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Research Article

Exploring the influence of different *Froriepia subpinnata* drying methods on its essential oils components

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ABSTRACT- Anarijeh (*Froriepia subpinnata*) is a species in the Apiaceae family, endemic to northern Iran, where it is widely distributed. In this study, we subjected the aerial parts of Anarijeh to various drying methods, including sun drying, shade drying, oven drying at 45 and 65 °C, microwave drying, and freeze drying. Essential oils (EOs) were extracted from both fresh and dried samples using hydro-distillation and analyzed via gas chromatography–mass spectrometry (GC-MS). The results showed that oven drying at 45 °C (2.83%), oven drying at 65 °C (2.81%), sun drying (2.79%), and shade drying (2.74%) produced the highest EOs yields. In contrast, freeze-dried (1.42%), microwave-dried (1.39%), and fresh (1.34%) samples yielded lower percentages. A total of 53 components were identified in the EOs, with the major compounds being p-cymen-7-ol (10.3–19.95%), durenol (20.49–28.88%), and terpinolene (5.63–20.47%). These findings suggest that oven drying may be a suitable method for processing Anarijeh aerial parts, offering both shorter drying times and higher EOs yields. However, as no statistical analyses were conducted, we cannot definitively conclude that the differences between oven drying and shade drying are significant.

INTRODUCTION

Froriepia subpinnata, commonly known as Anarijeh, is primarily found in southern and central Europe and northern Iran, particularly in the provinces of Guilan, Mazandaran, and Golestan (Vaseghi et al., 2018). In Iran, this species is represented by a single taxon (Amiri and Joharchi, 2016). Anarijeh is a perennial plant characterized by white flowers and a height that ranges from 20 to 110 cm. Locally, the leaves are traditionally used as herbs, flavoring agents, and spices in the preparation of aromatic regional dishes, particularly in early spring (Mohammadzadeh et al., 2018). In traditional medicine, *F. subpinnata* is employed for treating liver disorders and is valued for its carminative, antispasmodic, diuretic, sedative, and tonic effects (Mohammadzadeh et al., 2018; Mirzania et al., 2019). Its essential oils (EOs) has also demonstrated anticancer and antioxidant properties (Vaseghi et al., 2018). Drying is a common method for preserving herbs, as it reduces moisture content, thereby inhibiting microbial growth and slowing chemical degradation during storage (Díaz-Maroto et al., 2002). Dried herbs are widely used as flavor enhancers, contributing their distinctive aromas to culinary applications (Bor et al., 2016).

Beyond culinary uses, EOs derived from herbs are utilized for their antimicrobial properties and are found in medicinal products, hygiene items, and perfumes (Embuscado, 2015; Bor et al., 2016). The quality of dried herbs depends on their intended use. For medicinal purposes, the concentration of bioactive compounds is the key quality indicator (Ebadi et al., 2015), whereas for culinary herbs, desirable attributes include vibrant color and a fresh, characteristic aroma (Rahimmalek and Goli, 2013). Various drying pretreatments such as differences in temperature, duration, environmental conditions, and equipment can influence the content and profile of volatile compounds in both food and medicinal products (Calín-Sánchez et al., 2013). However, drying treatments can also negatively affect herb quality. Heat exposure can lead to nutrient loss, color changes, and the generation of oxidation products, which collectively impact aroma and appearance (Ozdemir et al., 2018). Although the exact factors contributing to variability in volatile content are not fully understood, the type of herb and the specifics of the drying method are believed to play major roles. For example, Calvo-Irabien et al. (2009) studied the effects of various drying methods including shade drying, sun drying, and oven drying at 20 and 40 °C on the EOs content of *Lippia graveolens*, but reported no significant differences among treatments. Despite their small quantities, EOs are the principal contributors to the fragrance of herbs (Rao et al., 1998). These versatile compounds are used across multiple industries, including pharmaceuticals, cosmetics, medicine, and food (Orphanides et al., 2016). In fresh plants, EOs are stored in trichomes specialized structures composed of one or more cells that extend from the surface of plant organs such as leaves, roots, or bark. The retention of EOs in dried leaves depends on the structural integrity of these oil glands (Ebadi et al., 2015). Therefore, minimizing trichome

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damage during the drying process may help preserve EOs content and improve the aromatic quality of dried herbs. However, the application of heat during drying can generate free radicals, which promote the oxidation of EOs compounds (Choe and Min, 2006). Consequently, higher drying temperatures tend to increase the degradation of volatile constituents, leading to diminished fragrance quality in the final dried product.

In general, drying herbs leads to a reduction in volatile compounds, although some drying methods are more effective than others at preserving these constituents (Chua et al., 2019). Among the various factors influencing the retention of volatile components, drying temperature plays a particularly critical role. Elevated temperatures can accelerate the loss of volatile chemicals by promoting the rupture of trichomes specialized glandular structures that store EOs thereby facilitating the evaporation of their contents. Additionally, high drying temperatures can degrade heat-labile constituents within the EOs, further reducing their overall concentration (Argyropoulos and Müller, 2014). Since aroma compounds are highly heat-sensitive, they tend to evaporate rapidly during the drying of plant tissues (Khangholil and Rezaeinodehi, 2008). Interestingly, in some cases, higher drying temperatures may enhance the preservation of certain compounds. For instance, hot-air drying of lemon myrtle leaves at 50 °C resulted in greater citral concentrations in comparison with drying at 30 or 40 °C. This effect may be attributed to the formation of a crust layer on the leaf surface, which hinders the diffusion of high-molecular-weight volatiles from the internal tissues (Buchaillot et al., 2009).

Sun drying, one of the oldest and most commonly used drying techniques in tropical and subtropical regions, is often employed for agricultural crops. However, it may be unsuitable for certain herbs due to the quality concerns. Sun drying is known to cause significant losses in color and aroma and may also damage the epidermal surface and shrink glandular trichomes, as observed in *Vernonia amygdalina*, which also showed reduced mineral content under this method (Alara et al., 2018). Shade drying, a variation of sun drying, avoids direct exposure to sunlight by placing herbs in shaded, well-ventilated areas with low humidity (22–27% for *Lippia citriodora*) (Ebadi et al., 2015). This method better preserves light-sensitive compounds and minimizes light-induced oxidative reactions. However, it is even more time-consuming than sun drying, which is already considered as a prolonged process (Ropelewska et al., 2023). Thus, while shade drying offers quality advantages, its long duration remains a limitation for large-scale or commercial use. In industrial settings, oven (or hot-air) drying is the most commonly used method, particularly in non-tropical regions (Orphanides et al., 2016). Its popularity stems from the ability to precisely control drying parameters such as temperature, duration, and air velocity. Nevertheless, hot-air drying often leads to a substantial reduction in volatile components, along with noticeable losses in herb aroma and color (Chua et al., 2019; Fennell et al., 2004). Microwave drying has emerged as a promising alternative due to its rapid drying capability and lower energy requirements. This technique evaporates water quickly, significantly shortening drying time (Karimi et al., 2021). Another technique, freeze drying, is widely recognized for its ability to preserve a fresh-like aroma in dried herbs. Studies, including those on spearmint, have shown that freeze drying results in lower degradation of aroma compounds compared to hot-air drying, making it ideal for preserving the olfactory quality of dried herbs (Antal et al., 2011). Given that *F. subpinnata* (Anarijeh) contains EOs, the present study aims to evaluate how different drying methods influence the retention and composition of these oils.

MATERIALS AND METHODS

Experimental design and treatment

Fresh Anarijeh (*F. subpinnata*) samples were collected from northern Iran. The leaves and roots were thoroughly cleaned to remove any physical contaminants such as dirt and gravel. The plants, approximately two years old, were harvested prior to flowering. Table 1 summarizes the morphological characteristics of the plant. The lengths of the longest leaf, leaflets, terminal leaflets, petiole, and root were measured using a ruler with a precision of 1 mm. Leaf width, leaflet width, terminal leaflet width, and petiole width were also measured using a ruler. Calipers were employed to determine the diameters of the plant stems and roots. The number of leaves, leaflets, leaflet lobes, and major root branches was recorded through direct counting. Following these measurements, the Anarijeh plants were stored at 4 °C to preserve freshness prior to drying. A total of 600 g of stems and leaves were subjected to six different drying methods: sun drying, shade drying, oven drying at 45 and 65 °C, microwave drying, and freeze drying. For sun drying, the fresh plant materials were spread on a cloth under direct sunlight at an ambient temperature of 24 °C and allowed to dry for two days. This process resulted in a final moisture content of 4.66%. Shade drying was conducted in a well-ventilated, dimly lit room at a constant temperature of 20 °C for four days, with minimal light exposure to prevent overheating and photodegradation. This method yielded a moisture content of 5.83%. Oven drying was carried out using a laboratory oven (Osk, Japan). Plant samples were spread in a single layer on trays and dried at temperatures of 45 and 65 °C, resulting in moisture contents of 1.42% and 1.41%, respectively. Microwave drying was performed using a domestic digital microwave oven (Nikai, NMO-518N, Japan) operating at 230 V and 800 W. Samples were placed on small Petri dishes on a 29 cm rotating glass plate and exposed to microwave radiation for 15 minutes. The moisture content after drying was measured at 4.59%. For freeze drying, samples were placed in a Helto Holten DW8 freeze dryer. The frozen material was subjected to vacuum drying for six hours, with the condenser temperature maintained at -15 °C. This method yielded dried samples with a moisture content of 2.30%.

Table 1. Information of growth characteristics of the *Froriepia subpinnata* plants

Plants	Number of leaves	Length of leaves (cm)	Width of leaves (mm)	Number of leaflets	Length of leaflets (mm)	Width of leaflets (mm)	Length of the terminal leaflet (mm)	Width of the terminal leaflet (mm)	Number of leaflet lobes	Number of terminal leaflet lobes	Diameter of plant (mm)	High of rosette (cm)	Diameter of root (cm)	Length of root (cm)	Number of main root branches	Width of petiole (mm)	Length of petiole (cm)
1	21	24.50	27.31	27	13.68	11.75	11.04	10.67	7	3	17.98	59.94	11.22	23.30	1	5.69	55.80
2	12	18.10	35.02	17	17.94	15.70	10.71	10.06	7	3	12.87	27.17	6.49	12.74	1	3.26	36.04
3	15	25.10	31.12	21	16.13	13.19	9.84	9.24	7	3	12.20	45.52	6.79	11.60	1	5.54	57.99
4	16	26.20	38.89	21	18.52	16.25	9.43	9.31	7	3	13.83	49.52	8.10	19.60	1	4.24	78.85
5	12	20.30	31.62	17	15.57	14.58	9.57	8.10	7	3	11.55	48.79	7.01	11.50	1	5.58	52.96
6	10	21.50	26.97	21	13.47	11.41	8.84	7.19	7	3	10.40	70.90	6.15	13.40	1	5.40	61.82
7	14	22.00	30.47	21	14.67	11.27	11.44	12.11	7	5	12.22	119.11	9.04	23.40	1	4.29	42.27
8	8	19.50	30.36	15	15.31	12.29	11.71	14.75	7	5	7.77	55.33	4.66	14.10	1	4.97	64.43
9	8	19.60	27.01	15	13.80	11.67	9.94	9.30	7	3	6.52	31.82	5.86	11.20	1	4.08	74.00
10	10	20.40	34.32	19	19.70	13.34	11.92	16.32	9	5	7.23	24.49	5.65	11.80	1	3.60	64.80
11	12	22.00	31.62	21	17.62	14.25	10.10	9.91	9	3	8.27	64.39	8.30	21.00	1	5.23	68.60
12	6	21.50	32.42	19	16.48	14.42	11.11	11.30	7	3	6.32	47.54	4.08	8.50	1	4.62	78.27
13	16	20.00	24.10	23	12.20	11.14	8.32	8.02	7	3	12.23	65.70	7.01	17.00	1	4.40	52.35
14	8	20.00	30.47	21	17.48	12.90	9.72	13.40	9	5	7.89	56.91	4.42	11.50	1	4.60	68.95
15	14	26.30	32.37	21	16.37	16.96	13.82	10.91	7	3	12.16	150.00	9.76	15.00	1	4.63	54.37
16	13	22.50	25.00	25	13.34	9.60	10.90	12.46	9	7	9.09	52.76	7.18	15.50	1	4.57	59.38
17	18	23.50	27.83	21	13.08	12.54	10.84	11.61	9	5	12.68	55.13	6.72	14.50	1	5.48	53.41
18	8	28.30	38.25	17	18.67	16.31	12.19	16.83	7	5	7.37	50.60	6.41	10.50	1	5.25	9.98
19	11	19.00	26.58	21	13.97	14.30	8.89	9.32	7	3	9.56	77.28	6.02	14.00	1	5.83	56.60
20	10	19.00	34.52	15	15.24	16.88	11.90	12.66	7	3	9.51	42.37	6.73	15.50	2	6.21	86.18
21	13	15.60	37.62	15	17.47	14.86	13.35	20.03	9	7	10.42	40.30	7.66	9.70	4	5.57	39.64
22	14	24.10	39.33	21	20.61	16.24	11.01	14.52	9	5	11.31	58.75	9.01	12.00	1	7.37	73.47
23	11	23.50	26.28	19	13.54	11.21	9.99	11.20	9	5	10.12	90.51	4.96	10.50	1	4.86	66.99
24	13	18.50	34.43	21	16.84	13.97	8.32	8.82	9	3	9.13	58.14	4.91	10.50	3	4.80	63.72
25	11	20.00	25.50	19	13.63	11.31	11.79	12.19	7	5	11.38	48.89	6.48	13.00	4	5.13	65.41
26	9	17.70	21.49	23	11.15	9.24	7.44	6.75	7	3	7.48	45.46	5.03	11.20	1	4.31	44.77
27	9	19.50	28.94	21	14.32	11.61	10.31	9.11	9	5	9.43	43.84	5.17	14.00	1	5.14	50.04
28	11	20.00	25.56	19	13.45	10.95	10.59	11.86	9	5	10.59	69.02	5.30	15.50	1	4.99	63.30
29	14	26.50	33.73	21	16.71	13.26	10.97	10.35	9	5	14.84	54.05	6.12	14.00	3	6.42	57.74
30																	

Essential oils yield (%)

EOs extraction was carried out using hydro-distillation with a Clevenger-type apparatus (MS-E104, Korea) (Fig. 1). This device is equipped with a heating element capable of reaching temperatures up to 450 °C, regulated by an analog temperature controller integrated with a K-type sensor. The unit features high thermal insulation using ceramic fiber and is built with a powder-coated aluminum case and a polypropylene body. It includes a support rod clamp compatible with rods of 12.7 mm diameter and can accommodate flasks ranging from 100 to 5000 mL. The system operates on a power supply of 220 or 110 V at 50/60 Hz. The apparatus comprises three main components: a glass flask (or balloon), an oil separation tube, and a cooling unit. For the distillation process, dried plant samples obtained from the six drying methods were pulverized and placed into a one-liter distillation flask, to which approximately 500 mL of distilled water was added. The system was then activated and operated for 5 hours until the EOs appeared as a yellow oily layer on the surface of the condensed water within the separation tube (Sarfaraz et al., 2023). Following extraction, the EOs was carefully removed from the oil separation tube using a pipette to prevent contamination. The oil was transferred to a pre-weighed, clean, and dry container. The final yield of EOs was determined gravimetrically by subtracting the weight of the empty container from that of the EOs -containing container. After weighing the EOs, the weight percentage was computed using the following equation:

EOs yield (%) = [(weight of EOs/dry weight of raw material) ×100] Eq. (1)

Fig. 1. Extracting essential oils from the *Froriepia subpinnata* plant using a Clevenger apparatus.

Gas chromatography-mass spectrometry (GC-MS)

The chemical composition of the EOs was analyzed using an Agilent 7890 gas chromatograph (Agilent Technologies, Palo Alto, CA) equipped with an HP-5ms capillary column (30 m × 0.25 mm i.d., 0.25 μm film thickness). The oven temperature program was initiated at 70 °C (held for 2 minutes), then increased to 200 °C and held for 1 minute. The injector and detector temperatures were maintained at 210 °C and 220 °C, respectively. The ion source temperature was set at 230 °C, while the mass quadrupole was maintained at 150 °C. The mass scan range was set between 35 and 500 m/z. Data acquisition and instrument control were performed using the GC-MSD ChemStation software. Retention indices (RIs) were calculated using the Kovats index method, based on the retention times of n-alkanes (C8–C21) injected under the same chromatographic conditions. EOs components were identified by comparing their RIs with those reported in the literature and by matching the mass spectra to reference spectra in the NIST 08 and Wiley 275 libraries. The ChemStation data system (Adams, 2007) was employed for spectral interpretation.

RESULTS AND DISCUSSION

Impact of various drying techniques on the EOs components of F. subpinnata

The EOs extracted from Anarijeh leaves was analyzed using GC-MS, revealing a total of 53 components across both fresh and dried samples. These compounds represented between 74.58% and 98.41% of the total volatile content. The composition analysis yielded several noteworthy findings. Among the drying methods, microwave treatment resulted in the highest concentration of citronellol, whereas this compound was undetectable in the samples dried via sun, shade, or oven at 65 °C. The lowest citronellol content was observed in sample dried in the oven at 45 °C and those subjected to freeze-drying. Dornol was identified as a major EOs constituent and was present in all treatments except shade and microwave drying. The highest concentrations of dornol were found in the control (28.88%) and freeze-dried (28.77%) samples, while the lowest was observed in the oven-dried samples at 65 °C (20.49%). Another significant compound, p-Cymen-7-ol, was presented in the most drying treatments, except for oven and microwave drying at 65 °C. The control exhibited the highest concentration of p-Cymen-7-ol (19.95%), while the lowest amount was found in the shade-dried samples (10.3%). Some EOs constituents were treatment-specific. For instance, the methyl ester of 10,12-tricosadiynoic acid was detected only in the control, while octanal, isoneral, β-ionon-5,6-epoxide, and trans-β-ionone were exclusive to the sun-dried samples. The compound p-Menthan-8-ol appeared only in the 45 °C oven-dried samples, whereas 2-nonenal (E)-, methyl caprate, β-eudesmene, geranyl isovalerate, and valerenol were only detected in the 65 °C oven-dried treatment. The microwave treatment uniquely included ascaridole epoxide, nerolidyl acetate, caryophyllene, α-guaiene, humulene, and 1-heptatriacotanol. In contrast, freeze-dried samples exclusively contained 2-(2-methyl-2-propenyl), β-myrcene, D-limonene, γ-terpinene, terpinolene, and thymol. All drying methods except microwave retained 2-Decenoic acid methyl ester, β-elemene, and (E)-β-farnesene. In terms of EOs yield, oven drying resulted in the highest extraction percentages, with 2.83% and 2.81% for 45 and 65 °C treatments, respectively. Conversely, the control (1.34%) and microwave drying (1.39%) yielded the lowest EOs contents (Table 2)

Table 2. Essential oils composition (%) of *Froriepia subpinnata* as affected by different drying methods

No.	Compound	Retention index	Fresh plant (Control)	Drying method					
				Sun	Shade	Oven 45 ºC	Oven 65 ºC	Microwave	Freeze-drier
1	3-Carene	1018	nd	0.98	0.01	nd	0.56	nd	0.76
2	Sabinen	956	2.24	1.60	nd	1.48	0.87	nd	nd
3	β-Pinene	979	nd	0.32	0.50	nd	nd	nd	nd
4	β-Myrcene	1022	1.80	0.94	2.88	0.87	0.38	nd	0.39
5	Octanal	979	nd	0.36	nd	nd	nd	nd	nd
6	α-Phellandrene	969	1.36	0.56	2.23	1.11	0.24	nd	nd
7	Isoneral	1239	nd	0.38	nd	nd	nd	nd	nd
8	p-Cymene	1026	nd	0.68	nd	nd	0.33	nd	nd
9	**D-Limonene**	**1030**	**8.95**	**6.98**	**8.34**	**6.52**	**3.96**	**nd**	**3.77**
10	γ-Terpinene	1072	1.45	0.87	2.66	0.74	0.46	nd	0.44
11	**Terpinolen**	**1088**	**11.27**	**8.65**	**20.47**	**7.78**	**5.63**	**nd**	**5.79**
12	Linalool	1099	nd	0.64	nd	3.46	4.29	nd	3.74
13	**Durenol**	**1309**	**28.88**	**24.9**	**nd**	**26.30**	**20.49**	**nd**	**28.77**
14	Terpinen-4-ol	1177	nd	1.77	0.81	0.47	0.68	nd	0.78
15	p-Cymen-8-ol	1185	nd	2.81	0.45	nd	nd	nd	1.09
16	**p-Cymen-7-ol**	**1290**	**19.95**	**10.30**	**16.10**	**15.50**	**nd**	**nd**	**15.09**
17	(S)-(-)-Citronellic acid, methyl ester	1295	nd	0.29	0.09	nd	0.39	nd	nd
18	Thymol	1290	1.03	3.08	1.25	3.00	2.61	nd	4.96
19	2-Decenoic acid, methyl ester	1479	2.30	3.79	1.98	3.77	6.03	nd	5.08
20	**β-Elemene, (-)-**	**1600**	**5.01**	**3.85**	**3.84**	**7.47**	**8.73**	**nd**	**8.66**
21	β-Ionon-5,6-epoxide	1999	nd	0.41	nd	nd	nd	nd	nd
22	(E)-β-Famesene	1453	2.82	2.08	2.66	5.15	5.89	nd	6.82
23	trans-β-Ionone	1493	nd	0.57	nd	nd	nd	nd	nd
24	(-)-Globulol	1584	nd	0.38	nd	nd	nd	5.75	nd
25	Myristicin	1523	nd	0.60	0.25	0.36	0.68	nd	nd
26	Caryophyllene oxide	1565	0.24	0.31	nd	nd	0.39	nd	nd
27	Neointermedeol	1655	nd	0.71	0.06	nd	0.67	nd	0.72
28	Neophytadiene	1821	1.08	1.68	2.14	5.54	5.80	nd	6.12

Table 2. Continued

No.	Compound	Retention index	Fresh plant (Control)	Drying method					
				Sun	Shade	Oven 45 ºC	Oven 65 ºC	Microwave	Freeze-drier
29	Phytol	2119	nd	0.48	0.62	nd	nd	nd	nd
30	β-Phellandrene	1029	nd	nd	4.35	nd	nd	nd	nd
31	β-Ocimene	1032	0.17	nd	0.32	nd	nd	6.05	nd
32	α-Terpinolen	1060	nd	nd	10.29	nd	nd	nd	nd
33	l-Verbenone	1208	3.29	nd	8.73	nd	nd	nd	nd
34	β-Citronellal	923	nd	nd	0.23	nd	nd	nd	nd
35	α-Ylangene	1365	nd	nd	0.11	nd	nd	nd	nd
36	Aromandendrene	1439	nd	nd	0.19	0.60	nd	6.29	0.62
37	Falcarinol	2026	nd	nd	0.28	nd	nd	nd	nd
38	α-Pinene	935	0.75	nd	nd	0.62	nd	nd	nd
39	m-Cymen-8-ol	1183	0.19	nd	nd	1.36	2.03	nd	nd
40	**Citronellol**	**1225**	**nd**	**nd**	**nd**	**1.52**	**nd**	**52.37**	**2.96**
41	p-Menthan-8-ol	1132	nd	nd	nd	0.75	nd	nd	nd
42	Valencen	1489	nd	nd	nd	0.41	1.00	nd	nd
43	2-Nonenal, (E)-	1135	nd	nd	nd	nd	0.37	nd	nd
44	Methyl caprate	1604	nd	nd	nd	nd	0.59	nd	nd
45	β-Eudesmene	1499	nd	nd	nd	nd	0.78	nd	nd
46	Geranyl isovalerate	1450	nd	nd	nd	nd	0.38	nd	nd
47	Valerenol	1869	nd	nd	nd	nd	0.35	nd	nd
48	10, 12-Tricosadiynoic acid, methyl ester	2832	0.32	nd	nd	nd	nd	nd	nd
49	Ascaridole epoxide	1296	nd	nd	nd	nd	nd	1.83	nd
50	Nerolidyl acetate	1687	nd	nd	nd	nd	nd	1.60	nd
51	Caryophyllene	1418	nd	nd	nd	nd	nd	7.96	nd
52	α-Guaiene	1440	nd	nd	nd	nd	nd	9.94	nd
53	Humulene	1454	nd	nd	nd	nd	nd	5.58	nd
54	1-Heptatriacotanol	3938	nd	nd	nd	nd	nd	1.04	nd
	Total identified (%)		93.1	80.97	91.84	94.78	74.58	98.41	96.56
	EOs yield (%)		1.34	2.74	2.79	2.83	2.81	1.39	1.42

The data were sorted on the basis of the retention index (RI) of components. nd: Not detected. Literature indexes were obtained from the NIST database (2009).

Impact of sun-drying technique on the EOs components of F. subpinnata

The selection of appropriate drying methods for aromatic plants is critical, as drying can substantially affect both the yield and chemical profile of EOs (Caputo et al., 2022). This is the first study investigated the impact of different drying techniques on the EOs yield and composition of *F. subpinnata*. Previous findings by Hazrati et al. (2021) demonstrated that drying can enhance the concentration of EOs components relative to the fresh materials, likely due to the biochemical processes such as glycoside hydrolysis, esterification, and oxidation. These transformations can lead to the formation of new compounds while also contributing to the degradation or loss of pre-existing ones (Bhatt et al., 2018). However, the effectiveness of drying methods varies considerably. Thamkaew et al. (2021) reported that solar drying, while traditional and accessible, may not be suitable for some herbs due to the significant losses in color and aroma, thus compromising product quality. Consistent with these observations, Rasekh et al. (2023) found that sun drying yielded the lowest EOs output in spearmint. Similarly, in our study, sun drying resulted in an EOs yield of only 2.74%, supporting previous conclusions regarding its limited efficiency. Nevertheless, despite the lower yield, sun drying preserved certain valuable constituents; notably, dorenol was the predominant compound in this treatment, accounting for 24.9% of the total detected volatiles. This underscores the nuanced relationship between drying methods and EOs composition suggesting that although sun drying may not maximize total oil yield, it could selectively preserve or even concentrate specific bioactive components, meriting further investigation.

Impact of shade drying technique on the EOs components of F. subpinnata

Previous studies have consistently emphasized the advantages of shade drying in preserving both the quality and yield of EOs in aromatic plants (Hashemi Moghaddam et al., 2020; Hazrati et al., 2021). For example, Abbas et al. (2021) demonstrated that shade drying led to superior oil quality, marked by elevated levels of α-pinene and δ-3-carene. These findings suggest that, despite potentially lower overall yields, shade drying can maintain or even enhance specific volatile constituents that contribute to the oil's therapeutic and aromatic properties. The results of the present investigation on Anarijeh EOs are consistent with these observations. Shade drying produced a moderate EOs yield of 2.79%, while preserving key volatile compounds such as terpinolen and p-Cymen-7-ol, which accounted for 20.47% and 16.1% of the oil, respectively. This indicates that shade drying may be particularly effective in retaining compounds associated with fragrance and bioactivity. Moreover, Ebadi et al. (2015) found that shade drying substantially minimized trichome damage in *L. citriodora* compared to the oven drying at 60 °C and vacuum drying at 40 °C. This preservation is significant, as trichomes are the primary sites for the synthesis and storage of EOs, and their integrity directly affects oil quality. The reduced structural damage associated with shade drying likely contributes to the improved composition and quality of EOs extracted from such materials. Although trichome integrity was not assessed in our study, incorporating such analysis in future research could provide deeper insights into the mechanisms underlying the effectiveness of various drying methods.

Impact of oven drying technique on the EOs components of F. subpinnata

The findings of Gangwar et al. (2024) suggest that oven drying at 30 °C is an effective method for obtaining a high EOs yield. However, our investigation on Anarijeh demonstrates that higher oven temperatures specifically 45 and 60 °C produced the greatest EOs yields among all treatments, at 2.83% and 2.81%, respectively. In our study, the moisture content of oven-dried samples was significantly reduced, measuring 1.42% and 1.41% at 45 and 60 °C, respectively. This substantial decrease in moisture content likely contributes to a higher EOs concentration, as water loss results in the relative enrichment of volatile compounds within the plant matrix. Furthermore, the controlled and uniform drying environment of an oven may reduce the risk of volatile compound degradation or loss, thereby enhancing the overall potency and composition of the resulting EOs. Among the identified compounds, durenol was the most abundant in the EOs from oven-dried samples, constituting 26.3% at 45 °C and 20.49% at 60 °C. This indicates that moderate oven drying not only enhances EOs yield but also preserves key bioactive constituents such as durenol, which is recognized for its aromatic and therapeutic properties. However, it is important to note that the impact of drying temperature on EOs yield and composition is highly species-dependent. Factors such as variations in secretory structures, environmental adaptation, and intrinsic EOs content influence the plant's response to drying conditions (Rahimmalek and Goli, 2013). Therefore, a comprehensive understanding of plant-specific physiological characteristics is essential for optimizing drying protocols to achieve maximal preservation of desirable phytochemicals.

Rahimmalek and Goli (2013) further emphasized that elevating the drying temperature from 50 °C to 70 °C can result in the significant loss of monoterpene compounds. This observation is consistent with our findings on Anarijeh EOs, wherein oven drying at 60 °C led to a lower concentration of durenol (20.49%) compared to the control samples (28.88%). These results suggest that although higher drying temperatures may enhance the total EOs yield, they can simultaneously reduce the relative abundance of specific bioactive constituents such as durenol. This highlights the critical need to carefully select drying

conditions to strike a balance between maximizing yield and maintaining the desired phytochemical profile. While elevated temperatures may increase the extraction efficiency by facilitating water removal and disrupting cellular structures, they may also promote the volatilization or thermal degradation of thermolabile compounds. Supporting this notion, Altay et al. (2024) reported a reduction in monoterpene hydrocarbons during convective drying at 45 and 55 °C in purple basil. Their study indicated that the drying process not only accelerates moisture loss but may also induce chemical transformations, wherein monoterpenes and phenylpropanoids are converted into sesquiterpenes under thermal stress. This transformation underscores the complex interplay between drying temperature and EOs composition, reinforcing the importance of optimizing drying protocols to preserve valuable volatile compounds. Collectively, these findings advocate for a nuanced approach to drying method selection—one that considers not only the quantity of EOs extracted but also the preservation of its bioactive quality.

Impact of microwave drying technique on the EOs components of F. subpinnata

Caputo et al. (2022) reported that oregano samples subjected to low-power microwave drying for 35 minutes achieved a notable EOs yield of 2.20% (w/w). In alignment with these findings, our current study demonstrated that microwave drying yielded the highest concentration of citronellol (52.37%) compared to all other drying methods. Citronellol, a naturally occurring monoterpene alcohol, is a prominent compound in the EOs of several aromatic plants, including *Cymbopogon winterianus* (Quintans-Júnior et al., 2008) and *C. citratus* (Abegaz et al., 1983). This compound is particularly valued for its therapeutic potential, with reviews highlighting its analgesic and anti-inflammatory properties, thereby underscoring its relevance in both aromatherapy and pharmacological applications (De Sousa, 2011). Further support for the efficacy of microwave drying comes from the study by Qin et al. (2022), who demonstrated that microwave treatments at 150 and 500 W increased the monoterpene hydrocarbon content in Amomum tsao-ko pericarps. However, they also observed that higher microwave power eventually led to a decline in monoterpene levels, suggesting a nonlinear relationship between microwave intensity and the retention of volatile constituents. This complexity is echoed in the findings of Nazari et al. (2024), who reported a consistent decline in EOs content of Shirazi thyme as drying temperatures increased. Their results suggest that elevated thermal exposure may compromise cellular integrity, particularly by damaging plasma membranes and cell walls, thereby accelerating the evaporation and degradation of EOs components and reducing their retention due to the destruction of storage structures. The divergence between our findings and those of Nazari et al. (2024) may be attributed to the interspecies differences, as well as variations in microwave power settings and drying durations. While our results demonstrate that lower microwave power can enhance the concentration of valuable components such as citronellol, other studies caution that excessive heat input may compromise EOs integrity. These findings collectively highlight the necessity of optimizing microwave drying parameters to balance efficient moisture removal with the preservation of essential phytochemicals.

Impact of freeze-drying technique on the EOs components of F. subpinnata

Sobatinasab et al. (2024) reported that freeze drying yielded optimal results in terms of both EOs yield and composition in *Trachyspermum ammi* (ajowan). Similarly, Pirbalouti et al. (2013) demonstrated that freeze drying effectively preserved a higher EOs output and superior chemical composition in both purple and green basil leaves compared to other drying methods. These studies highlight the efficacy of freeze drying in maintaining the integrity of volatile compounds, primarily due to its low-temperature operation and the rapid sublimation of water, which minimizes thermal degradation. Contrary to these findings, our study revealed that freeze drying resulted in a comparatively low EOs yield of only 1.42%, making it one of the least effective methods tested. This discrepancy may be attributed to the several factors, including species-specific differences in EOs storage structures, variations in freeze-drying parameters (e.g., freezing rate, chamber pressure, and drying duration), or the unique physicochemical properties of the EOs constituents in *F. subpinnata*. The relatively low yield observed may suggest that freeze drying, while beneficial for certain species, may not be universally suitable for all aromatic plants. Furthermore, the outcome could indicate that some volatile compounds in Anarijeh EOs are particularly sensitive to the sublimation phase, potentially resulting in their partial or complete loss. These findings underscore the importance of species-specific optimization of freeze-drying protocols to maximize EOs retention and composition. Future studies should explore the fine-tuning of freeze-drying variables to determine their influence on EOs recovery, particularly in plants with delicate or highly volatile oil profiles.

Heat map and principal component analysis (PCA) of F. subpinnata EOs constituents after various drying techniques

In our study, a total of 54 compounds were identified in the EOs of *F. subpinnata*, of which 12 dominant compounds were selected for PCA due to their significantly higher contributions across the different drying treatments. The PCA biplot effectively visualizes the distribution patterns of these major compounds in relation to the various drying

methods employed. The chart is distinctly divided into two sides: the right and the left. The majority of EOs compounds were located on the right side, indicating that treatments associated with this region particularly the control (fresh) samples exhibited elevated levels of compounds such as terpinolene, D-limonene, and p-Cymen-7-ol. These components were especially prominent in the control group, suggesting that the absence of drying preserves or enhances their concentration. In contrast, the left side of the PCA chart was dominated by citronellol and β-myrcene, which were most abundant in samples subjected to microwave and shade drying, respectively. This distribution indicates that these particular methods selectively enhance the accumulation of certain volatile constituents. Additionally, the lower region of the PCA biplot corresponded to the samples treated with oven drying at 45 and 60 °C, as well as freeze drying. These treatments were associated with increased concentrations of other key EOs compounds, including durenol, thymol, (E)-β-farnesene, neophytadiene, and methyl 2-decenoate. This suggests that these drying techniques result in a distinct EOs profile compared to the control or microwave/shade drying methods. Overall, the PCA provided a clear and comprehensive overview of the variation in EOs composition induced by different drying methods, highlighting the unique phytochemical signatures associated with each technique (Fig. 2).

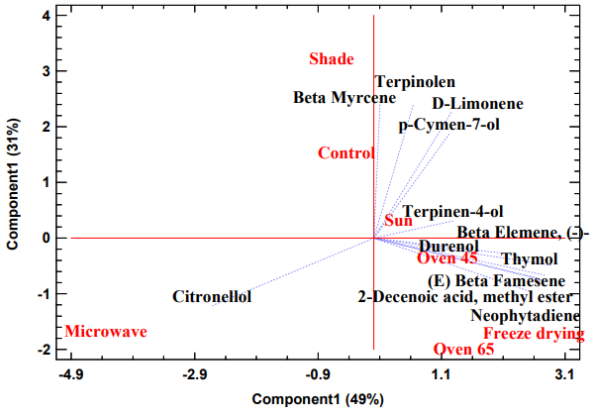

Fig. 2. Principal component analysis graph of the effect of different drying methods on the essential oils composition of the *Froriepia subpinnata*. Treatments included control, drying with sun, shade, oven at 45 and 65 ºC, microwave, and freeze dryer.

The heat map analysis highlighted citronellol as one of the most prominent constituents in the EOs of *F. subpinnata*. This compound was distinctly separated from the other EOs components, with its highest concentration observed in the microwave-drying treatment, as indicated by the intense red coloration on the heat map. Citronellol was also detected, albeit at lower concentrations, in samples subjected to freeze drying and oven drying at 45 °C, but it was absent in the remaining drying methods. Durenol, another key EOs component, formed a separate cluster within the heat map. This compound was present in the majority of drying methods except for shade and microwave drying. The highest concentrations of durenol were recorded in the control (fresh) samples and those subjected to freeze drying, whereas oven drying at 60 °C yielded the lowest amount. In contrast, p-Cymen-7-ol and terpinolene were grouped within the same cluster and were most abundant in the control treatment. These two compounds were not detected in samples dried using microwave or oven drying at 65 °C, indicating a significant impact of high-temperature and microwave drying on their stability. Microwave drying was positioned in an independent cluster, reflecting its unique chemical profile, where citronellol was the only EOs constituent detected in notable quantities. Freeze drying and oven drying at 45 °C were grouped together in another distinct cluster, suggesting a similarity in their EOs composition profiles (Fig. 3).

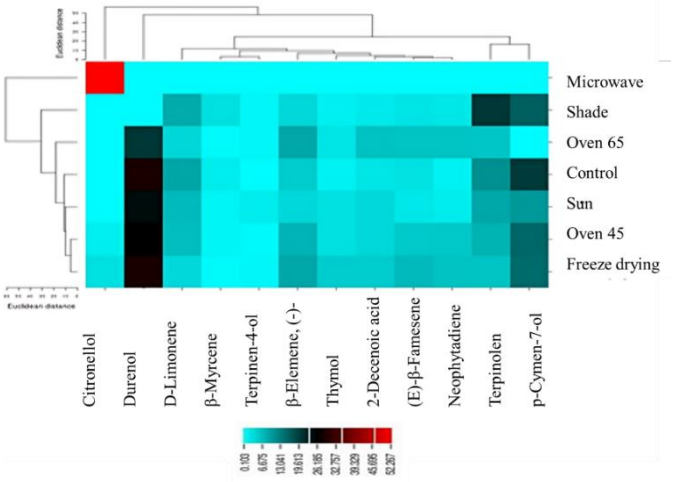

Fig. 3. Heat map graph of the effect of different drying methods on the essential oils composition of the *Froriepia subpinnata*. Treatments included control, drying with sun, shade, oven at 45 and 65 ºC, microwave, and freeze dryer.

CONCLUSION

The present study demonstrated that all drying procedures increased the EOs yield of *F. subpinnata* compared to the fresh (control) samples. Among the treatments, oven drying at 45 and 60 °C resulted in the highest EOs yields. However, due to the absence of statistical analysis, it is not possible to assert significant differences between the oven and shade drying treatments. In contrast, the control and microwave-dried samples produced the lowest EOs yields. Citronellol was identified as a primary constituent of Anarijeh EOs, with its highest concentration found in the microwave-dried samples, whereas it was undetected in the control treatment. Another important compound, durenol, was

present in the most drying treatments, except for shade and microwave drying. Notably, the highest levels of durenol were observed in the control and freeze-dried samples. Overall, the findings indicate that the chemical composition of Anarijeh EOs is significantly influenced by the drying method employed. Although oven drying at both 45 and 60 °C achieved the greatest total EOs yield, microwave drying selectively enriched citronellol, the major component. This underscores the differential impact of drying techniques not only on EOs yield but also on the qualitative composition of individual constituents. It is important to note that these results are based solely on EOs percentages and chemical profiles. Therefore, further biochemical and physiological studies are warranted to elucidate the mechanisms through which various drying methods influence the EOs composition in Anarijeh.

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CRediT AUTHORSHIP CONTRIBUTION STATEMENT

Conceptualization: Maryam Haghighi; Methodology: Maryam Haghighi; Software: Saeed Sedaghat; Validation: Maryam Haghighi; Formal analysis: Maryam Haghighi; Investigation: Saeed Sedaghat; Resources: Saeed Sedaghat; Data curation: Maryam Haghighi; Writing original draft preparation: Saeed Sedaghat; Writing review and editing: Maryam Haghighi; Visualization: Saeed Sedaghat; Supervision: Maryam Haghighi; Project administration: Saeed Sedaghat; Funding acquisition: Maryam Haghighi.

DECLARATION OF COMPETING INTEREST

The authors declare no conflicts of interest.

ETHICAL STATEMENT

"The conducted research is not related to either human or animals use. Author is aware of the content of the manuscript and consented to submit it to Iran Agricultural Research journal."

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