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Changes in the essential oil content and composition of *Thymus daenensis* Celak. under different drying methods

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ABSTRACT

Some active ingredients of the medicinal plant are changed during a drying process. This study was aimed for the effect comparison of the various drying methods on quantity and quality of *Thymus daenensis* essential oil. The factorial experiment treatments included pre-drying under sun as the first factor and various drying methods (fresh plant, sun drying, shade drying, oven drying at 35, 45, and 55 °C, vacuum drying at 35, 45, and 55 °C, and microwave drying at 100, 500, and 1000 W) as the second factor. The results showed that various drying methods and their interaction with the pre-drying had a significant effect on the essential oil content and its components. In present study an adverse relation was found between the essential oil content and thymol or carvacrol amount in the dried plant. The highest essential oil amount was observed in the oven and vacuum oven-drying at 35 °C without the pre-drying operation. While, the greatest content of thymol as the predominant oxygenated monoterpenes was acquired in the vacuum oven-drying at 55 °C. Generally, the oven and vacuum oven-drying at 35 °C without the pre-drying are recommended for *Thymus daenensis* drying due to possessing the highest quantity of essential oil and appropriate quality.

1. Introduction

Thyme (*Thymus daenensis* Celak.) is an herbaceous, perennial, and bushy herb which belong to Thymus genus of Lamiaceae family. The Persian and local names of *Thymus daenensis* is “Avishan-e-denaee” (Mozaffarian, 1996). Thymus genus includes about 350 species that are rich in essential oils. The major components in *Thymus daenensis* essential oil are thymol and carvacrol. These compounds have antioxidant activity (Dorman and Deans, 2004; Miguel et al., 2004; Sokmen et al., 2004; Youdim et al., 2002). It has been reported that the essential oil obtained from leaves and flowers have antispasmodic, carminative, anti-rheumatic, anti-sciatica, and strong antiseptic (Pirbalouti et al., 2013). As well as, this oil was used for producing mouthwash and cough syrups in the pharmaceutical industry.

Drying is one of the oldest techniques for storage food, meats, and herbs. This process involves water removal by evaporation into a certain threshold to prevent the activities of enzymes, microorganisms, and yeasts to increase the shelf life (Prusinowska and Śmigielski, 2015). Thus, biochemical reactions attributed to post-harvest decay due to lack of enough water impaired and herbs protected from degradation. Moreover, weight and volume of herbs reduced by a drying process, therefore the costs of packaging, storage and transportation will be minimal (Chakraborty and Dey, 2016).

In current, the variety of natural, artificial, and combined methods were used for plant matter drying. Natural methods are drying by sun or shade, while artificial drying procedures include warm air drying or modern technologies such as drying under vacuum and microwave radiation (Ekechukwu, 1999a, 1999b). Natural drying is an important method for drying crops because of lower costs. The natural drying has disadvantages such as not being able to move a large amount of plant matter and achieve the stable standards of quality (Soysal and Öztekin, 2001). Drying with hot air also due to lessen costs is often used for industrial production of dried herb. Albeit, the low energy efficiency and time-consuming operations are notable disadvantages for the warm air drying (Soysal and Öztekin, 2001).

Microwave radiation is used for drying plants. Short drying time is including important benefits to this approach. In addition, microwave energy helps to maintain the color of dried herbs and improving the plant active ingredient. Microwave radiation spread to plant matters quickly and impressive, thus energy consuming reduced due to rapid

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drying (Diaz et al., 2003).Unlike other drying methods that the heat penetrated from a surface to depth, in microwave method the heat was produced within the plant material and then distributed to the outside (Blose, 2001; Alibas, 2007). Heindl and Müller (2002) reported that valerian roots dried by microwave radiation possessed the high quality and low microbial contamination.

Drying under vacuum is another method, which conducted out in sub-atmospheric pressures. This is a very effective procedure for dehydration of sensitive products because a high degree of drying was obtained without the need to increase the temperature. Thus, this possibility is provided that susceptible materials to high temperature were dried by vacuum drying technique in short time. Moreover, vacuum drying is the best method for material which exposed to air oxygen rapidly oxidized (Jaya and Das, 2003; Mitra et al., 2011).

The active ingredients composition in the fresh medicinal plant may be changed by enzymatic processes during the drying process (Jambor and Czosnowska, 2002). Also, some studies have been reported that drying method has a significant impact on the essential oil content and constitutes (Basver, 1993; Deans and Svoboda, 1992). However, drying methods effect on essential oil yield and its components vary depending on using temperature, drying time, and plant species (Yazdani et al., 2006). Sefidkon et al. (2006) reported that the highest essential oil content in *Satureja hortensis* L. was respectively obtained in drying by oven 45 °C, the sun, and shade. Ahmadi et al. (2007) expressed that various drying methods include the sun, shade, oven 30 and 40 °C did not have a significant effect on quantity and quality of *Rosa damascena* Mill. essential oil. However, the maximum amount of citronellol and geraniol (the most important components for improving Rosa essential oil) was observed in plants dried by the shade.

Thus, an assessment regarding the effect of various drying methods on medicinal plants essential oil changes is an important issue. This study was aimed for the effect comparison of natural and artificial drying methods on quantity and quality of *Thymus daenensis* essential oil. Moreover, different temperatures effect in artificial methods on the essential oil was evaluated.

2. Materials and methods

This study was performed to evaluate the effect of different drying methods on quality and quantity of *Thymus daenensis* essential oil in the Institute of Medicinal Plants, ACECR. A factorial experiment was conducted based on the randomized complete block design (RCBD) with 3 replications. The first factor was pre-drying included exposing to the sun for 4 h and without the pre-drying that the samples dried immediately after harvest under related drying methods. The samples in the drying delay were exposed to the sun for 4 h; thereafter, placed under related drying methods. The second factor was various drying methods, which were included control (freshly harvested plant), sun drying, shade drying, oven drying at 35, 45, and 55 °C, vacuum drying at 35, 45, and 55 °C, and microwave drying at 100, 500, and 1000 W.

2.1. Sample preparation

The same and identical cuttings were collected from a *Thymus daenensis* plant. The cuttings were embedded in growth media that included sand, coco peat, and perlite (3:1:1) for rooting. Approximately 45 days later, the rooted cuttings were sown in the farm at October 2015. All agronomy practices include irrigation, fertilization; weed management, etc. conducted out on the base of plant requirement. Fresh plants in the flowering stage were harvested as randomly. Samples were collected at 10 June 2016 in early hours of a sunny morning (8–9 am). The samples were divided into two groups. A group exposed under the sun for 4 h in the farm. Another group immediately transported to the laboratory. Then, the samples in each group were dried by the various methods.

2.2. The sun and shade drying

For the sun drying method, a white clean cloth was flattened on the open area and then the plant samples were spread on the cloth beneath the sun. In sun-drying conditions, the pre-drying operation was to place the plant sample after harvest for 4 h in the shade and then in the sun. Also, the samples without the pre-drying operation were dried immediately after harvest at sun.

Also in the shade drying method, the samples were spread inside the room without direct sunlight penetration at room temperature. In both methods, the drying process was continued until the moisture content reached to 10%.

2.3. Oven drying

The sample was spread in a thin layer on the tray. Oven temperature was regulated on operating temperatures including 35, 45 and 55 °C. The samples were removed from the oven after their moisture reached to 10 percent. Then they were ready for essential oil extraction.

2.4. Microwave drying

A microwave oven (Samsung, model MC35J8055CK) with 2250 W power output at 2450 MHz, which equipped to a swivel tray plus digital setting for power and time was used for the sample drying. The plant material was poured into a glass container and then placed inside the microwave cavity. Radiations power were regulated on 100, 500 and 1000 W. These operations have been continued as long as the moisture content was reduced to 10%.

2.5. Vacuum drying

The samples in vacuum drying method were dried in a vacuum-dryer (Memmert Inc. VO 500 model, Germany) with technical features of 230 V, 60 Hz, and 2400 W. The vacuum oven temperature was sensitive to temperatures ranging from 5 to 200 °C. The various temperatures included 35, 45, and 55 °C under vacuum condition (600 mbar) was adjusted for every 5 h.

2.6. Essential oils (EO) analysis

Each dried sample was ground to a fine powder using a Moulinex food processor and was passed through a 20 mesh sieve to remove large pieces of debris. Oils were extracted by hydrodistillation for 3 h, from 50 g the ground tissue in 1 l of water contained in a 2 l flask using Clevenger-type apparatus, according to producers outlined British Pharmacopoeia. The oils were dried over anhydrous sodium sulphate and kept at 4 °C until it was analyzed. The experiment was repeated three times and its mean was reported as EO percent on the dry plant.

GC/MS analysis was performed on an Agilent instrument coupled with a 5973 Mass system equipped with flame ionization detector (FID) and a BPX5 capillary column (30 m × 0.25 mm; 0.25 μm film thicknesses). Temperature program includes oven temperature held for 2 min at 60 °C and was enhanced to 150 °C with 3 °C per min rate. Then, temperature enhancement was programmed up to 270 °C as 5 °C per min rate and this temperature held for 15 min. Other operating conditions include: carrier gas was He with a flow rate of 1.1 ml min^{-1}; injector and detector temperatures were 300 °C, and split ratio, 1:50. Mass spectra were taken at 70 eV. The mass spectra and retention indices of essential oil components were identified by comparison to published literature and presented the MS computer library (Adams, 2001).

2.7. Statistical analysis

All the data were analyzed by SAS software to GLM procedure. The

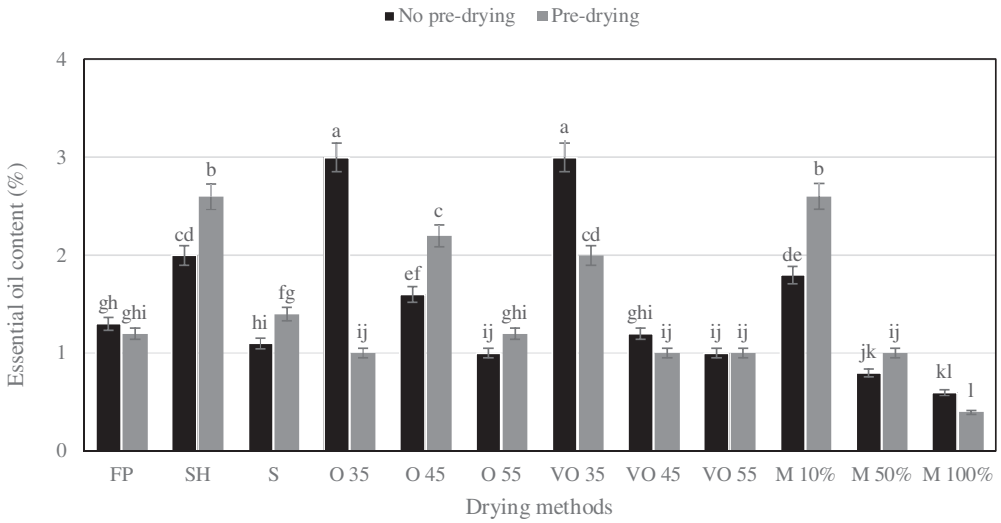

Fig. 1. The comparison mean of the essential oil content changes by the different drying methods in both the pre-drying conditions. FP: fresh plant; SH: shade drying; S: the sun drying; O 35, 45 and 55: oven drying at 35, 45 and 55 °C, respectively; VO 35, 45, 55: vacuum oven drying at 35, 45 and 55 °C, respectively; M 10%, 50% and 100%: microwave drying at 100, 500 and 1000 W, respectively.

difference among drying method means was compared by Duncan's multiple range tests at 5% confidence interval.

3. Results and discussion

3.1. Effect of drying method on the EO content

The results showed that the drying methods had significant effect on EO content (Fig. 1) and the highest EO contents were obtained by oven-drying and vacuum oven-drying at 35 °C without a pre-drying treatment. Of course, the EO contents were increased by the pre-drying along with shade-drying, sun-drying, oven-drying at 45 °C, and microwave-drying at 100 W drying. The lowest amount of EO was related to microwave-drying at 1000 W drying. This result was in accordance with other studies (Rahimmalek and Goli, 2013; Sarosi et al., 2013; Soares et al., 2007; Sefidkon et al., 2006). The changes of essential oil content during drying process depended on the kind of tissue, temperature, time, and drying method (Lewicki and Pawlak, 2003). However, some aromatic compounds removed from plant tissue along with the water by the evaporation process (Asekun et al., 2007). In general, the EO contents were decreased with increasing temperature and microwave power.

The current study ascertained that the essential oil content of *Thymus daenensis* reduced by increasing temperature in the oven and vacuum oven. This finding was in agreement with another study on peppermint and dill (Ayyobi et al., 2014), *Artemisia dracunculus* L. (Arabhosseini et al., 2006) and *Salvia officinalis* L. (Hamrouni Sellami et al., 2013). However, the pre-drying is the barrier to reduce essential oil in sample dried at high temperatures of the oven (Fig. 1). Glandular trichomes as secretory structures are responsible for production and accumulation essential oils in plants (Ebadi et al., 2015). Some studies revealed that increased drying temperature can harm to glandular trichomes in medicinal herb (Diaz-Maroto et al., 2003; Yousif et al., 2000). Also, it has been expressed that some essential oil components decomposed via autoxidation and hydro peroxidation in high temperature (Turek and Stintzing, 2013). Moreover, increased temperature causes more degradation of the cell wall and plasma membrane and it may affect plasma membrane permeability (Lewicki and Pawlak, 2003). According to the evidence, enhancing the temperature due to accelerating evaporation, essential oil components decomposition, and destruction of storage cells could lead to reducing the essential oil content.

3.2. Effect of drying method on the EO components

The drying methods had significant effect on EO components (Table 1). In this study, nineteen components were identified in the fresh and dried *Thymus daenensis*. Also, thymol, carvacrol, *para*-cymene, γ- terpinene and *E*-caryophyllene were main components of *T. daenensis* (Table 2). Previously, several studies have been reported that thymol, carvacrol, and β-caryophyllene are main compounds of *T. daenensis* (Bahreininejad et al., 2010; Rustaiee et al., 2010).

The results of present study showed that the main components in the fresh samples were α-thujene (2.61%), α-pinene (2.06%), myrcene (3.60%),α and γ-terpinene (2.29 and 10.75%, respectively), para-cymene (10.60%), 1, 8- cineole (2.09%), thymol (47.18%), carvacrol (2.27%), and caryophyllene (4.58%). Although, the highest thymol contrent was obtained by vacuum-drying at 55 °C in both the pre-drying conditions, the other detectable EO components were strongly diminished (Table 2).

Without pre-drying process, the different drying methods reduced contents of the main monoterpene hydrocarbons such as α-thujene, α-pinene, myrcene, α and γ-terpinene, para-cymene, 1, 8- cineole. However, these percentages strongly increased in the oven 35 °C after doing the pre-drying. The greatest amount of them was recorded for the oven 35 °C after the pre-drying beneath the sun and the minimum was obtained in the vacuum oven at 55 °C (Table 2). Similar results were reported by Rahimmalek and Goli (2013).

The thymol and carvacrol contents (as major components of oxygenated monoterpenes) were significantly different in various drying methods. In pre-drying and no-pre drying conditions, the highest thymol content was obtained in the vacuum oven-drying at 55 °C (84.72% and 84.84%, respectively). Its lowest (25.26%) was observed in the oven 35 °C after the pre-drying (Table 2). The maximum content of carvacrol (6.31%) was obtained by sun-drying after the pre-drying and its minimum (0.98%) was gained in the oven 35 °C after the pre-drying (Table 2). These results were confirmed by the findings of Sellami et al. on *Laurusnobilis* L. (2011). They reported that contents of oxygenated monoterpeneswere increased by drying procedures.

At the thymol and carvacrol biosynthesis pathway, geranyl pyrophosphate is converted to the sabinene hydrate via cyclization reaction and it eventually is transformed to the α-thujene. Terpinene is synthesized from the α-thujene and it converted to the para-cymene through the aromatization process. At the final, the thymol and carvacrol were synthesized from para-cymene (Alizadeh et al., 2013; Taherian et al., 2009). Hence, it was revealed that the drying processes have resulted in more conversion of the monoterpene hydrocarbons to the oxygenated monoterpenes including thymol and carvacrol. However, the oven 35 °C after the pre-drying operation was different from other drying methods. It increased the monoterpene hydrocarbons contents and reduced the oxygenated monoterpenes including thymol

Table 1
Variance analysis of essential oil content and composition of *Thymus daenensis*.

		Mean of square								
S.O.V	df	Essential oil	α-Thujene	α-Pinene	β-Pinene	Camphene	Myrcene	Phellandrene	α-Terpinene	*para*-Cymene
R	2	0.0006 ns	0.156 **	0.0001 ns	0.001 ns	0.0002 ns	0.141 ns	0.00007 ns	0.022 ns	0.22 ns
P	1	0.078 *	0.129 **	0.290 **	0.033 **	0.072 **	0.692 **	0.0002 ns	0.03 ns	13.47 **
D	11	2.5 **	3.155 **	2.364 **	0.203 **	0.721 **	7.695 **	0.039 **	2.915 **	58.02 **
P × D	11	0.89 **	0.926 **	0.981 **	0.057 **	0.344 **	1.725 **	0.009 **	0.867 **	10.93 **
Error	46	0.019	0.024	0.018	0.002	0.010	0.051	0.0004	0.011	0.638
CV		9.28	11.51	12.08	12.81	15.79	14.90	16.53	9.52	14.08

		Mean of square							
S.O.V	df	Limonene	Cineole	γ-Terpinene	Linalool	Borneol	Terpinene-4-ol	α-Terpineol	Thymoquinone
R	2	0.008 ns	0.013 ns	0.795 ns	0.0004 ns	0.04 ns	0.0003 ns	0.0001 ns	0.044 ns
P	1	0.05 **	0.294 **	10.38 **	0.133 **	0.301 **	0.033 *	0.025 **	1.87 **
D	11	0.579 **	1.352 **	75.16 **	0.281 **	0.775 **	0.286 **	0.028 **	1.12 **
P × D	11	0.145 **	0.534 **	21.92 **	0.094 **	0.422 **	0.112 **	0.035 **	1.14 **
Error	46	0.006	0.037	0.548	0.006	0.036	0.010	0.001	0.017
CV		15.53	16.94	16.99	10.85	12.73	13.50	13.57	18.57

		Mean of square							
S.O.V	df	Thymol	Carvacrol	Caryophyllene	Bisabolene	Caryophyllene oxide	MH	MO	SH
R	2	25.81 ns	0.03 ns	0.004 ns	0.00025 ns	0.03 ns	5.4 ns	34.7 ns	0.28 ns
P	1	105.38 *	2.06 ns	0.003 ns	0.00028 ns	0.035 *	88.7 **	111.6 *	0.002 ns
D	11	689.46 **	4.42 **	9.19 **	0.014 **	0.39 **	630.7 **	680 **	9.67 **
P × D	11	245.92 **	2.52 **	3.08 **	0.005 **	0.16 **	165.1 **	223.7 **	3.25 **
Error	46	23.965	0.180	0.204	0.001	0.011	3.554	24.714	0.226
CV		7.37	11.88	9.94	13.88	11.90	11.30	6.60	10.19

ns: no significant differences; *: significant at $p \leq 0.05$: **: significant at $p \leq 0.01$; R: Replicate; P: Pre-drying; D: Drying methods; CV: Coefficient of variation; df: Degree of freedom; S.O.V: Source of variation; MH: Monoterpene Hydrocarbons; MO: Oxygenated Monoterpenes; SH: Sesquiterpene Hydrocarbons.

and carvacrol contents.

Although the thymol content was increased by vacuum oven-drying with increasing temperature at both per-drying conditions, this trend was not observed in the oven and microwave-drying methods. The carvacrol content increased by enhancing temperature in the oven and vacuum oven after doing the pre-drying (Table 2). But the carvacrol content was enhanced by increasing microwave power at without pre-drying condition. Some studies have reported that thymol percentage in *Thymus vulgaris* L and *Thymus daenensis* increased at higher temperatures (Venskutonis et al., 1996; Rahimmalek and Goli, 2013).

Change of the sesquiterpene hydrocarbons amounts (*E*-caryophyllene and β-bisabolene) depended on the drying methods. So the highest amount of *E*-caryophyllene was obtained in the oven 55 °C, microwave 500 W without the pre-drying (7.52 and 7.06%, respectively), and also the oven 35 °C after the pre-drying (7.02%). Also, their lowest were observed in the vacuum oven-drying at 55 °C in the both pre-drying conditions (Table 2). This result was in accordance with other studies (Ebadi et al., 2015; Rahimmalek and Goli, 2013; Pirbalouti et al., 2013).

The sun and shade drying reduced the contents of monoterpene hydrocarbons and sesquiterpene hydrocarbons compared to the fresh plant. On the contrary, the oxygenated monoterpenes content increased in the sun and shade drying compared to the fresh plant (Table 2). Other authors obtained similar results to our findings (Rahimmalek and Goli, 2013; Pirbalouti et al., 2013).

With increasing oven temperature without the pre-drying, the contents of monoterpene hydrocarbons and sesquiterpene hydrocarbons had not significant changes. Of course, with increasing the oven temperature after the pre-drying, the contents of monoterpene hydrocarbons were significantly reduced, while the oxygenated monoterpenes amounts were significantly increased. The changes in the sesquiterpene hydrocarbons contents with increasing the oven temperature depended on performing the pre- drying. Although the content of sesquiterpene hydrocarbons did not change with increasing the oven temperature from 35 to 55 °C without the pre-drying process, their contents were decreased by increasing oven temperature after pre-drying. With raising the temperature at vacuum oven-drying methods, the contents of monoterpene and sesquiterpene hydrocarbons in both the pre-drying treatments were decreased, but the oxygenated monoterpenes content were increased (Table 2).

In present study, increasing the microwave radiation power owing to rising input energy has resulted in reducing the essential oil content. In both the pre-drying groups, the contents of all the monoterpenes such as α-thujene, α-pinene, β-pinene, camphene, and myrcene were reduced by increment the microwave radiation from 100 to 1000 W. Albeit, this reduction was more distinguished in the plant which was exposed to the sun before doing microwave drying. Also, the percentages of some oxygenated monoterpenes such as carvacrol increased, while content of sesquiterpene hydrocarbons decreased by boosting the microwave radiation power. These findings concurred with other research results (Di Cesare et al., 2003; Kubra and Rao, 2012). The microwave radiation rapidly diffused in plant matters and moreover, the water molecules absorb the microwave energy rapidly. Therefore water quickly evaporated and drying rates accelerated. The microwave radiation in addition to energy savings reduced drying times without increasing the temperature of the material surface (McLoughlin et al., 2003). Hence, the microwave radiation does not damage the external surface of plant tissues and thus improves the exterior characteristic of herbs (Szumny et al., 2009).

4. Conclusion

The drying methods had a significant effect on the essential oil content and composition of *Thymus daenensis*. Although the highest essential oil content was obtained by oven-drying and vacuum-drying at 35 °C without pre-drying, the highest content of thymol and

Table 2
Effect of various drying methods on essential oil composition.

Pre-drying	Drying methods	α-Thujene	α-Pinene	β-Pinene	Camphene	Myrcene	α-Phellandrene	α-Terpinene	*para*-Cymene
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
No exposing to the sun before drying	Fresh plant	2.61 ± 0.4 b	2.06 ± 0.2 b	0.67 ± 0.07 b	1.16 ± 0.12 b	3.6 ± 0.65 b	0.2 ± 0.03 cd	2.29 ± 0.17 b	10.6 ± 1.30 b
	Shade	1.6 ± 0.3 def	1.13 ± 0.03 efg	0.36 ± 0.04 efg	0.72 ± 0.02 e–h	2.17 ± 0.33 cd	0.11 ± 0.01 g	1.41 ± 0.10 d	4.9 ± 0.21 gh
	Sun	1.4 ± 0.2 fg	0.89 ± 0.1 hi	0.27 ± 0.04 h–k	0.56 ± 0.06 h–k	1.72 ± 0.11 e	0.34 ± 0.05 a	1.37 ± 0.09 d	5.08 ± 0.4 fgh
	Oven 35 °C	1.4 ± 0.03 fg	1.14 ± 0.03 efg	0.31 ± 0.03 g–j	0.61 ± 0.04 g–j	1.79 ± 0.14 e	0.09 ± 0.01 g	1.24 ± 0.04 def	7.25 ± 0.92 de
	Oven 45 °C	1.4 ± 0.1 fg	1.15 ± 0.1 efg	0.36 ± 0.03 fg	0.64 ± 0.03 g–j	1.96 ± 0.23 de	0.12 ± 0.02 efg	1.35 ± 0.08 de	5.21 ± 0.1 fgh
	Oven 55 °C	1.7 ± 0.2 de	1.54 ± 0.18 cd	0.44 ± 0.04 cd	0.98 ± 0.13 c	1.23 ± 0.14 f	0.12 ± 0.01 efg	1.34 ± 0.08 de	5.96 ± 0.8 efg
	VO 35 °C	1.4 ± 0.1 fg	1.2 ± 0.05 ef	0.32 ± 0.06 f–i	0.57 ± 0.06 h–k	1.27 ± 0.12 f	0.11 ± 0.01 g	1.19 ± 0.07 ef	7.43 ± 0.71 d
	VO 45 °C	1.04 ± 0.2 hi	0.95 ± 0.12 ghi	0.25 ± 0.05 ijk	0.55 ± 0.07 i–k	0.35 ± 0.04 ijk	0.11 ± 0.01 g	0.39 ± 0.02 j	3.01 ± 0.50 j
	VO 55 °C	0 l	0 m	0 n	0 n	0.07 ± 0.01 k	0 h	0.07 ± 0.00 k	0.77 ± 0.12 k
	MW 100 W	1.4 ± 0.01fg	0.84 ± 0.07 hij	0.33 ± 0.06 fgh	0.6 ± 0.04 g–j	1.31 ± 0.1 f	0.21 ± 0.03 cd	0.97 ± 0.05 g	4.59 ± 0.41 hi
	MW 500 W	0.8 ± 0.06 ij	0.83 ± 0.09 ij	0.25 ± 0.03 jkl	0.36 ± 0.05 lm	0.64 ± 0.09 ghi	0.09 ± 0.01 g	0.58 ± 0.07 hi	3.44 ± 0.56 ij
	MW 1000 W	0.6 ± 0.09 jk	0.51 ± 0.06 kl	0.17 ± 0.04 lm	0.36 ± 0.05 lm	0.48 ± 0.04 hij	0 h	0.49 ± 0.04 ij	2.42 ± 0.50 j
Exposing to the sun before drying	Fresh plant	2.2 ± 0.24 c	1.61 ± 0.18 c	0.67 ± 0.06 b	0.86 ± 0.13 cde	3.4 ± 0.27 b	0.18 ± 0.02 d	2.23 ± 0.20 b	11.6 ± 1.81 b
	Shade	1.4 ± 0.02 fg	1.05 ± 0.07 gh	0.31 ± 0.03 g–j	0.53 ± 0.05 ijk	2.01 ± 0.25 de	0.11 ± 0.01 fg	1.27 ± 0.05 de	5.03 ± 0.2 fgh
	Sun	1.2 ± 0.09 gh	0.91 ± 0.14 hi	0.24 ± 0.03 jkl	0.43 ± 0.07 kl	0.96 ± 0.07 fg	0.23 ± 0.04 c	1.09 ± 0.07 fg	7.17 ± 0.58 de
	Oven 35 °C	3.85 ± 0.4 a	3.76 ± 0.3 a	0.96 ± 0.07 a	2.09 ± 0.35 a	5.27 ± 0.49 a	0.28 ± 0.03 b	3.39 ± 0.33 a	15.3 ± 1.97 a
	Oven 45 °C	1.72 ± 0.2 de	1.33 ± 0.07 de	0.43 ± 0.04 cde	0.75 ± 0.03 efg	2.39 ± 0.45 c	0.14 ± 0.00 ef	1.58 ± 0.16 c	6.31 ± 0.5 def
	Oven 55 °C	1.1 ± 0.16 h	1.19 ± 0.1 ef	0.37 ± 0.02 d–g	0.92 ± 0.12 cd	0.81 ± 0.09 gh	0.1 ± 0.01 g	0.94 ± 0.06 g	6.17 ± 0.4 d–g
	VO 35 °C	1.82 ± 0.19 d	1.62 ± 0.2 c	0.47 ± 0.06 c	0.81 ± 0.06 def	1.71 ± 0.1 e	0.12 ± 0.01 efg	0.38 ± 0.04 j	8.79 ± 1.13 c
	VO 45 °C	0.71 ± 0.15 j	0.79 ± 0.05 ij	0.25 ± 0.05 jk	0.48 ± 0.04 ijk	0.23 ± 0.04 jk	0 h	0 k	3.26 ± 0.61 j
	VO 55 °C	0 l	0 m	0 n	0 n	0 k	0 h	0.09 ± 0.01 k	0.72 ± 0.05 k
	MW 100 W	1.49 ± 0.2 efg	1.15 ± 0.2 efg	0.39 ± 0.01 def	0.66 ± 0.01 f–i	1.75 ± 0.12 e	0.12 ± 0.02 efg	1.19 ± 0.03 ef	5.24 ± 0.1 fgh
	MW 500 W	0.84 ± 0.06 ij	0.64 ± 0.08 jk	0.23 ± 0.04 kl	0.34 ± 0.07 lm	0.83 ± 0.12 gh	0.15 ± 0.00 e	0.71 ± 0.04 h	3.04 ± 0.39 j
	MW 1000 W	0.41 ± 0.04 k	0.34 ± 0.03 l	0.15 ± 0.02 m	0.25 ± 0.03 m	0.52 ± 0.05 hij	0 h	0.49 ± 0.06 ij	2.6 ± 0.27 j

Pre-drying	Drying methods	Limonene	1,8-Cineole	γ-Terpinene	Linalool	Borneol	Terpinene-4-ol	α-Terpineol	Thymoquinone
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
No exposing to the sun before drying	Fresh plant	1.06 ± 0.14 b	2.09 ± 0.5 b	10.75 ± 1.6 b	0.85 ± 0.07 def	1.76 ± 0.14 bc	1.28 ± 0.19 a	0.24 ± 0.02 c–g	0.217 ± 0.03 jkl
	Shade	0.56 ± 0.09 de	0.93 ± 0.08 f–i	6.5 ± 0.64 cd	0.68 ± 0.02 h–k	1.4 ± 0.05 def	0.68 ± 0.06 e	0.19 ± 0.02 g–k	0.59 ± 0.04 gf
	Sun	0.34 ± 0.05 gh	1.08 ± 0.13 e–h	6.07 ± 0.89 de	0.54 ± 0.09 lm	1.42 ± 0.07 def	0.59 ± 0.05 ef	0.68 ± 0.08 a	0 l
	Oven 35 °C	0.58 ± 0.07 de	0.84 ± 0.12 ghi	4.9 ± 0.30 e	0.71 ± 0.04 ghi	1.25 ± 0.12 e–h	0.51 ± 0.07 f	0.18 ± 0.03 h–k	0.28 ± 0.02 ijk
	Oven 45 °C	0.57 ± 0.03 de	0.86 ± 0.11 ghi	5.61 ± 0.66 de	0.73 ± 0.04 fgh	0.96 ± 0.07 hij	0.59 ± 0.05 ef	0.15 ± 0.02 k	0.12 ± 0.01 kl
	Oven 55 °C	0.54 ± 0.05 de	1.67 ± 0.18 c	3.17 ± 0.56 f	0.93 ± 0.11 cd	2.26 ± 0.38 a	1.096 ± 0.15 b	0.28 ± 0.02 bc	0.36 ± 0.03 hij
	VO 35 °C	0.56 ± 0.02 de	1.13 ± 0.03 efg	1.93 ± 0.62 gh	1.04 ± 0.09 bc	1.34 ± 0.08 d–g	0.57 ± 0.06 ef	0.18 ± 0.03 ijk	0.81 ± 0.03 def
	VO 45 °C	0.27 ± 0.07 hij	0.67 ± 0.07 i	0.55 ± 0.07 ijk	0.704 ± 0.05 g–h	2.34 ± 0.42 a	0.96 ± 0.1 bcd	0.33 ± 0.05 b	0.828 ± 0.04 de
	VO 55 °C	0 k	0.22 ± 0.04 j	0.08 ± 0.01 k	0.396 ± 0.06 no	1.53 ± 0.09 cde	0.59 ± 0.05 ef	0.23 ± 0.02 c–h	1.99 ± 0.32 b
	MW 100 W	0.41 ± 0.05 fg	1.34 ± 0.13 de	3.01 ± 0.64 fg	0.59 ± 0.06 i–l	0.73 ± 0.08 j	0.54 ± 0.06 ef	0.26 ± 0.03 cde	0.15 ± 0.03 jkl
	MW 500 W	0.28 ± 0.05 hij	0.97 ± 0.05 f–i	1.87 ± 0.41 gh	0.43 ± 0.07 mno	2.24 ± 0.37 a	0.56 ± 0.05 ef	0.21 ± 0.02 e–j	0.26 ± 0.04 ijk
	MW 1000 W	0.27 ± 0.07 hij	0.76 ± 0.06 i	1.52 ± 0.27 hi	0.506 ± 0.05 lmn	1.57 ± 0.24 cd	0.54 ± 0.06 ef	0.22 ± 0.01 d–h	0.167 ± 0.02 jkl

(*continued on next page*)

Table 2 (*continued*)

Pre-drying	Drying methods	Limonene	1,8-Cineole	γ-Terpinene	Linalool	Borneol	Terpinene-4-ol	α-Terpineol	Thymoquinone
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
Exposing to the sun before drying	Fresh plant	0.91 ± 0.10 c	2.19 ± 0.34 ab	10.15 ± 1.01 b	1.153 ± 0.13 b	1.27 ± 0.20 d–h	1.28 ± 0.21 a	0.24 ± 0.04 c–g	0.18 ± 0.02 jkl
	Shade	0.54 ± 0.03 de	0.83 ± 0.13 ghi	6.61 ± 0.66 cd	0.82 ± 0.06 d–g	1.01 ± 0.14 hij	0.49 ± 0.05 f	0.17 ± 0.03 ijk	0.91 ± 0.08 d
	Sun	0.33 ± 0.05 ghi	0.8 ± 0.14 hi	1.81 ± 0.24 gh	0.56 ± 0.08 kl	1.26 ± 0.12 e–h	0.49 ± 0.10 f	0.17 ± 0.03 ijk	0.52 ± 0.05 gh
	Oven 35 °C	1.61 ± 0.25 a	2.42 ± 0.47 a	16.55 ± 2.06 a	1.42 ± 0.15 a	1.78 ± 0.14 bc	1.08 ± 0.15 bc	0.16 ± 0.03 ijk	1.79 ± 0.32 b
	Oven 45 °C	0.66 ± 0.05 d	1.17 ± 0.04 ef	7.41 ± 0.78 c	0.86 ± 0.08 de	1.24 ± 0.13 e–h	0.67 ± 0.06 e	0.204 ± 0.02 f–j	0.62 ± 0.05 efg
	Oven 55 °C	0.42 ± 0.05 fg	1.21 ± 0.06 ef	1.45 ± 0.29 hij	0.85 ± 0.06 def	2.03 ± 0.27 ab	0.57 ± 0.06 ef	0.21 ± 0.01 e–i	0.46 ± 0.04 ghi
	VO 35 °C	0.64 ± 0.06 de	1.65 ± 0.28 cd	3.24 ± 0.53 f	0.81 ± 0.05 e–h	1.44 ± 0.03 def	0.87 ± 0.09 d	0.27 ± 0.02 cd	2.43 ± 0.31 a
	VO 45 °C	0.21 ± 0.06 ij	0.93 ± 0.07 f–i	0.25 ± 0.02 jk	0.57 ± 0.07 jkl	1.84 ± 0.17 bc	0.92 ± 0.06 cd	0.25 ± 0.02 c–f	0.81 ± 0.03 def
	VO 55 °C	0 k	0.13 ± 0.03 j	0 k	0.32 ± 0.10 o	1.08 ± 0.11 ghi	0.54 ± 0.07 ef	0.21 ± 0.04 d–i	0.36 ± 0.05 hij
	MW 100 W	0.52 ± 0.03 ef	1.11 ± 0.01 e–h	6.28 ± 0.60 cd	0.74 ± 0.03 e–h	1.17 ± 0.12 fgh	0.54 ± 0.06 ef	0.19 ± 0.02 f–k	1.29 ± 0.27 c
	MW 500 W	0.29 ± 0.03 g–j	0.79 ± 0.06 hi	2.52 ± 0.49 fgh	0.48 ± 0.12 lmn	0.85 ± 0.12 ij	0.56 ± 0.06 ef	0.208 ± 0.01 e–j	0.23 ± 0.02 jk
	MW 1000 W	0.19 ± 0.03 j	1.31 ± 0.08 e	2.31 ± 0.42 fgh	0.84 ± 0.04 def	1.92 ± 0.15 b	0.89 ± 0.05 d	0.24 ± 0.02 c–g	1.304 ± 0.20 c

Pre-drying	Drying methods	*E*-Caryophyllene	β-Bisabolene	Carvacrol	Thymol	Caryophyllene oxide	Monoterpene Hydrocarbons	Oxygenated Monoterpenes	Sesquiterpene Hydrocarbons
		(%)	(%)	(%)	(%)	(%)	(%)	(%)	(%)
No exposing to the sun before drying	Fresh plant	4.58 ± 0.31 cde	0.16 ± 0.017 gh	2.27 ± 0.48 jk	47.18 ± 5.01 i	1.155 ± 0.15 bc	35.03 ± 4.63 b	56.09 ± 4.61 i	35.03 ± 0.39 b
	Shade	3.87 ± 0.35 e–h	0.135 ± 0.020 hi	3.32 ± 0.36 ghi	65.61 ± 4.8 fgh	0.825 ± 0.05 e–h	19.63 ± 1.01 cd	73.48 ± 5.42 gh	19.63 ± 0.29 cd
	Sun	3.53 ± 0.52 gh	0.168 ± 0.004 fgh	4.43 ± 0.4 bcd	68.41 ± 5.6 c–g	0.187 ± 0.02 k	18.11 ± 0.62 de	77.16 ± 6.14 d–h	18.11 ± 0.44 de
	Oven 35 °C	3.42 ± 0.57 h	0.11 ± 0.003 ij	4.22 ± 0.29 b–e	67.07 ± 5.93 e–h	0.77 ± 0.05 fgh	18.04 ± 1.45 de	75.44 ± 5.82 e–h	18.04 ± 0.52 de
	Oven 45 °C	4.07 ± 0.24 e–h	0.16 ± 0.004 fgh	3.79 ± 0.48 d–g	66.74 ± 3.77 e–h	1.16 ± 0.15 bc	18.39 ± 0.78 de	74.19 ± 3.99 fgh	18.39 ± 0.17 de
	Oven 55 °C	7.52 ± 0.58 a	0.26 ± 0.043 bc	3.39 ± 0.12 fgh	59.16 ± 4.03 h	1.34 ± 0.24 a	18.36 ± 0.67 de	69.43 ± 3.36 h	18.36 ± 0.73 de
	VO 35 °C	4.46 ± 0.05 c–f	0.18 ± 0.003 efg	4.14 ± 0.25 b–e	66.76 ± 4.78 e–h	0.72 ± 0.07 gh	16.01 ± 1.23 ef	76.09 ± 4.95 e–h	16.01 ± 0.07 ef
	VO 45 °C	5.68 ± 0.56 b	0.22 ± 0.026 7 cd	3.88 ± 0.31 d–g	71.40 ± 2.1 b–f	1.15 ± 0.15 bc	7.46 ± 0.58 g	81.68 ± 3.02 c–f	7.46 ± 0.69 g
	VO 55 °C	2.15 ± 0.43 i	0.086 ± 0.009 j	4.06 ± 0.21 c–f	84.72 ± 6.76 a	0.46 ± 0.04 j	1.01 ± 0.13 h	94.2 ± 7.29 a	1.01 ± 0.39 h
	MW 100 W	4.37 ± 0.42 c–f	0.17 ± 0.004 fgh	2.75 ± 0.44 h–k	71.69 ± 2.24 b–f	0.84 ± 0.05 efg	13.44 ± 1.38 f	79.85 ± 1.99 c–g	13.44 ± 0.34 f
	MW 500 W	7.06 ± 0.45 a	0.301 ± 0.063 a	4.15 ± 0.26 b–e	70.37 ± 1.58 b–f	1.02 ± 0.08 cd	9.39 ± 1.39 g	79.61 ± 1.99 c–g	9.39 ± 0.64 g
	MW 1000 W	3.78 ± 0.39 fgh	0.135 ± 0.020 hi	4.80 ± 0.58 b	77.46 ± 5.13 ab	1.074 ± 0.11 cd	6.82 ± 1.02 g	86.25 ± 5.75 abc	6.82 ± 0.33 g
Exposing to the sun before drying	Fresh plant	4.18 ± 0.65 c–g	0.15 ± 0.033 gh	2.07 ± 0.9 k	49.18 ± 4.34 i	0.94 ± 0.07 de	33.84 ± 2.42 b	57.82 ± 3.29 i	33.84 ± 0.71 b
	Shade	3.55 ± 0.50 gh	0.136 ± 0.020 hi	3.37 ± 0.13 fgh	67.67 ± 4.23 d–g	0.66 ± 0.06 hi	18.84 ± 0.60 de	75.57 ± 3.93 e–h	18.84 ± 0.44 de
	Sun	4.01 ± 0.27 d–h	0.27 ± 0.048 ab	6.31 ± 0.58 a	68.89 ± 6.16 c–g	0.47 ± 0.06 j	14.42 ± 0.13 f	79.71 ± 6.03 c–g	14.42 ± 0.13 f
	Oven 35 °C	7.02 ± 0.23 a	0.21 ± 0.018 de	0.98 ± 0.13 l	25.26 ± 4.97 j	0.82 ± 0.02 e–h	53.13 ± 6.07 a	35.05 ± 3.94 j	53.13 ± 0.35 a
	Oven 45 °C	4.64 ± 0.54 cd	0.178 ± 0.003 efg	2.90 ± 0.37 hij	62.25 ± 6.48 gh	0.72 ± 0.07 gh	22.73 ± 2.09 c	70.04 ± 6.99 h	22.73 ± 0.63 c
	Oven 55 °C	5.84 ± 0.64 b	0.231 ± 0.028 cd	4.01 ± 0.19 c–g	66.57 ± 5.68 e–h	0.8 ± 0.03 e–h	13.47 ± 0.26 f	76.14 ± 6.17 e–h	13.47 ± 0.77 f
	VO 35 °C	3.94 ± 0.31 d–h	0.147 ± 0.014 ghi	2.64 ± 0.5 ijk	61.48 ± 3.86 gh	1.29 ± 0.22 ab	19.22 ± 1.27 d	71.93 ± 3.63 gh	19.22 ± 0.25 d
	VO 45 °C	6.06 ± 0.65 b	0.2 ± 0.014 def	3.71 ± 0.54 efg	73.97 ± 3.38 b–e	1.29 ± 0.19 ab	6.56 ± 1.02 g	83.01 ± 4.06 cde	6.56 ± 0.76 g
	VO 55 °C	1.88 ± 0.34 i	0.078 ± 0.009 j	4.61 ± 0.49 bc	84.84 ± 5.82 a	0.49 ± 0.05 j	0.81 ± 0.05 h	92.16 ± 5.90 ab	0.81 ± 0.30 h
	MW 100 W	4.01 ± 0.27 d–h	0.133 ± 0.022 hi	2.74 ± 0.45 h–k	66.60 ± 7.3 e–h	0.93 ± 0.05 def	18.79 ± 0.68 de	74.63 ± 7.63 fgh	18.79 ± 0.22 de
	MW 500 W	4.87 ± 0.36 c	0.185 ± 0.005 efg	4.17 ± 0.27 b–e	75.99 ± 4.39 bc	0.53 ± 0.04 ij	9.58 ± 1.29 g	83.29 ± 4.27 cde	9.58 ± 0.46 g
	MW 1000 W	4.47 ± 0.22 c–f	0.162 ± 0.009 gh	2.98 ± 0.33 hi	75.66 ± 5.23 bcd	1.012 ± 0.05 cd	7.33 ± 0.94 g	85.23 ± 4.00 bcd	7.33 ± 0.10 g

The means with the same letters in each column indicates no significant difference between treatments at the 5% level of probability. VO: Vacuum oven; MW: Microwave.

carvacrol were observed in vacuum-drying at 55 °C in both the pre-drying conditions and sun-drying along with pre-drying, respectively. Generally, the highest amounts of monoterpene and sesquiterpene hydrocarbons were obtained by oven-drying at 35 °C along with pre-drying and also, the maximum content of oxygenated monoterpenes was related to oven-drying at 55 °C without pre-drying.

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