

GC/MS Analysis of essential oil composition of *Lobularia maritima* (L.) Desv. (Brassicaceae) from Western Algeria

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ABSTRACT

For the first time in Algeria, the quality and variability of the chemical composition of the essential oil isolated from the aerial parts of *Lobularia maritima* (L.) Desv. (Brassicaceae) were studied in two ecologically different regions of Western Algeria. The yields obtained by hydrodistillation are 0.11% for the region of Tessala (wilaya of Sidi Bel Abbes), 0.19% for Beni-Saf (wilaya of Ain-Temouchent). Analysis by GC and GC/MS allowed us to identify 62 compounds in the essential oil from the Beni-Saf region and 54 in that of Tessala. The majority compounds in two regions show quantitative variability and are, respectively, linalol (24.78%; 26.95%), eucalyptol (9.12%; 11.57%), linalyl acetate (18.90%; 20.85%), cis-dihydro-terpinyl acetate (5.38%; 9.73%), estragole (4.26%; 3.99%), α -terpineol (3.39%; 4.48%), geranyl formate (1.93%; 2.21%), terpineol, 4-o1 acetate (1.88%; 2.32%), β -myrcene (1.84%; 1.89%), β -pinene (1.24%; 1.05%) and e- β -ocimene (1.16%; 1.94%).

KEY WORDS

Lobularia maritima; aerial part; essentials oils; chemical composition; Western Algeria.

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INTRODUCTION

Lobularia maritima (L.) Desv. (Brassicaceae) is a perennial, ornamental and halophytic herbaceous plant widespread on dunes and coastal rocks in the Mediterranean basin. It has a slightly woody base, and grows spontaneously in the Western Algerian region (Lim, 2014; Tassin, 2012). According to Bellakhdar (1997), the Brassicaceae family is the object of a big use in traditional medicine. Plants of this family are well known for their anti-fungal (Terras et al., 1993; Schreiner & Koide, 1993), anti-inflammatory (Saito et al., 1996; Yu-Ling Ho & Yuan-Shiun Chang, 2002; Stocker et al., 2004), antirheumatic and antidiabetic (Kir-

tikar & Basu, 1975) and anti-oxidants properties (Saito et al., 1996; Marles et al., 2003).

Another medicinal property attributed to the Brassicaceae family is its anticancer effect (Fahey et al., 2001; Shukla et al., 2004; Font et al., 2005). This activity is mainly due to the presence of glucosinolates and in particular Indol-3-Carbinol compound resulting from glucobrassicinin hydrolysis (Seong-Joong et al., 2003).

In addition, Brassicaceae contain compounds called isothiocyanates, these substances have several biological and pharmacological properties explaining their benefits on human health (Adjele Eli Wilson, 2011).

Lobularia maritima is commonly used in

Spain as an antiscorbutic, diuretic and an astringent (Ram Nath Chopra, 1956), used in Essaouira as a febrifuge (Gattefossé, 1921). It is used in the treatment of lung diseases (Louis, 1984).

This annual bears many small, fragrant white flowers and has much branched vegetation, usually in abundant tufts. Its small, deep green leaves with grey, alternating and lanceolate reflections are more or less thick and hairy depending on the climate (Coste, 1937; Burnie et al, 2011). *Lobularia maritima* has flowers almost 10 months out of 12 with a flowering peak in autumn (Picó & Retana, 2001).

This research project aims to broaden knowledge on the chemical level, especially as a taxon reservoir, of diverse and important active substances in traditional medicinal uses on the *L. maritima*.

The choice of this plant was guided at first on the characterization of *L. maritima* native to Algeria, through the analysis of essential oil chemical composition extracted from aerial parts of this species, to highlight its chemical profile and to specify its chemical type(s). Sampling was carried out in two ecologically different regions of western Algeria.

Very little research has been done on the extraction and identification of the phytochemical composition of *L. maritima* essential oils. However, this species is being studied in Egypt and Tunisia. The various chemical compositions were probably provided depending on the environmental conditions, the stage of maturity and the harvesting period.

MATERIAL AND METHODS

Study area and collecting samples

The plant material was randomly collected in December 2018 in two Western Algeria localities: the region of Tessala (Sidi Bel Abbes) and the region of Beni-Saf (Ain Temouchent).

The analytical samples consist on the flowering aerial parts of *Lobularia maritima*, the quantity of 50 g was dried in the open air, protected from light for a full week. The essential oil was prepared by hydrodistillation, during 4 hours, of the plant aerial parts using Clevenger type equipment.

Essential oils extracting method

The extraction method adopted to extract essential oils from the flowering aerial parts is hydrodistillation with Clevenger type equipment. The assembly used consists of a metal tank containing 1 liter of water, placed above a heat source and surmounted by an enclosure containing the plant material placed on a grid. The enclosure is in turn connected to refrigerants which condense the vapours collected in the form of a distillate in a separating flask. This phase is dried on magnesium sulphate. The resulting essential oil sample is stored at -18 °C until analyzed. The essential oil yield of the two samples is expressed in %, by the amount of essential oil obtained for 50 g of dry matter.

Essential oils analysis

For the *L. maritima* essential oils analysis, we implemented the gas chromatographic method coupled with mass spectrometry (GC-MS). This analysis was carried out at the laboratory of Catalysis and Synthesis in Organic Chemistry LCSCO, Center for Scientific and Technical Research in Physico-Chemical Analyses (Crapc-Expertise of Tlemcen). The use of CM/MS was favoured under the following operating conditions.

Gas chromatographic analyses were carried out using a Bruker Scionsq device, a single-quadrupole mass spectrometer (SQ: single quadrupole), a divider injector, a capillary column of 25 m x 0.22 mm internal diameter (film thickness: 0.25 µm), The temperature of the ion source is set to 280 °C. The fragmentation is carried out in electronic impact under a field of 70 eV, scanning 35–600 Da.

The analytical conditions are as follows: a capillary column of type DB-5, polymethylsiloxane; the carrier gas is helium (1 ml/min). The injector temperature is 250 °C, the detector temperature is 250 °C; the linear temperature is programmed 50 °C for 10 min, after 50 °C to 250 °C, at 2 °C/min and then it is held at 250 °C for 15 min. Manual injection: 0.2 µl in Split mode (1/100).

Compounds identification

The essential oils constituents in the samples of the two regions were identified referring to their Kovats index at the Catalysis and Synthesis in Or-

ganic Chemistry LCSCO (Crapc-Expertise, Tlemcen) laboratory with the collaboration of the Physicochemical Analysis (Crapc) Research Center depending on the delegated ministry in charge of scientific research (Bou Ismaïl, Algiers).

The Kovats indexes (KI) are calculated in relation to the retention times of the different constituents (easily identified on chromatograms) and the retention times of a series of linear alkanes (C10–C11–C12–...–C28) (Van Den Dool & Kratz, 1963) according to the following formula:

$$KI = (100 \times z) + 100 \cdot \frac{tr_x - tr_z}{(tr_z + 1) - tr_z}$$

KI: Kovats index; z: alkane carbon number; tr_x : retention time of compound x of the essential oil; tr_z : retention time of the alkane preceding tr_x ; ($tr_z + 1$): retention time of the alkane following tr_x .

RESULTS AND DISCUSSION

Quality and average yield of the isolated essential oil

The isolated essential oil from *L. maritima* aerial flowering parts has pale yellow color and has a strong odor. The yield is calculated in relation to the dry matter; the most important yield is that of Beni-Saf region with 0.19%, while the yield of essential oil of the region of Tessala is 0.11%. Comparable results describing yield variability are present in scientific literature, particularly in Tunisia. Thus, Ben Hsouna et al. (2019) obtained a yield of 2.4g/1000g for the essential oil extracted from the aerial parts of the Chebba region, Mahdia (Tunisia).

Essential oils chemical composition

The GC/MS identified the main aerial parts essential oil constituents of the Maritime Alyssum from two study localities. The results of this analysis are summarized in Table 1.

The GC/MS identified the main aerial parts essential oil constituents of *L. maritima* from the two localities in Western Algeria. This analysis identified 62 compounds in the essential oil from the Beni-Saf region, representing 81.34% of the total composition, and 54 compounds in that of Tessala, representing 94.31% of the overall composition.

The global and detailed study of the aerial parts essential oils chemical composition of Algerian *L. maritima*, led us to divide the identified compounds into hydrocarbon monoterpenes, oxygenated monoterpenes, hydrocarbon sesquiterpenes, oxygenated sesquiterpenes, carbonyl compounds and phenylpropanoids. This analysis is marked by oxygenated monoterpenes significant presence with 67.11% for the Beni-Saf region and 80.14% for the Tessala region.

The chemical composition of the essential oils in both regions is more or less similar, with a few degrees of difference. Instead, the essential oil of the Beni-Saf region contains eleven hydrocarbon monoterpenes with a percentage of 6.40%. On the other hand, oxygenated monoterpenes represent 23 compounds with the highest rate (67.11%), followed by phenylpropanoids with a percentage of 4.32%, hydrocarbon sesquiterpenes (2.16%) (Table 2). Sesquiterpenes are only 0.95% of the total composition of this essential oil.

The essential oil of the Tessala region is richer in oxygenated monoterpenes with twenty-two compounds representing 80.14%; hydrocarbon monoterpenes, hydrocarbon sesquiterpenes and phenylpropanoids have a percentage of 7.27%, 1.62% and 3.99% respectively (Table 2). While carbonyl compounds and oxygen-containing sesquiterpenes are only 0.10% and 0.58% respectively.

From the results of Table 3, a complex of eleven compounds was detected as the majority of EHs in the western Algerian aerial parts of *L. maritima*. These identified compounds are almost the same in the two studied essential oils; however, the difference is quantitative. Thus, linalol (24.78%), linalyl acetate (18.90%), eucalyptol (9.12%) together makes up 52.8% of the Beni-Saf region essential oil total composition. The other identified compounds have a lower percentage: cis-dihydro-terpinyl acetate (5.38%), estragole (4.26%) and α -Terpineol (3.39%). The most important monoterpenes are geranyl formate (1.93%), terpineol, 4-ol acetate (1.88%) and β -Myrcene (1.84%). β -pinene and E- β -ocimene have a non-negligible amount of about 1.24% and 1.16% respectively.

The majority compounds of Tessala region essential oil are linalool (26.95%), linalyl acetate (20.85%). They represent 47.8% of the overall composition. Significant amounts of cis-dihydro-terpinyl acetate (9.73%) and eucalyptol (11.57%) are

RT	Compounds	Beni-Saf (EO%)	Tessala (EO%)	KI
7.136	α -Thujene	0.032	0.026	931
7.433	α -Pinene	0.443	0.645	939
8.226	Camphene	0.077	0.091	953
10.082	Sabinene	ND	0.144	976
10.154	β -Pinene	1.247	1.054	980
11.113	(3 E)-Octen-2-ol	0.009	0.032	982
11.889	β -Myrcene	1.843	1.894	991
12.737	3- δ -Carene	0.046	0.054	1011
14.068	1-para-menthene	0.121	0.115	1023
14.970	o-Cymene	0.047	0.253	1026
15.687	Eucalyptol	9.128	11.570	1032
17.105	(Z)- β -Ocimene	0.871	0.637	1040
18.159	E)- β -Ocimene	1.162	1.942	1050
18.830	γ -Terpinene	0.213	0.163	1062
19.795	cis-Sabinene hydrate	0.023	0.027	1068
20.451	cis-Linalool oxide (furanoid)	0.032	0.055	1072
21.727	para-Mentha-3.8-diene	0.351	0.311	1072
21.995	trans-Linalool oxide (furanoid)	0.125	0.238	1086
23.737	linalool	24.782	26.950	1100
25.017	1-Octen-3-yl-acetate	0.174	0.267	1110
26.049	3-Octanol acetate	0.166	0.285	1123
28.538	2-endo-norbornyl acetate	0.188	0.244	1128
28.845	Terpin-1-ol	0.177	0.254	1133
28.992	Nopinone	0.038	0.066	1140
29.438	cis- β -Terpineol	0.02	0.035	1144
29.573	Terpinen-4-ol	0.124	0.180	1177
30.600	para-Cymen-8-ol	ND	0.011	1182
30.909	α -Terpineol	3.391	4.482	1188
31.508	o-Methyl chavicol	4.265	3.995	1196
31.651	Carvone	ND	0.051	1243
31.800	Carvotanacetone	ND	0.013	1247
33.125	Fenchyl acetate(isomer)	0.037	ND	1249
34.164	geraniol	0.408	0.548	1252
34.405	Piperitone	0.031	ND	1252
36.531	Linalyl acetate	18.907	20.850	1257
37.257	geranial	ND	0.037	1267
38.041	bornyl acetate	0.158	0.08	1288
38.977	Lavandulyl acetate	0.085	0.123	1289
39.851	thymol	0.042	ND	1290
42.603	Cis-dihydro-terpinyl acetate	5.382	9.737	1298
42.781	trans-Pinocarvyl acetate	0.126	0.061	1298
43.881	Geranyl formate	1.938	2.214	1298
45.190	Terpineu.4-ol acetate	1.882	2.325	1299

45.674	β -dehydro-Elsholtzia ketone	ND	0.042	1302
45.714	Myrtenyl acetate	0.072	ND	1323
46.380	Melhyl eugenol	0.062	ND	1403
46.583	(Z)-Caryophyllene	0.589	0.678	1408
48.005	Trans- α -Bergamotene	0.293	ND	1432
48.391	cis-Muurolo-3.5-diene	0.018	ND	1444
48.700	alpha-Humulene	0.078	ND	1454
49.150	allo-Aromadendrene	0.013	0.029	1461
49.330	cis-Cadina-1(6).4-diene	0.049	0.017	1463
50.460	trans-Cadina-1(6).4-diene	0.730	0.632	1476
50.720	α -Bulnesene	0.004	ND	1503
50.910	E.E- α -farnesene	0.02	ND	1506
51.420	α -alaskene	0.16	0.100	1512
52.380	cis-Dihydroagarofuran	0.053	0.09	1520
53.002	trans-Calamenene	0.008	ND	1522
53.227	δ -Cadinene	0.168	0.172	1524
53.954	α -Cadinene	0.030	ND	1538
55.547	Spathulenol	ND	0.064	1578
56.301	Caryophyllene oxide	0.022	0.04	1581
56.889	Viridiflorol	0.053	0.041	1590
57.197	γ -Eudesmol	0.045	0.074	1632
59.053	Cubenol	ND	0.009	1642
59.245	β -Eudesmol	0.029	ND	1650
59.815	α -Cadinol	0.526	0.275	1653
60.343	7-epi- α -eudesmol	0.075	ND	1658
61.081	bulnesol	0.141	ND	1671
74.229	(E.E)-Farnesol	0.013	ND	1722
Total		81.34%	94.31%	

Table 1. Chemical constituents of *Lobularia maritima* essential oil (LmEO) from two study locations. RT: retention time; ND: not detected; KI: Kovats indices on an HP-5MS capillary column in reference to C10-C22 n-alkanes injected under the same conditions.

present; smaller amounts of estragole (3.99%), α -Terpineol (4.48%), Geranyl formate (2.21%), terpineu,4-ol acetate (2.32%) are present. In contrast, β -Myrcene (1.89 %), β -Pinene (1.05 %) and E- β -Ocimene (1.94 %) have the lowest monoterpenes (Table 3).

The results of the studied *L. maritima* aerial parts essential oils chemical composition extracted from the two localities showed a qualitative homogeneity and stability, with the exception of a few compounds present in some samples and absent in others.

However, their contents, especially those of the majority compounds, show apparent variability. As

a result, the origin, geographical origin and ecological conditions could be the cause of this variability, which explains the similarity of the contents of the majority compounds of the essential oil of the coastal region of Beni-Saf (Ain Témouchent) and that of the semi-continental region of Tessala (Sidi Bel Abbès), 90 km apart with almost the same ecological conditions, in particular altitude (68 m and 486 m respectively), bioclimate (semi-arid), annual precipitation (357 and 353 mm respectively) and annual average temperatures (17 and 18 °C respectively) (Bouterfas et al., 2014).

Also, the soil type, the bioclimatic stage and the sampling period (season and month) are parameters

Classes	Beni-Saf		Tessala	
	EO (%)	Components number	EO (%)	Components number
Hydrocarbon monoterpenes	6.40	11	7.27	12
Oxygenated monoterpenes	67.11	23	80.14	22
Hydrocarbon sesquiterpenes	2.16	13	1.62	6
Oxygenated sesquiterpenes	0.95	9	0.58	7
Carbonyl compounds	0.03	1	0.10	3
Phenylpropanoides	4.32	2	3.99	1
Other	0.34	3	0.58	3

Table 2. composition of essential oil of *Lobularia maritima* aerial parts from two study localities.

Major components	Beni-Saf (%)	Tessala (%)
Linalool	24.78	26.95
Linalyl acetate	18.90	20.85
Eucalyptol	9.12	11.57
Cis-dihydro-terpinyl acetate	5.38	9.73
Estragol	4.26	3.99
α -Terpineol	3.39	4.48
Geranyl formate	1.93	2.21
Terpineu.4-ol acetate	1.88	2.32
β -Myrcene	1.84	1.89
β -Pinene	1.24	1.05
(E)- β -Ocimene	1.16	1.94

Table 3. Percentage of major components of *Lobularia maritima* essential oil (LmEO) from two study locations.

not to be overlooked in the differences found between sampling locations.

A number of studies have shown the presence of some interesting flavonoids, such as kaempferol, kaempferol-7-rhamnoside, kaempferol 3-glucoside-7-rhamnoside, kaempferol-3-diglucoside, quercetin-7-glucoside (Matlawska, 1989), cyanidin

3-sambubioside-5-acylated glycosides (Tatsuzawa et al., 2006), kaempferol glycosides and cis-11-eicosenoic acid (Fiorentino et al., 2009) in *L. maritima* species.

We note that, our results are more or less identical to those obtained by other researchers, in particular Ben Hsouna et al. (2019) results in Tunisia which characterized the chemical composition of essential oils of *L. maritima*, using the same analytical technique and identified almost the same majority compounds such as; linalol (22.43%), α -terpineol (3.9%), globulol (4.32%), α -cadinol (4.91%), ledol (3.59%), α -pinene (3.51%) and pulegone (3.33%). The Gaara et al. (2010) published abstract reveals that Egyptian (Cairo) *L. maritima* flowers volatile oil contains twenty-six compounds, identified by GC/MS of which methyl eugenol, 4-methyl-2, 6-bis (1, 1- ethyl dimethyl) phenol and eugenol were found as majority compounds.

A chemical study carried out on 15 species of Brassicaceae, revealed the richness of these latter in fatty acids such as: erucic acid (22:1) (50-60 %) (*Sinapis*) and linolenic acid (18:3) (*Matthiola*, *Lepidium*) (Yaniv et al., 1991). The major compound of *Lepidium meyenii* is the phenyl acetonitrile (Tellez et al., 2002). Grove et al. (1978) have identified the palmitic acid 6-O-glucopyranosyl esters, oleic, linoleic and linolenic as majority component in *Brassica napus* L. pollen. The Isothiocyanates,

sulfur compounds and glucosinolates have been identified in *Moricandia arvensis* (L.) DC. (Fahey et al., 2001). The major component in oil of *Raphanus sativus* is the phytol (Blazevic and Mastelic, 2009). The study of Zeraib (2011) allowed the isolation of fatty acids, glucosinolates and sulfur compounds from *Moricandia arvensis*.

Therefore, the majority compounds vary quantitatively and qualitatively according to ecological conditions, geographical origin, the part of analyzed species (Bourkhiss et al., 2007; Toumi et al., 2013), the extraction method and the inter-specific hybridization effect (El Abed & Kambouche, 2003; Satrani, 2006).

CONCLUSIONS

Essential oils come in the form of complex mixtures. The chemical composition of these natural complex mixtures was determined using the gas chromatographic method coupled with mass spectrometry (GC-MS). The chemical profile studied makes it possible to say that the essential oil extracted from *L. maritima* aerial parts of Algeria belongs to the linalols class. The extraction yield is quite high. It can therefore be useful on an industrial scale. This essential oil is rich in oxygenated monoterpenes and less rich in hydrocarbon monoterpenes and sesquiterpenes. It is also rich in phenylpropanoides, allowing an interesting biological activity (El Abed, 2003).

In fact, the biological activities of an essential oil are not due to the majority compounds only, but to the whole of the compounds contained in this oil (El Abed, 2003). The study on the microbiological activities of essential oil of *L. maritima* is therefore in progress and will be the subject of future publications.

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