A new chemotype of Lantana rhodesiensis Moldenke essential oil from Côte d'Ivoire: chemical

composition and biological activities

Fatimata Nea^{a,b,*}, Evelyne Amenan Tanoh^{a,b}, Esse Leon Wognin^{a,c}, Tierry Kenne Kemene^b, Manon Genva^b, Matthew Saive^b, Zanahi Felix Tonzibo^a, Marie-Laure Fauconnier^b

^aLaboratory of Biological Organic Chemistry, UFR-SSMT, University Félix Houphouët-Boigny, 01 BP 582 Abidjan 01, Côte d'Ivoire

^bLaboratory of Chemistry of Natural Molecules, University of Liège, Gembloux Agro-Bio Tech, 2, Passage of Déportés, B-5030 Gembloux, Belgium

^cLaboratory of Instrumentation Image and Spectroscopy, National Polytechnic Institute Felix Houphouët-Boigny, BP 1093 Yamoussoukro, Côte d'Ivoire

*Corresponding author.

E-mail address: neafatima@gmail.com (F. Nea).

ABSTRACT

Lantana rhodesiensis Moldenke is a plant native to subtropical and tropical regions, which is widely used in traditional medicine. The composition of essential oils hydrodistillated from leaves, fruits and stems of plants growing in two different localities of Côte d'Ivoire, Kapélé and Nyanbélégé, were analysed over four months. Essential oil composition was determined by gas chromatography–mass spectrometry (GC-MS), and the main constituents of leaf essential oils were β -caryophyllene (20.3–27.1%), α -copaene (9.5–11.9%), δ -cadinene (7.3–9.9%) and α -humulene (7.5–9.6%). Thymol was also found in two samples from Kapélé (7.7 and 13.4%) and in one sample from Nyanbélégé (17.4%). Fruit essential oil was characterised by a predominance of β -caryophyllene (22.8–25.2%), α -copaene (8.7–11.7%), α -humulene (8.0–9.9%) and δ -cadinene (7.3–10.6%). In addition, stem oil was dominated by β -caryophyllene (14.3–22.4%), followed by α -copaene (7.4–14.7%), δ -cadinene (10.6–12.6%), α -humulene (5.0–8.0%) and caryophyllene oxide (5.2–8.0%). Statistical analysis showed that leaf and fruit essential oils exhibited very similar compositions, but differed from those of the stem. Essential oils of the leaves showed antioxidant and anti-inflammatory activities. Samples with a high thymol concentration exhibited high antioxidant activities, while those containing low quantities of thymol displayed elevated anti-inflammatory properties.

Keywords: Lantana rhodesiensis, Essential oil, GC-MS, Cluster analysis, Antioxidant activity, Anti-inflammatory activity.

1. Introduction

The Verbenaceae family consists of 3,000 plant species belonging to 75 genera (Mwanasiti, 2013). Among these, the genus *Lantana* was described by Linnaeus in 1753 as containing seven species, six from South America and one from Ethiopia (Munir, 1996). Nowadays, *Lantana* spp. are found in subtropical and tropical regions, mostly in America, but also tropical Asia and Africa (Ghisalberti, 2000). The most common species are *Lantana camara L., Lantana trifolia L., Lantana rhodesiensis* Moldenke and *Lantana viburnoide* (Forssk.) Schweinf (Mwanasiti, 2013). *L. rhodesiensis* has a number of synonyms, including *Lantana ukambensis* (Vatke) Verdc. and *Lippia ukambensis* (Vatke) (Piero et al., 2015).

L. rhodesiensis is a multi-stemmed grass or small shrub, which reaches approximately two metres in height. Leaves have an opposite arrangement along the stems, and flowers vary from mauve to purple. Blue to purple berries (Piero et al., 2015), are collected during the rainy season (Peters, 1992). In many African countries, the plant is used locally as a food, in traditional medicine, as an insect repellent and for biofuel (Peters, 1992; Sawadogo et al., 2015). Traditional medicine uses of *L. rhodesiensis* include the treatment of malaria, measles, smallpox (Fratkin, 1996), cancer (Sawadogo et al., 2012), diabetes (Piero et al., 2015), coughs, fever, oral sores,

rheumatism (Peters, 1992; Piero et al., 2015), arterial hypertension (Bangou et al., 2011), parasitic diseases and cardiac arrhythmia (Bangou et al., 2017). It also shows antimicrobial activities. *L. rhodesiensis* has been reported to contain non-volatile compounds, with significant amounts of triterpenes, steroids, phenols, alkaloids, flavonoids and tannins (Bangou et al., 2011; Fratkin, 1996; Bangou et al., 2017; Piero et al., 2015; Sawadogo et al., 2012). Previous studies demonstrated that essential oil extracted from *L. rhodesiensis* leaves from Tanzania was dominated by camphor, 4-thujanol and 1,8-cineole (Chogo and Crank, 1982), whereas that from whole aerial parts of plants harvested in Kenya exhibited camphor and camphene as the major compounds (Omolo et al., 2004).

In this current study, the first chemical analysis of *L. rhodesiensis*, growing in two different sites of Côte d'Ivoire, was presented. Essential oil hydrodistillated from leaves, fruits and stems of these plants were characterised by gas chromatography–mass spectrometry (GC-MS). The *in vitro* antioxidant and anti-inflammatory activities of essential oils from leaves were also evaluated.

2. Materials and methods

2.1. Chemicals

Trolox, 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical, thymol, limonene, diclofenac sodium and bovine serum albumin (BSA) were obtained from Sigma–Aldrich (St Louis, MO, USA). Anhydrous sodium sulphate, potassium ferricyanide, trichloroacetic acid (TCA), ferric chloride, sodium chloride, potassium chloride, disodium hydrogen phosphate, potassium dihydrogen phosphate and hydrochloric acid were bought from VWR Chemicals (Radnor, PA, USA). Methanol was purchased from VWR International (Fontenay-sous-Bois, France).

Plant Material

Fresh leaves and stems of *L. rhodesiensis* were collected from June 2017 to September 2017 in Kapélé (9.437056, -5.701292) and Nyanbélégé (9.467, -5.693), Department of Korhogo, Northern region of Côte d'Ivoire. Weather data from year 2017 were obtained from the *Société d'Exploitation et de Développement Aéroportuaire, Aéronautique et Météorologique* (SODEXAM). The localities were selected for their accessibility and for the abundance of the plant in the local area. As fresh fruits could only be harvested in July in sufficient quantities for hydrodistillation, two fruit samples were collected during this month. Plant material was authenticated at the Centre National de Floristique (CNF), Abidjan, Côte d'Ivoire (herbarium number: UCJ017435). To standardise conditions, plants were harvested in the morning, at the beginning of each month.

2.2. Essential oil hydrodistillation

Fresh material (1.5 kg) was submitted to hydrodistillation for 4 h using a Clevenger-type apparatus. Essential oils were collected, dried over anhydrous sodium sulphate and then stored in sealed vials at 4 °C until analysed. The yield of essential oil (%) was calculated using the following formula (Zhang et al., 2015):

Essential oil mass (g) Fresh material mass (g)

2.3. Essential oil analysis

Essential oil yield (%) =

Ten mg of essential oil was dissolved in 100 mL hexane and analysed by GC-MS. All samples were analysed three times.

2.4. GC-MS and data analysis

Essential oils were analysed according to the procedure described by Rebey et al. (2018) and Tanoh et al. (2019), with some modifications. An Agilent 7890B GC system coupled to a MSD 5977B detector (Agilent, Santa Clara, CA, USA) and fitted with a HP-5MS capillary column (5% phenyl-95% methyl siloxane, 30 m x 0.25 mm, x 0.25 μ m) was used, with helium as the carrier gas (1.2 mL/min). One μ l essential oil solution in hexane was

injected in splitless mode. The oven temperature program had an increase in temperature from 50 °C (1 min) to 300 °C (5 min), at a rate of 5 °C/min. The mass selective detector was operated with an ionisation energy of 70 eV used over a scan mass range of 40–400 atomic mass units. The source and quadrupole temperatures were fixed at 230 °C and 150 °C, respectively. Data were analysed using MassHunter Workstation Software, Qualitative Analysis Navigator and Qualitative Analysis Workflows (Version B.08.00, Agilent Technologies, Inc. 2016), with identification of the individual components based on their chromatographic retention index (RI) and comparison of spectra with a library (Pal 600K®). The RIs were experimentally determined using a series of C_7 – C_{30} n-alkanes, and were compared with those reported in the literature (Babushok et al., 2011). Identifications were also made by reference to authentic standard compounds (Sigma, Darmstadt, Germany) analysed under the same conditions as the essential oils, when they were commercially available.

Comparison of essential oil composition was performed using multivariate statistical procedures as previously described (Soilhi et al., 2019). Principal component analysis (PCA) and hierarchical cluster analysis (HCA) were carried out with XLSTAT 2016.02.27444 software (Addinsoft, Paris, France). HCA used Euclidean distance coefficient functions based on the dissimilarity between two samples. A dendrogram was constructed based on square Euclidean distance measurements (Ward's method).

2.5. Antioxidant activity

DPPH radical scavenging assay

DPPH radical scavenging activity was determined as described by Bicas et al. (2011), with slight modifications. One mL of a methanolic solution of essential oils (25, 50, 75 and 100 μ g/mL) was mixed with 1 mL of a methanolic DPPH solution (0.004% w/v). The mixture was incubated at room temperature for 30 minutes, and the absorbance was measured at 517 nm using an Ultrospec 7000 UV-visible dual beam spectrophotometer (GE Healthcare, Chicago, IL, USA). For comparison, inhibition of the free radical DPPH by Trolox, thymol and limonene was also analysed under the same conditions. Trolox was used as a positive standard. All tests were carried out in triplicate.

The inhibition percentage of the DPPH free radical (I%) was calculated as described by Hazzit et al. (2009):

$$I\% = [(A_b - A_a)/A_b] \ge 100$$

where A_b is the absorbance of the reaction media without essential oil and A_a is the absorbance of the test sample.

I% was plotted against sample concentrations to obtain the IC_{50} index, which was defined as the concentration of antioxidant required to decrease the initial DPPH concentration by 50% (Sharififar et al., 2007).

Reducing power determination

The reducing power of essential oils, thymol, limonene and the standard (Trolox) were determined according to the protocol of Lamia et al. (2018). In brief, 1 mL of sample (25, 50, 75 and 100 μ g/mL) was mixed with 1 mL 0.2 M sodium phosphate buffer pH 6.6 and 1mL 1% potassium ferricyanide solution, and was incubated at 50 °C for 20 minutes. One mL 10% (v/v) TCA was then added to the solution, followed by centrifugation at 3000g for 10 minutes. The supernatant was recovered and mixed with 1.5 mL distilled water and 150 μ l 0.1% FeCl₃. The absorbance at 700 nm was then measured. Absorbance increases of the reaction mixture, relative to the blank, indicated an increase in reducing power.

2.6. Anti-inflammatory activity

The *in vitro* inhibitory effect of essential oils from *L. rhodesiensis* leaves was determined using the protein denaturation method described by Rahman et al. (2015), with some modifications. The protein used was 5% (w/v) BSA in water. Sodium phosphate buffer pH 6.3 contained 136 mM NaCl, 2.68 mM KCl, 10.1 mM Na₂HPO₄ and 1.76 mM KH₂PO₄ in distilled water. The pH was adjusted to 6.3 using 1M HCl.

Methanolic solutions of essential oil (0.05 mL) at various concentrations (25, 50, 75 and 100 μ g/mL) and standard drug, diclofenac sodium (25, 50, 75 and 100 μ g/mL), were individually mixed with 0.45 mL of 0.5% (w/v) BSA. The samples were incubated at 37 °C for 20 minutes and then the temperature was increased, keeping the samples at 57 °C for 3 minutes. After cooling, 2.5 mL phosphate buffer was added to the solutions. The absorbance was then measured at 255 nm using a spectrophotometer. The results for the essential oils were compared with those for the diclofenac sodium standards.

The percentage of inhibition was calculated by the following formula (Thanh et al., 2017):

% Inhibition = $[(Abs C - Abs T) / Abs C] \ge 100$

where Abs C is the absorbance of the reactional media without inhibitor or essential oils, and Abs T is the absorbance of the test sample.

2.7. Statistical analysis of biological activity measurements

Statistical analysis of biological activity results was performed by one-factor analysis of variance (one-way ANOVA) using Minitab 18 software (Minitab LLC, State College, PA, USA). The threshold of significance (α) was set at 0.05. When a significant difference was observed, Tukey's multiple comparison test was performed.

3. Results and Discussion

3.1. Essential oil yields and chemical composition

Kapélé and Nyanbélégé are two villages separated by 8.8 km and located in the Department of Korhogo $(9^{\circ}27'28" \text{ N}, 5^{\circ}37'46" \text{ W}; 360 \text{ m} above sea level})$ in Côte d'Ivoire. Eighteen essential oil samples were obtained from *L. rhodesiensis* collected in both locations (nine samples from each locality), over four months. The fresh plant materials produced pale yellow essential oils, the hydrodistillation yields of which are reported in Table 1, as w/w calculated on a fresh weight basis.

	Jı	ıne		July		Au	gust	September		
Yield (%)	Leaves	Stems	Leaves	Fruits	Stems	Leaves	Stems	Leaves	Stems	
Kapélé	0.20	0.09	0.32	0.13	0.14	0.28	0.12	0.34	0.15	
Nyanbélégé	0.27	0.10	0.30	0.09	0.12	0.33	0.13	0.40	0.17	

 Table 1. Lantana rhodesiensis essential oils yields

Regardless of the locality, the highest essential oil yields from leaves and stems were obtained with plants collected during the month of September. Humidity and rainfall were particularly high in Korhogo during that month in 2017 (Fig. 1). The temperature was lower in comparison to June, but the duration of solar insolation was also important. The high essential oil yields obtained in September could be explained by high temperatures and precipitation that might have stimulated plant photosynthesis. Indeed, essential oils components are secondary metabolites, and their production can be affected by the plant photosynthetic activity and water availability (Croteau et al., 1987).

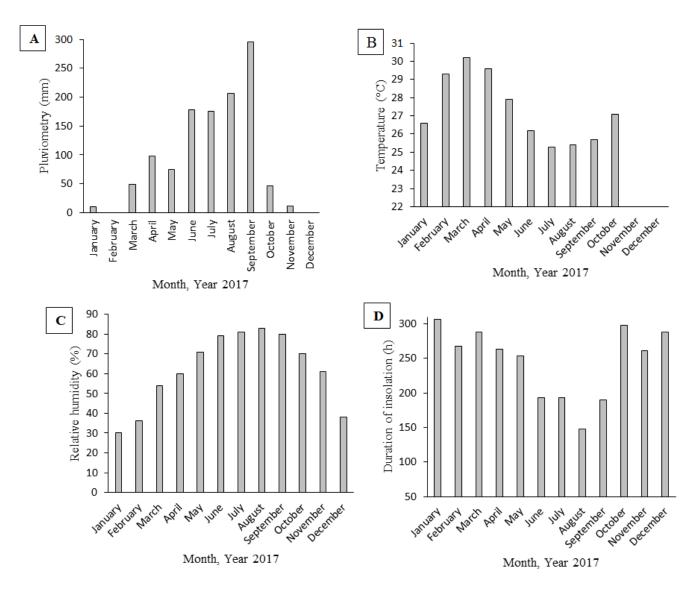


Figure 1. Korhogo weather data from year 2017: A. Pluviometry (mm), B. Temperature (°C), C. Relative humidity (%), D. Duration of insolation (h)

The relative concentrations of volatile organic compounds identified in essential oils are reported in Tables 2 (Kapélé) and 3 (Nyanbélégé). Sixty-five and sixty-one components, respectively, were identified in essential oils from these two sites. Results showed that the essential oil of *L. rhodesiensis* mainly consisted of sesquiterpenes and monoterpenes. Indeed, β -caryophyllene was the major compound in all the essential oil samples. Other sesquiterpenes were also present at high levels, such as α -copaene, α -humulene, δ -cadinene and caryophyllene oxide. All samples contained high quantities of non-oxygenated sesquiterpenes. In addition, only four samples contained smaller amounts of oxygenated monoterpenes in comparison with non-oxygenated monoterpenes. Among all identified monoterpenes, limonene, α -pinene and thymol were the major compounds.

Leaf essential oils: Thirty-seven compounds were identified in leaf oil samples, which were notable for high levels of sesquiterpene hydrocarbons (59.7–73.9%). Sesquiterpene proportions were the highest in August in samples from Kapélé (85.5%), while the highest proportions in samples from Nyanbélégé were obtained in July (85.8%). The most prevalent components were β -caryophyllene (20.3 ± 0.2% to 27.1 ± 0.3%), α -copaene (9.5 ± 0.2% to 11.9 ± 0.0%), α -humulene (7.5 ± 0.1% to 9.6 ± 0.1%), and δ -cadinene (7.3 ± 0.0% to 9.9 ± 0.1%). Thymol, limonene and α -pinene were the monoterpenes present at the highest levels in some samples. Thymol was identified in July (13.4 ± 0.3%) and August (7.7 ± 0.1%) samples from Kapélé and in the September sample (17.4 ± 0.2%) from Nyanbélégé. Limonene and α -pinene were present in high quantities in samples from Kapélé in June

at 7.0 \pm 0.1% and 5.2 \pm 0.1%, and in September at 6.5 \pm 0.1% and 4.7 \pm 0.1%, respectively. By contrast, only the June sample contained limonene (6.3 \pm 0.1%) and α -pinene (4.0 \pm 0.1%) at high quantities in Nyanbélégé. The essential oils from Nyanbélégé also differed from those of Kapélé due to the abundance of caryophyllene oxide (5.3 \pm 0.1% to 7.5 \pm 0.2%) in three samples.

Fruit essential oils: GC-MS analysis of fruit essential oils from Kapélé and Nyanbélégé allowed the identification and quantification of 34 and 29 constituents, respectively. β-caryophyllene ($22.8 \pm 0.1\%$ to $25.2 \pm 0.4\%$), α-copaene (8.7 ± 0.1 to $11.7 \pm 0.1\%$), α-humulene (8.0 ± 0.0 to $9.9 \pm 0.2\%$) and δ-cadinene (7.3 ± 0.1 to $10.6 \pm 0.1\%$) were the main constituents in oils from both locations. The essential oil collected from *L. rhodesiensis* fruits from Nyanbélégé was lower in monoterpenes (17.8%) than that of Kapélé (29.7%). In particular, limonene, present in essential oil from Nyanbélégé ($6.5 \pm 0.19\%$), was absent in fruit oil from Kapélé, which was characterised by higher levels of thymol ($17.2 \pm 0.4\%$). Thus, the chemical composition of essential oils from fruit was related to plant location. Indeed, the major compounds in essential oil samples from Kapélé were β-caryophyllene ($22.8 \pm 0.4\%$) and thymol ($17.2 \pm 0.4\%$), while β-caryophyllene ($25.2 \pm 0.4\%$), α-copaene ($11.7 \pm 0.1\%$) were the most abundant in essential oil samples from Nyanbélégé.

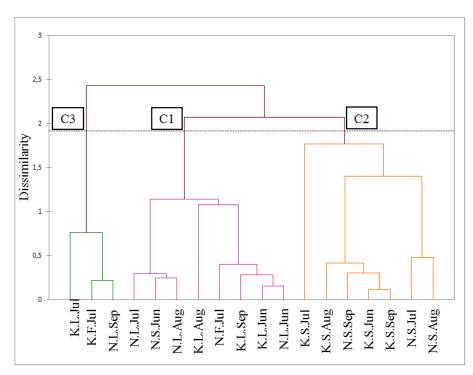
Stem essential oils: Fifty-three compounds accounting for more than 99.9% of the total composition were identified in the eight stem essential oil samples of *L. rhodesiensis*. These oils were dominated by sesquiterpenes (70.4–86.2%), among which β-caryophyllene (14.3 ± 0.6% to 22.4 ± 0.0%), followed by α-copaene (7.4 ± 0.3% to 14.7 ± 0.2%), δ-cadinene (10.6 ± 0.3% to 12.6 ± 0.1%), α-humulene (5.0 ± 0.2% to 8.0 ± 0.1%) and caryophyllene oxide (5.2 ± 0.0% to 8.0 ± 0.1%) were the major compounds. The level of sesquiterpenes, for samples from each month was higher in essential oils from Nyanbélégé compared to those from Kapélé. Monoterpenes (6.9–17.4%) were found in lower proportions than sesquiterpenes. Essential oil samples from Kapélé were rich in thymol (8.3 ± 0.2% – 15.3 ± 0.6%), whereas only the September sample from Nyanbélégé contained that molecule (8.5 ± 0.1%). A variety of sesquiterpenes was detected at lower levels: α-muurolene (up to 5.3 ± 0.15%), nerolidol (up to 3.0 ± 0.03%), γ-elemene (up to 3.8 ± 0.12%) and β-elemene (up to 1.1 ± 0.05%).

3.2. Chemical variability of essential oil composition

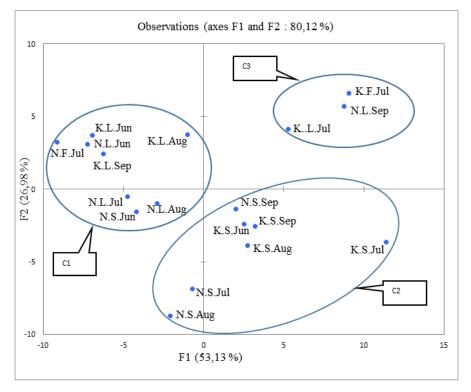
The dendrogram of the HCA (Fig. 2a) divided the 18 essential oil samples into three groups, C1, C2 and C3. Sample distribution obtained by PCA was in agreement with these three groups (Fig. 2b). Indeed, the two principal axes accounted for 80.12% of the total variability (F1, 53.13% and F2, 26.98%). The C1 and C3 clusters were mainly composed of essential oil samples from leaves and fruits, while almost all stem oil samples were clustered in C2. Indeed, leaves and fruits of *L. rhodesiensis* showed two distinct chemical composition profiles:

- Cluster C1 (six leaf, one fruit and one stem oil samples) was dominated by sesquiterpenes, such as β -caryophyllene (23.9 ± 2.1%), α -copaene (12.2 ± 2.1%), δ -cadinene (9.8 ± 1.3%) and α -humulene (9.0 ± 0.8%). Caryophyllene oxide (4.8 ± 1.4%), limonene (4.4 ± 2.5%) and α -muurolene (4.5 ± 0.4%) were also present in significant amounts in these essential oils.
- Cluster C3 (two leaf and one fruit oil samples) mainly consisted of β -caryophyllene (22.0 ± 1.0%), thymol (16.0 ± 2.3%) and α -copaene (9.5 ± 0.8%). However, this cluster also showed high levels of α -humulene (7.9 ± 0.4%), δ -cadinene (7.6 ± 0.5%) and caryophyllene oxide (4.5 ± 0.8%).

Based on the major compounds, profiles of the chemical composition of leaves and fruits essential oils could be differentiated by their content of thymol. This phenolic monoterpene, which was present at $3.5 \pm 1.9\%$ in cluster C1, reached $16.0 \pm 2.3\%$ in cluster C3, becoming the second most abundant component in this cluster after β -caryophyllene. As clusters C1 and C3 included leaf and fruit samples from Kapélé and Nyanbélégé collected over different months, there was low essential oil variability between the locations and the harvesting periods. Finally, the results showed that the chemical compositions of fruit oil samples were similar to those from leaves.



2a. Dendrogram



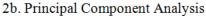


Figure 2. 2a. Dendrogram and 2b. Principal Component Analysis distribution of the 18 essential oil samples of L. rhodesiensis

K: Kapélé, N: Nyanbélégé, S: Stems, F: Fruits, L: Leaves, Jun: June, Jul: July, Aug: August, Sep: September

By contrast, seven stem essential oil samples clustered in C2 were characterised by a unique chemical profile, dominated by β -caryophyllene (18.6 ± 2.4%), α -copaene (12.0 ± 2.3%) and δ -cadinene (11.2 ± 0.7%), followed, in lower proportions by thymol (8.1 ± 4.1%), caryophyllene oxide (6.8 ± 1.0%), α -humulene (6.1 ± 0.7%) and α -muurolene (4.7 ± 0.4%).

Table 2. Essential oil composition of Lantana rhodesiensis from Kapélé (mean of triplicates)	
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Commente	No GAG		T. 4	T		une	T	July	C (1,		igust	September		
ompounds	N° CAS	Id	Ir th	Ir cal	Leaves	Stems	Leaves	Fruits	Stems	Leaves	Stems	Leaves	Stems	
pinene	80-56-8	MS,STD,RI	930	930	5.2 ± 0.1	-	0.4 ± 0.1	2.2 ± 0.0	-	-	-	4.7 ± 0.1	-	
amphene	79-92-5	MS,STD,RI	943	945	0.9 ± 0.0	-	-	$0.3\ \pm 0.0$	-	-	-	0.6 ± 0.1	-	
pinene	18172-67-3	MS,STD,RI	975	973	-	-	1.3 ± 0.0	-	-	-	-	1.1 ± 0.1	-	
-octen-3-ol	3391-86-4	MS,STD,RI	978	977	-	1.0 ± 0.0	-	-	-	0.9 ± 0.0	1.0 ± 0.0	-	0.6 ± 0.1	
octanol	589-98-0	MS,STD,RI	997	996	-	-	-	-	-	-	0.7 ± 0.0	-	-	
-cymene	25155-15-1	MS,STD,RI	1026	1021	-	-	0.3 ± 0.0	1.1 ± 0.0	-	-	-	-	-	
monene	138-86-3	MS,STD,RI	1028	1026	7.0 ± 0.1	1.0 ± 0.0	2.0 ± 0.1	tr	tr	0.9 ± 0.0	0.2 ± 0.0	6.5 ± 0.1	tr	
-terpinene	98-85-4	MS,STD,RI	1060	1055	-	-	-	0.4 ± 0.0	-	-	-	-	-	
-octanol	112-32-3	MS,STD,RI	1068	1066	-	0.1 ± 0.0	-	0.4 ± 0.0	-	-	-	0.6 ± 0.0	-	
nalool	78-70-6	MS,STD,RI	1099	1099	2.3 ± 0.0	1.2 ± 0.0	2.2 ± 0.0	$4.0\ \pm 0.0$	0.8 ± 0.0	$1.8\ \pm 0.0$	1.6 ± 0.0	2.3 ± 0.1	1.2 ± 0.1	
onanal	124-19-6	MS,STD,RI	1100	1100	-	0.2 ± 0.0	-	-	-	-	-	-	0.2 ± 0.2	
loocimene	673-84-7	MS,RI	1125	1126	0.4 ± 0.0	-	-	-	-	-	-	0.4 ± 0.0	-	
tronellal	106-23-0	MS,STD,RI	1143	1145	-	0.2 ± 0.0	-	-	-	-	-	-	-	
orneol	507-70-0	MS,STD,RI	1166	1163	-	-	_	0.4 ± 0.0	_	_	-	_	-	
-terpineol	20126-76-5	MS,STD,RI	1177	1178	-	_	0.3 ± 0.0	0.4 ± 0.0 0.7 ± 0.0	0.2 ± 0.0	_	_	_	_	
-terpineol	98-55-5	MS,STD,RI MS,STD,RI	1190	1178	-	_	-	0.7 ± 0.0 0.4 ± 0.0	-	_	_	_	_	
ethyl salicylate	119-36-8	MS,STD,RI	1193	1191	0.3 ± 0.0	0.9 ± 0.0	0.3 ± 0.0	-	-	_	0.5 ± 0.0	0.3 ± 0.0	-	
ecanal	112-31-2	MS,STD,RI MS,STD,RI	1200	1202	0.3 ± 0.0	0.9 ± 0.0 0.2 ± 0.0	0.3 ± 0.0	-	-	-	0.5 ± 0.0	0.5 ± 0.0	-0.2 ± 0.1	
citronellol	106-22-9	MS,STD,RI MS,STD,RI	1200	1202	-0.3 ± 0.0	0.2 ± 0.0 1.1 ± 0.0	-	-	0.2 ± 0.0	-	-	-	0.2 ± 0.0 0.3 ± 0.0	
	106-22-9	MS,STD,RI MS,STD,RI	1223	1224	0.5 ± 0.0 0.6 ± 0.0	1.1 ± 0.0 2.0 ± 0.1	-0.4 ± 0.0	-0.4 ± 0.0	0.2 ± 0.0 0.6 ± 0.0	-	-	-0.4 ± 0.0	0.3 ± 0.00 0.2 ± 0.00	
eraniol	5392-40-5		1250	1250		2.0 ± 0.1	0.4 ± 0.0			-	-	0.4 ± 0.0	0.2 ± 0.2	
eranial		MS,STD,RI			-	-	-	2.7 ± 0.0	0.4 ± 0.0	-	-	-	-	
iymol	89-83-8	MS,STD,RI	1290	1291	2.9 ± 0.0	8.4 ± 0.3	13.4 ± 0.3	17.2 ± 0.4	15.3 ± 0.6	7.7 ± 0.1	8.3 ± 0.2	3.1 ± 0.1	$9.6\pm0.$	
elemene	20307-84-0	MS,RI	1337	1336	0.7 ± 0.0	0.3 ± 0.0	0.7 ± 0.0	0.5 ± 0.0	0.2 ± 0.0	$0.8\ \pm 0.0$	0.4 ± 0.0	-	-	
-cubebene	17699-14-8	MS,RI	1350	1349	0.3 ± 0.0	-	-	-	0.2 ± 0.0	-	-	0.3 ± 0.0	0.3 ± 0.1	
ıgenol	97-53-0	MS,STD,RI	1356	1355	-	-	-	-	0.3 ± 0.0	-	-	-	-	
-copaene	3856-25-5	MS,STD,RI	1379	1379	11.7 ± 0.1	11.8 ± 0.0	10.2 ± 0.1	8.7 ± 0.1	7.4 ± 0.3	11.2 ± 0.1	12.4 ± 0.2	11.1 ± 0.2	13.3 ±	
bourbebene	5208-59-3	MS,RI	1385	1385	0.9 ± 0.0	0.9 ± 0.0	1.6 ± 0.1	$0.9\ \pm 0.0$	tr	1.9 ± 0.1	1.0 ± 0.1	0.9 ± 0.0	$0.7 \pm 0.$	
elemene	515-13-9	MS,STD,RI	1388	1389	tr	1.3 ± 0.0	1.7 ± 0.1	1.2 ± 0.0	1.2 ± 0.0	$1.5\ \pm 0.0$	1.1 ± 0.1	1.3 ± 0.0	0.8 ± 0.1	
gurjunene	489-40-7	MS,RI	1413	1410	0.3 ± 0.0	0.3 ± 0.0	-	-	-	-	-		-	
-caryophyllene	87-44-5	MS,STD,RI	1424	1424	24.5 ± 0.3	18.6 ± 0.4	22.4 ± 0.3	22.8 ± 0.4	14.3 ± 0.6	27.1 ± 0.3	19.1 ± 0.3	22.7 ± 0.5	21.0 ± 0	
-elemene	3242-08-08	MS,RI	1434	1436	2.9 ± 0.0	2.6 ± 0.1	3.2 ± 0.0	$2.8\ \pm 0.0$	3.3 ± 0.1	3.6 ± 0.0	3.8 ± 0.1	1.0 ± 0.0	$2.2 \pm 0.$	
eranyl acétone	3796-70-1	MS,RI	1447	1449	-	-	-	-	$0.2\ \pm 0.0$	-	-	-	0.5 ± 0.5	
-humulene	6753-98-6	MS,STD,RI	1456	1458	9.2 ± 0.2	6.8 ± 0.1	8.3 ± 0.1	8.0 \pm 0.0	5.0 ± 0.2	9.5 ± 0.1	6.8 ± 0.1	$\textbf{8.8} \pm \textbf{0.1}$	5.6 ± 0.1	
loaromadendrene	25246-27-9	MS,RI	1462	1462	1.3 ± 0.2	1.0 ± 0.1	1.2 ± 0.3	1.2 ± 0.0	1.0 ± 0.0	1.2 ± 0.0	1.4 ± 0.2	1.5 ± 0.1	$1.0 \pm 0.$	
ermacrene D	23986-74-5	MS,RI	1480	1480	0.8 ± 0.3	0.2 ± 0.0	0.9 ± 0.2	0.7 ± 0.1	-	1.0 ± 0.1	0.7 ± 0.1	0.8 ± 0.1	0.4 ± 0.1	
ionone	14901-07-6	MS,STD,RI	1485	1484	-	-	0.3 ± 0.0	-	$0.2\ \pm 0.0$	0.4 ± 0.1	-	-	0.3 ± 0.1	
selinene	17066-67-0	MS,RI	1488	1487	0.3 ± 0.0	1.6 ± 0.1	1.4 ± 0.1	1.3 ± 0.0	$1.8\ \pm 0.1$	0.7 ± 0.1	1.2 ± 0.0	0.4 ± 0.0	1.6 ± 0.1	
tridecanone	593-08-8	MS,STD,RI	1491	1496	-	2.1 ± 0.1	1.3 ± 0.0	-	2.8 ± 0.1	-	-	-	2.3 ± 0.1	
alencene	4630-07-3	MS,STD,RI	1495	1496	-	-	-	$1.0\ \pm 0.0$	-	-	-	0.7 ± 0.1	-	
-muurolene	483-75-0	MS,RI	1499	1503	4.1 ± 0.1	4.4 ± 0.4	3.9 ± 0.0	3.4 ± 0.2	4.1 ± 0.2	4.7 ± 0.2	5.0 ± 0.1	4.1 ± 0.1	4.8 ± 0.1	
-cadinene	39029-41-9	MS,RI	1511	1514	0.6 ± 0.0	0.7 ± 0.1	tr	0.5 ± 0.0	0.9 ± 0.2	0.5 ± 0.1	0.8 ± 0.1	0.6 ± 0.0	0.7 ± 0	
cadinene	483-76-1	MS,RI	1519	1524	9.4 ± 0.1	10.6 ± 0.2	8.2 ± 0.0	7.3 ± 0.1	10.6 ± 0.3	9.9 ± 0.1	11.1 ± 0.2	9.3 ± 0.2	12.6 ±	
-calacorene	21391-99-1	MS,RI	1546	1549	-	0.7 ± 0.1	0.4 ± 0.0	-	0.7 ± 0.0	0.4 ± 0.1	0.7 ± 0.0	0.4 ± 0.1	1.0 ± 0.0	
emol	639-99-6	MS,RI	1550	1553	0.5 ± 0.0	2.0 ± 0.1	0.4 ± 0.0 0.6 ± 0.0	$0.6\ \pm 0.1$	1.9 ± 0.1	-	0.7 ± 0.0 0.2 ± 0.0	1.3 ± 0.1	1.0 ± 0.1 1.7 ± 0.1	
erolidol	7212-44-4	MS,RI	1565	1565	2.3 ± 0.0	1.8 ± 0.1	3.3 ± 0.1	1.9 ± 0.2	1.9 ± 0.1 1.9 ± 0.1	4.0 ± 0.1	0.2 ± 0.0 2.0 ± 0.2	3.7 ± 0.1	0.9 ± 0.1	
athulenol	6750-60-3	MS,STD,RI	1582	1583	3.7 ± 0.0	0.3 ± 0.0	0.8 ± 0.1	0.5 ± 0.2	1.0 ± 0.1 1.0 ± 0.1	0.5 ± 0.1	0.5 ± 0.1	0.3 ± 0.0	0.3 ± 0.0	
aryophyllene oxide	1139-30-6	MS,STD,RI	1582 1589	1585 1589	3.7 ± 0.0 3.7 ± 0.1	5.2± 0.0	4.5 ± 0.0	3.7 ± 0.2	7.9 ± 0.2	4.3 ± 0.1	7.0 ± 0.3	4.0 ± 0.1	7.0 ± 0	
ridiflorol	552-02-3	MS,STD,RI MS,STD,RI	1593	1593	0.3 ± 0.1	0.7 ± 0.1	0.5 ± 0.1	0.5 ± 0.1	1.1 ± 0.1	0.4 ± 0.1	0.7 ± 0.3	0.5 ± 0.1	0.6 ± 0.0	
								0.5 ± 0.1	1.1 ± 0.1				0.0 ± 0.	
-cudesmon	1207-/1-0	1/13,1/1	1031	1032	-	0.5±0.0		-	-	-	-	-	-	
-eudesmol	12	209-71-8	209-71-8 MS,RI	209-71-8 MS,RI 1631	209-71-8 MS,RI 1631 1632	09-71-8 MS,RI 1631 1632 -	09-71-8 MS,RI 1631 1632 - 0.5±0.0	209-71-8 MS,RI 1631 1632 - 0.5±0.0 - 8						

55	ζ-cadinol	5937-11-01	MS.RI	1639	1636	0.6 ± 0.1	1.3 ± 0.0	0.8 ± 0.1	0.8 ± 0.1	2.1 ± 0.4	0.8 ± 0.1	1.1 ± 0.3	0.7 ± 0.0	1.2 ± 0.1
56	δ-cadinol	19435-97-3	MS.RI	1643	1645	-	1.1 ± 0.1	-	-	1.6 ± 0.4	0.6 ± 0.1	1.4 ± 0.3	0.6 ± 0.0	0.9 ± 0.1
57	β-eudesmol	473-15-4	MS,RI	1651	1652	-	0.6 ± 0.0	-	0.3 ± 0.1	0.7 ± 0.3	-	-	0.5 ± 0.0	-
58	α-eudesmol	473-16-5	MS,RI	1652	1655	-	-	-	-	-	-	-	1.0 ± 0.02	-
59	a-cadinol	481-34-5	MS,RI	1652	1656	-	1.9 ± 0.0	1.1 ± 0.1	1.3 ± 0.1	3.0 ± 0.7	1.0 ± 0.1	2.0 ± 0.4	-	1.8 ± 0.1
60	2-pentadecanone.	502-69-2	MS,RI	1842	1840	-	1.5 ± 0.1	0.4 ± 0.0	-	1.7 ± 0.1	0.4 ± 0.0	1.1 ± 0.0	-	2.5 ± 0.1
	6.10.14-trimethyl													
63	palmitic acid	57-10-3	MS,STD,RI	1942	1947	-	0.7 ± 0.1	-	-	$3.7\ \pm 0.0$	-	3.2 ± 0.3	-	0.4 ± 0.1
64	isophytol	505-32-8	MS,RI	1944	1948	-	-	-	-	-	0.3 ± 0.0	-	-	-
65	phytol	150-86-7	MS,RI	2112	2116	2.1 ± 0.0	2.1 ± 0.1	1.9 ± 0.1	tr	$2.0\ \pm 0.1$	2.1 ± 0.0	3.0 ± 0.0	3.5 ± 0.1	0.7 ± 0.0
Hye	lrocarbons monoterpen	es (%)				13.6	1.0	3.9	4.0	0.0	0.9	0.2	13.4	0.0
Oxy	genated monoterpenes	(%)				6.1	13.2	16.3	25.7	17.4	9.5	10.0	5.8	11.4
Hyd	rocarbons sesquiterpen	es (%)				66.7	61.8	64.2	60.3	50.6	73.9	65.5	63.9	66.2
Oxy	genated sesquiterpenes	(%)				11.2	15.4	11.5	9.6	21.2	11.6	14.9	12.6	14.6
Dite	rpenes (%)					2.1	2.1	1.9	0.0	2.0	2.4	3.0	3.5	0.7
Oth	ers (%)					0.3	6.8	2.3	0.4	8.9	1.7	6.5	0.8	7.1
Ider	ntified compounds (%)					> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9

 Table 3. Essential oil composition of Lantana rhodesiensis from Nyanbélégé (mean of triplicates)

						J	une		July		A	ugust	September	
\mathbf{N}°	Compounds	N° CAS	Id	Ir th	Ir cal	Leaves	Stems	Leaves	Fruits	Stems	Leaves	Stems	Leaves	Stems
1	α-pinene	80-56-8	MS,STD,RI	930	933	4.0 ± 0.1	1.0 ± 0.0	0.2 ± 0.0	4.9 ± 0.2	tr	1.4 ± 0.0	tr	1.5 ± 0.0	tr
2	camphene	79-92-5	MS,STD,RI	943	948	0.5 ± 0.0	-	-	0.7 ± 0.0	-	0.2 ± 0.0	-	-	-
3	β-pinene	18172-67-3	MS,STD,RI	975	976	-	-	-	1.0 ± 0.2	-	-	-	-	-
4	sabinene	3387-41-5	MS,STD,RI	976	976	1.1 ± 0.0	-	-	-	-	-	-	-	-
5	1-octen-3-ol	3391-84-4	MS,STD,RI	978	977	-	-	0.7 ± 0.0	-	0.5 ± 0.0	-	0.6 ± 0.0	-	0.9 ± 0.0
7	p-cymene	25155-15-1	MS,STD,RI	1026	1026	-	-	-	-	-	-	-	1.2 ± 0.1	-
8	d-limonene	138-86-3	MS,STD,RI	1028	1029	6.3 ± 0.1	2.3 ± 0.1	1.5 ± 0.1	6.5 ± 0.2	tr	4.5 ± 0.0	0.6 ± 0.0	3.6 ± 0.2	0.5 ± 0.0
9	1.8-cineol	470-82-6	MS,STD,RI	1033	1031	-	-	-	-	0.5 ± 0.0	-	-	-	-
10	Y-terpinene	98-85-4	MS,STD,RI	1060	1059	-	-	-	-	-	-	-	0.4 ± 0.02	-
11	1-octanol	112-32-3	MS,STD,RI	1068	1069	0.5 ± 0.0	0.4 ± 0.0	0.3 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.2 ± 0.0	-	-
12	linalool	78-70-6	MS,STD,RI	1099	1099	2.3 ± 0.0	2.4 ± 0.1	2.4 ± 0.0	2.8 ± 0.0	1.8 ± 0.1	2.0 ± 0.0	1.4 ± 0.1	2.9 ± 0.1	1.4 ± 0.0
14	alloocimene	673-84-7	MS,RI	1125	1129	0.3 ± 0.0	-	-	0.4 ± 0.0	-	-	-	-	-
15	camphor	76-22-2	MS,STD,RI	1143	1145	tr	-	-	-	-	0.6 ± 0.0	-	0.3 ± 0.0	-
16	borneol	507-70-0	MS,STD,RI	1166	1167	-	-	-	-	0.2 ± 0.0	-	-	-	-
17	4-terpineol	20126-76-5	MS,STD,RI	1177	1178	-	-	-	-	-	-	-	0.5 ± 0.0	-
18	a-terpineol	98-55-5	MS,STD,RI	1190	1191	tr	-	0.2 ± 0.0	-	0.3 ± 0.0	-	-	0.3 ± 0.0	-
19	methyl salicylate	119-36-8	MS,STD,RI	1193	1195	0.3 ± 0.0	0.5 ± 0.1	-	-	0.2 ± 0.0	-	-	0.4 ± 0.0	0.4 ± 0.0
20	decanal	112-31-2	MS,STD,RI	1195	1205	-	0.3 ± 0.0	-	-	-	-	0.2 ± 0.0	-	-
21	β-citronellol	106-22-9	MS,STD,RI	1225	1228	-	0.4 ± 0.0	-	-	-	1.5 ± 0.1	0.9 ± 0.1	-	-
22	geraniol	106-24-1	MS,STD,RI	1258	1254	-	0.8 ± 0.0	-	0.3 ± 0.0	0.5 ± 0.0	2.3 ± 0.0	1.1 ± 0.0	-	-
23	geranial	5392-40-5	MS,STD,RI	1268	1267	-	-	0.4 ± 0.0	-	0.7 ± 0.1	-	-	-	-
25	thymol	89-83-8	MS,STD,RI	1297	1293	2.7 ± 0.0	3.8 ± 0.1	2.8 ± 0.1	1.2 ± 0.0	4.0 ± 0.0	4.0 ± 0.1	2.8 ± 0.0	17.4 ± 0.2	8.5 ± 0.1
26	δ-elemene	20307-84-0	MS,RI	1337	1340	-	-	-	-	-	-	-	0.5 ± 0.0	-
27	α-cubebene	17699-14-8	MS,RI	1350	1353	-	0.4 ± 0.0	-	0.5 ± 0.0	0.2 ± 0.0	0.3 ± 0.0	0.4 ± 0.0	0.1 ± 0.0	-
28	eugenol	97-53-0	MS,STD,RI	1356	1359	-	-	-	-	0.3 ± 0.0	-	-	0.7 ± 0.0	-
29	α-copaene	3856-25-5	MS,STD,RI	1379	1379	11.9 ± 0.0	14.7 ± 0.2	11.6 ± 0.1	11.7 ± 0.1	12.3 ± 0.1	$\textbf{10.8} \pm \textbf{0.1}$	14.7 ± 0.2	9.5 ± 0.2	12.3 ± 0.1

30	β-bourbebene	5208-59-3	MS,RI	1385	1389	0.9 ± 0.0	0.7 ± 0.0	1.8 ± 0.1	-	-	3.3 ± 0.1	0.7 ± 0.0	2.8 ± 0.1	-
31	β-elemene	515-13-9	MS,STD,RI	1388	1389	1.5 ± 0.1	0.9 ± 0.0	1.3 ± 0.0	-	1.1 ± 0.1	1.8 ± 0.0	-	1.7 ± 0.1	0.9 ± 0.0
32	α-gurjunene	489-40-7	MS,RI	1413	1415	0.3 ± 0.0	0.3 ± 0.0	-	-	0.2 ± 0.0	-	0.2 ± 0.0	-	-
33	β-caryophyllene	87-44-5	MS,STD,RI	1424	1424	24.7 ± 0.2	$\textbf{22.4} \pm \textbf{0.2}$	$\textbf{23.9} \pm \textbf{0.2}$	$\textbf{25.2} \pm \textbf{0.4}$	18.0 ± 0.1	$\textbf{20.3} \pm \textbf{0.2}$	$\textbf{17.8} \pm \textbf{0.4}$	$\textbf{20.9} \pm \textbf{0.4}$	$\textbf{21.4} \pm \textbf{0.2}$
34	Y-elemene	3242-08-08	MS,RI	1434	1436	1.3 ± 0.0	1.2 ± 0.0	0.9 ± 0.0	-	1.4 ± 0.1	0.9 ± 0.0	0.5 ± 0.0	2.0 ± 0.1	2.3 ± 0.0
35	α-humulene	6753-98-6	MS,STD,RI	1456	1458	9.6 ± 0.1	$\textbf{8.0} \pm \textbf{0.1}$	9.2 ± 0.2	9.9 ± 0.2	6.5 ± 0.2	$\textbf{7.8} \pm \textbf{0.1}$	6.2 ± 0.1	7.5 ± 0.1	6.0 ± 0.0
36	alloaromadendrene	25246-27-9	MS,RI	1462	1465	1.5 ± 0.1	1.4 ± 0.1	1.7 ± 0.0	tr	1.5 ± 0.0	1.6 ± 0.1	1.6 ± 0.1	1.4 ± 0.1	1.1 ± 0.2
37	germacrene D	23986-74-5	MS,RI	1480	1480	1.0 ± 0.1	0.4 ± 0.1	1.1 ± 0.2	2.1 ± 0.1	0.3 ± 0.0	-	0.4 ± 0.0	0.7 ± 0.1	-
38	β-ionone	14901-07-6	MS,STD,RI	1485	1484	-	-	-	-	-	0.3 ± 0.0	-	0.3 ± 0.1	-
39	β-selinene	17066-67-0	MS,RI	1488	1487	0.6 ± 0.0	0.7 ± 0.0	0.5 ± 0.0	0.5 ± 0.1	1.5 ± 0.1	0.3 ± 0.0	0.4 ± 0.0	0.6 ± 0.1	0.6 ± 0.0
40	2-tridecanone	593-08-8	MS,STD,RI	1491	1496	-	-	-	-	1.6 ± 0.1	1.6 ± 0.1	-	-	8.1 ± 0.2
42	valencene	4630-07-3	MS,STD,RI	1495	1496	-	-	1.0 ± 0.0	-	-	-	-	-	-
43	α-muurolene	483-75-0	MS,RI	1497	1503	4.2 ± 0.3	4.8 ± 0.1	4.5 ± 0.1	5.1 ± 0.0	4.6 ± 0.1	4.1 ± 0.1	5.3 ± 0.2	3.4 ± 0.1	4.5 ± 0.2
44	Y-cadinene	39029-41-9	MS,RI	1511	1514	tr	0.7 ± 0.1	0.6 ± 0.1	0.9 ± 0.1	0.6 ± 0.1	0.8 ± 0.1	0.8 ± 0.1	0.7 ± 0.0	0.6 ± 0.2
45	δ-cadinene	483-76-1	MS,STD,RI	1519	1524	9.7 ± 0.4	12.2 ± 0.1	9.5 ± 0.2	10.6 ± 0.1	11.5 ± 0.3	7.5 ± 0.1	11.3 ± 0.0	7.3 ± 0.2	10.9 ± 0.0
46	α-calacorene	21391-99-1	MS,RI	1543	1546	-	0.7 ± 0.1	0.7 ± 0.0	0.6 ± 0.1	0.9 ± 0.1	0.7 ± 0.1	1.2 ± 0.1	0.5 ± 0.1	0.9 ± 0.0
47	elemol	639-99-6	MS,RI	1550	1553	1.0 ± 0.0	1.8 ± 0.1	0.9 ± 0.0	1.4 ± 0.1	1.1 ± 0.2	3.0 ± 0.1	3.5 ± 0.0	0.3 ± 0.0	-
48	nerolidol	7212-44-4	MS,RI	1565	1565	4.3 ± 0.1	3.0 ± 0.0	4.6 ± 0.0	4.5 ± 0.3	2.8 ± 0.4	2.3 ± 0.2	1.9 ± 0.1	2.5 ± 0.0	1.3 ± 0.0
49	spathulenol	6750-60-3	MS,RI	1582	1583	-	0.3 ± 0.0	0.5 ± 0.0	0.4 ± 0.1	0.5 ± 0.3	-	-	-	-
50	caryophyllene	1139-30-6	MS,STD,RI	1589	1589	$\textbf{4.3} \pm \textbf{0.1}$	$\textbf{4.3} \pm \textbf{0.0}$	6.4 ± 0.1	3.6 ± 0.1	6.1 ± 0.1	7.5 ± 0.2	$\textbf{8.0} \pm \textbf{0.1}$	5.3 ± 0.1	6.3 ± 0.0
	oxide													
51	viridiflorol	552-02-3	MS,STD,RI	1593	1597	0.5 ± 0.0	0.7 ± 0.1	0.7 ± 0.1	0.6 ± 0.1	0.9 ± 0.1	0.6 ± 0.1	1.0 ± 0.1	0.4 ± 0.1	0.8 ± 0.0
52	Y-eudesmol	1209-71-8	MS,RI	1631	1632	-	1.3 ± 0.0	-	-	1.3 ± 0.0	-	0.6 ± 0.1	-	1.8 ± 0.0
53	ζ-cadinol	5937-11-01	MS,RI	1639	1636	0.7 ± 0.1	1.1 ± 0.1	1.0 ± 0.1	1.0 ± 0.1	1.4 ± 0.0	1.1 ± 0.0	1.9 ± 0.1	0.6 ± 0.1	1.4 ± 0.1
54	δ-cadinol	19435-97-3	MS,RI	1643	1645	0.5 ± 0.0	0.9 ± 0.1	0.8 ± 0.1	0.8 ± 0.1	1.0 ± 0.0	0.8 ± 0.0	1.3 ± 0.1	-	1.3 ± 0.1
55	β-eudesmol	473-15-4	MS,RI	1651	1652	0.4 ± 0.0	1.5 ± 0.0	0.8 ± 0.1	0.8 ± 0.1	1.6 ± 0.0	0.8 ± 0.0	1.4 ± 0.1	-	-
56	α-eudesmol	473-16-5	MS,RI	1652	1655	0.9 ± 0.0	2.2 ± 0.1	1.6 ± 0.1	1.4 ± 0.1	2.5 ± 0.1	1.7 ± 0.1	2.5 ± 0.0	-	1.9 ± 0.2
57	myristic acid	544-63-8	MS,RI	1759	1759	-	-	-	-	-	-	0.4 ± 0.02	-	-
58	2-pentadecanone.	502-69-2	MS,RI	1842	1844	0.3 ± 0.0	0.7 ± 0.0	0.6 ± 0.0	-	0.9 ± 0.0	0.5 ± 0.0	1.0 ± 0.0	0.5 ± 0.0	1.5 ± 0.0
	6.10.14-trimethyl													
59	palmitic acid	57-10-3	MS,STD,RI	1942	1947	-	-	-	-	5.3 ± 0.2	-	5.3 ± 0.3	-	-
60	isophytol	505-32-8	MS,RI	1944	1940	-	-	0.4 ± 0.0	-	-	0.3 ± 0.0	-	-	-
61	phytol	150-86-7	MS,RI	2112	2116	2.0 ± 0.1	1.4 ± 0.1	4.6 ± 0.0	0.3 ± 0.0	3.3 ± 0.3	2.5 ± 0.3	1.5 ± 0.1	1.3 ± 0.1	2.4 ± 0.0
Hve	drocarbons monoterp	oenes (%)				12.2	3.2	1.8	13.5	0.0	6.1	0.6	6.6	0.5
•	genated monoterpen	. ,				5.0	7.3	5.8	4.3	7.9	10.3	6.3	21.5	9.9
Hydrocarbons sesquiterpenes (%)						67.1	69.3	68.4	67.1	60.5	60.2	61.7	59.7	61.5
-	genated sesquiterpen					12.5	16.9	17,4	14.4	19.1	18.7	22.1	9.0	14.8
Diterpenes (%)						2.0	1.4	5.0	0.3	3.3	2.8	1.5	1.3	2.4
	ers (%)					1.1	1.8	1.6	0.3	9.1	1.9	7.8	1.9	10.9
	tified compounds (%	5)				> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9	> 99.9

 N° CAS: Cas Number; Id: Identification methods; MS: comparison of mass spectra with those of Pal 600K \circledast librairies; STD: comparison of retention time and mass spectra of available standards; RI: comparison of retention index with those reported in the literature; Ir th: retention index taken from NIST 08 or PubChem; Ir cal: retention index on HP-5MS capillary column, experimentally determined using series of C7–C30 alkanes; tr: trace; -: not found.

3.3. Essential oil composition and variability

Generally, essential oil composition can be affected by internal (developmental and genetic) and external (environmental) factors (Rajabi et al., 2014; Rebey et al., 2019). Thus, weather parameters such as temperature and rainfall, and ecological conditions such as soil composition, may noticeably affect essential oil yield and composition (Rebey et al., 2018; Zhang et al., 2015). On the basis of the current study, sesquiterpenes were predominant in all essential oils obtained from *L. rhodesiensis* from the Korhogo area in Northern Côte d'Ivoire. β -caryophyllene was the major compound in essential oils hydrodistillated from leaves, fruits and stems. This sesquiterpene hydrocarbon, which exhibits several biological activities, is used as a hepatic protector (Cho et al., 2015), anxiolytic, antidepressant (Bahi et al., 2014), and neural protector (Chang et al., 2013). It is also used as a functional food and dietary supplement due to its antioxidant properties (Calleja et al., 2013; Pant et al., 2014). The high β -caryophyllene content of essential oils hydrodistillated from *L. rhodesiensis* organs encourages a more extensive study of the properties of this plant in traditional medicine.

The present study showed a considerable difference in the chemical composition of essential oils of *L*. *rhodesiensis* from Côte d'Ivoire, compared to those from Tanzania and Kenya. Notably, many prevalent compounds from Ivorian leaf oil, such as β -caryophyllene, α -copaene and thymol, were absent from the essential oil studied in Tanzania. Indeed, the Tanzanian oil was dominated by camphor (36.5%), 4-thujanol (18.5%) and cineole (11.0%; Chogo et al., 1982), which were absent or present in very low proportions in samples from Côte d'Ivoire. In addition, β -cubebene (6.5%), the most abundant sesquiterpene in Tanzania, was not detected in Ivorian leaf oil. Similarly, essential oil obtained from whole aerial parts from Kenya was dominated by camphor (39.84%) and camphene (8.63%), compounds which were only detected in trace amounts in the present study.

Slight variations in chemical composition can be frequently observed between samples from different geographical areas (Benini et al., 2012a; Dodoš et al., 2019); however, when the differences are significant, the term chemotype can be used. These are defined as plants of the same species that produce essential oils of markedly different chemical composition, and thus, biological properties (Torras et al., 2007). On this basis, a new chemotype of *L. rhodesiensis* essential oil from Côte d'Ivoire was defined here and characterised by its high levels of β -caryophyllene, α -copaene, α -humulene, δ -cadinene and thymol. This chemotype was different to those from Tanzania (camphor, 4-thujanol and 1,8-cineole) and Kenya (camphor and camphene). Many studies have focused on essential oil chemotypes obtained from diverse plants; for example, common thyme, *Thymus vulgaris*, is a source of polymorphism, with six different chemotypes containing thymol, geraniol, linalool, carvacrol, 4-thujanol/terpinen-4-ol and borneol (Amorese et al., 2018; Glamoclija, 2017; Kaloustian et al., 2005; Rota et al., 2008; Thompson et al., 2003; Torras et al., 2007). Another intensively studied plant species with respect to essential oil polymorphism is tansy (*Tanacetum vulgare*). The chemotype of essential oil extracted from Belgian plants of this species is β -thujone, chrysanthenyl acetate, camphor and thujone (de Pooter et al., 1989), whereas that from Norway is thujone, chrysanthenyl acetate, camphor, and thujone (Dragland et al., 2005; Roholff et al., 2004).

The essential oils hydrodistillated from organs of Ivorian *L. rhodesiensis* exhibited compounds that were not identified in the Kenyan oil. These compounds included: δ -cadinene, nerolidol, thymol and α -humulene, which is a sesquiterpene with an anti-inflammatory effect (Rogerio et al., 2009). The observed chemical composition difference between essential oils from Côte d'Ivoire and those from Kenya and Tanzania was probably related to various factors, such as environmental conditions, plant genotype, geographic origin, organs harvested, harvesting period and extraction method (Benini et al., 2012a; Filly et al., 2016). For example, leaves, flowers and whole aerial parts used for essential oil production in Kenya were dried in the shade for one week before hydrodistillation (Omolo et al., 2004) while hydrodistillation was performed the day after harvesting in the present study. A previous study demonstrated the effect of harvesting site on essential oil composition, as ylang-ylang (*Cananga odorata*) essential oils with different origins (Grande Comore, Mayotte, Nossi Bé and Ambanja) displayed significantly different compositions (Benini et al., 2012b). It would be interesting to study *L. rhodesiensis* essential oils elsewhere, in order to find other chemotypes in other countries. In addition, an extensive genetic and enzymatic study of terpene synthesis pathways would help to elucidate whether *L. rhodesiensis* essential oils variability was due to environmental conditions and/or plant genotype. On a similar note, vegetatively propagated *L. rhodesiensis* from Kenya and Tanzania could be cultivated in Côte d'Ivoire, to allow an analysis of essential oil composition under the same environmental conditions.

The chemical composition of essential oils hydrodistillated from *L. rhodesiensis* stems was different from that of leaves and fruits, which had similar compositions. The variability in the chemical composition of *L. rhodesiensis* essential oils was largely organ specific. Similar variability has already been established for other plants, such as *Cinnamomum zeylanicum*. The major component of leaf essential oil extracted from this plant is eugenol (Singh et al., 2007; Wang et al., 2009), while *trans*-cinnamaldehyde and camphor are the main constituents of bark (El-Baroty et al., 2010; Moarefian et al., 2013) and root essential oils (Paranagama et al., 2001; Wijesekera et al., 1974), respectively.

3.4. Biological activities

As previously mentioned, *L. rhodesiensis* is currently used in traditional medicine for the treatment of various diseases, such as rheumatism and diabetes. In order to understand the basis for these traditional uses of *L. rhodesiensis*, antioxidant and anti-inflammatory activities of essential oils produced from leaves were studied. Two essential oil samples from *L. rhodesiensis* leaves were chosen for each location, on the basis of having the most different profiles in major components. Kapélé samples consisted of leaves collected in June (K.L.Jun) and July (K.L.Jul), while the Nyanbélégé samples were thymol and limonene proportions (Fig. 3).

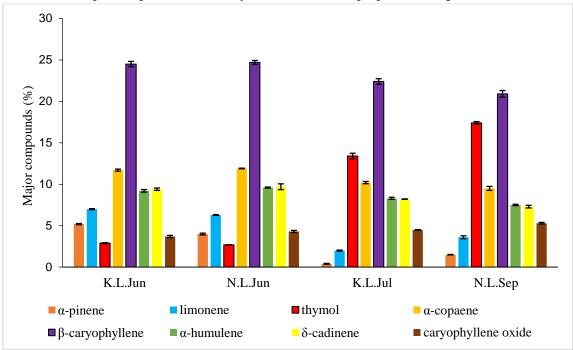


Figure 3. Major compounds of *L. rhodesiensis* leaves essential oils used for the study of biological activities. K.L.Jun: June leaves essential oil of Kapélé; K.L.Jul: July leaves essential oil of Kapélé; N.L.Jun: June leaves essential oil of Nyanbélégé; N.L.Sep: September leaves essential oil of Nyanbélégé.

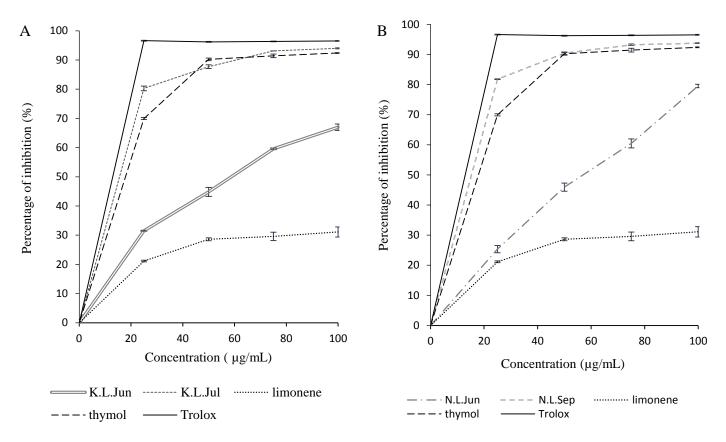


Figure 4. A. Inhibition percentage of DPPH radicals by essential oil from leaves of *L. rhodesiensis* from Kapélé, limonene, thymol and Trolox as a function of concentration; B. Inhibition percentage of DPPH radicals by essential oil from leaves of *L. rhodesiensis* from Nyanbélégé, limonene, thymol and Trolox as a function of concentration.

DPPH assay

Antioxidant activity is dependent on the mobility of the hydrogen atom of hydroxyl groups from phenolic compounds. Transfer of a H atom to a free DPPH radical results in a stable DPPH molecule and a decrease in the concentration of free radicals (Barkat and Laib, 2011).

Results showed that the free radical I% increased with increasing concentrations of Trolox, thymol, limonene and essential oils from *L. rhodesiensis* leaves (Fig. 4). Notably, the inhibition percentage of samples K.L.Jul and N.L.Sep was higher than that of samples K.L.Jun and N.L.Jun. The main differences between these selected oil samples were the proportions of thymol and limonene, with K.L.Jul and N.L.Sep containing high thymol quantities, while limonene was found at higher levels in K.L.Jun and N.L.Jun. Limonene inhibited the DPPH radical very weakly, with an inhibition percentage of $31.10 \pm 1.73\%$ at a concentration of 100 µg/mL. By contrast, the thymol standard showed a high inhibition ability. On the basis of these observations, the high inhibition percentage of samples K.L.Jul and N.L.Sep is probably due to their high content of thymol; however, synergetic effects could not be excluded.

Essential oil hydrodistillated from leaves harvested in September in Nyanbélégé (N.L.Sep) showed the highest antioxidant activity (IC₅₀ 22.57 ± 0.06 µg/mL), which was almost similar to essential oil from leaves harvested in July from Kapélé (IC₅₀ 23.83 ± 0.43 µg/mL; Fig. 4 and 5). Thymol (IC₅₀ 27.21 ± 0.06 µg/mL) displayed lower antioxidant activities than Trolox (IC₅₀ 15.24 ± 0.07 µg/mL), the positive standard with well-known antioxidant properties. By contrast, limonene showed low antioxidant properties, failing to inhibit 50% DPPH at the studied concentrations ($\leq 100 \mu g/mL$).

Essential oils collected in June at the two locations (K.L.Jun and N.L.Jun) had high IC_{50} values, and therefore, low antioxidant activities. This was probably due to low levels of some compounds, such as thymol or carvacrol, which have been proposed to play an important role in the antioxidant activity of essential oils (Güllüce

et al., 2003; Kulisic et al., 2004; Sokmen et al., 2004; Tepe et al., 2005). Indeed, it has been shown that thymol purified from *Carum copticum* oil has a high antioxidant activity (Sameeran and Shamin, 2017).

Results also showed that September oil from Nyanbélégé and July oil from Kapélé had high antioxidant activities (Fig. 5), which could be related to their higher thymol contents. This compound has well-known antioxidant properties (Esmaeili and Khodadadi, 2011), and ANOVA and Tukey's comparison test showed that the high IC_{50} value of limonene was significantly different from that of thymol, which was very low. β -caryophyllene is a sesquiterpene, which has also been shown to possesses free radical scavenging activity using the DPPH assay (Dahham et al., 2015; Dar et al., 2011). As this molecule was not present in higher proportions in K.L.Jul and N.L.Sep oils in comparison with K.L.Jun and N.L.Jun oils, it might not be the cause of the difference in antioxidant activities.

Reducing power determination

The reducing abilities of Trolox, thymol, limonene, K.L.Jun, N.L.Jun, K.L.Jul, and N.L.Sep were investigated by Fe^{3+} to Fe^{2+} transformation. Higher absorbance measurements from the assay indicated greater reducing power. The high reducing power of thymol, K.L.Jul and N.L.Sep correlated well with their marked antioxidant activities (Fig. 6), indicating a possible contribution of reducing power to this activity. Indeed, the reducing power of K.L.Jun and N.L.Jun was lower than that of K.L.Jul and N.L.Sep, which had abundant levels of thymol.

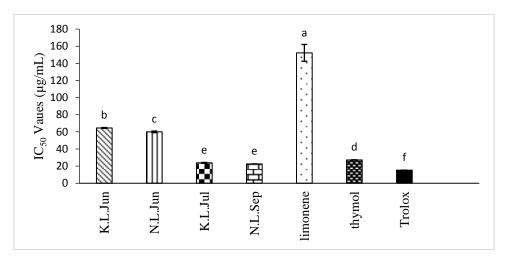


Figure 5. IC_{50} values ($\mu g/mL$) obtained with DPPH assay of leaves essential oils of *L. rhodesiensis*, limonene, thymol and Trolox. Histograms with different letters are significantly different (p-value < 0.05) (n=3).

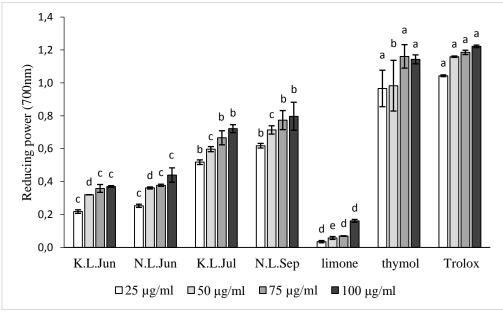


Figure 6. Reducing power of leaves essential oils of *L. rhodesiensis*, limonene, thymol and Trolox at various concentrations. K.L.Jun: June leaves essential oil of Kapélé; K.L.Jul: July leaves essential oil of Kapélé; N.L.Jun: June leaves essential oil of Nyanbélégé; N.L.Sep: September leaves essential oil of Nyanbélégé. Histograms that do not share any letters are significantly different (p-value < 0.05) (n=3).

Essential oils are complex mixtures; however, it appears that associated antioxidant activity is usually related to the presence of phenolic compounds (Hazzit et al., 2009; Yang et al., 2011). Previous reports have shown that oxygenated monoterpenes, such as thymol, carvacrol and α -terpineol, are mainly responsible for the antioxidant potential of plant essential oils (Bicas et al., 2011). In the present study, ANOVA and Tukey's comparison test showed that the assay absorbance for thymol was not significantly different from that of the positive control, Trolox, both of these compounds having high antioxidant activities.

 β -caryophyllene and other molecules with well-known antioxidant properties are also present in essential oils and may contribute to their global antioxidant activity. All of those compounds may interact synergistically or antagonistically to create an effective system against free radicals (Barkat and Laib, 2011).

Anti-inflammatory activity

Arthritis is a generic term used to describe an inflammatory condition that affects one or more joints. Protein denaturation is one of the well-documented causes of inflammation, leading to a variety of inflammatory diseases. Consequently, the ability of a substance to inhibit protein denaturation demonstrates its potential anti-inflammatory activity (Osman et al., 2016; Rahman et al., 2015).

To evaluate the anti-inflammatory activity of essential oils produced from leaves of *L. rhodesiensis*, the model of the denaturation of BSA was used. The principle of this method was based on the ability of oil samples to prevent the thermal denaturation of BSA. Indeed, production of auto-antigens in certain rheumatic diseases may be due to *in vivo* protein denaturation. Mechanisms of denaturation probably involve alterations in electrostatic, hydrogen, hydrophobic and disulphide bonding. Several anti-inflammatory drugs have been shown to have a dose-dependent ability to inhibit thermally induced protein denaturation (Chandra et al., 2012; Grant et al., 1970).

In this current study, the effect of leaf essential oils on the thermal denaturation of BSA was evaluated at various concentrations (Fig. 7), in comparison with a standard drug, diclofenac.

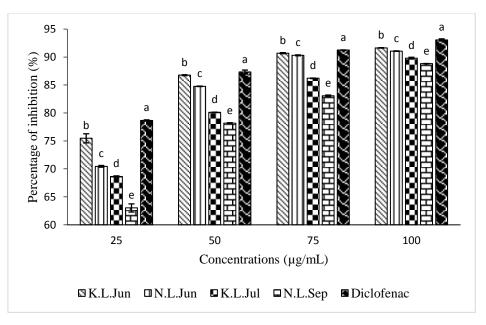


Figure 7. Inhibition percentage of bovine albumin denaturation of leaves essential oils of *L. rhodesiensis* and Diclofenac. K.L.Jun: June leaves essential oil of Kapélé; K.L.Jul: July leaves essential oil of Kapélé; N.L.Jun: June leaves essential oil of Nyanbélégé; N.L.Sep: September leaves essential oil of Nyanbélégé. Histograms that do not share any letters are significantly different (p-value < 0.05) (n=3).

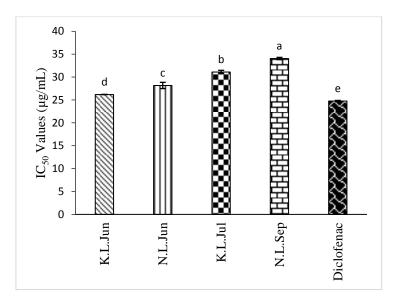


Figure 8: IC₅₀ values (μ g/mL) obtained with the bovine albumin denaturation of leaves essential oils and Diclofenac. K.L.Jun: June leaves essential oil of Kapélé; K.L.Jul: July leaves essential oil of Kapélé; N.L.Jun: June leaves essential oil of Nyanbélégé; N.L.Sep: September leaves essential oil of Nyanbélégé. Histograms that do not share any letters are significantly different (p-value < 0.05) (n=3).

The results showed that the percentage of inhibition rose with increasing essential oil concentrations. Moreover, inhibition percentages for each concentration were higher for K.L.Jun and N.L.Jun in comparison to K.L.Jul and N.L.Sep. The essential oils from Kapélé leaves denatured the protein at 91.64 \pm 0.04% for the June sample and 89.80 \pm 0.16% for the July sample at a concentration of 100 µg/mL. Likewise, samples of essential oils at the same concentration from Nyanbélégé leaves showed an inhibition percentage of 91.06 \pm 0.04% for leaves harvested in June and 88.83 \pm 0.03% for those harvested in September. IC₅₀ values of the essential oil samples and diclofenac were determined (Fig. 8).

Comparison of the IC₅₀ values showed that the effect of diclofenac was close to that of N.L.Jun and K.L.Jun. Diclofenac had an IC₅₀ of 24.76 \pm 0.11 µg/mL, while that for N.L.Jun and K.L.Jun was 28.16 \pm 0.69 µg/mL and 26.18 \pm 0.05 µg/mL, respectively. Those results were confirmed by ANOVA followed by Tukey's multiple comparison test (Fig. 8). K.L.Jun and N.L.Jun had significantly higher proportions of α -humulene and β -caryophyllene in comparison to K.L.Jul and N.L.Sep. Anti-inflammatory properties of these molecules have previously been demonstrated in essential oil from *Cordia verbenacea* (Fernandes et al., 2007), and the higher anti-inflammatory activity of K.L.Jun and N.L.Jun was probably due to their high content of α -humulene and β -caryophyllene. Also, K.L.Jun and N.L.Jun have higher proportions of limonene than K.L.Jul and N.L.Sep. As it has already been shown that limonene extracted from *Citrus junos* Tanaka displays high anti-inflammatory activities, it is possible that essential oil anti-inflammatory properties are also due to the presence of this molecule (Hirota et al., 2010). The results of this study demonstrated that the essential oils of *L. rhodesiensis* leaves were promising candidates for inhibiting protein denaturation in inflammatory diseases, such as rheumatic diseases. As *in vitro* effects are sometimes different to *in vivo* activities (Donadu et al., 2018; Niki, 2010), *in vivo* assays of the antioxidant and anti-inflammatory activities of these essential oils will be needed.

4. Conclusions

The variability in the composition of essential oils hydrodistillated from leaves stems and fruits of *L. rhodesiensis* growing in two Ivorian locations was studied over four months. Sesquiterpenes were the predominant molecules in all samples, with β -caryophyllene as the major compound. In this study, a new chemotype of *L. rhodesiensis* essential oils was discovered and was characterised by a mixture of β -caryophyllene, α -copaene, α -humulene, δ -cadinene and thymol. This chemotype differed from essential oils for this same species in other countries. Statistical analysis showed that the the variability in the volatile composition of essential oils mainly depended on the plant organ from which it was extracted. Results also highlighted the high antioxidant and anti-inflammatory activities of essential oils extracted from leaves. Based on these activities, essential oils hydrodistillated from

leaves of Ivorian *L. rhodesiensis* have a high potential for a use in traditional medicine for the treatment of many conditions. However, the toxicology of essential oils also needs to be evaluated before they are used in human care. In addition, if this wild plant is extensively used for the distillation of essential oils, it will be important to set up conservation measures, such as the establishment of plant nurseries.

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