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Impact of different drying approaches on VOCs and chemical composition of *Mentha spicata L.* essential oil: A combined analysis of GC/MS and E-nose with chemometrics methods

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A R T I C L E I N F O

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A B S T R A C T

Given the significance spearmint (*Mentha spicata L.*), there is an increased demand for dry medicines and high-quality aromatic mint. In this study, a non-destructive technique based on electronic nose was used in combination with a GC/MS method and chemometrics to determine the quality of spearmint essential oil under eight drying methods. It was found that hot air drying (HAD) method yielded the highest amount of essential oil and its compounds. So that its highest amount was obtained in HAD1a method and equal to 1.380%. However, the amount decreased with increasing drying temperature and air velocity. In addition, sun drying performed worst its value was equal to 0.663%. The highest percentage of the obtained essential oil compounds was related to the three main components of carvone (64.30–7.45%), limonene (24.21–6.59%), and carveol (18.34–1.92%). Furthermore, the Nu-SVM classification algorithm with the sigmoid function provided an accuracy of 0.975 for classifying eight essential oil groups. In light of the limited knowledge about the effects of drying on the qualitative and quantitative characteristics of medicinal plants, a new method for essential oil evaluation was proposed in this study. The results could provide a theoretical reference to choose the best drying method and quickly determine the quality of mint essential oils.

1. Introduction

Plant-derived compounds are commonly used in medicine and preventive health care, and the scope of some substances is constantly expanding. Over 200 genera and 3000 species comprise the mint family, which is economically and medicinally very important. A mint genus consists of 25–30 species found in the temperate regions of Asia, Europe, Australia, and South Africa (ref). Mint species differ significantly in their chemical composition. The essential oil of spearmint (*Mentha spicata L.*) contains large amounts of carvone, which is responsible for its special aroma (Zhao et al., 2013).

The essential oil yield of *M. spicata* is lower than *M. piperita.* Carvone is the main constituent of *M. spicata* and *M. longifolia*, while this compound is not found in *M. piperita*, *M. aquatic*, *M. arvensis*, and *M. pulegium* (Mahboubi, 2021). The essential oils and extracts of spearmint have a variety of applications in the pharmaceutical, cosmetic, and food industries. For example, the essential oil and leaves of *M. spicata* are used therapeutically. Their general properties are analgesic, tonic, stomachic, cough suppressant, antispasmodic, astringent, analgesic, and sedative (Đurović et al., 2022). Spearmint oil has also been used medicinally since ancient times, especially for headaches, colds, and neuralgia. It also relieves skin irritation and digestive problems and has antispasmodic properties (Balakrishnan, 2015). Despite contradictory findings about the chemical composition of *M.spicata* essential oil, many studies have confirmed carvon and limonene as the main constituents (Mahboubi, 2021). Spearmint essential oil contains

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carvone. The high price of this compound has prompted breeders to develop mint varieties with high carvone content. In the aromatic and pharmaceutical industries, the different chemotypes are characterized by specific odors and biological activities. Europeans, for instance, enjoy the scent of carvone (Zhao et al., 2013). The effectiveness of a medicinal plant in both food and pharmaceutical industries depends on the amount and composition of its biologically active constituents (Velička et al., 2022).

The genetic factors play a fundamental role in determining the chemical composition of essential oils (EO). However, various biotic and abiotic elements comprising environmental diversity can induce significant alterations in the constituents' synthesis. These elements have the capacity to redirect metabolic pathways, ultimately resulting in the biosynthesis of different compounds. Among these influential factors, climatic elements such as temperature, precipitation, solar irradiation, and relative air humidity hold prominence. Their impact on EO production can fluctuate depending on the season and even the specific time of plant harvesting. Furthermore, the numerous interspecific interactions that occur between plants and various entities in their geographic environment, such as microorganisms, insects, and other plants, must not be overlooked (Sá Filho et al., 2022).

Several factors affect the concentration of volatile compounds in mint during drying, including drying conditions (temperature, air velocity), humidity, plant variety and age, climate, soil, and harvesting methods. Essential oils can be affected by drying and storage conditions (Karami et al., 2017). Both agricultural products and food quality are preserved by drying, an important preservation technique in the food industry. In addition, drying reduces the water activity of the product to suppress the growth of microorganisms and inhibit chemical reactions to extend shelf life. Additionally, drying facilitates transportation and saves storage space (Kaveh et al., 2020). The most common drying methods include hot air drying (HAD), vacuum drying (VD), vacuum freeze drying (VFD), and microwave hot air alternating drying (MW-HAD). In the HAD method, food is dried in an oven with a constant hot air flow. Despite its ease of operation and low cost, this method requires a long drying time and consumes little energy as an optimal method for drying raw plant foods (Karami et al., 2021).

During drying, mass and heat are transferred simultaneously, resulting in adverse changes, including a reduction in the quality of the final product. When the drying process is carried out under different conditions, enzymatic and non-enzymatic reactions can occur, resulting in significant variations in the composition of secondary metabolites. Thus, since not all developed drying methods are suitable for different biological materials, it is important to select an appropriate drying method to optimize the productivity of the targeted phytochemicals (Park et al., 2021).

Various drying methods have been investigated to improve the quality of dry mint leaves by evaluating their effects on the polyphenol content, antioxidant capacity, and essential oil performance (Zhu et al., 2015). Guo et al. (2022), investigated the drying kinetics, textural, and aromatic characteristics of mint leaves during hot air thin layer drying. The results showed that hot air thin film drying at 35 °C improved the final quality while maintaining the flavor of dried *M. haplocalyx* leaves.

Gas chromatography-mass spectrometry (GC/MS) is widely used for quantitative and qualitative analysis of aromatic compounds in food. Using GC/MS, the high separation performance of a GC system can be complemented by the high sensitivity of an MS system. These systems can identify compounds based on fragmentation patterns with a sensitivity of one part in a billion. Columns for gas chromatography can have multiple polarities, allowing them to analyze compounds with different polarities. To comprehensively determine the composition of a sample, chromatography columns with different polarities should be used (Fan et al., 2018). Whereas, the use of electronic noses (e-noses) based on electronic sensors is becoming more common as an alternative to the GC/MS technique. With the better ease of use, high sensitivity, real-time detection, and non-destructive properties, e-nose has shown better performance compared to other analytical techniques such as GC/MS (Rusinek et al., 2022). It also analyzes volatile compounds in the gas phase without separating individual aromatic components from the odor (Aghili et al., 2023). This system consists of arrays of gas sensors and can identify simple or complex odors and aromatic profiles with a pattern recognition system (Wardencki et al., 2013). Moreover, the e-nose is fast, easy to use and does not interfere with the analyzed sample. It has already been used in various applications to identify aromas in food and agricultural products (Żytek et al., 2023). The olfactory system of the E-Nose machine is capable of imitating the human nose and recognizing complex patterns like the human olfactory system. The system consists of three components: (1) a sample transport system, (2) a sensor array with partial characteristics for gas detection, and (3) a data processing system for processing odor data. Regardless of how much or how little odor is present, the e-nose device can detect the presence of VOCs in a variety of molecular structures (Karami et al., 2020b, 2020c).

The study process consists of two phases. In the first phase, different drying methods were studied and their effects on essential oil quantity were investigated. It should be noted that successful completion of this task is a prerequisite for beginning the next phase of the study. GC/MS and E-nose methods were used in the second stage to investigate the quality of the essential oil. Moreover, in light of the limited knowledge about the effects of drying on the qualitative and quantitative characteristics of medicinal plants, a new method for essential oil evaluation was proposed in this study. Although e-nose technology has made great strides in other applications, there are few reports on its application to medicinal plant quality control. With e-nose technology, the odor could become a new quantitative indicator for medicinal plant quality control.

2. Materials and methods

The study process consists of two phases. In the first phase, different drying methods will be studied and their effects on oil quantity will be investigated. It should be noted that successful completion of this task is a prerequisite for beginning the next phase of the study. GC/MS and E-nose methods will be used in the second stage to investigate the quality of the essential oil.

2.1. Sample preparation

For drying experiments, fresh mint samples were harvested daily from Miandarband region (34°36'52.3"N 46°56'47.4"E) of Kermanshah in September 2022, both leaves and stems of mint were used for experiments. After transferring them to the laboratory, a cleaning step was performed on them (removing excess parts such as unusable leaves and roots). To determine the initial moisture content of the mint samples, the standard oven method was applied for 1 h at 105 °C (Parhizi et al., 2022). Finally, the average initial moisture of basil leaves obtained 82 ± 0.5% (w.b.).

2.2. Drying equipment and empirical method

Hot air drying (HAD) method with the help of hybrid solar-electrical dryer was used at three temperatures of 40, 50, and 60 °C denoted by symbols (1, 2, and 3) and at two inlet air velocities of 1 and 2 m/s, respectively, as shown in symbols (A, B). In addition, two samples were dried in the sun and shade. Drying experiments were performed with a solar electric hot air dryer that heats the air after it passes over and draws it into the trays with a fan. The temperature was controlled by an electric element at the output of the collector, powered by a solar panel that stores the energy needed for the element and the control parts of the device. In addition, this dryer is equipped with temperature and humidity sensors to control the drying process. All experiments were conducted 30 min after the dryer was turned on to reach a steady state. A thin layer of mint was placed on the trays and placed in the drying chamber.

2.3. Extraction, identifying the compounds and odor essential oil

2.3.1. Hydro-distillation

After the drying process, the essential oil was extracted from the dried product by hydro-distillation using a clevenger apparatus. Essential oils can be extracted by distillation with water, which is cheap because water is usually used as a solvent. EO was extracted by heating 500 mL of distilled water and 50 g of dried spearmint leaves up to 100 °C, followed by liquefaction of the vapors in a condenser. The setup consists of a condenser and a decanter to collect the condensate and separate EO from the water. In each treatment, the extraction of the essential oil after boiling was considered for three hours, and the yield of essential oil was calculated as the volume in mL (v/w%) of essential oil. According to Eq. (1), the yield was calculated based on the fresh weight from the relationship between EO and the dry leaf weight (Djamila et al., 2021):

$$\text{EOyield}(\%) = \frac{\text{Q1}}{\text{Q2}} \times 100 \tag{1}$$

Where Q1 is quantity of EO (g), and Q2 is Quantity of leaves (g).

To remove moisture, the essential oil was dried over anhydrous sodium sulfate. The sample was then sealed in a glass bottle and stored at −18 °C until analysis.

2.3.2. Gas chromatography/mass spectrometry (GC/MS) analyses of EO

Qualitative GC/MS analysis of the extracted essential oils was performed using a HP 6890 gas chromatograph coupled to a HP 5973 mass selective detector (Agilent Technologies, Foster City, CA, USA) operating in 70 eV mode. The sample (20 μL, essential oil) was diluted to 1 mL with hexane (≥99%) (provided by Sigma Aldrich, Germany). Compounds were separated on a capillary column (HP −5MS), (30 m long × 0.25 mm diameter) using a 0.25 μm thick stationary phase film ((5% phenyl) methylpolysiloxane). The flow rate of helium carrier gas was set to 1.23 mL/min. The oven temperature was held at 40 °C for 2 min after injection and then programmed at 3 °C/min to 210 °C, where the column was held for 10 min. The split ratio was 1:10. The electron ionization of the mass detector was 70 eV. The volatile compounds were identified by searching on the stored Wiley 7 n,l mass computer library, and NIST 05a,l (National Institute of Standards and Technology) and comparing them with mass spectra data (Adams, 2007; Matulyte et al., 2019).

2.3.3. E-nose

Analysis of spearmint essential oil samples was performed using a portable E-nose (Rasekh et al., 2021b) consisting of an array of multiple gas sensors, a signal acquisition unit, and pattern recognition software. Nine chemical metal oxide semiconductor sensors are included in the array.

A sample of essential oil (1 mL) was placed in a sealed glass vial and kept at equilibrium at 40 °C for 30 min. The volatiles in the headspace of the sealed glass vial was transported with clean ambient air into the temperature- and humidity-controlled sensor chamber. The conductance change in the sensor array is expressed by the normalized sensor response Xs(t)-Xs(0)/Xs(0), where Xs(t) and Xs(0) represent the unprocessed sensor response and the baseline, respectively. Each measurement cycle lasted 100 s, allowing the sensor to reach a steady state. The interval for data acquisition with a computer was 1 s. Between measurement cycles, the sensor was purged with purge gas filtered through activated carbon for 200 s to return the sensor signal to baseline. For each spearmint essential oil sample, 15 measurements were made.

2.4. Data processing

An MSD Chemstation (Agilent Technologies, Inc., Santa Clara, CA, USA) first processed the GC/MS data and structural identification was performed using NIST 2014 library search and retention index (RI) validation. The dataset consists of preprocessed signals obtained from 9 MOS gas sensors installed in the e-nose during 120 measurements corresponding to 8 independent samples evaluated with 15 replications. Unsupervised statistical tools such as principal component analysis (PCA) and supervised (linear and quadratic discriminant analysis (LDA and QDA, respectively), and support vector machines (SVM)) were used along with feature extraction (pre-processing) methods. To build the best supervised multivariate classification models, the dataset was divided into a training dataset for learning purposes and an internal validation using the cross-validation method.

To reduce the dimensionality of the experimental data, PCA was used as an unsupervised pattern recognition method (Hidayat et al., 2019). The purpose of this analysis was to show how the values of spearmint essential oils can be grouped based on their volatile composition profiles. Therefore, PCA provides a preliminary evaluation of the potential of e-nose as an unsupervised classification model for spearmint essential oil samples.

Three supervised statistical methods were used to evaluate the performance of e-nose on spearmint essential oil samples: LDA, ANN, and SVM with four linear, radial, sigmoid, and RBF kernels. A confusion matrix was created to derive cross-validity predictions. The spearmint essential oils were divided into a calibration set, which included 70% of all samples, and a validation set, which included 30% of all samples. Data from the electronic noses were analyzed using TheUnscrambler software.

2.5. Statistical analysis

Analysis of variance was performed as an ANOVA −1 design based on completely random. Duncan's test was used to determine statistically significant differences between spearmint essential oil samples under different drying conditions ($p \leq 0.01$). Statistical analysis was performed using Mstatc software. Data were expressed as mean and standard deviation (SD).

3. Result and discussion

3.1. Effect of drying methods on the essential oil yield

For the effect of different drying methods on the performance of essential oil, first, the essential oil of dried samples was extracted by hydro-distillation method. Hydro-distillation yielded dried mint leaves ranging from 0.663% to 1.380%. The statistical analysis revealed that different drying methods significantly affected the performance of essential oils ($p \leq 0.01$) (Table 1).

An average of 0.772% spearmint essential oil was obtained from all drying methods. Beigi et al. (2018) obtained an average oil yield of about 15.16 mL/kg dry weight. Chi et al. (2016) confirmed an average oil yield of 8 mL/kg dry matter. According to research conducted by Moghaddam et al. (2013), the essential oil content was 13.8 g/kg of dry matter. Also, Scavroni et al. (2005) reported an average oil yield of about 13.9 mL/kg dry matter. Various parameters may contribute to the differences between the reported values, such as the environmental and experimental conditions to which the plants were exposed.

According to Fig. 1, Duncan's multi-range mean comparison results at a 1% level compared extracts of mint essential oil from different

Table 1
Analysis of variance of essential oil of *Mentha spicata L.*

	Degrees of Freedom	Sum of Squares	Mean Square
Drying methods	7	1.210	0.173*
Error	16	0.008	0.0005
Total	23	1.217	

* Significant at $p \leq .01$; Cv = 2.31%.

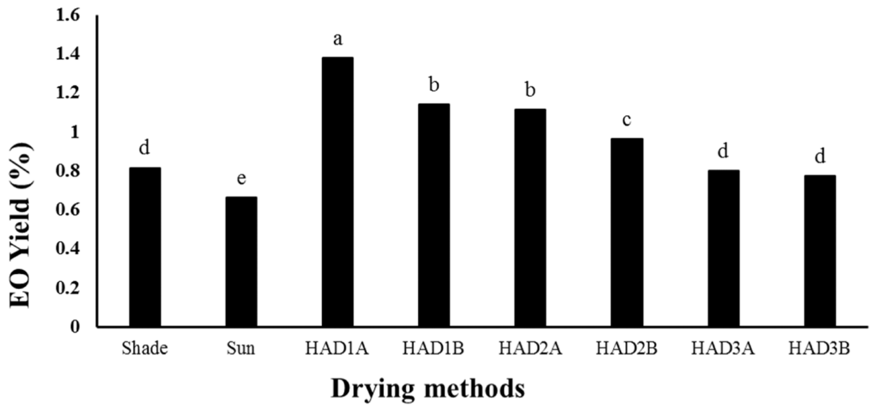


Fig. 1. The average effect of drying methods on the extracted essential oil (α = 0.01). (LSD Value = 0.05333).

drying methods.

The yield of essential oil from dried leaves for methods HAD1A, HAD2A, and HAD3A was 1.380, 1.114, and 0.799, respectively. Furthermore, the same value for HAD1B, HAD2B, and HAD3B was 1.141, 0.9640, and 0.7720, respectively. Also, the performance of extracted essential oil yield in the shade and sun drying methods was 0.8160 and 0.6630, respectively. The HAD1A method extracted the highest amount of essential oil, while the sun-drying method yielded the lowest amount.

The results are in agreement with Rohloff et al. (2005) who reported that mint harvested at different growth stages, including early flowering, full flowering, and late flowering, continuously yielded less essential oil when the drying temperature was increased from 30 to 70 °C. Blanco et al. (2000) determined the essential oil content in dried spearmint leaves at 40, 60, and 80 °C to be 1.0, 0.14 and 0.12 (%V/W), respectively. Similarly, Chi et al. (2016) reported that increasing the drying temperature of mint leaves in the range of 40–80 °C resulted in a decrease in essential oil content. Similar results were reported for various medicinal and aromatic plants (Karami et al., 2017). Aromatic plants should be dried between 30 and 50 °C to obtain excellent EO yields (Djamila et al., 2021; Karami et al., 2020a).

3.2. Chemical compositions of mint essential oil

The results of GC/MS analysis of mint leaves obtained from fresh and dried leaves are shown in Table 2 and Fig. 2. Twenty-four components were identified in the mint leaves after different drying methods, which accounted for 98.15% of the total volatiles. Shade and sun drying identified 16 and 15 compounds, respectively, while hot air drying identified 18 compounds. Fig. 2 shows the main components of the dried samples: carvone (64.30–7.45%), limonene (24.21–6.59%), and carveol (18.34–1.92%). The effects of the considered methods and drying conditions on the chemical composition of mint leaves can be discussed according to Table 2. Results showed that drying treatments can significantly change the chemical profiles of essential oils. Different processes, such as oxidation, glycoside hydrolysis, esterification and others, lead to the loss or increase of some essential oils (Pirbalouti et al., 2013).

Drying produces new compounds that cannot be identified when dried in the shade or sun, such as sabinene, myrcene, linalool, and alloocimene. While drying in the shade or sunlight preserves some compounds, including pulegone, a monoterpene ketone found in the leaves and flower parts of several members of the mint family. Besides, the 3 compounds piperine, bornyl acetate, and isopulegone were present only in shade-dried samples. This phenomenon can be explained by the fact that the drying method activates the hydrolase enzymes, which change the proportion of volatile compounds in certain ways (Rios-Estepa et al., 2008). The changes in oil composition depend on several factors, including plant species, drying method, plant age, and cultivation conditions.

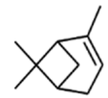
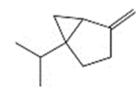
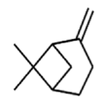
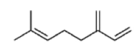
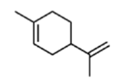
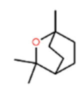
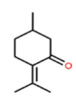
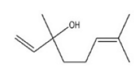
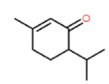
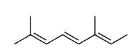
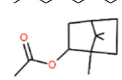
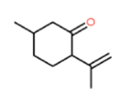
The carvone content of a mint plant varies greatly depending on climatic conditions, its vegetative or reproductive state, and its processing method. The carvone chemotype is grown commercially. Spearmint essential oil should contain more than 50% carvone (Zheljazkov et al., 2010). Table 2 shows that the proposed drier was able to maintain more than 50% carvone in drying methods HAD1A, HAD1B, HAD2A, and HAD2B. In comparison, this value decreased with increasing drying temperature and was 49.38% and 24.53% in shade and sun drying, respectively. The highest amount of carvone was associated with HAD1A at 64.30%, while the lowest amount was associated with HAD3B at 7.45%. Another main compound was limonene, whose highest amount was obtained at 24.21% with HAD1B and the lowest amount at 6.59% with the sun-drying method. The third compound is carveol, a limonene monoterpenoid with a minty odor that appears pale yellow or colorless liquid. With a value of 18.34%, HAD3A obtained the highest value, while sun-drying gave the lowest value of 1.92%.

Zekri et al. (2019) performed a similar study on mint. They dried mint in the shade for different periods up to six days using the shade drying method. Their results showed that carvone and limonene were the most abundant essential oils. The values ranged from 74.91% and 13.17% for Meknes and 71.56% and 10.50% for Azro. Chauhan et al. (2009) investigated the chemical composition of the essential oil of *Mentha spicata L.* from the northwestern region of Himalayas, India. They reported that carvone was the main constituent, ranging from 49.62% to 76.65%, followed by limonene, which ranged from 9.57% to 22.31%.

The results show that hot air drying strongly affects the volatile compounds in the leaves of *M. spicata* L., especially when the content of these volatile compounds is very low. Nevertheless, we found that the mint-derived bioactive compounds were mostly well preserved in the hot air-dried samples, as shown in Table 2.

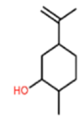
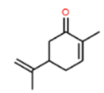
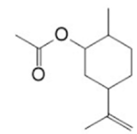
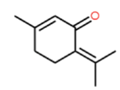
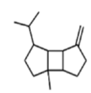
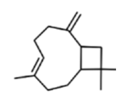
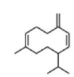
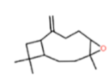
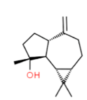
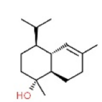
The volatile components of spearmint essential oil were classified by cluster analysis and affected by different drying methods. Fig. 3 shows the results in the form of a heat map. Heat maps provide an alternative to visual representations of high-dimensional data. On the left side of Fig. 3, the cluster lines represent clusters of volatile compounds. Three main clusters of volatile compounds were identified. The first two, starting at the top of the map, are represented by carvone compounds and are the major compounds most abundant in spearmint essential oil. The third cluster refers to the compounds with the lowest amounts, most of which were in this cluster. Fig. 3 depicts different drying methods as cluster lines. There were two main groups of drying methods. HAD2B, HAD1A, HAD1B, and HAD2A are in the first group, on the left. Four methods preserved the most essential oil, while the second group, located on the right side, included methods such as HAD3A, HAD3B, shade, and sun, which had the least amount of extracted essential oil.

Table 2
The active ingredient content of the *Mentha spicata L.* at various drying methods by GC/MS.

Compound	Synonym	Classification	Formula	Molecular weight (g/mol)	Chemical structure	RT (min)	RI	Drying methods (%)							
								Shade	Sun	HAD1A	HAD1B	HAD2A	HAD2B	HAD3A	HAD3B
α-pinene	(1S,5S)-2,6,6-Trimethylbicyclo[3.1.1]hept-2-ene ((−)-α-Pinene)	Bicyclic monoterpene	$C_{10}H_{16}$	136.23		5.67	931	-	0.47	-	-	-	2.30	1.68	5.99
Sabinene	1-isopropyl-4-methylenebicyclo[3.1.0] hexane	Bicyclic monoterpene	$C_{10}H_{16}$	136.23		6.31	972	-	-	0.32	0.66	0.47	1.78	0.87	1.56
β-pinene	Pseudopinene Nopinene 2(10)-Pinene	Bicyclic monoterpene	$C_{10}H_{16}$	136.23		6.40	975	0.97	0.94	0.81	0.74	1.14	2.45	2.08	8.26
Myrcene	β-myrcene	Acyclic monoterpene	$C_{10}H_{16}$	136.23		6.62	988	-	-	0.14	0.71	0.58	5.25	3.28	5.42
Limonene	Dipentene *p*-mentha-1,8-diene	Monocyclic monoterpene	$C_{10}H_{16}$	136.23		7.50	1030	10.5	6.59	16.01	24.21	18.04	18.94	13.26	16.3
1,8 Cineole	Eucalyptol	Bicyclic monoterpenoid / Ether	$C_{10}H_{18}O$	154.23		7.80	1032	-	0.71	-	-	-	-	-	1.12
Pulegone	*P*-menthen-3-one	Monocyclic monoterpenoid / Ketone	$C_{10}H_{16}O$	152.23		8.39	1214	13.53	20.62	-	-	-	-	-	-
Linalool	3,7-Dimethylocta-1,6-dien-3-ol	Acyclic monoterpenoid / Alcohol	$C_{10}H_{18}O$	154.23		8.78	1099	-	-	-	-	-	0.58	0.87	2.54
Piperitone	α-Piperitone 2-Cyclohexen-1-one, 3-methyl-6-(1-methylethyl)	Monocyclic monoterpenoid / Ketone	$C_{10}H_{16}O$	152.23		8.82	1228	3.01	-	-	-	-	-	-	-
Alloocimene	2,6-Dimethyl-2,4,6-octatriene	Acyclic monoterpene	$C_{10}H_{16}$	136.23		9.42	1131	-	-	0.45	0.54	0.57	0.61	0.99	4.61
Bornyl acetate	2-Camphanol acetate 1,7,7 trimethylbicyclo [2.2.1] heptan-2-yl acetate		$C_{12}H_{20}O_2$	196.23		10.44	1290	0.74	-	-	-	-	-	-	-
Isopulegone	*p*-menth-8-en-3-one	Monocyclic monoterpenoid / Ketone	$C_{10}H_{16}O$	152.23		10.59	1178	2.73	-	-	-	-	-	-	-

(*continued on next page*)

Table 2 (*continued*)

Compound	Synonym	Classification	Formula	Molecular weight (g/mol)	Chemical structure	RT (min)	RI	Drying methods (%)							
								Shade	Sun	HAD1A	HAD1B	HAD2A	HAD2B	HAD3A	HAD3B
Carveol	Dihydrocarveol Cyclohexanol, 2-methyl-5-(1-methylethenyl)- 8-*p*-menthen-2-ol	Monocyclic monoterpenoid / Alcohol	$C_{10}H_{18}O$	154.23		11.22	1250	10.44	1.92	2.66	1.37	15.46	2.42	18.34	5.32
Carvone	Carvol *p*-mentha-6,8-dien-2-one	Monocyclic monoterpenoid / Ketone	$C_{10}H_{14}O$	150.23		12.46	1262	24.53	49.38	64.30	58.83	53.65	50.12	34.25	7.45
Dihydrocarvyl acetate	Dihydrocarveol acetate Tuberyl acetate Carhydrine p-Menth-8-en-2-yl acetate		$C_{12}H_{20}O_2$	196.23		14.18	1338	1.45	2.13	1.93	1.18	2.67	1.36	4.17	4.65
Piperitenone	3-Terpinolenone *p*-menth-1,4(8)-dien-3-one	Monocyclic monoterpenoid / Ketone	$C_{10}H_{14}O$	150.23		14.69	1340	1.19	0.34	0.22	0.19	0.66	0.23	0.29	0.6
β-bourbonene	4(15)-Bourbonene	Tricyclic sesquiterpene	$C_{15}H_{24}$	204.35		15.44	1387	0.75	0.11	2.25	1.88	0.96	2.13	2.08	3.46
β-caryophyllene	trans-Caryophyllene	Bicyclic sesquiterpene	$C_{15}H_{24}$	204.35		16.19	1422	1.83	1.15	2.33	1.58	2.31	1.75	2.64	0.63
Germacrene-D		Monocyclic sesquiterpene	$C_{15}H_{24}$	204.35		16.70	1482	0.36	0.17	2.82	1.18	0.37	0.57	1.41	9.52
Caryophyllene oxide	β-Caryophyllene oxide Epoxy Caryophyllene	Tricyclic sesquiterpenoid / Ether	$C_{15}H_{24}O$	220.35		19.25	1574	5.09	2.83	0.50	0.38	0.85	0.30	0.23	0.69
Spathulenol	Spatulenol	Tricyclic sesquiterpenoid / Alcohol	$C_{15}H_{24}O$	220.35		19.50	1575	0.27	0.12	0.45	0.30	0.27	0.35	0.36	0.81
α-cadinol	Cadin-4-en-10-ol	Tricyclic sesquiterpenoid / Alcohol	$C_{15}H_{26}O$	222.35		20.95	1655	0.21	0.19	0.29	0.29	0.15	0.18	0.45	0.97
Total								77.6	87.67	95.48	94.04	98.15	91.32	87.25	79.9
Other compounds								22.4	12.33	4.52	5.96	1.85	8.68	12.75	20.1

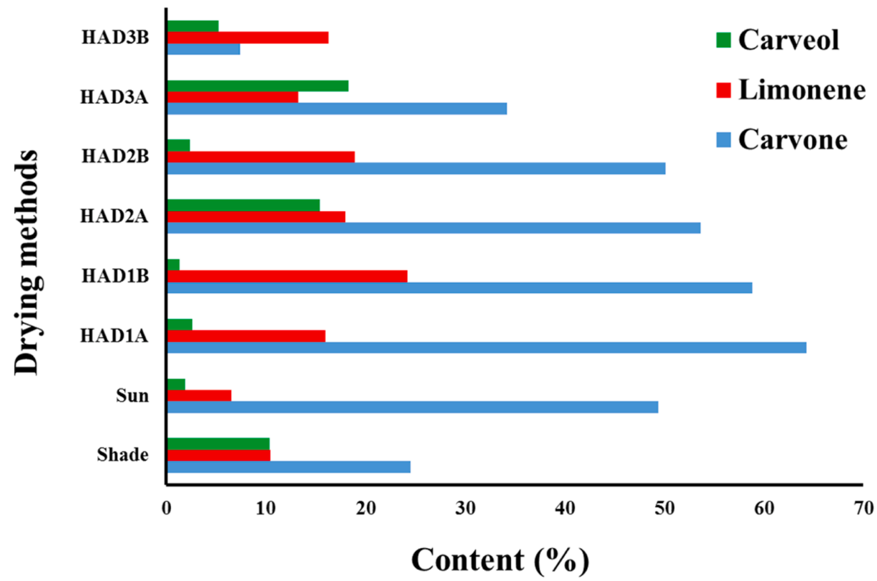


Fig. 2. Contents of three common components in *Mentha spicata L.* volatile oil at different drying methods.

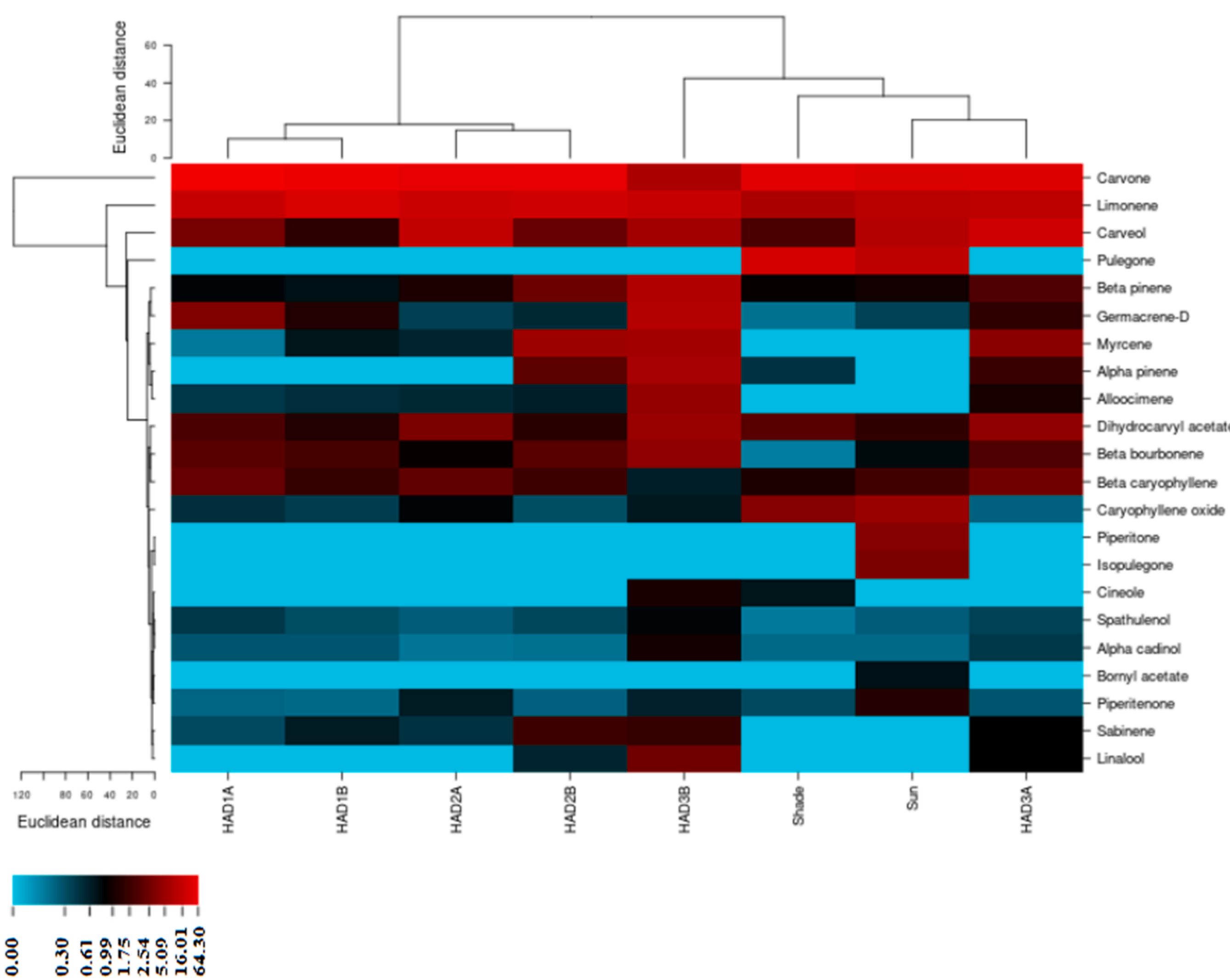


Fig. 3. Heat map of the diversity of volatile compounds in the different drying methods. Volatile compounds were plotted on the Y-axis, while the X-axis corresponds to different drying methods.

3.3. Principal component analysis

The effects of drying methods on the volatiles of the dried mint leaves were investigated using PCA analysis, as shown in Fig. 4a. The variance of PC1 and PC2 in PCA were 72% and 19%, respectively. The cumulative variance contribution was 91%, indicating that PCA was able to reflect the sensory information of volatile compounds in dried mint leaves under different drying methods. The PCA result showed that the aroma profiles analyzed by E-nose were differentiated among the different drying methods. As shown in Fig. 4a, the samples obtained by drying with a hot air dryer are mainly located in the first and second areas, while those obtained by shading and sun drying are found in the fourth area. Consequently, there was no clear overlap between the samples dried by different methods, indicating that the mint leaf samples have a unique profile of volatile aromatic compounds. Based on this result, electronic noses can identify volatile components that define sensory properties.

The loading plot (Fig. 4b) also shows the predicted values of the variables. The chemical compounds located in the area marked by the two circles have a great influence on the possibility of indicating the drying method (Huo et al., 2014; Kang et al., 2014). According to Fig. 4b, the compounds that are exclusive to solar and shade drying are

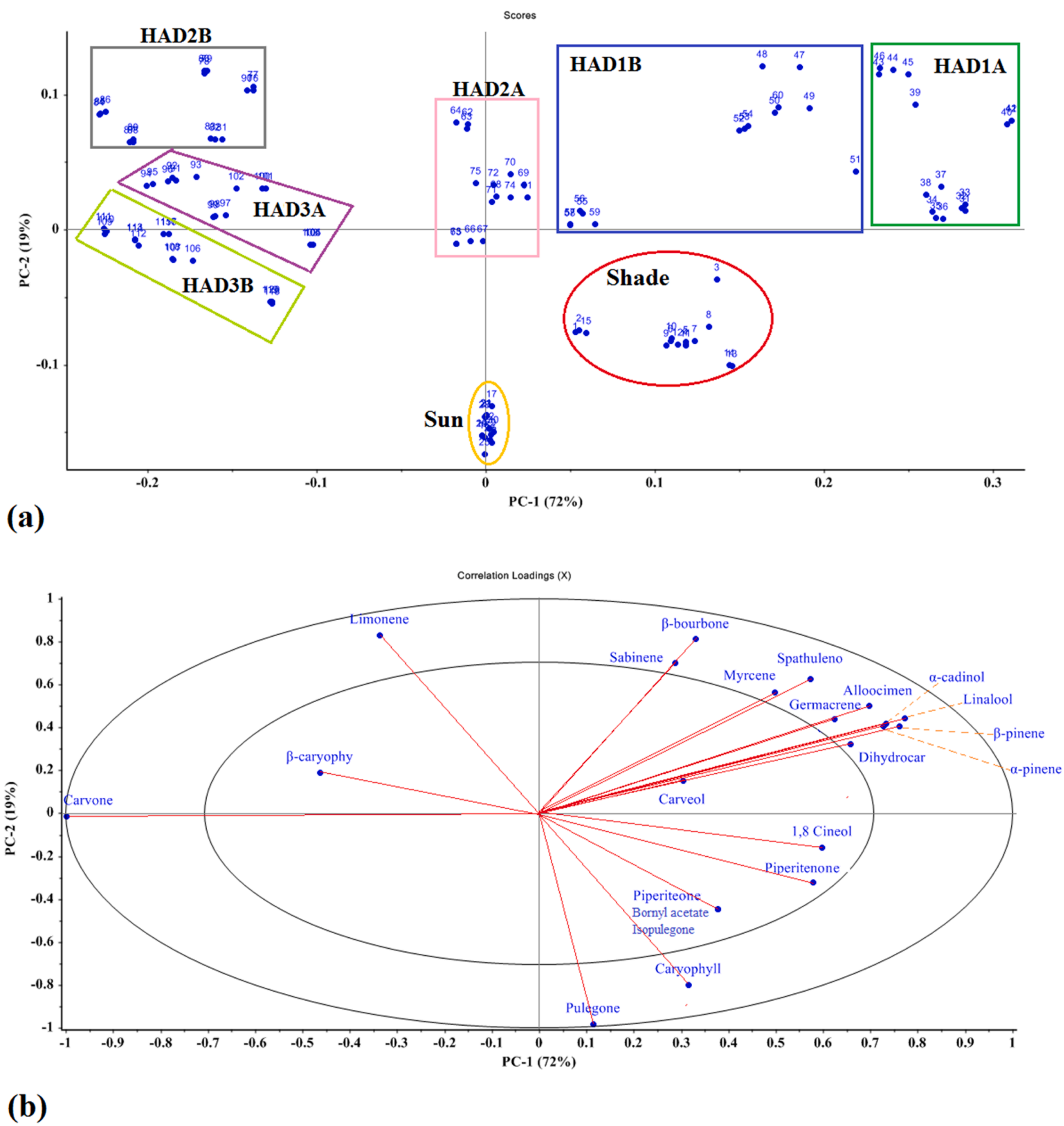


Fig. 4. (a) PCA of the electronic nose of Mentha haplocalyx volatile components under various drying methods and (b) loading diagram of the compounds identified by GC/MS method.

in the fourth region, based on the loading diagrams for the results of GC/MS. Pulegone, piperitone, bornyl acetate, isopulegone, and 1,8 cineole are among these compounds. Based on these results, the electronic nose can accurately identify samples based on their drying method. Caryophyllene oxide and piperitenone, which were present in all drying methods, were also included here because their amounts were higher in the sun and shade drying methods than in the HAD drying method. Therefore, these compounds can be used to exclusively identify the sun and shade drying methods.

Using the HAD drying method, most of the compounds are in the first and second regions (Fig. 4b), which is consistent with the PCA plot (Fig. 4a). The linalool composition, which was present only at HAD drying and high temperatures, was in the second region. Also, Fig. 4A, the three drying methods, HAD2B, HAD3A, and HAD3B, are also in the same range.

It can be concluded that the results of the electronic nose have a very strong correlation with the results of GC/MS, which makes it an acceptable substitute for GC/MS for quality control. In addition to wine, tea, and herbal extracts, the electronic nose has also been successfully used to investigate the sensory profile of a variety of food products (Huo et al., 2014; Kang et al., 2014; Laureati et al., 2010; Rasekh et al., 2021a).

Guo et al. (2022) evaluated the textural and aromatic properties of oregano leaves during the hot air thin film drying process using an electronic nose. The results showed that drying the samples at 35 °C tended to show a higher response in volatile organic compounds, while drying at 45 °C or 55 °C tended to show a higher response. Accordingly, as the temperature and velocity of the drying air increase, the samples shift from the right to the left side of the PCA graph, which means that they exhibit less odor as the temperature and velocity increases. Since volatile organic compounds are sensitive to heat, our results suggest that low drying temperatures are beneficial for preserving essential oils. This is because some of the active compounds of the plant escape when dried at high temperatures. During drying, moisture is released from the surface of the leaves by diffusion, and since essential oil glands are located on or near the surface, some of the plant's oil is lost. This mechanism may explain the reason for the decrease in the amount of compounds and odor of the essential oil during drying (Karami and Rasekh, 2018).

3.4. Supervised multivariate classification methods

3.4.1. To validate the ability of E-nose to predict quality grade in real-time using data collected data, supervised multivariate classification methods such as LDA, QDA, linear SVM, and radial SVM were used

Eight groups of essential oils were classified using the LDA model as the first supervised method. Data from 9 metal oxide sensors were used as input to the model with the same weight. The results are shown in Fig. 5. Using the first two unique functions, the samples were classified into 8 groups with 95% variance, as shown in Fig. 5. Table 3 shows the confusion matrix and the performance parameters of the LDA methods. In 120 cases measured with the electronic nose, only 6 were incorrectly classified. So in the HAD1A drying method, one item was wrongly classified in the HAD1B class, in the HAD1B drying method, two samples were classified in the HAD2A method, and in the HAD3A drying method, three samples were classified in the HAD3B method. Based on the functional parameters obtained, the electronic nose was able to identify all samples of essential oils obtained by shade and sun drying methods with 100% accuracy. Overall, it can be noted that the average values of Accuracy, Precision, and Recall were 0.988, 0.956, and 0.950, respectively, and the average values of F and AUC parameters were 0.950 and 0.974, respectively. The high accuracy of the electronic nose in detecting shade and sun samples can be attributed to the uniqueness of their compounds. In contrast, in the methods of HAD, there was an overlap between the compounds, as only their quantity changed. Consequently, the electronic nose has a higher response than the shadow and sun methods, which can be used for a variety of products due to its high efficiency in identifying unique compounds in negligible amounts.

Two support vector machine methods, namely C and Nu, were used to classify the essential mint oil samples by drying method. Nu is always between (0,1), while C ranges from 0 to infinity. The parameters of this method, Nu, C, and γ, were validated by trial and error through minimization. The algorithm was trained with 70% of the total data and tested with 30%. It was assumed that the input data had the same weight. Four sigmoid, radial, and linear polynomial functions were also used. The results are shown in Table 6. As shown in Table 4, all models

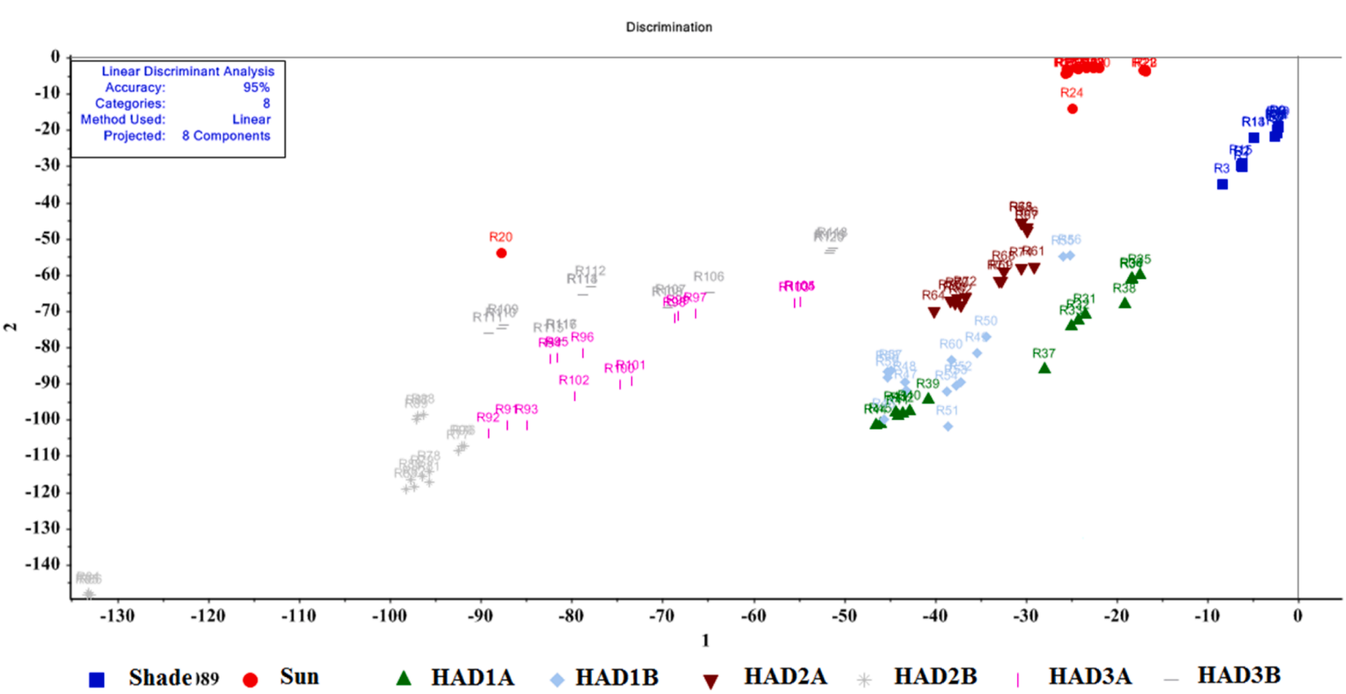


Fig. 5. Results of Linear discriminant analysis (LDA) to detect essential oil in the different drying methods.

Table 3
The confusion matrix and performance parameters obtained from LDA method to detect essential oil in the different drying methods.

Confusion matrix									Performance Parameters					
Drying Methods	Shade	Sun	HAD											
			1A	1B	2A	2B	3A	3B	Accuracy	Precision	Recall	Specificity	AUC	F
Shade	15	0	0	0	0	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
Sun	0	15	0	0	0	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD1A	0	0	14	0	0	0	0	0	0.992	1.000	0.933	1.000	1.000	0.966
HAD1B	0	0	1	13	0	0	0	0	0.975	0.929	0.867	0.990	0.960	0.897
HAD2A	0	0	0	2	15	0	0	0	0.983	0.882	1.000	0.981	0.932	0.938
HAD2B	0	0	0	0	0	15	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD3A	0	0	0	0	0	0	12	0	0.975	1.000	0.800	1.000	1.000	0.889
HAD3B	0	0	0	0	0	0	3	15	0.975	0.833	1.000	0.971	0.902	0.909
					Average per class				**0.988**	**0.956**	**0.950**	**0.993**	**0.974**	**0.950**

Table 4
Results and comparison of Nu-SVM and C-SVM models subjected to the kernel functions.

Kernel function	C-SVM				Nu-SVM			
	C	γ	Train	Validation	Nu	γ	Train	Validation
linear	100	1	96.17	91.50	0.255	0.1	96.17	91.50
Polynomial	1	100	95.83	90.00	0.255	1	94.50	92.00
Radial basis function	1	100	95.50	90.33	0.745	0.1	96.67	91.67
sigmoid	0.1	100	86.67	83.33	**0.5**	**0.1**	**97.50**	**94.17**

The bolded value in the table means the Nu-SVM model with linear kernel function showed the best performance in detecting lemon fraud.

had relatively good accuracy, but the sigmoid model of the Nu-SVM method had a training accuracy of 97.50% and a validation accuracy of 94.17% (Fig. 6). Table 5 also shows the confusion matrix and performance parameters for the sigmoid function in the Nu-SVM method, which provided the highest classification accuracy. Of the total 120 samples measured, only the data of the HAD1A group overlapped with those of the HAD1B group. Also, the average values of the network performance parameters, namely accuracy, precision, recall, and specificity were 0.994, 0.979, 0.975, and 0.996, respectively, and the AUC and F values were 0.988 and 0.975, respectively.

The last supervised method for classifying essential oils was the Artificial Neural Network (ANN) method. An ANN consists of an input layer, several hidden layers, and an output layer. In this study, 9 metal oxide sensors were used for the input layer and 8 different drying groups were used for the output layer. The hidden layer was also obtained by trial and error from the hidden layer data. The nodes in each layer are connected to the nodes in the next layer, which becomes deeper as the number of hidden layers' increases. Approximately 70% of the total data was used for training, 15% for validation, and 15% for testing. The developed models were then evaluated using R-squared (R^2) and Root Mean Square Error (RMSE). Therefore, the 9–10–8 topology was the best topology for classifying 8 groups of spearmint essential oil in different drying methods with R^2 values for training and testing of 0.986 and 0.908, respectively. In addition, the RMSE value for training and testing

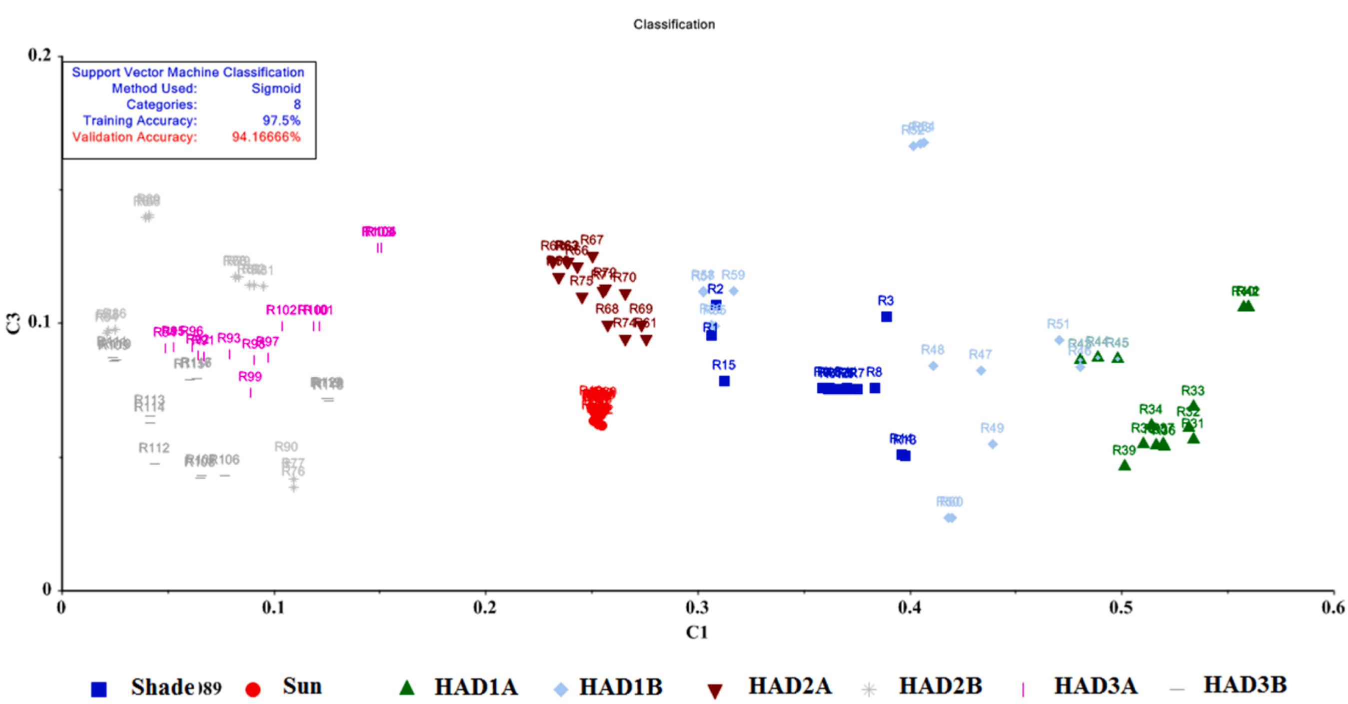


Fig. 6. Results of the Nu-SVM method with sigmoid kernel function to classification of essential oil.

Table 5
The confusion matrix and performance parameters obtained from Nu-SVM method with sigmoid kernel function to detect essential oil in the different drying methods.

Confusion matrix									Performance Parameters					
Drying Methods	Shade	Sun	HAD											
			1A	1B	2A	2B	3A	3B	Accuracy	Precision	Recall	Specificity	AUC	F
Shade	15	0	0	0	0	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
Sun	0	15	0	0	0	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD1A	0	0	12	0	0	0	0	0	0.975	1.000	0.800	1.000	1.000	0.889
HAD1B	0	0	3	15	0	0	0	0	0.975	0.833	1.000	0.971	0.902	0.909
HAD2A	0	0	0	0	15	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD2B	0	0	0	0	0	15	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD3A	0	0	0	0	0	0	15	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD3B	0	0	0	0	0	0	0	15	1.000	1.000	1.000	1.000	1.000	1.000
					Average per class				**0.994**	**0.979**	**0.975**	**0.996**	**0.988**	**0.975**

were 0.036 and 0.104, respectively. The model had an overall recognition accuracy of 96.7%. Table 6 shows the functional parameters and disturbance matrix of this network. According to this, the neural network misclassified only 4 data out of 120 measured samples. Moreover, the average values of the performance parameters of the network, namely accuracy, precision, recognition, and specificity were 0.992, 0.971, 0.967, and 0.995, respectively, and the AUC and F values were 0.983 and 0.966.

The performance of the algorithms was evaluated using functional parameters (Fig. 7). This figure shows that all models had an accuracy and specificity higher than 0.99. The Nu-SVM method with the sigmoid kernel function had the highest recall of 0.975, while the LDA method had the lowest with a value of 0.95. Overall, all models had good accuracy in classifying essential oils.

Different drying methods have been shown to affect the aroma compounds of foods and plants differently. Overall, the results showed that the amount of essential oil produced and the composition of leaves depended on the drying methods, drying times, and drying temperatures (Karami et al., 2021). Guo et al. (2022) evaluated the textural and aromatic properties of oregano leaves using an electronic nose. Based on the obtained results, the taste fingerprint and PCA showed that the aroma profiles changed significantly with drying. The results exhibited that hot air thin film drying at 35 °C improved the final quality of dried *M. haplocalyx* leaves while retaining their flavor. HS-GC/MS combined with E-Nose GC was used by Yu et al. (2022) to compare different drying methods on the volatiles of ginger. They found that GC E-Nose samples of HAD-dried ginger contained more pungent aromas than other methods. Makarichian et al. (2021) used the electronic nose to investigate how different drying methods affect garlic aroma. According to their data, the LDA and BPNN methods classified the aroma of samples based on different drying methods with 96.67% and 100% accuracy, respectively. Therefore, these two volatilization techniques combination provide not only a comprehensive aroma profile of mint essential oils under different drying methods, but also present a flavor description for drying, storage, and processing of dried mint or its products.

4. Conclusion

To preserve herbal products, drying is the most appropriate method. In the production and commercialization of medicinal plants, choosing a specific drying method is an important cost factor. This study aimed to determine the effects of different drying methods on the quality and quantity of spearmint essential oil. According to the results, the HAD1A drying method yielded the highest essential oil yield, while the sun drying method yielded the lowest. In addition, the essential oil compounds were determined by the GC/MS method, and 18 compounds were determined by the HAD drying method, some of which decreased significantly with increasing temperature. In the dried samples, the main components were carvone (64.30–7.45%), limonene (24.21–6.59%), and carveol (18.34–1.92%). In addition, E-nose was used to evaluate the aroma characteristics of the mint essential oil. A total of three classification algorithms, LDA, SVM, and ANN, were used. The Nu-SVM method achieved the highest classification rate using the sigmoid function with 0.975, while ANN and LDA were accurate with 0.967 and 0.95, respectively. The results of this study provided a theoretical basis for developing hot-air thin-layer drying processes of medicinal plants and improving their sensory quality. Overall, based on the results of the current study, future research could focus on continuously improving the technology for in situ drying medicinal plants, as well as developing a suitable monitoring system to control the sensory quality of the final products.

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CRediT authorship contribution statement

Mansour Rasekh: Formal analysis, Funding acquisition, Conceptualization, Project administration. **Hamed Karami:** Investigation,

Table 6
The confusion matrix and performance parameters obtained from ANN method with topology 9–10–8 to detect essential oil in the different drying methods.

Confusion matrix									Performance Parameters					
Drying Methods	Shade	Sun	HAD											
			1A	1B	2A	2B	3A	3B	Accuracy	Precision	Recall	Specificity	AUC	F
Shade	15	0	0	0	0	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
Sun	0	15	0	0	0	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD1A	0	0	15	1	0	0	0	0	0.992	0.938	1.000	0.990	0.964	0.968
HAD1B	0	0	0	14	0	0	0	0	0.992	1.000	0.933	1.000	1.000	0.966
HAD2A	0	0	0	0	15	0	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD2B	0	0	0	0	0	15	0	0	1.000	1.000	1.000	1.000	1.000	1.000
HAD3A	0	0	0	0	0	0	15	3	0.975	0.833	1.000	0.971	0.902	0.909
HAD3B	0	0	0	0	0	0	0	12	0.975	1.000	0.800	1.000	1.000	0.889
					Average per class				**0.992**	**0.971**	**0.967**	**0.995**	**0.983**	**0.966**

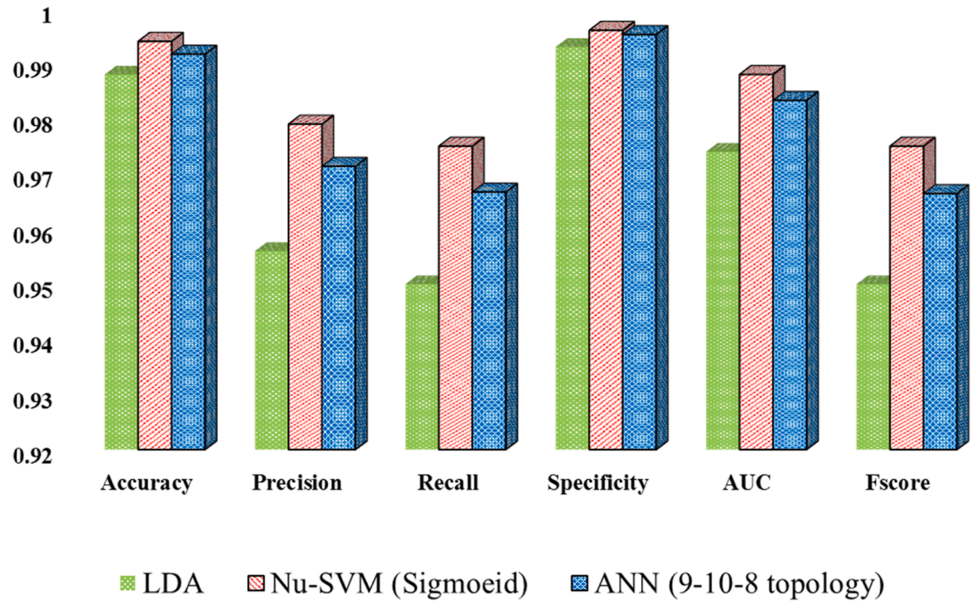

Fig. 7. Average performance parameters of the algorithms used in essential oil classification in the different drying methods.

Conceptualization, Supervision, Methodology, Formal analysis, Software, Writing – original draft, Data curation, Writing – review & editing, Project administration. **Mohammad Kamruzzaman:** Formal analysis, Supervision, Writing – review & editing. **Vahid Azizi:** Formal analysis, Data curation, Writing – review & editing. **Marek Gancarz:** Formal analysis, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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