamenene (15.7%), α-copaene (14.8%), α-cubebene (10.4%), cadina-3,5-diene (10.3%), (*E*)-β-caryophyllene (6.3%) and cadina-1,4-diene (6.1%). $^{13}$C NMR spectroscopy was very useful in the identification of *trans*-calamenene, 7-hydroxycalamenene, cadina-3,5-diene and cadina-1,4-diene. Moreover, monitoring the evolution of the leaf essential oil composition and the yield on a 12-month period (one sample per month) was achieved. The twelve essential oil samples exhibited a chemical homogeneity but the yield varied from sample to sample (0.26–0.60%).



## Introduction

Generally distributed in tropical and subtropical regions, mainly in tropical Africa and Eastern Asia, the genus *Artabotrys*, family Annonaceae, contains more than 100 species (Sagen et al., 2003). In Côte d'Ivoire, five species grow wild in dense rain forests, namely *Artabotrys hispidus* Sprague & Hutch., *A. insignis* Engler & Diels, *A. jollyanus* Pierre, *A. oliganthus* Engler & Diels and *A. velutinus* Sc. Elliot. They are either climbing and evergreen shrubs or woody lianas.

*Artabotrys jollyanus* is a climbing shrub with persistent elliptic oblong leaves, 15–21 cm in length, 7–9 cm width. The limb base is rounded to cuneate and its apex acuminate. Flower petals are elliptical (25 mm in length), grouped in dense axillary inflorescences (http://www.plantes-botanique.org).

Ethnomedicinal uses of species of the genus *Artabotrys* have been reviewed (Tan and Wiart, 2014). Numerous papers reported on the phytochemistry of this genus and highlighted the presence of alkaloids in *A. uncinatus* (Lam.) Merr. (Hsieh et al., 1999), *A. crassifolius* Hook. f. & Thomson (Tan et al., 2015), *A. hexapetalus* (L. f.) Bhandare (Zhou et al., 2015), *A. odoratissimus* R.Br. (Kabir, 2010), or polyphenols in *A. hildebrandtii* O. Hffm. (Andriamadioa et al., 2015), *A. hexapetalus* (Li et al., 1997; Somanawat et al., 2012). Concerning the chemical composition of essential oils more than fifteen species have been investigated (Hung et al., 2014) and sesquiterpenes were often the major components (Menut et al., 1992; Fournier et al., 1999; Thang et al., 2014). To our knowledge no investigations have been undertaken to date on the chemical composition of *A. jollyanus* essential oil.

In continuation of our on-going work related to the characterization of aromatic and medicinal Annonaceae from Côte d'Ivoire (Yapi et al., 2012, 2013, 2014; Ouattara et al., 2011; Ouattara et al., 2013, 2014) the chemical composition of the essential oil isolated from leaves of *A. jollyanus* has been investigated by combination of chromatographic [GC-FID, GC(RI)] and spectroscopic techniques (MS, $^{13}$C NMR). Firstly, we report the detailed leaf essential oil composition of a selected sample. Secondly, the temporal variation of leaf oil composition was studied by analyzing eleven other leaf samples collected along the vegetative cycle.

## Materials and methods

### *Plant material*

Leaves from *Artabotrys jollyanus* Pierre, Annonaceae, have been harvested (April 2014 - March 2015) in the Adiopodoumé forest

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http://dx.doi.org/10.1016/j.bjp.2017.04.001


**Fig. 1.** Locality of harvest of leaves of *A. jollyanus* from Côte d'Ivoire.

on Abidjan-Dabou axis (southern Côte d'Ivoire, 5°19′50.074″ N, 4°7′41.109″ O, Fig. 1). The plant species was identified by M. Assi Jean, technician at the Herbarium of the Centre National of Floristique (Félix Houphouët-Boigny University, Abidjan-Cocody/Côte d'Ivoire) where voucher specimen was deposited with number LAA 7650.

*Essential oil isolation*

Essential oil (EO) samples were obtained by hydrodistillation from the fresh leaves (300 g) with a Clevenger-type apparatus for a period of 3.5 h; the essential oils (S1-S12) were dried over anhydrous sodium sulphate ($Na_2SO_4$), and then stored in a freezer until analysis. The yields, calculated on the fresh weight basis (w/w), were comprised between 0.26% and 0.60%. All oil samples were light yellow coloured.

*Gas chromatography (GC) analyses*

Analyses were performed on a Clarus 500 PerkinElmer (PerkinElmer, Courtaboeuf, France) system equipped with a FID and two fused-silica capillary columns (50 m × 0.22 mm, film thickness 0.25 μm), 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: helium (0.8 ml/min); split: 1/60; injected volume: 0.5 μl. The relative proportions of the oil constituents were expressed as percentages obtained by peak-area normalization, without using correction factors. Retention indices (RI) were determined relative to the retention times of a series of *n*-alkanes (C7-C28) with linear interpolation (Target Compounds software from Perkin Elmer).

*Gas chromatography–mass spectroscopy (GC/MS) analyses*

The essential oils were analyzed with a Perkin-Elmer TurboMass detector (quadrupole), directly coupled to a Perkin-Elmer Autosystem equipped with a fused-silica capillary column (50 m × 0.22 mm i.d., film thickness 0.25 μm), BP-1 (dimethylpolysiloxane). Carrier gas, helium at 0.8 ml/min; split, 1/60; injection volume, 0.5 μl; injector temperature, 250 °C; oven temperature programmed from 60 °C to 220 °C at 2 °C/min and then held isothermal (20 min); Ion source temperature, 250 °C; energy ionization, 70 eV; electron ionization mass spectra were acquired over the mass range 40–400 Da.

*$^{13}C$ NMR analyses*

$^{13}C$ NMR analysis were performed on a Bruker AVANCE 400 Fourier Transform spectrometer operating at 100.63 MHz for $^{13}C$, equipped with a 5 mm probe, in deuterated chloroform ($CDCl_3$), with all shifts referred to internal tetramethylsilane (TMS). $^{13}C$ NMR spectra were recorded with the following parameters: pulse width (PW), 5 μs (flip angle 45°); acquisition time, 2.7 s for 128 K data table with a spectral width (SW) of 25,000 Hz (250 ppm); digital resolution 0.183 Hz/pt. The number of accumulated scans was 2500 for each sample (about 50 mg of essential oil in 0.5 ml of $CDCl_3$).

*Identification of individual components*

Component identification was based on: (a) comparison of their GC retention indices (RI) on polar and apolar columns determined relative to the retention times of a series of *n*-alkanes with linear interpolation with those of authentic compounds or literature data; (b) on computer matching with laboratory-made and commercial mass spectral libraries (König et al., 2001; Adams, 2007; US National Institute of Standards and Technology, 1999); and (c) on comparison of the signals in the $^{13}C$ NMR spectra of essential oils with those of reference spectra compiled in the laboratory spectral library with the help of laboratory-developed software (Ouattara et al., 2014). Indeed the $^{13}C$ NMR spectrum of a molecule may be considered as its fingerprint. In other words, two compounds, such as sesquiterpenes, exhibit always enough chemical shift values of their carbons sufficiently differentiated to allow their identification. Therefore, taking into account various parameters (the number of observed signals, the number of overlapped signals, the difference of chemical shift measured in the mixture and in the reference spectra) the identification of an individual component of a complex mixture is possible without individualization of the compound (Bighelli and Casanova, 2010).

## Results and discussion

Detailed analysis of a leaf oil sample (S4) from *A. jollyanus* has been carried out by GC(RI), GC–MS and $^{13}C$ NMR. In total, 37 compounds (12 monoterpenes, 5.2% and 25 sesquiterpenes 91.7%) that accounted for 96.9% of the whole composition, have been identified in the sample (Table 1, Fig. 2). The major components were sesquiterpenes, particularly sesquiterpene hydrocarbons. It could be highlighted that the five main components accounted only for 10–16% each: *trans*-calamenene (15.7%), α-copaene (14.8%), α-cubebene (10.4%), cadina-3,5-diene (10.3%) and 7-hydroxycalamenene (10.1%). Other sesquiterpenes present at significant levels were (*E*)-β-caryophyllene (6.3%), cadina-1,4-diene (6.1%), β-cubebene (3.1%), α-humulene (3.0%), δ-cadinene (1.9%) bicyclosesquiphellandrene (1.8%), bicyclogermacrene (1.5%) and spathulenol (1.1%). Finally, the monoterpene fraction was mostly represented by (*Z*)-β-ocimene (3.6%). The other hydrocarbon monoterpenes were present at very low levels not exceeding 0.5%.

Identification of some components needed special attention:

- Calamenene stereoisomers (*cis* or *trans*) display overlapped peaks on apolar and polar capillary chromatography columns (RIa: 1509; RIp: 1829) and insufficiently differentiated mass spectra (Joulain and König, 1998). Therefore, identification of the correct isomer was achieved by $^{13}C$ NMR analysis, the spectra of both compounds being fully differentiated (Nakashima et al., 2002).
- Cadina-1,4-diene (6.1%) and cadina-3,5-diene (10.3%) were suggested by MS and then confirmed by comparison of their $^{13}C$ NMR

**Table 1**
Chemical composition of *Artabotrys jollyanus* leaf oil.

| | Components[a] | RI[lit] [b] | RIa | RIp | *A. jollyanus* | Identification |
|---|---|---|---|---|---|---|
| 1 | α-Pinene | 936 | 929 | 1015 | 0.3 | RI, MS |
| 2 | Camphene | 950 | 942 | 1065 | tr | RI, MS |
| 3 | Sabinene | 973 | 964 | 1123 | tr | RI, MS |
| 4 | β-Pinene | 978 | 969 | 1112 | tr | RI, MS |
| 5 | Myrcene | 987 | 979 | 1160 | 0.2 | RI, MS |
| 6 | Limonene | 1025 | 1019 | 1201 | 0.2 | RI, MS |
| 7 | β-Phellandrene | 1023 | 1019 | 1211 | 0.4 | RI, MS |
| 8 | (*Z*)-β-Ocimene | 1029 | 1024 | 1233 | 3.6 | RI, MS, $^{13}$C NMR |
| 9 | (*E*)-β-Ocimene | 1041 | 1035 | 1250 | 0.4 | RI, MS, $^{13}$C NMR |
| 10 | Terpinolene | 1082 | 1077 | 1283 | tr | RI, MS |
| 11 | Linalool | 1086 | 1082 | 1545 | tr | RI, MS |
| 12 | *allo*-Ocimene | 1113 | 1116 | 1372 | 0.1 | RI, MS |
| 13 | α-Cubebene | 1355 | 1348 | 1456 | 10.4 | RI, MS, $^{13}$C NMR |
| 14 | α-Copaene | 1379 | 1375 | 1490 | 14.8 | RI, MS, $^{13}$C NMR |
| 15 | β-cubebene* | 1390 | 1385 | 1535 | 3.1 | RI, MS |
| 16 | β-Elemene* | 1389 | 1385 | 1587 | 0.5 | RI, MS, $^{13}$C NMR |
| 17 | α-Gurjunene | 1413 | 1408 | 1525 | 0.2 | RI, MS |
| 18 | (*E*)-β-Caryophyllene | 1421 | 1416 | 1593 | 6.3 | RI, MS, $^{13}$C NMR |
| 19 | Cadina-3,5-diene | 1448 | 1444 | 1627 | 10.3 | RI, MS, $^{13}$C NMR |
| 20 | α-Humulene | 1455 | 1448 | 1665 | 3.0 | RI, MS, $^{13}$C NMR |
| 21 | *allo*-Aromadendrene | 1462 | 1456 | 1640 | 0.3 | RI, MS |
| 22 | Cadina-1(6),11-diene | - | 1466 | 1655 | 0.8 | RI, MS, $^{13}$C NMR |
| 23 | Germacrene D | 1479 | 1474 | 1704 | 0.4 | RI, MS, $^{13}$C NMR |
| 24 | Bicyclosesquiphellandrene | 1487 | 1483 | 1708 | 1.8 | RI, MS, $^{13}$C NMR |
| 25 | 4-*epi*-Cubebol | 1490 | 1485 | 1883 | 0.8 | RI, MS, $^{13}$C NMR |
| 26 | Bicyclogermacrene | 1494 | 1489 | 1728 | 1.5 | RI, MS, $^{13}$C NMR |
| 27 | α-Muurolene | 1496 | 1491 | 1719 | 0.2 | RI, MS |
| 28 | Cubebol | 1514 | 1504 | 1935 | 0.8 | RI, MS, $^{13}$C NMR |
| 29 | *trans*-Calamenene | 1517 | 1509 | 1829 | 15.7 | RI, MS, $^{13}$C NMR |
| 30 | δ-Cadinene | 1520 | 1513 | 1751 | 1.9 | RI, MS, $^{13}$C NMR |
| 31 | Zonarene | 1521 | 1515 | 1752 | 0.4 | RI, MS |
| 32 | Cadina-1,4-diene | 1523 | 1523 | 1777 | 6.1 | RI, MS, $^{13}$C NMR |
| 33 | Spathulenol | 1572 | 1561 | 2117 | 1.1 | RI, MS, $^{13}$C NMR |
| 34 | Caryophyllene oxide | 1578 | 1567 | 1976 | 0.2 | RI, MS |
| 35 | *epi*-Cubenol | 1623 | 1612 | 2058 | 0.4 | RI, MS |
| 36 | Cubenol | 1630 | 1628 | 2051 | 0.7 | RI, MS |
| 37 | 7-Hydroxycalamenene | 1803[c] | 1775 | 2783 | 10.1 | RI, MS, $^{13}$C NMR |
| | Total | | | | 96.9 | |
| | Monoterpene hydrocarbons | | | | 5.2 | |
| | Oxygenated monoterpenes | | | | tr | |
| | Sesquiterpenes hydrocarbons | | | | 77.7 | |
| | Oxygenated sesquiterpenes | | | | 14.0 | |

[a] Order of elution and contents determined on the apolar column (BP-1), except for compounds with an asterisk, percentages on polar column (BP 20). RIa, RIp = retention indices measured on apolar (BP1) and polar (BP 20) column; tr = trace level (<0.05%).
[b] Literature indices on a DB-1 (http://massfinder.com by Hochmuth, 2017).
[c] Indice on a HP-5 column (Azevedo et al., 2014).

chemical shifts with those reported in the literature (Joulain and König, 1998);
- 7-Hydroxycalamenene (10.1%). The MS spectrum of this compound was not compiled in MS libraries at our disposal and in our home-made MS library. The compound was identified by comparison of its $^{13}$C NMR chemical shifts with those reported in the literature (Cambie et al., 1990).

The chemical composition of *A. jollyanus* leaf oil differed from that of Ivoirian *A. oliganthus* leaf oil, dominated by monoterpenes (δ-3-carene, 60.2%, myrcene, 10.6%) (Ouattara et al., 2011). It differed from those of bark oils from Gabonese *Artabotrys lastourvillensis* Pell (cyperene, 25.9%; cyperenone, 11.1%) (Menut et al., 1992) and from Beninese *Artabotrys velutinus* (benzyl benzoate, 61.2%; (*E*)-β-caryophyllene, 9.1%) (Yovo et al., 2016). It differed also from those of EOs isolated from *Artabotrys* species grown in other areas of the world. For instance, the compositions of Vietnamese species were mostly dominated by various sesquiterpenes: *A. petelotti* Merr. leaf oil (elemol, 19.4%, *cis*-β-guaiene, 9.2%, δ-cadinene, 8.4%); *A. intermedius* Hassk. leaf oil (δ-3-carene, 19.1%; α-gurjunene; 10.7%; α-zingiberene, 6.3%); *A. harmandii* Finet & Gagnep. (spathulenol, 17.4%; aromadendrene epoxide, 12.2%; γ-elemene, 7.1%; β-elemene, 5.0%; bicyclogermacrene, 5.0%) (Hung et al., 2014); *A. taynguyenensis* Ban leaf oil (valencene, 40.1%; δ-selinene, 8.8%; α-pinene, 6.7%; α-muurolene, 5.1%; α-panasinsene, 5.1%) (Thang et al., 2014). The flower oil of Vietnamese *A. hexapetalus* contained mainly caryophyllene oxide, 31.5%; β-caryophyllene, 11.4%; humulene epoxide, 10.0% and α-copaene, 8.1% (Phan et al., 2007).

The monitoring of the temporal evolution of the yield and the chemical composition of the leaf EO from *A. jollyanus* has been achieved over a period of 12 months, from April 2014 to March 2015 (Table 2). Yields varied substantially from 0.26% to 0.60% (mean value = 0.40%). Three domains may be distinguished. Higher yield has been observed in September (0.60%), during the dry period. Acceptable yields (0.39–0.52%, 7 oil samples out of 12) occurred in the period June-November and lowest yields (0.26–0.32%, 4 samples out of 12) have been obtained in May on the one hand and during the period December-March on the other hand.

The composition of the EO was always dominated by sesquiterpene hydrocarbons although the contents of the main components varied also substantially. For instance, *trans*-calamenene (mean value = 18.1%) accounted for 15.7–17.7% for 9 oil samples out of 12 (14.7%, 22.4% and 27.6% for the last three samples). In parallel,

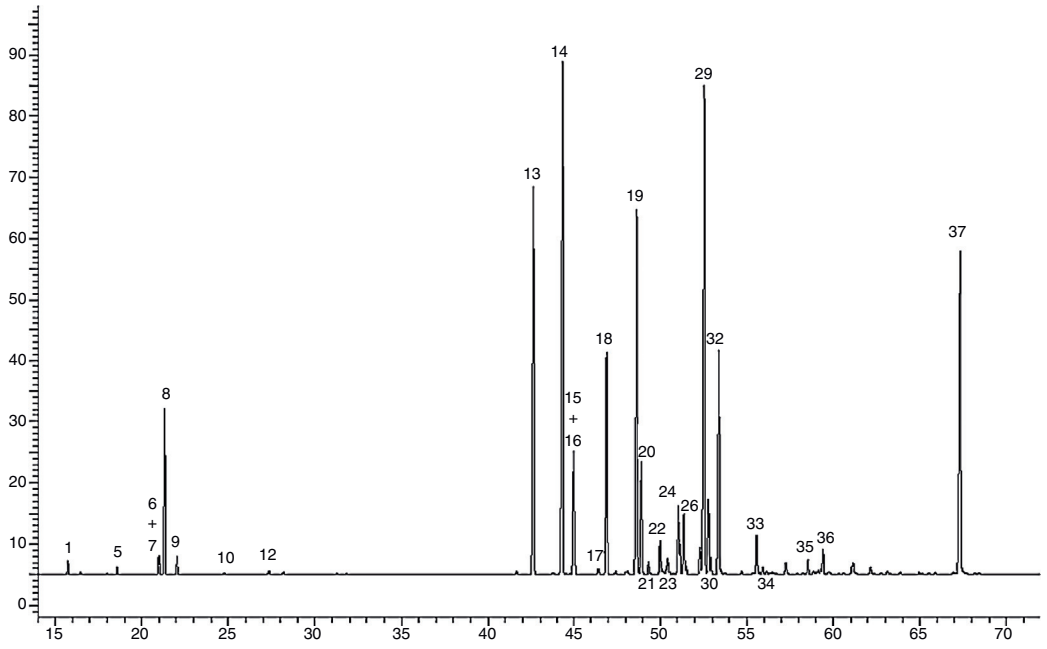


**Fig. 2.** Gas chromatogram on apolar column (BP-1) of leaf sample of *Artabotrys jollyanus* essential oil.

**Table 2**
Chemical composition of *Artabotrys jollyanus* leaf oil: main components of twelve samples.

| Sample<br>Month | S1<br>Apr | S2<br>May | S3<br>Jun | S4[a]<br>Jul | S5<br>Aug | S6<br>Sept | S7<br>Oct | S8<br>Nov | S9<br>Dec | S10<br>Jan | S11<br>Feb | S12<br>Mar |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| *Components* | | | | | | | | | | | | |
| (*Z*)-β-Ocimene | 5.5 | 5.3 | 5.3 | 3.6 | 4.7 | 3.4 | 4.6 | 3.4 | 5.5 | 3.5 | 4.6 | 2.4 |
| α-Cubebene | 10.7 | 9.2 | 10.6 | 10.4 | 10.7 | 11.0 | 8.8 | 9.7 | 14.5 | 12.9 | 8.8 | 10.2 |
| α-Copaene | 14.7 | 12.4 | 14.8 | 14.8 | 15.7 | 16.4 | 12.7 | 14.3 | 20.4 | 18.8 | 12.3 | 14.6 |
| β-Cubebene | 2.7 | 2.1 | 3.2 | 3.1 | 3.1 | 3.2 | 2.7 | 3.1 | 2.5 | 1.4 | 3.2 | 2.3 |
| (*E*)-β-Caryophyllene | 8.0 | 7.9 | 7.1 | 6.3 | 6.6 | 6.6 | 6.8 | 6.7 | 7.7 | 7.1 | 6.5 | 5.7 |
| Cadina-3,5-diene | 7.3 | 4.6 | 9.2 | 10.3 | 10.2 | 10.6 | 9.3 | 10.8 | 7.6 | 8.0 | 11.0 | 0.4 |
| α-Humulene | 3.5 | 3.6 | 3.2 | 3.0 | 3.1 | 3.0 | 3.4 | 3.2 | 3.2 | 3.1 | 3.3 | 2.9 |
| *trans*-Calamenene | 18.6 | 22.4 | 16.4 | 15.7 | 16.5 | 16.3 | 16.6 | 16.4 | 17.8 | 18.7 | 14.7 | 27.6 |
| Cadina-1,4-diene | 6.0 | 5.2 | 6.0 | 6.1 | 5.8 | 5.8 | 5.6 | 6.1 | 4.5 | 6.0 | 6.4 | 2.9 |
| Spathulenol | 1.4 | 1.9 | 1.1 | 1.1 | 1.0 | 0.9 | 1.3 | 1.1 | 1.0 | 0.8 | 0.8 | 2.4 |
| 7-Hydroxycalamenene | 4.8 | 6.5 | 7.6 | 10.1 | 7.0 | 7.9 | 10.4 | 9.1 | 1.3 | 4.9 | 10.5 | 10.4 |
| Yield | 0.39 | 0.26 | 0.48 | 0.40 | 0.42 | 0.60 | 0.45 | 0.52 | 0.28 | 0.32 | 0.39 | 0.32 |

[a] S4 is previously described in Table 1

α-copaene (mean value = 15.2%) displayed more diverse contents (12.3–12.7%, 3 samples; 14.3–14.8%, 5 samples; 15.7–20.4%, 4 samples). The content of 7-hydroxycalamenene (mean value = 7.5%) seemed to vary drastically from 1.3% to 10.5%. In fact, the contents of four samples (6.5–7.9%) were very close to the mean value, five samples displayed higher contents (9.1–10.5%), two samples exhibited lower contents (4.8 and 4.9%) and the last sample accounted for 1.3% only. Finally, the content of cadina-3,5-diene varied from 7.3% to 11.0% for ten samples, while it was only 4.6% for one sample and 0.4% for the last one. Similarly, the content of cadina-1,4-diene varied from 5.2% to 6.4% for ten samples, while it was only 4.5% and 2.9% for the two last ones.

It is noticeable that the sum of percentages of cadina-1,4-diene, cadina-3,5-diene, *trans*-calamenene and 7-hydroxy-calamenene were constant in all samples and close to 39%. Indeed, *trans*-calamenene may be obtained by deshydrogenation of cadina-1,4-diene and cadina-3,5-diene. Then, 7-hydroxy-calamenene was synthetized by hydroxylation of *trans*-calamenene. These results are in agreement with the proposed pathway for the biosynthesis of thymol from γ-terpinene *via* p-cymene (Poulose and Croteau, 1978).

## Conclusion

The chemical composition of *A. jollyanus* leaf essential oil was reported for the first time. The essential oil from Ivoirian species exhibited original composition dominated by hydrocarbon sesquiterpenes, mostly *trans*-calamenene, α-copaene, β-cubebene, cadina-3,5-diene, accompanied by 7-hydroxycalamenene. The composition of the EO remained dominated by the same sesquiterpenes during its phenological cycle of the plant, although the content of some components varied substantially.

## Authors' contributions

SGG (Ph.D. Student) contributed in collecting plant sample, running the laboratory work. ZAO and ATY contributed in collecting plant sample and supervised the laboratory work. YAB designed the study. AB and FT drafted the paper and contributed to critical reading of the manuscript. MP contributed to critical reading of the manuscript. All the authors have read the final version of the manuscript and approved the submission.

## Conflicts of interest

The authors declare no conflicts of interest.

## Acknowledgments

The authors are grateful to the CASES, (NGO) for financial support. They are indebted to Dr M. Gibernau for botanical advices.

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