



## GC/MS analysis of the essential oil composition of *Tourneuxia variifolia* Coss. growing in the Algerian Sahara

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### Abstract

The use of plant-derived compounds in medicine has a long history, especially essential oils for their recognized complex mixture of bioactive compounds. The purpose of this investigation was to determine the chemical composition of the essential oil extracted from *Tourneuxiavariifolia*Coss. (Asteraceae), a Saharan plant that has not previously been investigated for its phytochemical or biological properties. The essential oil was obtained by hydro-distillation and analyzed using gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS), resulting in the identification of 52 components out of a total of 78, which accounted for 99.91% of the oil. The major compounds identified were phthalate (14.19%), methyl linolenate (14.02%), germacrene-D (7.71%), methyl octadecan-10,13-dienoate (6.59%), and delta-cadinene (5.02%). This study provides valuable information about the chemical composition of the essential oil of *Tourneuxiavariifolia*. The presence of several bioactive compounds in high quantities, such as phthalate and germacrene-D, suggests the potential of this species as a source of natural products for various applications.

**Keywords:** Asteraceae, essential oil, GC-MS, germacrene-D, *Tourneuxia variifolia*.

## Introduction

Essential oils are highly complex natural mixtures with a strong odor produced by aromatic plants as secondary metabolites. They consist of organic volatile compounds derived from various chemical classes such as terpenes, sesquiterpenes, alcohols, ethers, aldehydes, ketones, esters, and phenols (Modzelewska *et al.*, 2005). The amount of essential oil components varies by species and parts of plants, as well as collecting time, growth stage, extraction procedures, and analysis methods (Sadgrove *et al.*, 2022; Kokkini *et al.*, 1997). Due to the great number of constituents, essential oils are well-known for their biological properties such as antimicrobial (Sonam *et al.*, 2017; MurbachTeles *et al.*, 2014), antispasmodic effects (CodrutaHeghes *et al.*, 2019), antiviral actions (Tariq *et al.*, 2019), anti-inflammatory (Schepetkin *et al.*, 2019), antioxidant, antidiabetic (Edris, 2007) and cancer chemo-protective action (Blowman *et al.*, 2018; Boussaha *et al.* 2023).

The Asteraceae family is one of the largest flowering plants, with over 1100 genera and more than 20000 species. Many species of Asteraceae family contain important phytochemical compounds such as terpenoids, polyphenols, flavonoids, as well as essential oils, which are known for their medicinal actions (Shing *et al.*, 2002). A literature survey shows that this family contains a high number of essential oil producers with biological activity (Okunade, 2002) which led us to investigate a species from the monophyletic Scorzonnerinae sub-tribe of the tribe Cichorieae (Mavrodiev *et al.*, 2004). The tribe Cichorieae contains around 98 genera and 1550 species (Kilian *et al.*, 2009). There are fourteen sub-tribes within the tribe Cichorieae, one of which is the monophyletic Scorzonnerinae sub-tribe with seven genera, the two largest of which are *Tragopogon* L. and *Scorzonera* L., while *Pterachaenia*, *Geropogon*, and *Tourneuxia* are monotypic. However, *Koelpinia* has four or five species, and *Epilasia* has three or four species, mostly from Europe and North Africa (Bremer, 1994).

*Tourneuxia* genus only consists of *T. variifolia* Coss., which is characterized by the annual life form. It is found throughout North Africa (Algeria, Morocco, Tunisia, and Libya). According to Mavrodiev (2004), *Tourneuxia* and the *Gelasia* clade have a relationship as sister groups (Mavrodiev *et al.*, 2004). The monospecific North African *Tourneuxia*, according to Zaika (2020), is resolved with significant evidence as sister to all other members of the subtribe, indicating its systematic location (Zaika *et al.*, 2020). Many species of the sub-tribe Scorzonnerinae contain important phytochemical compounds such as terpenoids, polyphenols, flavonoids, as well as essential oils, which are known for their medicinal actions.

Although the *Tourneuxia variifolia* species is known to nomads in the Northern Sahara as "Telma" and it is commonly consumed by camels and cattle, there is no information that it is used in traditional

or folk medicine. Even though many species of the Asteraceae family are well known for their precious essential oils, to our knowledge, there is no report about the chemical composition of *T. variifolia's* essential oil. Therefore, the objective of this research is to further continue our phytochemical studies on *T. variifolia* species (Zerrouki *et al.*, 2021) and investigate its essential oil composition by GC and GC-MS techniques for the first time.

## Material and methods

### *Plant materials*

The aerial parts of *Tourneuxia variifolia* were collected at the end of March 2016, during the flowering phase from Bechar province in the Southwest of Algeria and identified by Mr. Benabdelhakem (Director of the national agency for the preservation of natural resources, Bechar, Algeria). An authenticated voucher specimen with the identification number 03/2016/ATV was deposited at the Herbarium of the VARENBIOMOL unit research, University of Frères Mentouri Constantine 1.

### *Extraction of the essential oil*

The aerial parts (leaves and flowers) were first dried away from sunlight for two weeks. A quantity of 200g of sample dried aerial parts were cut into small pieces and subjected to hydro-distillation for 3 hours using Kaiser Lang apparatus (till no oil obtained). The essential oil was collected and dried over anhydrous sodium sulfate, then stored at 4°C for analysis. The oil yield was calculated as a percentage of the plant's dry weight.

### *GC and GC-MS analysis*

The essential oil was analyzed using an Agilent Model 6890 gas chromatograph (GC-FID) with an HP-5 MS fused silica capillary column with a (5%-phenyl) methylpolysiloxane stationary phase (25 m x 0.25 mm; 0.25 m film thickness), programmed from 50°C (5 mn) to 250°C at 3°C/mn and held for 10 minutes. The injector and flame ionization detector had temperatures of 280 and 300°C, respectively. The essential oil was diluted to 3.5 percent (v/v) in acetone and injected in split mode (1/60), with hydrogen serving as the carrier gas (1.0 mL/mn). The retention indices (RI) of standard alkanes (C8-C20) were calculated using the Van den Dool and Kratz equations under identical conditions. A Model 7890/5975 Agilent gas chromatograph-mass spectrometer (GC-MS) with an HP-5 capillary column (25 m x 0.25 mm, film thickness of 0.25 m) was programmed under the identical circumstances as the GC-FID and was used for mass spectrometry. The electron impact mode of the mass spectrometer (MS) was set to 70 ev, and the electron multiplier was set to 2200 V.

The temperatures of the ion source and MS quadrupole were 230°C and 180°C, respectively. In the  $m/z$  range of 33–450, mass spectral data was collected in scan mode. The mass spectra and retention indices (RI) of the essential oil constituents were compared to those of reference compounds from libraries such as Adams and McLafferty & Stauffer (Adams, 2007; McLafferty & Stauffer, 1991). Internal normalization was used to calculate the proportions of the detected substances.

## Results and discussion

The aerial parts of *Tourneuxia variifolia*, collected from the southeastern region of Algeria, were subjected to hydro-distillation to extract their essential oil. A yield of 0.05% (w/w) of yellowish oil with a distinctive aroma was obtained on a dry weight basis. The extracted oil was subsequently analyzed using GC and GC-MS.

By comparing their retention times to those of reference compounds from libraries, 52 components were identified. Seventy-eight constituents, accounting for around 99.91% of the total peak area, were detected, and only 87.614% of the plant's volatile compounds were identified and classified into five chemical classes. Table 1 illustrates all of the compounds that were identified and classified according to their experimental retention times and retention indices.

In terms of the number of identified components, carbonylic compounds represented the largest group (nine compounds) with a percentage of 39.853%. However, methyl linolenate (14.02%), phthalate (14.19%), and octadecan-10, 13-methyl dienoate (6.59%) were the most common ester compounds in the essential oil. Sesquiterpenes were also detected with a percentage of 19.81%. Germacren-D (7.711%), delta-cadinene (5.02%), and  $\beta$ -caryophyllene (1.67%) had the highest percentages. This study also indicated the presence of a notable quantity of oxygenated sesquiterpenes and alkanes (7.430% and 5.75%), respectively, while oxygenated monoterpenes were presented only with 1.28%.

**Table 1:** Retention times, Retention indices and percentage composition of *T. variifolia*'s essential oil

N°	RT	RI <sup>a</sup>	RI <sup>b</sup>	Compound	Area %	Ref
1	9.744	1192	1200	Para-cymen-8-ol	1.726	Adams 2007
2	9.865	1199	1198	Alpha-terpineol	0.591	Adams 2007
3	10.203	1223	1217	Trans carveol	0.401	Adams 2007
4	10.655	1254	1235	Chrysanthenyl acetate	0.210	Adams 2007
5	12.394	1380	1376	Alpha-copaene	0.726	Adams 2007
6	12.518	1390	1390	Beta-cubebene	1.905	Adams 2007
7	12.617	1397	1394	Jasmone Z	0.243	Adams 2007
8	12.973	1425	1428	beta-caryophyllene	1.673	Choi 2003
9	13.057	1432	-	beta-copaene	0.261	<b>N. Avai.</b>
10	13.118	1437	1434	Trans alpha-bermagoten	0.600	Priestap 2003a
11	13.425	1461	1461	Alloaromadendren	0.153	Adams 2007
12	13.69	1483	1478	Gamma curcumene	2.239	Priestap 2003b

13	13.744	1487	1480	Germacren-D	7.711	Adams 2007
14	13.816	1493	1485	E-beta ionone	0.288	Adams 2007
15	13.937	1503	1499	Alpha muurolen	0.458	Karioti 2003
16	13.986	1507	1508	E,E-alpha farnesen	0.302	Adams 2007
17	14.022	1510	1514	GeranylIsobutanoate	0.464	Adams 2007
18	14.122	1518	1513	Gamma cadinene	0.446	Adams 2007
19	14.179	1523	1524	Delta-cadinene	5.018	Adams 2007
20	14.46	1547	1548	Alpha calacorene	0.662	Kowalski 2005
21	14.669	1565	-	Beta calacorene	0.936	N. Avai.
22	14.763	1573	1581	Caryophyllene oxide	0.301	Adams 2007
23	14.869	1582	1585	$\beta$ -Oplopenone	1.388	Caredda 2002
24	14.955	1589	-	Salvia-4(14)-en-1-ol	0.826	N. Avai.
25	15.024	1595	1588	<i>epi</i> -globulol	0.310	Apel 2004
26	15.157	1606	1613	Geranyl isovalerate	1.356	Couladis 2001
27	15.253	1615	1613	1- <i>epi</i> -cubenol	0.625	Couladis 2001
28	15.624	1648	-	Alpha muurolol + <i>epi</i> alpha muurolol	1.490	N. Avai.
29	15.675	1653	1649	Beta eudesmol	0.401	Adams 2007
30	15.776	1662	1653	Alpha cadinol	1.394	Adams 2007
31	15.824	1666	1658	7- <i>epi</i> -alpha eudesmol	0.348	Pino 2003
32	15,965	1679	1674	Cadalene C15H18	0.854	Adams 2007
33		1691	-	Germacran-4(15),5,10(14)-triene-1-alpha-ol	0.547	N. Avai.
34	16,169	1697	1689	Pentadecan-2-one	0.152	Pino 2005
35	16,393	1718	1711	Pentadecanal	0.643	Pino 2005
36	16,876	1763	1770	14-Hydroxy- $\alpha$ -muurolene	1.863	Couladis 2001
37	17,099	1784	1688	Cedren-13-ol	0.859	Ghasemi 2003
38	17,212	1795	1800	Octadecane	0.444	Adams 2007
39	17,643	1837	-	Neophytadiene	0.291	N. Avai.
40	17,699	1843	1843	6,10,14-trimethyl-pentadecan-2-one	0.672	Sasaerila 2003
41	18,297	1902	-	Octadecan-2-one	0.731	N. Avai.
42	18,539	1927	1927	Methyl hexadecanoate	3.018	Adams 2007
43	18,958	1970	-	Phthalate	14.19	N. Avai.
44	20,171	2090	-	Octadecan-10,13-methyl dienoate	6.595	N. Avai.
45	20,277	2101	2098	Methyl linolenate	14.02	Tellez 2002
46	20,526	2132	-	Phytol diterpene	2.935	N. Avai.
47	21,925	2300	2300	Tricosane	1.136	Adams 2007
48	23,536	2500	2500	Pentacosane	0.578	Adams 2007
49	25,032	2700	2600	Hexacosane	0.711	Spiewok 2006
50	25,742	2800	2800	Octacosane	0.819	Sullivan 2002
51	26,433	2901	2900	Nonacosane	0.845	Sullivan 2002
52	27,743	3101	3100	Hentriacontane C31	0.259	Steiner 2006
<b>Oxygenated monoterpenes</b>					2.718	
<b>suesquiterpenes Hydrocarbons</b>					23.09	
<b>Oxygenated sesquiterpenes</b>					10.352	
<b>Hydrocarbons alkanes</b>					4.792	
<b>diterpenes Hydrocarbons</b>					0.291	
<b>Oxygenated diterpenes</b>					2.935	
<b>Carbonylic compound</b>					39.853	
<b>Others</b>					3.583	
<b>Total identified</b>					87.614	
<b>Total non-identified</b>					12.291	
<b>Total</b>					99.905	

The Compounds listed in order of their RI.

RI (retention index) measured relative to n-alkanes (C8-C20) using HP-5 ms.

RT: retention time, RI<sup>a</sup>: Kovats retention index calculated, RI<sup>b</sup>: Kovats retention index litterateur, Area %: quantitative percentage of the compounds, N.avai.: Not available.

A few studies on the chemical composition of the essential oils of the sub-trib Scorzonerea species have previously been reported. According to Boussaada (2008), the most abundant components detected in the essential oil obtained from the aerial parts of *Scorzonera undulata* collected in western Tunisia were methyl palmitate (methyl hexadecanoate) (30.4%) and methyl linolenate (23.9%) (Boussaada *et al.*, 2008). However, hexadecanoic acid (42.2%), *n*-tetradecanoic acid (16.1%), 9-octadecenoic acid (7.7%) and 9-hexadecenoic acid (4.5%) were the major compounds in the same species collected from Algeria (Harkati *et al.*, 2012). The dominant components of the essential oil obtained by free microwave extraction from the aerial parts of *Scorzonera calyculata* from Iran were 6,10,14-trimethyl-2-pentadecanone (27.73%), caryophyllene oxide (16.84%), neophytadiene (7.68%), and (E)- $\beta$ -ionone (6.77%) (Ayromlou *et al.*, 2020). Although the essential oil obtained from different parts of *Scorzonera papposa* collected in Turkey contained a high percentage of methyl palmitate (16.78%) in the root, phytone (30.44%) in the stem, phytone (40.17%) in the leaf, and phytol (31.84%) in the seed (Yurteri *et al.*, 2022).

Previous studies have found that species of the *Scorzonera* genus contained high amounts of fatty acids and aliphatic hydrocarbons and small amounts of sesquiterpenoids, monoterpenoids, and aromatic compounds, indicating significant differences from our identified compounds. We noticed that carbonylic compounds and hydrocarbon sesquiterpenes were the major components identified in *Tourneuxiavariifolia*'s oil, with methyl linolenate and Germacren-D being identified as major components. As indicated previously, Germacren-D was one of the most common plant volatiles and was considered to be a biogenetic precursor of many sesquiterpenes (Bülow *et al.*, 2000). On the other hand, it is interesting to note that fatty acids and their esters are the most abundant components of vegetable oils, and they are reported to be good antibacterial and antifungal agents (Desbois *et al.* 2010; Erdemoglu *et al.*, 2003), as well as antioxidants (Lima LARS *et al.*, 2012). Additional phytochemical and biological studies on this plant are needed to explore new compounds as well as their pharmacological properties.

## Conclusion

This report shows the first study on the chemical composition of the essential oil of *T. variifolia*, collected in the southeast of Algeria. This oil is characterized by the main presence of phthalate (14.19%), methyl linolenate (methyl hexadecanoate) (14.020%), Germacren-D (7.71%), octadecan-10, 13-methyl dienoate (6.595%), and  $\delta$ -cadinene (5.02%). Comparing with the literature, the chemical components of the Algerian sample, identified in this study, differ from those reported in other studies. Further research is needed to investigate the pharmacological properties and potential therapeutic applications of the identified compounds.

### Conflict of Interest

The authors declare no conflict of interest.

### Authors' Declaration

The authors hereby declare that the work presented in this article is original and that any liability for claims relating to the content of this article will be borne by them.

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