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Research Article

SPME/GC-MS analysis of *Artemisia campestris subsp. glutinosa, Lavandula angustifolia Mill.*, and *Zingiber officinale* volatiles

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Abstract

The identification and quantification of the volatile organic compounds profile of the plants is an important tool for food quality and authenticity assessment. In that context, herewith the study, we aimed at quantifying the volatile compounds of three plants *Artemisia campestris* L. subsp. *glutinosa, Lavandula angustifolia Mill.*, and *Zingiber officinale* Rosch using Gas Chromatography-Mass Spectrometry (SPME/GC-MS). Thirty-three volatile compounds were revealed with SPME/GC-MS. Of the identified compounds, α-curcumene (34.41%) and eucalyptol (20.91%), were predominant compounds in *Zingiber officinale* Rosch. For *Artemisia campestris* subsp. *glutinosa*, camphor (31.78%), and α-thujone (16.82%) were noted as the most abundant volatile compounds. Besides eucalyptol (15.10%), and linalool (11.98%) were recorded as major volatile compounds in the *Lavandula angustifolia Mill.*

Keywords: Essential oil, secondary metabolites, terpenoids, medicinal and aromatic plants

Artemisia campestris subsp. glutinosa, Lavandula angustifolia *Mill.* ve *Zingiber officinale* uçucu yağlarının SPME / GC-MS ile analizi

Öz

Bitkilerin uçucu organik bileşik profilinin belirlenmesi, gıda kalitesi ve özgünlüğünün değerlendirilmesinde önemli araçlardandır. Bu çalışmanın amacı, Artemisia campestris L. subsp. glutinosa, Lavandula angustifolia Mill., and Zingiber officinale Rosch. bitkilerinin gaz kromatografisi-kütle spektrometresi (SPME / GC-MS) kullanılarak organik uçucu bileşiklerinin tayini yapılmasıdır.

SPME / GC-MS ile toplamda 33 uçucu bileşik belirlenmiştir. Zingiber officinale Rosch'da ana bileşen olarak α-curcumene (% 34.41) ve eucalyptol (% 20.91) belirlenirken Artemisia campestris subsp. glutinosa 'de ise, camphor (%31.78) ve α-thujone (%16.82) ana bileşen olarak belirlenmiştir. Ayrıca, eucalyptol (%15.10) ve linalool (%11.98) ise L. angustifolia bitki ekstresinde başlıca uçucu bileşikler olarak tespit edilmiştir.

Anahtar Kelimeler: Uçucu yağlar, sekonder metabolitler, terpenoitler, tıbbi ve aromatik bitkiler.

1. Introduction

Artemisia campestris L. belonging to the Asteraceae family is a perennial herb, generally known as field wormwood and extensive in North Africa, North America, Europe, and Asia. This plant consists of several subtaxa. *A. campestris* L. which can be distinguished by their certain morphological characteristics (Dib et al., 2017). It has been reported that the composition of volatile isolated from various species of *Artemisia*. Volatile components of *A. campestris glutinosa* have been identified and reported, *viz.* caryophyllene oxide, ar-curcumene, β-pinene, p-cymene, agermacrene D, bicycle-germacrene β-pinene, and germacrene D (Juteau, Masotti, Bessière, & Viano, 2002). *Lavandula angustifolia* Mill. belonging to the Lamiaceae family is widely known as an aromatic herb (Omidbaigi, 2000) used in the perfumery industries (Trease & Evans, 1989). It is a potent medicinal herb and aromatic and commonly consumed in traditional and folk medicines worldwide for the treatment of several diseases such as gastrointestinal, nervous, and rheumatic disorders (Leung, 1980). *Lavandula angustifolia* Mill. has also been used in folk and traditional medicine as a diuretic, carminative, anti-rheumatic, anti-epileptic, and pain broker, particularly for migraine and headache (Hajhashemi, Ghannadi, & Sharif, 2003). *Zingiber officinale* L. belonging to the Zingiberaceae family that is mostly used as a spice includes several bioactive compounds such as shogaols, gingerdione, gingerdiol, andgingerol. It is consumed for its antioxidant properties, antitumorigenic, immunomodulatory, anti-inflammatory, anti-apoptotic (Hosseini et al., 2016; Lim, 2016). *Zingiber officinale* Rosch essential oil, extracted from it is rhizomes, is commonly used in medicine for its anti-cancer, antioxidant, antifungal, anti-inflammatory, and antibacterial, biological activities (dos Santos Reis et al., 2020). Volatile compounds such as ketones, terpenes esters, and alcohols are volatile compounds obtained from medicinal plants (Aziz et al., 2018). Some of these compounds are considered as inhibitor agents of pathogens, such as *Staphylococcus epidermidis, Salmonella, Escherichia coli,* and *Staphylococcus aureus* (Aziz et al., 2018; Tariq et al., 2019) and are extensively used for food preservation (Noori, Zeynali, & Almasi, 2018). The essential oils extracted from the ginger rhizomes are used as antibacterial (da Silva et al., 2018), antioxidant (An et al., 2016), anti-inflammatory (Funk et al., 2016), and anti-cancer (dos Santos Reis et al., 2020; Wang, Qi, & Yuan, 2015). *Artemisia campestris* L. is known for its medicinal, and pharmacological properties such as anti-hyperlipidemic (Barkat, Boumendjel, Saoudi, El Feki, & Messarah, 2015), anti-diabetic (Sefi, Fetoui, Makni, & Zeghal, 2010), anti-hypertensive (Hamed, Serria, Lobna, & Khaled, 2014), anti-inflammatory (Jaouadi et al., 2016), anti-venom (Hamed et al., 2014), wound healing (Essid et al., 2015; Ghlissi, Sayari, Kallel, Bougatef, & Sahnoun, 2016), anti-leishmaniasis (Dib & El Alaoui-Faris, 2019)) effects.

In this study, we determined the volatile compound of *Artemisia campestris subsp. glutinosa*, *Lavandula angustifolia Mill.*, and *Zingiber officinale* using SPME/GC-MS.

2. Material and Methods

Plant material

Zingiber officinale was purchased from local spice shops in Igdir. L. angustifolia plants were collected from experimental fields of Igdir University, Faculty of Agriculture. A. campestris plants were collected from Tuzluca, Igdir. The plants were identified by

Dr. Ramazan Gurbuz and Dr. Belkis Muca Yigit.

Essential oil extraction

30 g of A. campsetris, 30 g of L. angustifolia and 30 g of Zingiber officinale leaves dried in shadow and powdered by a blender added to 200 mL of distilled water (1/10: w/v) separately and extraction was performed in a Neo-Clevenger then filtered. The filtrate water sample was frozen and lyophilized in a lyophilizer (Labconco, Freezone 1 L) at 5 mm Hg at -50 °C.

Analysis of volatile compounds

Analysis of volatile compounds was performed with SPME/GC-MS method (Koyuncu & Tuncturk, 2017). 5 ml of the ultra-pure water (18.2 **Ω**) was added to 1.00 g of the sample in the glass vials (Supelco, USA) and allowed to equilibrate at 40 ºC for 30 min. A 2-cm (50/30 μm divinyl benzene/ carboxen/ polydimethyl siloxane) SPME fiber (Supelco Co., Bellefonte, PA, USA) was used for the extraction of volatile compounds from samples. The desorption of the extracted volatiles was performed using a Thermo Fisher Trace ISQ GC-MS gas chromatography-mass spectrometry system and run in split (ratio was 1:10) mode. During desorption, the SPME fiber remained in the injector for 2 min at a temperature of 200 ºC, with helium as the carrier gas at a flow rate of 1.0 mL/min. The volatile compounds were separated on the DB-5MS column (30 m × 0.25 mm × 0.25 μm; Agilent, USA). The oven was held at 40 ºC for 1 min, then increased at 5 ºC per min to 120 ºC, it was held for 2 min, then rose again at 10 ºC per min to 240 ºC and hold 3 min. The mass spectrometer was set to scan from 45 to 450 amu (threshold 1000) at a sampling rate of 1.11 scans/s.

3. Result and Discussion

Volatile composition analyses by SPME/GC-MS

Volatile compounds are emitted as gases and include the different structures of chemicals that have health effects (EPA, 2017). The characterization of the volatile profile is an important means for authenticity evaluation and food quality (Oliveira-Alves et al., 2020). The volatile compounds of the *A. campestris, L. angustifolia*, and *Z. officinale* were identified using SPME/GC-MS and presented in Table 1 and Figures 1- 3.

Concerning *Z. officinale*, chromatographic analysis revealed that borneol (2.72%), camphene (2.74%), citronella (2.86), E-citral (11.10%), eucalyptol (20.91%), linalool (2.87%), Z-citral (8.78%), and α-curcumene (34.41%) were of the common compounds defined in *Z. officinale*. According to the previous reports regarding essential oil composition of Zingiber officinale Roscoe, 2,6-dimethyl hepten-l-ol, a-gurjunene, linalool oxide, isovaler-aldehyde, 2-pentanone, cadinol, a- and g-calacorene, eremophyllene, t-muurolol, a-himachallene, a-cubebene acetic acid, pinanol, a-santalene, geranyl propionate, geranoic acid, (E,E)-a-farnesene, n-methyl pyrrole and geranic acid were observed (Onyenekwe and Hashimoto, 1999). In another research, citral (geranial 10.5% and neral 9.1%), α-zingiberene (17.4%), camphene (7.8%), α-farnesene (6.8%) and β-sesquiphellandrene (6.7%) were of the common and major components (Höferl et al., 2015).

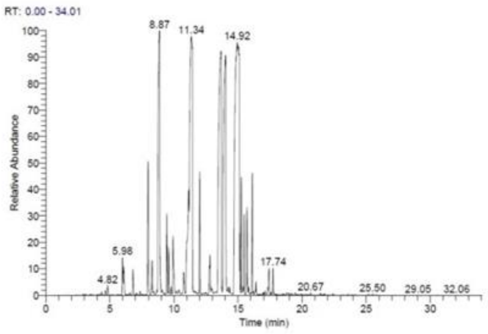

Figure 1. Chromatogram of *A. campestris* by SPME.

The major volatile compounds of the *A. campestris* were characterized as follows camphor (31.78 %), eucalyptol (23.11 %), and α-thujone (16.82 %). Also, 33 volatile compounds were identified in *L. angustifolia*. Camphor is used widely in medicine. Its oils are consumed to repel stored-products beetles including *Trilobium castaneum* and *Sitophilus granarius* (Ali & Ibrahim, 2018; Obeng-Ofori, Reichmuth, Bekele, & Hassanali, 1998).

The following components having quantities higher than 1% in volatile compounds were proved for *A. campestris*: 2-α-p (1.31%), ç-terpinene (1.52%), terpinen-4-ol (1.63%), (-)-myrtenal (1.81%), tricyclene (2.57%), sabinene (1.82%), and butanoic acid, 2-methyl-(1.33%), ethyl ester (1.33%) and volatile compounds with quantities higher than 10% camphene (11.66%), α-thujone (16.82%), camphor (31.78%), and eucalyptol (23.11%). Previous reports revealed that β-pinene (24.2–27.9%), p-cymene (17.4–22.3%) and α-pinene (4.1–11.0%) were of the dominants in *Artemisia campestris* L. (Akrout et al., 2001). In a similar reports by Juteau et al. (2002), γ-terpinene, capillene, 1-phenyl-2,4-pentadiyne, spathulenol, methyleugenol,p-cymene , and β-pinene were of the major compounds.

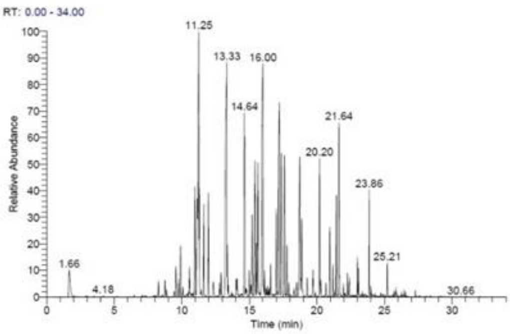

Figure 2. Chromatogram of *L. angustifolia* by SPME.

The relevant chromatograms of the extracts were presented in Figures 1-3. The major volatile compounds of the *L. angustifolia* were as follows eucalyptol (15.10 %), linalool (11.99 %), and butanoic acid, hexyl ester (10. 56 %). The following structures having quantities lower volatile compounds comparatively as proved for. *angustifolia* L: The following structures having quantities higher than 1% in volatile compounds were demonstrated by GC-MS: Neryl acetate (1.09%), geranyl acetate (1.64%), hexanoic acid, hexyl ester (3.61%), α-myrcene (1.09%), 1,3,6-octatriene, 3,7-dimethyl-, (E)-(1.47%), ç-terpinene (1.70%), camphor (5.51%), linalyl isobutyrate (2.03%), borneol (3.52%), hexyl 2-methyl butyrate (7.28%), hexyl isovalerate (2.92%), linalyl acetate (3.09%), lavandulyl acetate (6.09%), allyl tiglate (2.85%), and dimethylamine-D1 (1.54%), geranyl isovalerate (1.35%) and volatile compounds with quantities higher than 10% butanoic acid, hexyl ester (10.56%), linalool (11.98%), and eucalyptol (15.10%). Since their lower boiling point, no visible break-through was obtained for compounds such as ethylene, ethane, and acetylene in the GC chromatogram. The sensibility loss for such compounds might be associated with missing trapping in the pre-concentrator. According to the previous reports, caryophyllene (24.1%), β-phellandrene (16%)

and eucalyptol (15.6%) were reported to be major compounds in *Lavandula angustifolia* (Jianu et al., 2013). Verma et al. (2010) reported that the predominant compounds of *Lavandula angustifolia* were found to be as linalyl acetate (47.56 %), linalool (28.06 %), lavandulyl acetate (4.34 %), and α-terpineol (3.75 %). The differences in percentage and major components might be attributed to the cultivar and genotype of the species, harvesting time, distillation techniques and ecological conditions of the collected region.

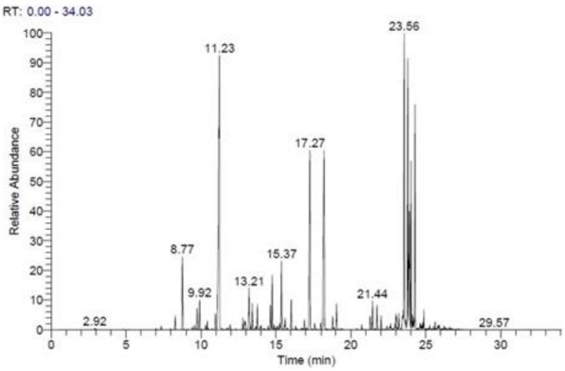

Figure 3. Chromatogram of Z. officinale by SPME.

4. Conclusion

Along with the present study, essential oil profiles in different plant species, *viz. Artemisia campestris* L. subsp. *glutinosa*, *Lavandula angustifolia* Mill., and *Zingiber officinale* Rosch were revealed. Those three plant species are of the well-known for their uses in medicine and cosmetics. We, herewith the study, used solid phase micro-extraction chromatographic analysis for profiling the compounds. The relevant data we obtained herein were compared with the previous reports. Substantial differences were noted regarding components and percentage of the relevant compounds. Those differences might be the consequences of the climatic conditions of the location, genotype of the species, harvesting times, storage conditions and chromatographic techniques.

Table 1. Volatile compounds of *L. angustifolia, A. campestris,* and *Z. officinale*.

No	Retention Time	Compound Name	CAS	Area %		
				L. angustifolia	*A. campestris*	*Z. officinale*
1	4.83	Hexanal	66-25-1	ND	0.18	ND
2	5.98	Butanoic acid, 2-methyl-, ethyl ester	7452-79-1	ND	1.33	ND
3	6.81	Cyclohexane, (1-methylethylidene)-	5749-72-4	ND	0.42	ND
4	7.34	2-Heptanol	543-49-7	ND	ND	0.12
5	7.98	Tricyclene	508-32-7	ND	2.57	ND
6	8.28	α-Pinene	80-56-8	0.34	0.70	0.50
7	8.77	Camphene	79-92-5	0.51	11.66	2.74
8	9.44	Sabinene	3387-41-5	ND	1.82	0.09
9	9.57	2-α-Pinene	127-91-3	0.78	1.31	0.14
10	9.91	α-Myrcene	123-35-3	1.09	ND	1.71
11	10.42	1-Phellandrene	99-83-2	ND	ND	0.47
12	10.57	Acetic acid, hexyl ester	142-92-7	0.92	ND	ND
13	11.25	Eucalyptol	470-82-6	15.10	23.11	20.91
14	11.62	1,3,6-Octatriene, 3,7-dimethyl-, (E)-	3779-61-1	1.47	ND	ND
15	11.98	ç-Terpinene	99-85-4	1.70	1.52	ND
16	12.34	Linalool oxide (Z)	5989-33-3	0.40	ND	ND
17	12.80	α-Terpinolene	586-62-9	ND	ND	0.85
18	13.32	Linalool	78-70-6	11.98	ND	2.87
19	13.68	α-Thujone	546-80-5	ND	16.82	ND
20	14.01	2,6,6-trimethylbicyclo[3.1.1]heptan-2-ol	98510-89-5	ND	ND	0.22
21	14.09	cis Sabinen hydrate	15826-82-1	0.63	ND	ND
22	14.64	Camphor	76-22-2	5.51	31.78	ND
23	14.75	Citronella	106-23-0	ND	ND	2.86
24	15.21	Linalyl isobutyrate	78-35-3	2.03	ND	ND
25	15.41	Borneol	10385-78-1	3.52	ND	2.72
26	15.64	Butanoic acid, hexyl ester	2639-63-6	10.56	ND	ND
27	15.70	Terpinen-4-ol	562-74-3	ND	1.63	ND
28	16.02	α-Fenchyl alcohol	470-08-6	ND	ND	1.07
29	16.11	(-)-Myrtenal	564-94-3	ND	1.81	ND
30	16.33	Decanal	112-31-2	0.23	ND	0.11
31	16.57	cis-P-2-Menthen-1-ol	35376-39-7	0.61	ND	ND
32	16.90	α-Citronellol	106-22-9	ND	ND	0.46
33	17.21	Hexyl 2-Methyl butyrate	10032-15-2	7.28	ND	ND
34	17.26	Z-Citral	106-26-3	ND	ND	8.78
35	17.37	Hexyl isovalerate	10032-13-0	2.92	ND	ND
36	17.42	L(-)-Carvone	6485-40-1	ND	0.67	ND
37	17.59	Nerol	106-25-2	ND	ND	0.27
38	17.60	Linalyl acetate	115-95-7	3.09	ND	ND
39	17.74	cis Piperitone oxide	35178-55-3	ND	0.47	ND
40	17.78	α-Fenchyl alcohol	470-08-6	0.90	ND	ND
41	18.21	E-Citral	141-27-5	ND	ND	11.10
42	18.73	Lavandulyl acetate	25905-14-0	6.09	ND	ND
43	18.79	Endobornyl acetate	76-49-3	ND	ND	0.61
44	19.05	2-Undecanone	112-12-9	ND	ND	0.98
45	20.19	Allyl Tiglate	7493-71-2	2.85	ND	ND
46	20.67	α-Terpinenyl acetate	80-26-2	0.24	ND	ND
47	20.95	Neryl acetate	141-12-8	1.09	ND	ND
48	21.43	Geranyl acetate	105-87-3	1.64	ND	ND
49	21.44	α-Copaene	3856-25-5	ND	ND	0.84
50	21.63	Hexanoic acid, hexyl ester	6378-65-0	3.61	ND	ND
51	21.75	α-elemene	515-13-9	ND	ND	0.90
52	21.96	Diphenyl ether	101-84-8	0.26	ND	ND
53	22.02	Zingiberene	495-60-3	ND	ND	0.37
54	22.66	α-Bergamotene	17699-05-7	ND	ND	0.31
55	23.00	trans-α-Farnesene	502-60-3	0.84	ND	ND
56	23.56	α-Curcumene	644-30-4	ND	ND	34.41
57	23.86	Geranyl isovalerate	109-20-6	1.35	ND	ND
58	24.87	Germacrene B	15423-57-1	ND	ND	0.96
59	26.23	α-Eudesmol	473-15-4	ND	ND	0.13

ND: Not detected

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