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Changes in the essential oil content and composition of *pelargonium graveolens* l'hér with different drying methods

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SUMMARY: In this study, the effect of various drying methods (fresh plant, shade-drying, sun-drying, and oven-drying at 30 and 60 °C) on the essential oil (EO) composition of rose-scented geranium were determined. Essential oil samples were extracted by hydrodistillation and analyzed by GC and GC-MS systems. The highest EO contents were obtained in the fresh plant (1.98%), followed by shade-drying (1.34 %) and oven-drying at 30 °C (1.20 %). The main components were citronellol (23.99-39.87%), geraniol (4.15-17.09%), menthone (4.48-8.34%), linalool (1.96-7.42%), β-caryophyllene (2.63-4.32%), geranyl tiglate (0.99-4.52%), citronellyl butyrate (0.53-5.31%) and cis-rose oxide (0.71-3.15%). The drying methods showed a marked impact on the constituents of the EO samples. The results demonstrated that drying the aerial parts of fresh geranium, and shade-drying and oven-drying at 30 °C were the best optimal methods to obtain the highest oil yield, and citronellol, geraniol, and linalool contents in the oil.

KEYWORDS: *Rose-scented geranium; Essential oil yield; Chemical composition; Drying methods; Citronellol; Geraniol*

RESUMEN: ***Cambios en el contenido y composición del aceite esencial de* pelargonium graveolens *l'hér con diferentes métodos de secado.*** Se estudió el efecto de varios métodos de secado (planta fresca, secado a la sombra, secado al sol y secado en horno a 30 y 60 °C) sobre la composición del aceite esencial (AE) de geranio con aroma a rosas. Los aceites esenciales de las muestras fueron extraídos por hidrodestilación y analizados mediante GC y GC-MS. Los mayores contenidos de AE los obtuvo la planta fresca (1,98%), seguido del secado a la sombra (1,34 %) y secado en estufa a 30 °C (1,20 %). Los principales componentes fueron citronelol (23,99-39,87 %), geraniol (4,15-17,09 %), mentona (4,48-8,34 %), linalol (1,96-7,42 %), β-cariofileno (2,63-4,32 %), geranil tiglato (0,99-4,52 %), butirato de citronelilo (0,53-5,31 %) y óxido de cis-rosa (0,71-3,15 %), los métodos de secado mostraron un marcado impacto en los constituyentes de las muestras de EO. Los resultados demostraron que el secado de las partes aéreas del geranio fresco, y el secado a la sombra y el secado en horno a 30 °C fueron los mejores métodos óptimos para obtener el mayor rendimiento de aceite y contenido de citronelol, geraniol y linalool en el aceite.

PALABRAS CLAVE: *Composición química; Genario; Geranio perfume a rosa; Rendimiento de aceite esencial; Secado.*

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1. INTRODUCTION

The genus *Pelargonium* is cultivated around the world for the production of essential oils and absolutes. *Pelargonium graveolens* L'Hér is an aromatic and hairy shrub from which oil is obtained from its leaves, flowers, and stems. Various factors such as cultivar, oil distillation method, distilled part of the plant, age of the material, growing location and seasonal changes in the region, as well as harvest season and time, affected the final essential oil composition of rose-scented geranium (Verma *et al.,* 2013; Szutt *et al.,* 2019). The main constituents of the essential oils of *P. graveolens* were reported as geraniol (14.1-34.6%), citronellol (15.2-31.3%), linalool (2.9-9.2%), citronellyl formate (4.4-9.2%), isomenthone (4.5-6.6%), 10-epi-γ-eudesmol (4.7-6.7%) and geranyl formate (3.8-6.2%) by Verma *et al.* (2013) citronellol (20.9-39.5%), geraniol (10.9-26.5%), linalool (2.9-14.2%), isomenthone (7.4-9.4%), citronellyl formate (5.5-9.1%) and 10-epi-γ-eudesmol (5.2-9.0%) by Singh *et al.* (2018); citronellol (22.3%), geraniol (15.5%), geranyl acetate (13.1%), limonene (9.3%), phenyl ethyl alcohol (5.9%) and linalool (5.6%) by Szutt *et al.* (2018); citronellol (27.0%), geraniol (20.7%), 10-*epi*-*γ*-eudesmol (13.1%), citronellyl formate (6.4%) and linalool (5.7%) by Ben ElHadj *et al.* (2020). According to ISO 4371-2012, *P. graveolens* essential oil from different geographical origins should have citronellol (18-43%), geraniol (5–20%), linalool (2-–11%), citronellyl formate (4-12%), isomenthone (4-10%), geranyl formate (1–8%), (*Z*)-r*ose* oxide (0.4-3.5%), menthone (0.0-2.5%) and geranyl tiglate (0.7-2.0%) as the main components (ISO, 2012). Considered one of the top 20 oils in the world, the essential oil of rose-scented geranium was extensively used as a flavoring agent in the food, soaps and beverages industry, cosmetic, perfumery, aromatherapy, traditional medicine, and pharmaceutical industries. Rose-scented geranium is famous for its strong rose-like pleasant fragrance; it is cultivated due to its high-value essential oil used in herbal medicine and aromatherapy and the production of high-quality perfumes and cosmetics. Also, *P. graveolens* essential oils or/and extracts are well known for their sensory attributes and pharmacological properties, antioxidant, antibacterial, antifungal, antimicrobial, insecticidal, allelopathic, anti-aflatoxin, anti-urease, anti-tyrosinase, therapeutic, repellent, fumigant and photoprotective effects (Lohani *et al.,* 2019; Ben ElHadj *et al.*, 2020; Kujur *et al.,* 2020). Different drying methods have been developed for the quality product and high-quantity products, and it has been observed that the essential oil content and components depend on the drying conditions, drying method, and plant species (Özgüven *et al.*, 2019). A previous report showed that drying methods and temperature had a significant effect on quality indicators such as organoleptic and sensory properties, oil content, and composition in medicinal and aromatic plants such as *Laurus nobilis* (Sekeroglu *et al.,* 2007), *Mentha longifolia* (Saeidi *et al.,* 2016), *Mentha pulegium* (Ahmed *et al.,* 2018), *Ocimum americanum* (Bhatt *et al.,* 2018), *Thymus daenensis* (Mashkani *et al.,*2018), *Lippia citriodora* (Aghdam *et al.,* 2019), *Lavandula angustifolia* (Sałata *et al.,* 2020) and *Dracocephalum moldavica* (Morshedloo *et al.,* 2021). In most cases, it has been reported for many plants that increasing drying temperature lowers the essential oil content, while keeping the temperature below 30-35 °C preserves more aromatic compounds (Mashkani *et al.,* 2018; Sałata *et al.,* 2020). Drying techniques affect the essential oil yield and composition, so it is very important to determine an appropriate drying method to achieve higher active substances in medicinal and aromatic plants. The drying methods may differ from one aromatic herb and spice to another. For medicinal and aromatic plants which are sensitive to the drying process, optimum drying is required to obtain a high-quality product, as some bioactive compounds change during the drying process. Therefore, the optimization of quality requires studying each specific pre-drying and drying method for each type of herb (Thamkaew *et al.,* 2021). Although a large number of herb-drying studies have been conducted in recent years, as far as we know, studies on the effectiveness of different drying methods on the quantity, quality, and composition of the essential oil of rose-scented geranium are scarce. The present study aimed to determine the influence of different drying methods, which included fresh plants (control), sun-drying, shade-drying, oven-drying at 30 °C, and oven-drying at 60 °C on dry herbage yield and essential oil content and composition of rose-scented geranium.

2. MATERIALS AND METHODS

2.1. Sample preparation

Pelargonium graveolens was grown in the Burhaniye Aromatic Plants Field Station, Balıkesir Metropolitan Municipality Rural Services Department during the 2019 growing season. The fresh aerial parts of *Pelargonium graveolens* (Geraniaceae) which were used in this research were harvested at the flowering phase during a one-year vegetative cycle. The harvested plants were then randomly divided into five groups containing three sets of 700 g of fresh weight in each method. While one of the sets was used as a fresh sample, different drying methods were applied to the others, including shade-drying, sun-drying, oven-drying at 30 °C, and oven-drying at 60 °C. The initial moisture content was determined at 105 °C for 7 h in the oven until there was no change in weight in two measurements.

2.2. Drying methods

The samples were divided into five batches containing 700 g of fresh weight in 3 replicates for each method. The methods were shade-drying at room temperature of 20-25 °C, sun-drying under direct sunlight at 24-27 °C, oven-drying at 30 °C, and oven-drying at 60 °C. In all drying methods, drying was continued until final moisture content reached approximately 10% on a wet basis. Then, when the constant weight was reached, they were ready for essential oil extraction. Shade-drying was carried out at a dark and dry room temperature under natural airflow, without exposure to direct sunlight, 5 cm layer thickness, and shelves on top of each other. For the sun-drying method, a clean white cloth was laid on a cage net 20 cm above the ground in an open area and the samples were dried under direct sunlight by mixing regularly. For the oven-drying method, samples were dried in a laboratory oven (Venticell, Germany) and two temperatures of 30 and 60 °C were used.

2.3. Extraction of essential oils and analysis

To obtain essential oil, 400 g of plant samples, which were subject to different drying methods, were used. The samples included in each application were divided into four as 100 g each. Three of these four samples were used in three replicates to obtain the essential oil. One was reserved as a spare. In each repetition of each method, 100 g of plant samples were ground to obtain essential oil, and immediately after grinding, using 400 mL of distilled water, it was distilled with a Clevenger device (S-H LTD., Ankara, Turkey) for three hours. The essential oil samples obtained were stored at 4 °C in the dark until analysis.

The essential oil analyses and identification were performed using Gas Chromatography-Mass Spectrometry analyses (GC/MS). GC/MS analyses were carried out on an Agilent 7890A GC system equipped with a J&W DB-Wax fused silica capillary 122-7061 column (250 °C: 60 m x 250 μm x 0.15 μm), and 5975C model MS and flame ionization detector (FID) were used simultaneously. The initial temperature of the column was kept at 50 °C, held for 1 min, and gradually increased from 25 °C/min to 200 °C, and then reached 230 °C at 3 °C/min, held for 15 min. The injection volume was 1 μL neat with a split ratio of 50:1. Helium was the carrier gas, used at a constant pressure of 10 psi and a flow rate of 1.0 mL/min. The compounds were identified using the Whiley and NIST Mass Spectral Library data of the GC/MS system, and by comparing the MS and retention index data with the mass spectral literature data (Adams, 2007). The percentages of each component were reported as raw percentages based on total ion current without standardization of each drying method. Changes in the essential oil composition of *Pelargonium graveolens* using different drying methods is summarized in Table 1.

2.4. Statistical analysis

The data were analyzed using the analysis of variance in SPSS. The mean of the main constituents for the essential oil values was compared using Duncan's multiple range test at 1% confidence interval. In order to visually evaluate the changes in terpene classes according to drying methods, a PCA biplot consisting of drying methods and terpenes was created. The biplot suggested by Yan and Rajcan (2002), was also applied to investigate variations within the different studies based on multi-traits data. The PCA biplot, the correlation coefficient between any two terpene classes is approximated by the cosine of the angle between their vectors. Acute angles indicate positive correlations, obtuse angles indicate negative correlation, and right angles indicate no correlations between two

Table 1: Chemical composition of essential oils obtained from aerial part of *Pelargonium graveolens* subjected to different drying methods (n=3)

RI[a]	Constituents[c]	Fresh plant	Methods[b]			
			Shade-drying	Sun-drying	Oven-drying at 30 °C	Oven-drying at 60 °C
1136	α-Pinene	0.48 ± 0.04	0.54 ± 0.03	0.85 ± 0.01	0.61 ± 0.05	0.20 ± 0.04
1161	2H-Pyran	0.07 ± 0.01	0.06 ± 0.01	ND	0.06 ± 0.01	ND
1168	Pentanoic acid. 4-methyl, methyl ester	0.10 ± 0.00	0.08 ± 0.02	0.16 ± 0.01	0.22 ± 0.03	ND
1169	Cyclopentasiloxane. decamethyl-	0.03 ± 0.02	0.06 ± 0.03	0.03 ± 0.01	ND	0.39 ± 0.09
1173	β-Pinene	0.05 ± 0.02	ND	0.19 ± 0.03	ND	ND
1175	Myrcene	0.16 ± 0.02	0.15 ± 0.01	0.12 ± 0.01	0.14 ± 0.01	0.14 ± 0.01
1177	α-Phellandrene	0.08 ± 0.01	0.04 ± 0.00	0.06 ± 0.01	0.10± 0.01	ND
1182	2-Butenoic acid. 2-methyl, methyl ester (E)	0.76 ± 0.02	0.20 ± 0.05	1.12 ± 0.06	0.70 ± 0.03	ND
1188	Limonene	0.15 ± 0.01	0.16 ± 0.01	0.17 ± 0.01	0.18 ± 0.01	ND
1192	Sabinene	0.08 ± 0.01		0.08 ± 0.00	0.12 ± 0.01	ND
1200	β-Ocimene. (E)-	0.17 ± 0.01	0.29 ± 0.02	0.03 ± 0.02	0.18 ± 0.01	ND
1209	β-Ocimene. (Z)-	0.18 ± 0.01	0.2 ± 0.01	0.21 ± 0.01	0.17 ± 0.01	ND
1210	p-Cymene	0.22 ± 0.02	0.18 ± 0.01	0.17 ± 0.01	0.21 ± 0.01	0.15 ± 0.02
1211	Heptadecanoic acid. methyl ester	0.04 ± 0.01	0.04 ± 0.01	0.08 ± 0.01	0.08 ± 0.01	ND
1214	Terpinolene	0.05 ± 0.01	0.07 ± 0.00	0.07 ± 0.01	ND	ND
1218	Cyclohexasiloxane	0.08 ± 0.01	0.27 ± 0.01	0.37 ± 0.03	0.08 ± 0.01	1.51 ± 0.07
1227	6-Methyl-5-hepten-2-one	0.12 ± 0.01	ND	ND	0.14 ± 0.01	ND
1240	*cis*-Rose oxide	0.71 ± 0.01 d	2.00 ± 0.29 c	3.15 ± 0.02 a	1.04 ± 0.12 d	2.46 ± 0.17 b
1254	Pentadecane	0.09 ± 0.01	ND	0.09 ± 0.00	ND	0.35 ± 0.01
1260	Linalool oxide	0.09 ± 0.24	ND	ND	ND	0.29 ± 0.01
1267	2-Ethyl-1-hexanol	5.4 ± 0.17	1.61 ± 0.40	4.02 ± 0.20	3.34 ± 0.48	1.43 ± 0.07
1281	Menthone	5.51 ± 0.11 b	4.48 ± 0.15 c	8.34 ± 0.30 a	6.52 ± 0.19 b	4.55 ± 0.03 c
1283	Linalool	6.27 ± 0.38 b	7.42 ± 0.44 a	2.88 d ± 0.07 d	3.78 ± 0.19 c	1.96 ± 0.01 e
1289	β-Bourbonene	1.22 ± 0.09 c	0.95 ± 0.05 d	1.83 ± 0.08 b	1.34 ± 0.16 c	2.23 ± 0.13 a
1291	α-Gurjunene	0.27 ± 0.01	ND	ND	ND	0.27 ± 0.01
1292	Aromadendrene	0.28 ± 0.00	ND	0.46 ± 0.01	0.32 ± 0.01	0.56 ± 0.01
1301	Isopulegol	0.07 ± 0.02	0.32 ± 0.01	0.09 ± 0.01	0.12 ± 0.02	0.06 ± 0.01
1303	α-Bergamotene	ND	ND	0.19 ± 0.01	ND	0.12 ± 0.01
1305	α-Guaiene	0.12 ± 0.01	ND	ND	0.13 ± 0.01	ND
1308	Citronellyl formate	ND	ND	ND	ND	4.94 ± 0.02
1310	β-Copaene	ND	ND	ND	ND	0.31 ± 0.1
1312	β-Elemene	0.97 ± 0.04	ND	0.65 ± 0.01	0.82 ± 0.06	0.25 ± 0.02
1316	β-Caryophyllene	4.32 ± 0.18 a	3.36 ± 0.2 b	3.04 ± 0.02 c	4.27 ± 0.05 a	2.63 ± 0.03 c
1327	α-Elemene	0.52 ± 0.03	0.49 ± 0.04	0.18 ± 0.02	ND	0.32 ± 0.02
1334	γ -Muurolene	0.73 ± 0.03	0.04 ± 0.01	0.49 ± 0.01	ND	1.52 ± 0.14
1327	trans-Muurola-3.5-diene	0.38 ± 0.01	ND	0.21 ± 0.01	0.23 ± 0.01	ND
1329	Germacrene D	0.19 ± 0.01	ND	0.20 ± 0.01	0.19 ± 0.01	0.15 ± 0.01
1333	Alloaromadendrene	0.12 ± 0.01	0.72 ± 0.01	ND	0.38 ± 0.01	0.35 ± 0.01
1334	Valencene	0.91 ± 0.02	0.16 ± 0.01	ND	1.04 ± 0.01	ND
1335	Geranyl formate	ND	0.45 ± 0.04	ND	ND	0.79 ± 0.02
1336	Neral	0.45 ± 0.05	0.27 ± 0.01	ND	0.43 ± 0.01	0 ± 0
1339	α-Terpineol	0.61 ± 0.01	0.72 ± 0.02	0.26 ± 0.01	0.39 ± 0.02	0.24 ± 0.01
1342	α-Humulene	1.15 ± 0.06	0.86 ± 0.02	0.85 ± 0.02	1.14 ± 0.03	0.73 ± 0.04
1343	Isoledene	0.54 ± 0.01	0.46 ± 0.01	0.28 ± 0.01	0.65 ± 0.02	ND
1348	Viridiflorene	3.16 ± 0.1 a	2.22 ± 0.05 c	2.69 ± 0.26 b	3.31 ± 0.01 a	3.28 ± 0.09 a
1356	Citronellol	33.06 ± 0.77 b	39.87 ± 0.23 a	27.84 ± 1.21 c	34.67 ± 0.46 b	23.99 ± 0.48 d
1364	Bicyclogermacrene	0.6 ± 0.03	0.33 ± 0	0.29 ± 0.01	0.47 ± 0.01	ND
1366	β-Selinene	ND	ND	ND	ND	0.64 ± 0.01
1367	δ-cadinene	1.17 ± 0.01 c	1.06 ± 0.02 c	1.71 ± 0.11 b	0.05 ± 0.23 d	3.30 ± 0.05 a
1369	*cis*-Muurola-3.5-diene	ND	0.18 ± 0.01	0.21 ± 0.02	1.36 ± 0.20	ND
1371	Nerol	1.31 ± 0.02	1.03 ± 0.01	1.30 ± 0.03	1.17 ± 0.01	1.23 ± 0.02
1372	γ -*Cadin*ene	0.18 ± 0.01	0.15 ± 0.01	0.98 ± 0.02	0.20 ± 0.01	ND

RI[a]	Constituent[c]	Fresh plant	Methods[b]			
			Shade-drying	Sun-drying	Oven-drying at 30 °C	Oven-drying at 60 °C
1374	Geranyl isobutyrate	ND	0.22 ± 0.01	0.10 ± 0.02	ND	0.44 ± 0.01
1377	Geranyl propionate	ND	ND	0.22 ± 0.01	ND	1.10 ± 0.03
1381	α-Cubebene	0.15 ± 0.01	0.17 ± 0.01	0.21 ± 0.01	0.16 ± 0.01	0.14 ± 0.01
1387	Geraniol	16.26 ± 0.07 a	17.09 ± 0.12 a	11.18 ± 0.59 c	14.05 ± 0.26 b	4.15 ± 0.05 d
1398	Aromadendrene. dehydro	0.08 ± 0.00	0.12 ± 0.02	0.09 ± 0.00	0.06 ± 0.01	0.3 ± 0.02
1399	(+)-Calamenene	0.32 ± 0.00	0.33 ± 0.02	0.46 ± 0.01	0.45 ± 0.01	0.95 ± 0.01
1410	Geranyl isovalerate	0.25 ± 0.01	0.22 ± 0.00	0.28 ± 0.04	0.30 ± 0.03	1.29 ± 0.01
1428	2-Phenylethyl alcohol	0.80 ± 0.06	0.28 ± 0.01	1.25 ± 0.04	0.76 ± 0.01	0.14 ± 0.01
1435	2.6-Octadiene. 2.6-dimethyl	0.41 ± 0.02	0.36 ± 0.01	0.46 ± 0.01	0.38 ± 0.01	1.14 ± 0.04
1442	α-Calacorene	0.06 ± 0.01	0.05 ± 0.00	0.21 ± 0.01	0.10 ± 0.01	0.37 ± 0.02
1446	10-epi-cubebol	0.06 ± 0.00	ND	ND	0.06 ± 0.01	ND
1449	5.11-Epoxycadin-1(10)-ene	0.27 ± 0.03	0.26 ± 0.01	0.90 ± 0.01	0.41 ± 0.01	1.35 ± 0.05
1452	(E.Z)-α-Farnesene	ND	0.15 ± 0.01	ND	0.06 ± 0.01	ND
1454	Alloaromadendrene oxide-(1)	ND	ND	0.22 ± 0	0.12 ± 0.01	0.21 ± 0.01
1459	Furopelargone A	0.14 ± 0.01	0.09 ± 0.00	0.36 ± 0.02	0.23 ± 0.01	ND
1472	Citronellyl butyrate	0.66 ± 0.06 bc	0.53 ± 0.02 c	0.81 ± 0.01 bc	1.06 ± 0.08 b	5.31 ± 0.11 a
1485	Geranyl butyrate	ND	0.27 ± 0.02	0.32 ± 0.01	ND	0.77 ± 0.02
1490	Caryophyllene oxide	0.40 ± 0.01	0.55 ± 0.02	1.40 ± 0.03	0.58 ± 0.01	0.76 ± 0.03
1493	Bicyclogermacrene	0.07 ± 0.22	ND	ND	ND	0.28 ± 0.00
1509	Ledol	0.23 ± 0.01	0.27 ± 0.01	0.60 ± 0.01	0.27 ± 0.00	0.79 ± 0.01
1511	1.10-di-*epi*-Cubenol	0.75 ± 0.01	0.57 ± 0.01	1.19 ± 0	1.37 ± 0.05	0.46 ± 0.01
1517	Cubenene	ND	ND	0.20 ± 0.01	ND	0.67 ± 0.01
1526	Cadina-1.4-diene	ND	0.43 ± 0.01	0.42 ± 0.01	ND	0.76 ± 0.01
1528	1-*epi*-Cubenol	0.35 ± 0.02	0.57 ± 0.02	0.45 ± 0.05	0.38 ± 0.03	0.51 ± 0.04
1531	Methyl cinnamate	ND	ND	0.4 ± 0.01	ND	ND
1537	Geranyl tiglate	1.17 ± 0.25 c	1.68 ± 0.07 b	0.99 ± 0.04 c	1.75 ± 0.15 b	4.52 ± 0.03 a
1541	Globulol	0.32 ± 0.01	ND	0.28 ± 0.02	ND	0.17 ± 0.01
1544	Hexahydrofarnesylacetone	ND	ND	0.88 ± 0.01	ND	1.36 ± 0.01
1554	α-Eudesmol	0.07 ± 0.01	0.08 ± 0.01	0.06 ± 0.00	0.09 ± 0.02	ND
1564	Spathulenol	0.52 ± 0.02	0.36 ± 0.01	0.98 ± 0.01	0.55 ± 0.02	1.69 ± 0.02
1575	Cedrol	ND	ND	0.73 ± 0.01	ND	ND
1583	Farnesol 2	ND	ND	0.19 ± 0.01	ND	0.12 ± 0.01
1588	Geranyl acetate	0.06 ± 0.01	0.07 ± 0.01	0.13 ± 0.01	0.08 ± 0.01	0.16 ± 0.01
1596	tau-Cadinol	ND	ND	0.55 ± 0.01	ND	0.24 ± 0.01
1598	*trans*-Cadina-1(6).4-diene	0.38 ± 0.02	0.15 ± 0.01	0.22 ± 0.01	ND	ND
1616	2-Phenylethyl tiglate	0.51 ± 0.02 e	1.34 ± 0.11 c	1.74 ± 0.29 b	0.96 ± 0.15 d	3.43 ± 0.05 a
1641	Isospathulenol	0.09 ± 0.25	0.14 ± 0.01	0.24 ± 0.02	0.16 ± 0.01	ND
1652	α-Cadinol	0.40 ± 0.03	0.39 ± 0.01	0.55 ± 0.07	0.75 ± 0.06	0.65 ± 0.04
1678	1.4-Benzenedicarboxylicaciddimethyl ester	1.38 ± 0.17	ND	1.18 ± 0.01	1.07 ± 0.01	0.32 ± 0.03
1723	Caryophylladienol I	ND	ND	0.25 ± 0.01	ND	0.31 ± 0.01
1757	Caryophylla-3.8(13)-dien-5β-ol	ND	ND	0.24 ± 0.01	0.26 ± 0.01	0.48 ± 0.01
	Monoterpenes (M)	66.37 ± 1.17 b	75.19 ± 2.72 a	57.62 ± 0.74 c	64.26 ± 1.22 b	40.53 ± 0.8 d
	Monoterpenes hydrocarbons (MH)	2.03 ± 0.03 b	1.99 ± 0.06 b	2.58 ± 0.06 a	2.09 ± 0.08 b	1.63 ± 0.04 c
	Oxygenated monoterpenes (OM)	64.34 ± 0.38 b	73.2 ± 1.32 a	55.04 ± 0.57 c	62.17 ± 2.01 b	38.9 ± 3.04 d
	Sesquiterpenes (S)	22.8 ± 0.41 c	18.29 ± 0.69 d	27.25 ± 0.97 b	24.27 ± 0.35 c	41.69 ± 0.45 a
	Sesquiterpenes hydrocarbons (SH)	18.88 ± 0.37 b	14.25 ± 0.56 c	18.62 ± 0.31 b	18.74 ± 0.75 b	29.19 ± 0.31 a
	Oxygenated sesquiterpenes (OS)	3.92 ± 0.05 d	4.04 ± 0.06 d	8.63 ± 0.26 b	5.53 ± 0.20 c	12.56 ± 0.33 a
	Others (O)	9.94 ± 0.16 c	4.45 ± 0.49 e	12.09 ± 0.29 b	8.28 ± 0.69 d	14.41 ± 0.13 a
	Total (%)	99.11 ± 0.33	98.23 ± 0.47	96.96 ± 0.29	96.81 ± 0.19	96.63 ± 0.29

[a]RI: retention Index, [b]: Averages of the same linear values (each section separately) followed by same letter did not differ significantly from Duncan's multiple range tests at 0.01% significance. [c]: Mean value ± standard deviation, and the mean values of the components of each drying method were based on the average of three replicates. ND: not detected.

classes. The length of the vector describes the discriminating ability of the terpene class. A terpene class with a short vector indicates that the class is not associated with other classes, lacks variation, or is not useful for drying method discrimination (Akçura, 2011). A PCA biplot analysis was performed using GGE biplot software (Yan, 2001).

3. RESULTS AND DISCUSSION

The results showed that the drying methods had a significant effect on EO content (Figure 1). Fresh plant and samples dried by shade-drying and by oven at 30 °C showed high EO contents (1.98, 1.34, and 1.20%, respectively). In contrast, low essential oil content of 0.70 and 0.42% was obtained from sun-drying and oven-drying at 60 °C. The methods fresh sample, shade-drying, and oven-drying at 30 °C resulted in higher EO, while increasing temperature (from 30 to 60 °C) showed a decrease in EO content. Similarly, Çalışkan *et al.* (2017) in *Mentha piperita*, found higher essential oil content in shade-drying and oven-drying at 38 °C than sun-drying. Although not as much as the fresh plant material, both shade-drying at ambient temperature and oven-drying at 30 °C gave feasible results in terms of oil yield. The changes in essential oil content during the drying process depended on temperature, time, and drying method. Similarly, Sourestani *et al.* (2014) in *Agastache foeniculum*, found higher essential oil content at room temperature (25 °C) than oven-drying at 40 °C. Shade-drying and oven-drying (at 30 °C) methods are considered efficient to achieve the best EO quality and quantity (Saeidi *et al.,* 2016). Some studies pointed out that increased drying temperature can damage glandular trichomes, decomposition of some essential oil components through high-temperature autoxidation and hydro peroxidation (Turek and Stintzing, 2006), and accelerated evaporation and decomposition of essential oil components (Mashkani *et al.,* 2018) can cause a decrease in essential oil content.

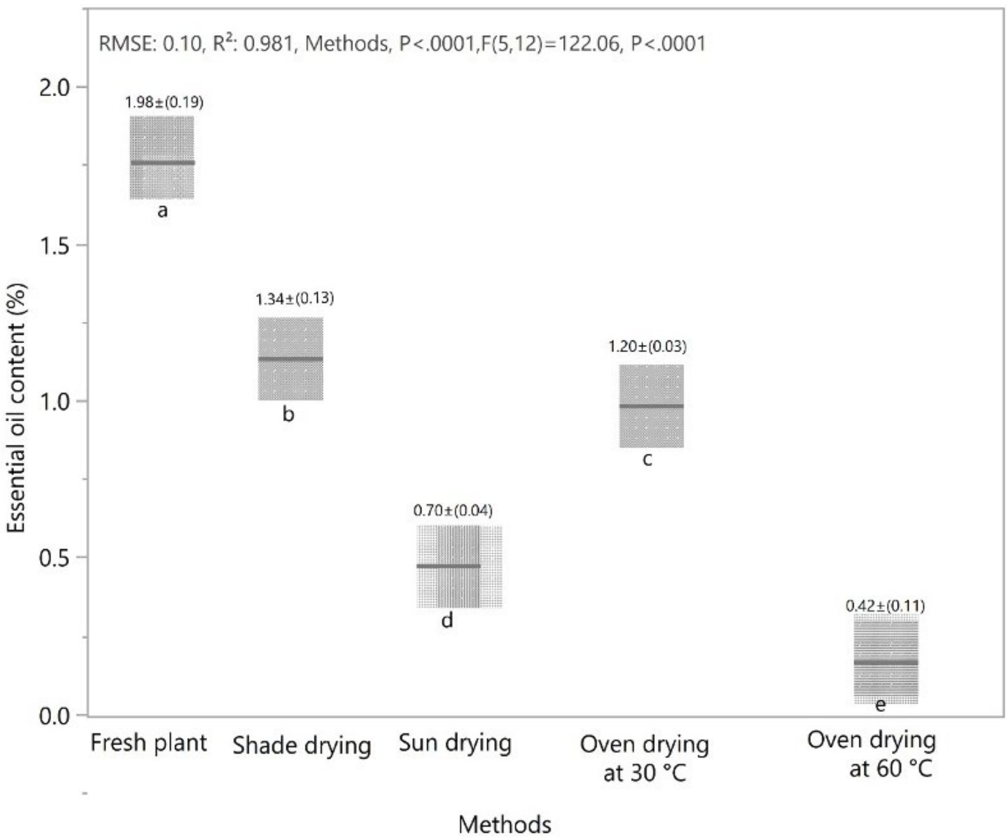

FIGURE 1. The comparison mean of the essential oil content (%) of *Pelargonium graveolens* changes according to the different drying methods (n=3). Results are expressed as means ± standard error indicated on the box plot. Means of essential oil content followed by similar letters in boxess are not significantly different at 1% probability level by the LSD test (LSD value= 0.1932). The results of the essential oil content for each drying method are based on the average of three replicates.

Ninety-three components were identified in EO samples of rose-scented geranium by using different drying methods comprising 96.63 to 99.11% of total EO (Table 1). The majority of them consisted of oxygenated monoterpenes and sesquiterpene hydrocarbons. The main components in the EO in all drying methods were citronellol (24.0-39.9%), geraniol (4.2-17.1%), menthone (4.5-8.3%), and linalool (2.0-7.4%). Other main components in the oil were found to be β-caryophyllene, *cis*-rose oxide, geranyl tiglate, citronellyl butyrate, viridiflorene, 2-phenylethyl tiglate, β-bourbonene, δ-cadinene and nerol. These results are consistent with previous reports (Juliani *et al.,* 2006), which demonstrated that commercial geranium oils are characterized by high citronellol (19-45%) and lower amounts of geraniol (less than 24%) and linalool (less than 14%) as the main components.

Citronellol and geraniol, which are oxygenated monoterpene, reached their highest values under natural shade-drying conditions; whereas the lowest value was achieved by artificial oven-drying at 60 °C. Since high temperatures cause a large loss in citronellol, geraniol, and linalool contents, these should be considered to be compounds which are sensitive to direct sunlight, high and low temperatures. The results demonstrated that the aerial parts of geranium with sun-drying, oven-drying at 30 °C, and oven-drying at 60 °C presented decreased citronellol content by about 12.03, 5.20, and 15.88% as compared to the shade-drying, with geraniol content by 5.91, 3.04, and 12.94% and linalool content by 4.54, 3.64, and 5.46%, respectively. Oxygenated monoterpenes with sweet rose-like (citronellol) and flowery rose-like (geraniol) odor in geranium oil are important reasons for the demand for perfumery.

The highest percentage of δ-cadinene, citronellyl butyrate, geranyl tiglate and 2-phenylethyl tiglate were obtained from samples dried in an oven 60 °C;, while the highest amount of linalool and β-caryophyllene were obtained from the sample dried by shade ambient temperature and the fresh sample. Increasing the drying temperature from 30 to 60 °C significantly reduced the contents in citronellol, geraniol, β-caryophyllene, menthone, and linalool in the dried aerial parts of geranium; whereas the contents in citronellyl butyrate, geranyl tiglate, 2-phenylethyl tiglate, δ-cadinene and spathulenol increased. Citronellyl formate, β-copaene, and β-selinene were detected only in oven-dried samples at 60 °C. The sun-drying method had a stimulative effect on some other compounds' biosynthesis and accumulation such as α-pinene, cis-rose oxide, menthone, δ-cadinene, γ-cadinene, and caryophyllene oxide. The drying method affected the geranium's chemical profiles and caused significant changes in the contents in citronellol, geraniol, linalool, menthone, and β-caryophyllene, which are the main compounds in the EO.

Factors such as plant species, drying method, drying conditions and time, amount of water evaporated during drying, temperature, the chemical structure of the compounds, oxidation, chemical reactions, degradation, isomerization, cyclization, dehydrogenation, glycoside hydrolysis, autoxidation of terpenoids, esterification and/or other processes could significantly change the chemical profiles of EO and some of the EO compounds may be lost, reduced and/or increased (Ahmed *et al.,* 2018; Bhatt *et al.,* 2018; Beigi *et al.,* 2018; Özgüven *et al.,* 2019; Thamkaew *et al.,* 2021). One of the most important chemical changes is due to the autoxidation of oil components that affect the deterioration process of terpenoids, and increasing drying temperature and exposure to direct sunlight causes further loss in aroma components and degradation of aroma quality (Başer and Demirci, 2011; Thamkaew *et al.,* 2021). Compared to shade drying, especially in an oven at 60 °C and sun drying, the volatile profile of the EO changed due to the formation of secondary aroma compounds such as terpene esters, sesquiterpenes, alcohols, aldehydes, and others. During drying, the EO composition and content increased, decreased and the production of new compounds occurred. During the drying process, the EO compositions of the plants changed, which may be a result of the release of components from the rupture of their cell walls, oxidation reactions, or hydrolysis of glycosylated volatile compounds (Xing *et al.,* 2018).

Among the compounds identified in EO, the percentage of oxygenated monoterpenes (OM) was the highest, ranging from 38.9 to 73.2%. EO extracted from plants dried at 30 and 60 °C and sun-dried contained more sesquiterpenes and fewer monoterpenes compared to fresh samples and drying in shady, natural conditions. Sun and oven drying most reduced the contents in compounds from the OM groups, while fresh and drying in shady, nat-

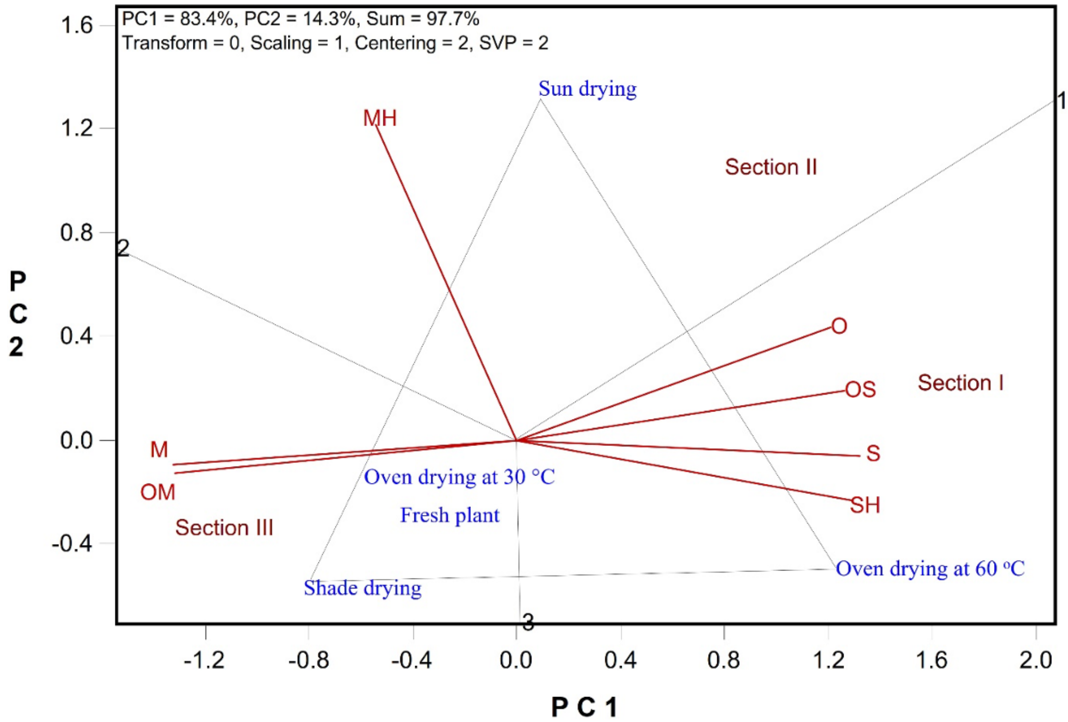

Figure 2. PCA biplot of the changes in active ingredient groups of geranium essential oil according to drying methods and the relations between groups. M: Monoterpenes, MH:Monoterpenes hydrocarbons, OM: Oxygenated monoterpenes, S: Sesquiterpenes, SH: Sesquiterpenes hydrocarbons, OS: Oxygenated sesquiterpenes, O: Others

ural conditions reduced the contents in compounds from the SH and OS groups. Drying of *Pelargonium graveolens* in the shade was most suitable for a high percentage of OM. Aghdam *et al.* (2019), who found high monoterpene content in fresh lemon verbena plants and sesquiterpene content in oven drying, presented similar conclusions.

Drying in direct sun resulted in a reduction in the contents in EO, citronellol, geraniol, linalool, and β-caryophyllene compared to fresh samples, shade-drying, and oven-drying at 30 °C, so it may not be an appropriate drying method for geranium. The shade-drying method preserved the EO content and the major volatile components in geranium better than the oven-drying and sun-drying methods. However, although the drying time of shade-drying is longer than sun-drying, it can provide advantages in terms of preserving light-sensitive substances and minimizing light-dependent chemical reactions such as oxidation. In terms of EO content and components, it was found that drying with hot air at 60 °C is not appropriate, but low drying temperature (30 °C) is appropriate for oil content and preservation of heat-sensitive compounds in geranium.

The PCA-biplot created to visually evaluate the changes in terpene classes according to drying methods is shown in Figure 2. The PCA biplot in this study captured 97.7% of the variations due to drying method and drying method by terpene group interactions. Polygons were created to evaluate drying methods in the biplot, and drying methods are presented with vectors. Terpenes, which had a positive relationship with drying methods, were located close to each other. In the three sections on the biplot, drying methods and terpene classes, which are positively related, formed three groups. Oxygenated sesquiterpenes (OS), sesquiterpenes (S), sesquiterpenes hydrocarbons (SH) and others (O) increased with oven-drying at 60 °C, monoterpenes (M) and oxygenated monoterpenes (OM) increased with sun-drying, monoterpenes hydrocarbons (MH) increased with shade-drying, fresh plant and oven-drying at 30 °C methods (Figure 2).

4. CONCLUSIONS

The quality of medicinal dried herbs is defined by the content in bioactive compounds. The drying methods had a significant impact on the essential oil content and composition, which is the quality indicator of *Pelargonium graveolens* L'Hér. While high temperature reduced the rate of EO in oven-drying, fresh plant and shade-drying were found to be more appropriate in terms of oil content and components compared to the other methods. A significant difference was noted in the percentage of main constituents such as citronellol, geraniol, linalool, β-caryophyllene geranyl acetate, geranyl tiglate, citronellyl butyrate and viridiflorene between the different natural and artificial drying methods, as well as between them and fresh samples. While the highest oxygen monoterpene contents were identified in shade-dried and fresh plants, oven-drying had a stimulating effect on the biosynthesis and accumulation of sesquiterpene compounds. The data can be used by pharmaceutical and perfumery industries in their post-harvesting programs. The results of this study showed that drying *Pelargonium graveolens* in natural shade is more suitable for high oil yield and oxygenated monoterpene content while drying this plant in the oven at 30 °C can be recommended to shorten the drying process.

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RESEARCH DATA POLICY DATA AVAILABILITY

The authors declare no conflict of interest relating to the article.

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