C. R. Chimie xxx (2016) 1–5

Contents lists available at ScienceDirect

Comptes Rendus Chimie

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Seasonal variations of volatile constituents of *Hemizygia bracteosa* (Benth.) Briq. aerial parts from Benin

Variations saisonnières de constituants volatils de la partie aérienne de Hemizygia bracteosa *(Benth.) Briq. du Bénin*

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ARTICLE INFO

Article history:
Received 30 October 2015
Accepted 17 March 2016
Available online xxxx

Keywords:
Hemizygia bracteosa
Lamiacea
Essential oil composition
Seasonal variations
(*E*)-β-Farnesene
β-Elemene.

ABSTRACT

Essential oils from fresh aerial parts of *Hemizygia bracteosa* (Benth.) Briq. were extracted by steam distillation. The oil yield from plants collected during the hot season (February) and during the cold season (August) were 0.12 ± 0.01% and 0.25 ± 0.02%, respectively. GC/FID and GC/MS analyses allowed us to identify a total of 65 compounds, representing 97% of the hydrodistillate. The main components of the oil from the hot period were (*E*)-β-farnesene (64 ± 0.04%), β-elemene (7.4 ± 0.05%), *trans*-nerolidol (6.2 ± 0.04%), and α-muurolene (2.7 ± 0.03%). The essential oil from the cold season was characterized by the presence, as major compounds, of (*E*)-β-farnesene (67 ± 0.04%) along with β-caryophyllene (3.6 ± 0.06%), β-elemene (3.3 ± 0.05%), 7-epi-α-selinene (3.1 ± 0.01%) and *p*-cymene (2.5 ± 0.04%). This is the first report of these components in the essential oil of *Hemizygia bracteosa* (Benth.) Briq.

Mots clés:
Hemizygia bracteosa
Lamiacea
Huile essentielle
Variation saisonnière
(*E*)-β-farnesène
β-élémène

RÉSUMÉ

Les huiles essentielles de parties aériennes fraîches de *Hemizygia bracteosa* (Benth.) Briq. ont été extraites par distillation à la vapeur. Les rendements en huile de la plante récoltée pendant la saison chaude (février) et la saison froide (août) étaient respectivement de 0,12 ± 0,01% et de 0,25 ± 0,02%. Les analyses GC/FID et GC/MS ont permis d'identifier 65 composés, représentant 97% de l'hydrodistillat. Les principaux composants de l'huile obtenue dans la période chaude étaient : (*E*)-β-farnésène (64 ± 0,04%), β-élémène (7,4 ± 0,05%), *trans*-nérolidol (6,2 ± 0,04%) et α-muurolène (2,7 ± 0,03%). L'huile essentielle de la saison froide est caractérisée par la présence, comme composés majoritaires, de

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http://dx.doi.org/10.1016/j.crci.2016.03.013
1631-0748/

(*E*)-β-farnésène (67 ± 0,04%), β-caryophyllène (3,6 ± 0,06%), β-élémène (3,3 ± 0,05%), 7-épi-α-selinène (3,1 ± 0,01%) et *p*-cymène (2,5 ± 0,04%). C'est la première observation de ces composants dans l'huile essentielle de *Hemizygia bracteosa* (Benth.) Briq.

1. Introduction

Hemizigia bracteosa (Benth.) Brip. (ex *Orthosiphon bracteatus* (Benth.) Baker) (Lamiaceae) is an erected annual and sometimes perennial herb of about 90 cm tall, widespread in tropical and South Africa, in marshy grasslands in Senegal, Benin (ex Dahomey), North and South Nigeria and West Cameroon [1]. The flowers are white and fairly inconspicuous but the colorful purple of its bracts makes the plant a striking feature [2].

In Southern Africa the plants are burnt and the smoke or vapors inhaled to treat mental illnesses, for narcotic or divination purposes [3]. The leaves are smoked or chewed by the San in Botswana to give energy for dancing and as a stimulant [4]. The Shonas of Zimbabwe are reported to use powdered leaves orally to treat fits [5]. In Zimbabwe, the plant is used in association with other plants for treating or preventing HIV infection in general and maybe to reduce the viral load of patients infected with HIV and/or exhibiting symptoms of acquired human immunodeficiency syndrome (AIDS) [6]. The plant is very useful in West Africa [7]. It is used to treat malaria, to foment the body of patients suffering from fever and its leaves are used as a mosquito repellent [8]. The fresh aerial parts of the plant are also traditionally used by fumigation in Benin and the decoction of the leaves in addition with *Dialium guineense*, *Pavetta corymbosa*, *Rytigynia canthiodes* and *Uvaria chamae* is used orally to treat malaria [9,10]. Leaves are also used in drink preparation and were shown to possess some antimicrobial activities [11].

To the best of our knowledge there are no reports concerning the volatile components of the aerial parts of *Hemizygia bracteosa* (Benth.) Briq. The aim of this study is to describe the chemical composition of essential oils extracted from fresh aerial parts of this plant from Benin and the impact of the harvesting period on this chemical composition and on the essential oil extraction yield.

2. Experimental

2.1. Plant material

Aerial parts of *Hemizygia bracteosa* (Benth.) Briq. were collected in the morning, in the Botanical Garden of the Abomey-Calavi University (Republic of Benin). The fresh aerial parts were harvested in February 2009 (sample I), a period of very hot weather (35 °C), and in August 2009 (sample II) (21 °C), a colder period with occasional light rain. A voucher specimen (n°AA6391/HNB) of these aerial parts has been deposited at the University of Abomey-Calavi Herbarium.

2.2. Essential oil isolation

Five hundred grams (500 g) of fresh aerial parts were steam distilled for 3 h in an improved Clevenger-type apparatus [12]. The extraction of each aerial part (I and II) was carried out in triplicate. The essential oil yields were based on the fresh material.

2.3. Chemical analysis

Analysis of the oils was performed by GC/FID and GC/MS [13,14].

2.3.1. GC/FID analysis

The GC/FID analysis was carried out on a FOCUS GC (ThermoFinigan; Milan, Italy) using the following operating conditions: A DB5 column (25 m × 0.25 mm, df: 0.25 μm) (J&W Scientific Column of Agilent Technologies, N° US167072Ã, USA); injection mode: splitless; injection volume: 1 μL (TBME solution); flow of split: 10 ml/min; splitless time: 0.80 min; injector temperature: 260 °C; oven temperature was programmed as following: 50 °C – 250 °C at 6 °C/min and maintained at 250 °C for 5 min; carrier gas was helium with a constant flow of 1.2 mL/min; FID detector temperature was: 260 °C. The data were recorded and treated with the ChromCard software. The quantification was completed by the calculation of the areas under curve of the peaks (GC/FID, by the normalization process) and the identification of compounds by comparison of the retention indices with references.

2.3.2. GC/MS analysis

GC-MS analysis was carried out on a TRACE GC 2000 series (Thermo-Quest, Rodano, Italy), equipped with an autosampler AS2000 Thermo-Quest. The GC system was interfaced to a Trace MS mass spectrometer (ThermoQuest) operating in the electronic impact mode. A HP5 column (30 m × 0.25 mm, df: 0.25 μm) was used under the same operating conditions as above. The coupling temperature of the GC was 260 °C. The energy of the electrons was 70 eV and the source of the electrons at 260 °C. The data were recorded and analyzed using the Xcalibur 1.1 software (Thermo-Quest). The mass spectra of the peaks were analyzed and compared with references and NIST/EPA/NIH database [15].

2.4. Identification of oil constituents

Individual components of the volatile oils were identified by comparison of their relative retention times with those of authentic standard references, computer matching against commercial library [15,16] and home-made library mass spectra made from pure substances and components of known oils [14]. Mass spectrometry literature data were also used for the identification, which was confirmed by comparison of the GC retention indices (RI) on a non-polar column (determined from the retention times of a series of *n*-alkanes "C8-C24" mixture). The Kovats indices (KI)

Please cite this article in press as: S. Kpoviessi, et al., Seasonal variations of volatile constituents of *Hemizygia bracteosa* (Benth.) Briq. aerial parts from Benin, Comptes Rendus Chimie (2016), http://dx.doi.org/10.1016/j.crci.2016.03.013