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Chemical Composition of the Essential Oil of *Warionia saharae* from Morocco

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Abstract: Air-dried aerial parts of wild *Warionia saharae* were collected in Tata region (south of Morocco) during June 2005. The plant samples were distillated by Clevenger apparatus and the obtained essential oils were analyzed by GC and GC-MS. The yield obtained varies greatly with a range of 0.5 to 1.1%. Thirty compounds amounting 91% of the oil, were identified. The major components of *Warionia saharae* essential oils were β-eudesmol (52.7%), *trans*-nerolidol (17.4%), linalool (5.1%), guaiol (2.4%), terpinen-4-ol (1.4%) and 1, 8-cineole (1.2%).

Key words: W*arionia saharae;* Compositae, Oil yields, Essential oil composition.

Introduction: The *Warionia saharae* which belongs to the important compositae's family is an endemic species of North Africa, characterized by a discerning odour[1-3]. *Warionia saharae* was reported for the first time in the Oranais sahara (Beni ounif in Algeria) by Dr. Warrion as a shrub of 1 to 3 m of height. The thick trunk, is covered of a gray peel, structural of very wavy terminal leaf bouquets, and of capitulate of yellow flowers. The picking of stems leafed of this bush, clear a very heady and spicy odour; the latex that flows out of injuries of the peel, glue to hands in a very tenacious way[4].

In Morocco, *Warionia saharae* is growing wild in various regions[5, 6]. The habitat is between schistose rocks[7,8]. *Warionia saharae* is known in Morocco by the berber vernacular names of "âfessas"; "âfezded" and "Tazart n-îfiss" [9]. In the Moroccan traditional medicine, the leaves of the plant are used to treat inflammatory diseases, such as rhumatoid arthritis, and for gastrointestinal disorders[9].

The chemical composition of *Warionia saharae* essential oils from the leaves was reported for the first time by Ramaut *et al*.[10]. The authors have isolated and identified only

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3 constituents: eudesmol (42.25%), linalool (8.63%) and nerolidol (17.26%). The chemical composition of the dichloromethane extract of *Warionia saharae* leaves has been investigated[11-14]. Twelve new guaianolide type sesquiterpene lactones were identified.

Recently, the chemical composition of the hexanoic extract prepared using Soxhlet apparatus from Moroccan *Warionia saharae* leaves was reported for the first time by Elamrani *et al*[15]. The authors found that the major components of the extract were hexadecanoïc acid (17.8%), ethenyloxy-1-octadecane (9.5%), tridecene (7.3 %), eicosene-9 (6.7%), octadecanoïc acid (6,7 %), (E)-2-decenol, (6.7%), eicosene-3 (5.1 %) and eicosane (4.5%).

In continuation of our investigation of Moroccan aromatic flora, the aim of this study is to examine particularly the detailed chemical composition of *Warionia saharae* essential oil because no information could be found, in which these chemical composition and physical properties were established before for this species. To the best of our knowledge, the detailed chemical composition of the essential oils of this species is being reported here for the first time.

Experimental

Plant Material: The samples of *Warionia saharae* were collected in June 2005 from Tata region (Douar Targant at 760 Km south of Rabat). In this region, fives places with a surface of 20 m x 20 m were delimited. In each place, six individual samples of the whole plant were collected and were mixed to obtain a heterogeneous sample (about 5Kg). Therefore, five main heterogeneous samples were obtained and were treated separately. The plant material was identified according to the flora of Morocco[16] and also by Pr. M. Rejdali and Pr. A. Achhal, Agronomic Institute and Veterinary Hassan II, Rabat (Morocco). A voucher specimen is deposited in the Herbarium of the Department of Botany and Ecology at the Agronomic Institute and Veterinary Hassan II, Rabat (Morocco) and also in our laboratory at the Chemistry Department at the Science Faculty Ain chock, Casablanca (Morocco)[15].

Isolation of Essential oil: After air-drying in the shade for a week, the plant material (leaves, flowers and twigs) was subjected to hydrodistillation using a Clevenger-type apparatus for 3 hours. In parallel to each distillation we determined the moisture of the air dried leaves: two samples (5g each) were oven dried (104°C) for 4 h. The oil yields were expressed in dry matter percentage.

The oils, which were separated from water by decantation, were dried by filtration over anhydrous sodium sulphate. The oils obtained were light yellow with a strong odour. The physicochemical characteristics of the oils were determined according to the AFNOR[17] standards at 20^0C. The specific gravity, the refractive index, the optical rotation and the solubility in alcohol, were determined using respectively, the AFNOR norms: NF T 75-111, NF T 75-112, NF T 75-113, NF T 75-101.

Oil Analysis: The different *Warionia saharae* essential oils were analysed by gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS).

GC: The analysis was carried out using an HP 5980 gas chromatography apparatus equipped with FID and two capillary columns DB-5 and CW20M (25 m x 0.25 mm, film thickness 0.25 μm). Analytical conditions were: injector and detector temperature 240 and 260^{0}C respectively, oven temperature programmed from 50 to 250^{0}C at 4^{0}C/min. Isothermal temperature at 250^{0}C for 30 min; carrier gas 1 ml N2/min. Relative concentrations were calculated using peak areas as given by HP 3396A integrator, without correction for response factors. Retention indexes were obtained by injection of the homologous hydrocarbons series C_8 - C_{30} in the same conditions (programmed temperature) in two capillary columns (DB-5 and Carbowax 20M).

GC-MS: The analysis was done using an HP 5980 Series II gas chromatograph equipped with HP-5 capillary column (25m x 0.3mm; film thickness 0.25 μm) and an HP 5772A mass selective detector. Analytical conditions were: injector and detector temperatures: 240 and 260^{0}C respectively; oven temperature programmed from 50 to 250^{0}C at 4^{0}C/min, then isothermal at 250^{0}C for 10 min; carrier gas 2 ml He/min; source 70 eV. The constituents were identified by the combination of retention index data and mass spectra data using NBS library and other literature data[18, 19.]

Results and discussion: The oil yields and physicochemical properties of the Moroccan *Warionia saharae* species can be seen in Table 1. Examination of these results shows that the oil content varies greatly with a range of 0.5 to 1.1%. A survey of the literature reveals that our results are similar to those reported by Ramaut *et al*.[10]. The authors studied the essential oil of *Warionia saharae* and reported that the yield was 0.9 %.

Table 2 lists the retention indices and percentage composition of the compounds identified in the Moroccan *Warionia saharae* essential oils. A total of 30 compounds, amounting 91% of the oils, were identified. The major components of *Warionia saharae* essential oils were β-eudesmol (52.7 %), *trans*-nerolidol (17.4%), linalool (5.1%), guaiol (2.4%), terpinen-4-ol (1.4%) and 1,8-cineole (1.2%).

In comparison to the alone result previously reported in the literature for the oils of *Warionia saharae*, there were considerable differences qualitatively and quantitatively. Ramaut *et al*.[10] identified only 3 compounds such as eudesmol (42.25%), linalool (8.63%) and nerolidol (17.26%). The same components were also found in our oils. The percentage of β-eudesmol (52.7 %) in our oils was particularly higher than those mentioned by Ramaut *et al*.[10] . In addition, twenty-seven constituents were found in our essential oils and have not been identified by Ramaut et *al*.[10]. As far as we know, these twenty-seven components have not been previously described in the *Warionia saharae* oil in the literature.

The percentage composition of the different groups of compounds in *Warionia saharae* essential oils was also studied (Table 2). We observed that the hydrocarbon fraction represented 8.7% of the total oil composition, while the oxygenated fraction represented 82.4%.

We conclude from this study that the Moroccan *Warionia saharae* essential oil is characterized by higher percentage of oxygenated components particularly the β-eudesmol (52.7%).

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Table 1. Physicochemical properties of Moroccan *Warionia saharae* essential oils

Determination at 20°C Yield (%)	0.5 to 1.1
Specific gravity	0.954
Refractive index	1.5601
Optical rotation	+ 9° 30’
Solubility in alcohol	0.43 vol at 70%

- The values of the yield given were obtained from 10 determinations: 5 samples and two distillations each
- The values of the physicochemical properties given represent the average of two determinations

Table 2: Chemical composition of Moroccan *Warionia saharae* essential oils

Compounds	Retention Index IK (DB-5)	Retention Index IK (CW 20M)	Mode of identification	Percentage (%)
α-Thujene	928	1030	a,c	0.7
α-Pinene	938	1033	a,c	0.8
Camphene	952	1085	a,b,c	0.9
Sabinene	976	1370	a,c	0.2
β-Pinene	980	1113	a,c	0.3
β-Myrcene	993	1150	a,c	0.6
α-Terpinene	1018	1176	a,c	0.3
p-Cymene	1026	1270	a,c	0.5
Limonene	1031	1210	a,c	0.8
β-Phellandrene	1032	1216	a,b,c	0.6
1,8-Cineole	1033	1230	a,c	1.2
γ-Terpinene	1062	1250	a,b,c	0.1
Menth 2-en-1-ol (*cis*-para)	1068	-	a,c	0.5
Terpinolene	1089	1299	a,c	0.1
Linalool	1098	1550	a,c	5.1
Terpinen-4-ol	1178	1613	a,c	1.4
α-Terpineol	1189	1710	a,c	0.5

table 1. (continued)

Compounds	**Retention Index IK (DB-5)**	**Retention Index IK (CW 20M)**	**Mode of identification**	**Percentage (%)**
cis-Piperitol	1194	-	a,b,c	0.5
Nerol	1228	1804	a,c	0.4
α-Terpinyl acetate	1351	1705	a,c	0.2
β-Caryophyllene	1419	1607	a,c	0.5
β-Farnesene	1460	-	a,b	0.2
allo-Aromadendrene	1462	1650	a,c	0.1
β-Selinene	1485	1737	a,b,c	0.3
β-Bisabolene	1509	-	b,c	0.6
δ-Cadinene	1524	-	a,c	0.8
Nerolidol (*trans*)	1564	2000	a,c	17.4
Caryophyllene oxide	1581	-	a,c	0.4
Guaiol	1595	-	a,b,c	2.4
β-Eudesmol	1636	2185	a,b,c	52.7
Oxygenated fraction				82 .4
Hydrocarbon fraction				8 .7
Total				91.1

a = Comparison of our MS data with NBS75K library data;
b = Comparison of our MS data with literature data (Adams);
c = Comparison of our RI data with literature data (Lasseve data base, Chicoutimi Univ., Quebec, Canada).
- Compounds are listed in order of their elution from a DB5 column