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Volatile Compounds, Bioactive Properties and Chlorophylls Contents in Dried Spearmint (*Mentha spicata L.*) as Affected by Different Drying Methods

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ABSTRACT

This study presents a comparison of the quality characteristics of spearmint (*Mentha spicata L.*) dried by a photovoltaic thermal dryer (PVT), the shade dried spearmint (SDS), and an oven dried spearmint (ODS). The obtained samples were evaluated with respect to volatile compounds (VC), total phenolic content (TPC) and flavonoid content (TFC), antioxidant capacity (AC) and chlorophylls (Chl) contents. PDS had the highest amount of TPC, TFC and AC, while SDS and ODS did not differ significantly from each other in terms of these features. SDS exhibited the highest Chl a and Chl b contents, whereas ODS showed the lowest. The composition of VC in the dried spearmints was significantly affected by the drying methods used. The total amount of terpenoids, especially carvone, responsible for spearmint's characteristic aroma in SDS was higher than those of the others, while the concentrations of most VC were lowest in ODS. According to the results, the PVT can be recommended for drying spearmint.

Keywords: Mint drying, Photovoltaic thermal collector, Dven drying, Shade drying, Drying procedure, Quality of dried spearmints

1. Introduction

Spearmint (*Metha spicata L.*) is one of the most extensively cultivated species of the *Mentha* genus and is commonly referred to as garden mint. This aromatic and herbal spice is utilized globally in flavoring, pharmaceutical, cosmetic and fragrance applications. Additionally, they can be used in fresh or dried forms, and as essential oils (Nalawade et al. 2019). Spearmint is often used in many cuisines and the food industry mainly due to its distinct aroma. In addition, mint species are well-known for their antioxidant (Hinneburg et al. 2006) and antimicrobial (Shah & Mello 2004) characteristics. Therefore, it is estimated that the commercial significance of these plants will increase further owing to the spicing and the other diverse benefits.

Aroma is a primary factor in evaluating the quality of spices as an organoleptic characteristic. The aroma of spices primarily characterized by their contents of volatile compounds (VC) (Govindarajan & Salzer 1986). The specific aroma of spearmint is generally derived from certain terpenoid compounds (Da Porto & Decorti, 2009). The qualitative and quantitative composition of these compounds are affected by pre- and post-harvest conditions (Nalawade et al. 2019). Drying processes particularly change the profile of VC depending on temperature, humidity and time conditions (Korkmaz et al. 2020; İzli & Polat 2020; Polat et al. 2021). Nowadays, the content of bioactive substances in herbs and spices has been considered as another parameter of their quality, with a rising trend (Uribe et al. 2016). These phytochemical compounds have various beneficial effects on human physiology such as anticancer, antimicrobial, anti-diabetic, anti-inflammatory, cytotoxicity, and cytoprotective properties (Tafrihi et al. 2021). Phenolic acids and flavonoids are the primary bioactive components in mints (Lv et al. 2012). The amount of these compounds in genus *Mentha* can also differ depending on drying conditions as well as other factors including growing conditions, cultivar, harvest time (Mahendran & Rahman 2020).

Drying is one of the main processes applied to spice herbs and has critical effects on the overall acceptance of the final products. It is generally used to reduce the moisture content in the plant tissues in order to extend their shelf life. Additionally, drying treatments also facilitate the processing of spices into different forms such as powders, extracts and oils (Uribe et al. 2016). Spice herbs are dried using various methods, frequently by sun and shade drying. These traditional ways are economical, but have some disadvantages such as being time-consuming, requiring a large area, sacrificing some biochemical attributes (Mokhtarian et al. 2020), and allowing microbial (Mokhtarian et al. 2017) and other environmental (Arslan et al. 2010) contaminations. Therefore, several drying methods have been used in order to minimize the loss of quality as well as the time and energy cost of drying techniques. Herbal plants are typically dried using oven drying and hot air drying on an industrial scale (Mokhtarikhah et al. 2020). Furthermore, solar dryers are proposed as an alternative drying method for mints (Akpinar 2010; Mokhtarian et al. 2020). They are categorized as hot air convection mode (natural or forced) and energy search (direct, indirect, mixed and hybrid solar) (El-Sebaii & Shalaby 2013). The hot air from solar drying can be generated by flat plate collectors, vacuum tubes, photovoltaic (PV) and photovoltaic thermal (PVT) collectors. PVT collectors supply both thermal and electrical energy (Kovacı et al. 2020). The solar dryers designed by these collectors are recommended by several researchers to dry *Mentha* plants due to their low cost (El-Sebaii & Shalaby 2013), high efficiency (Arslan et al. 2020; Arslan & Aktaş 2020) and short drying time (Koşan et al. 2020).

The majority of published research related to dried *Mentha* genus have been primarily focused on the essential oils of these herbs (Baranauskienė et al. 2007). There are also several studies about determining some properties of dried mints using various methods. (Mokhtarian et al. 2020) found that both the total phenolic content (TPC) and chlorophylls (Chl) content of peppermint (*Mentha piperita L.*) dried using a solar collector were higher than those that were traditionally (sun and shade) dried. (Díaz-Maroto et al. 2003) demonstrated that spearmints (*Mentha spicata L.*) dried using oven (at 45 °C) and air (at ambient temperature) methods presented the best composition regarding VC compared to freeze drying. Although dried leaves of spearmint are extensively used in the food industry or in many culinary practices, the number of studies on aroma compounds and bioactive properties of this spice dried via different drying methods is limited. Moreover, to the best of our knowledge, no extensive investigations have been carried out on the VC composition and bioactive attributes of spearmint dried with a solar dryer designed with a PVT collector. Hence, the purpose of present study was to evaluate the VC composition, TPC, TFC, antioxidant capacity (AC) and Chl content of dried spearmint obtained by ODS, SDS and PDS drying methods. Additionally, the thermal and electrical efficiencies of the PVT collector were calculated.

2. Material and Methods

2.1. Plant material

Fresh spearmint (*Mentha spicata* L.) was obtained from a local market (Ankara, Turkey) and used in the experiments on the same day. A total of 300 g of fresh spearmint leaves were separated from their stems and then divided into three equal parts after being washing with water. These parts were used immediately for each drying procedure.

2.2. Standards and chemicals

Methanol, acetone, formic acid, Folin-Ciocalteau reagent, sodium carbonate, toluene, sodium nitrite, aluminum chloride, sodium hydroxide, 2,2 Diphenyl-1-picrylhydazyl (DPPH), and analytical standards of trolox, GA and catechin were purchased from Sigma Aldrich (Taufkirchen, Germany).

2.3. Photovoltaic thermal dryer (PVTD)

The PVTD used was designed as a system consisting of a PVT collector and a drying chamber (DC). The schematic view of this system is given in Figure 1.

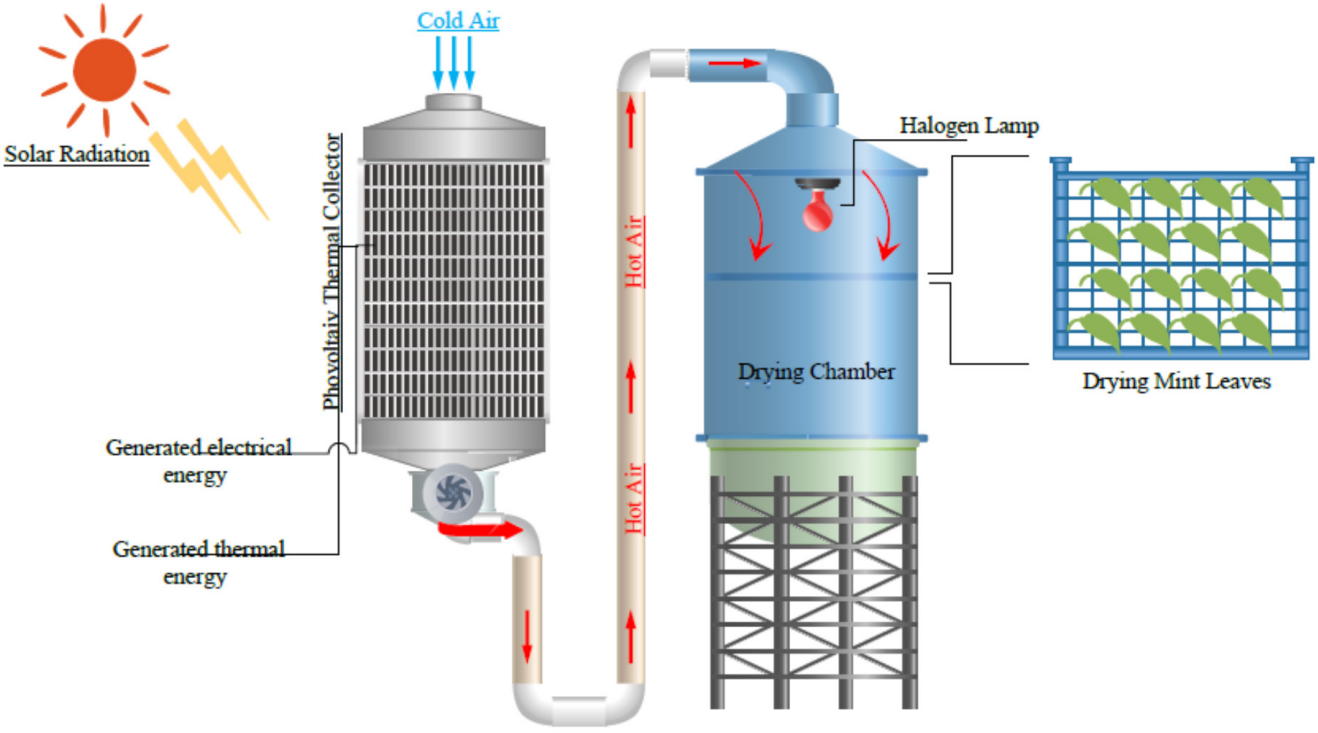

Figure 1- Schematic illustration of photovoltaic thermal collector drying system

2.3.1. Photovoltaic thermal panel

In the PVT panel design, it has been aimed to generate both heat and electricity energy from solar radiation for a sustainable solar drying system. In this way, a sustainable drying system capable of generating its own energy has been produced. The design has been created in such a way that fresh air enters from the upper part of the PVT panel and extracts the heat energy from the panel before entering the DC. Fans that pulled in fresh air were connected in series at the entrance of the PVT panel. The backside of the PV module was enclosed with an aluminum sheet, leaving 10 cm of space for the fresh air to flow homogeneously. In addition, the sides and back of the PVT panel were covered with insulation material so that no heat loss occurred. The electricity generated from the PVT panel was stored in an accumulator. It fed the system's fans and halogen lamps, which served as the auxiliary heat source in the DC, via a converter (Figure 1). Five thermocouples were located at the back of the PVT panel to observe the temperature distribution. Technical specifications of the PVT panel and other equipment used in the experimental setup are given in Table 1.

Table 1- Technical specifications of the apparatus used in the drying system

Apparatus	***Specifications***
PVT panel	275W, Vm=31.79 V, Im=8.66 A, 1640x992 mm
Charge regulator	6.9V-17.2V, 20 A, 30 Hz, -10 to+50 °C
Accumulator	Yigit battery YD12-26 12V26AH, running temperature -15 - +40 °C
Halogen lamp	50W, 12 V, AC
Fan	12 V DC - 0.32 A, -10 to +55 °C
Thermal insulation	Expanded polystyrene, 50 mm thick, 0.035 W/mK

PVT: Photovoltaic thermal, W: Watt, Vm: Maximum voltage, Im: Maximum current, V: Voltage, DC: Direct current, AC: Alternative current, mm: Millimeter, A: Amper, Hz: Hertz, mK: meter Kelvin

2.3.2. Drying chamber

The DC was constructed with 40x40x60 cm dimensions and was covered with insulation material to prevent heat loss. The heated air in the PVT panel entered from the upper part of the DC. In the DC, 50Wx4 halogen lamps, which are an auxiliary heat source, were used in order to provide sustainable drying even under conditions with no or insufficient solar radiation. Halogen lamps were

homogeneously installed on the upper part of the DC. A tray was placed 15 cm below the halogen lamps and one fan was attached to the entrance of the DC. A load cell was placed just below the tray in order to observe the amount of moisture lost by the product. Technical characteristics of the devices used in the DC are indicated in Table 2. In addition, the properties of the measurement devices used in the experimental setup are given in Table 2.

Table 2- Equipment in experimental setup and their specification's

Devices	*Brand*	*Qualification*	*Accuracy*	*Quantity (ea)*
Anemometer	Kimo, VT 200	0–20 m/s, 0.3-35 m/s, -20 to +80 °C	±0.03 m/s, ±0.1 °C	1
Data Logger	Elimko, E-680	-200 + 1200 °C	±0.5 °C	1
Load cell	Zemic, L6D, OIMLC3	Measurement capacity max. 5000 g	±0.01 g	1
Solar meter	Kimo, SL 100	0-1300 W/m^2	±5 %	1
Thermocouple	Elimko, K type NiCr-Ni	-200 + 1200 °C	±0.5 °C	9
Thermohygrometer	Kimo, HD 100	-20 to + 70 °C, 5-95 % RH	±1.8 %RH, ±0.3 °C	1

RH: Relative humidity, W: Watt, m: Meter, s: Second, g: Gram

2.4. Drying procedures

The drying experiments were performed in Ankara (39.93° N latitude and 32.86° E longitude). Using each of the three drying methods, the leaves were dried until their moisture content was below 10% (w/w) in accordance with the Turkish commercial dried spices standard. This level was achieved when the difference between two consecutive weight measurements was less than 1%. The initial and the final moisture contents of the leaves were measured using the AOAC method (1990) (method number 934.06) (Williams 1984). The moisture content in fresh leaves was 82.50% (w/w) based on wet weight. Following the drying treatments, the leaves were ground to a 200-500 µm particle size with a mill and stored in screw-capped vials at -18 °C until analysis. Each drying was performed in triplicate.

2.4.1. Oven drying

The fresh spearmint leaves were thinly spread onto a stainless-steel wire tray (38x38 cm) and dried in an oven (Memmert UN110, Schwabach, Germany) at 65 °C for 8 h.

2.4.2. Shade drying

The shade drying was carried out in the shade at room temperature. Fresh spearmint leaves were thinly spread onto a stainless-steel wire tray (38x38 cm) and left in a well-ventilated room. The leaves were turned twice to ensure a uniform drying. The drying process was complete after approximately 36 hours. The average temperatures of the room in the day and night were 22±2 and 18±2 °C, respectively, during drying. The relative humidity of the room during drying was 41±3%.

2.4.3. Photovoltaic thermal drying

Fresh spearmint leaves were evenly distributed on a stainless-steel wire tray (38x38 cm) and placed in the DC. Thereafter, all PVT experiments were initiated at 10 am and the drying process began. The experiments continued until the drying was completed (5.5 h). The DC temperature was set to 35° with the assistance of a thermostat. When the temperature dropped below 35°, halogen lamps started to operate automatically. After the drying process started, the surplus energy generated from the PVT panel continued to be stored in the accumulator. In the absence of sufficient solar radiation, the electrical energy required for the DC was provided by the accumulator. The schematic view of the experiment set is given in Figure 1. Accordingly, the ambient air enters the PVT collector from the upper side with the help of the fan and exits as hot air from the lower side. The hot air is allowed to enter the drying room, and its exhaust is delivered to the exterior as hot and humid air.

2.5. Extraction procedure of phenolics

The extraction of phenolic was carried out according to the procedure described by Capanoglu et al. (2013) with some modifications. Two hundred mg of sample was extracted with 3 mL of 75% methanol (containing 1% formic acid) using a homogenizer (Ultra-Turrax T25 Basic, IKA, Staufen, Germany) at 15000 rpm for one minute. The extract was then sonicated for 15 min at the room temperature and centrifuged (Universal 320 Hettich, Westphalia, Germany) at 1200 g for 10 min at 10 °C. The supernatant was collected and the

residue was extracted twice more. All supernatants were combined in a tube and the final volume was adjusted to 10 mL with the acidified methanol. This methanolic extract was used for TPC, TFC and AC analyses of dried spearmint samples.

2.6. Total phenolic and total flavonoid content analysis

The TPC was determined by Folin-Ciocalteau method (Singleton & Rossi 1965). Briefly, 100µL of methanolic extract was mixed with 900 µL of distilled water and 5 mL of Folin-Ciocalteu reagent (0.2 mol/L). This mixture was shaken vigorously, and left to rest for 8 min. Thereafter, 5 mL of 7.5% Na_2CO_3 solution was added, and vortexed for 20 s. This solution was left in the dark for 2 h at room temperature, and the absorbance was measured at 765 nm with a spectrophotometer (Biochrom Libra S70 Dual; Harvard Bioscience Co. Shanghai, China). TPC was calculated in terms of mg gallic acid equivalent (GAE) per g dry weight (DW) using a standard curve prepared by GA (0.0625-1 mg mL^{-1}).

TFC was determined (Uribe et al. 2016) with some modifications. 0.4 mL of the methanolic extract was added to a mixture of 4 mL distilled water and 0.3 mL of 5% $NaNO_2$. After 5 min, 0.3 mL of 10% $AlCl_3$ solution was added, and 6 min later, 2 mL of NaOH (1 mol/L) was added. The resulting mixture was stirred and the absorbance was recorded at 510 nm with a ultraviolet-visible (UV-VIS) spectrophotometer (Biochrom Libra S70 Dual; Harvard Bioscience Co., Shanghai, China). The calibration curve was plotted using quercetin (0,03125-1 mg mL^{-1}) and TFC was expressed in terms of mg quercetin equivalent per g DW.

2.7. Antioxidant capacity analysis

AC was estimated by the DPPH radical scavenging method using the protocol of Lingua et al. (2016) with minor modifications. One hundred µL of the methanolic extract was added to 3.9 mL of 60 µM DPPH solution in methanol and stirred (Lingua et al. 2016). The mixture was left in the dark for 30 min. The absorbance was then measured at 517 nm against methanol using a UV-VIS spectrophotometer (Biochrom Libra S70 Dual; Harvard Bioscience Co., Shanghai, China). The DPPH solution without the sample was used as a control. The DPPH scavenging capacity was calculated in terms of mg Trolox equivalent (TE) per g DW.

2.8. Chlorophylls content analysis

The Chl content was determined according to (Lichtenthaler 1987), with a slight modification. Briefly, 100 mg of sample was extracted via a homogenizer (Ultra-Turrax T25 Basic, IKA, Staufen, Germany) at 16500 rpm for two minutes with 10 mL of 100% acetone. The homogenate was centrifuged (Universal 320 Hettich, Westphalia, Germany) at 400 g for 5 min at room temperature. The supernatant was transferred into a tube and the extraction was repeated twice more to yield a colorless residue. Then, all supernatants were collected, filtered by Whatman filter paper, and the final volume was adjusted to 30 mL by adding 100% acetone. The absorbance of extract was measured by a UV-VIS spectrophotometer (Biochrom Libra S70 Dual; Harvard Bioscience Co. Shanghai, China) at wavelengths 645 and 663 nm. CC was calculated by the following equations [(1), (2)] and expressed in terms of mg per g DW.

$$\text{Chlorophyll a (Chl a)} = 12.25 \times A_{663} - 2.79 \times A_{645} \quad (1)$$

$$\text{Chlorophyll b (Chl b)} = 21.50 \times A_{645} - 5.10 \times A_{663} \quad (2)$$

2.9. Volatile compound analysis

VC in samples were analyzed by solid-phase microextraction (SPME) followed by a GC-MS system (Shimadzu QP2020 GC-MS; Shimadzu Corp., Kyoto, Japan), according to Korkmaz et al. (2020) with some modification. 500 mg of sample were placed in a 20 mL SPME screw cap vial (Supelco, Bellefonte, PA, USA). 10 µL toluene (84 mg L^{-1}) was added as internal standard (IS) and the vial was immediately closed with a polytetrafluoroethylene/silicon septum (Supelco). Following this stage, all SPME operations were conducted by an auto sampler (AOC 5000 Plus; AOC 5000 Plus, CTC, Switzerland) coupled with the GC-MS. The vial was kept at 40 °C for 30 min. with an agitating speed of 220 rpm, and then a 2 cm SPME fiber (Supelco, Bellefonte, PA) coated with DVB/CAR/PDMS (50/30 µm) injected into the vial. The fiber was exposed to the headspace of the vial for 30 min at the same temperature and stirring speed. Thereafter, the fiber was withdrawn and transferred to the injection port of GC-MS for desorption at 250 °C for 5 min in splitless run. Each sample was analyzed in triplicate.

VC were separated on a DB-Heavy Wax column (60 m x 0.25 mm, 0.25 µm; Agilent J&W Scientific, Folsom, CA, USA). The oven temperature program was set to 40 °C for 3 min, increased to 80 °C at 3 °C/min (held for 1 min), and then raised to 240 °C at 5 °C/min (held for 6 min). Helium was used as the carrier gas with a flow of 1.07 mL/min. Mass spectrometry was applied by a 201°C ion source temperature with a scanning range of m/z 20-450 and 70 eV electron ionization.

Identification was carried out by comparing mass spectra with the standard Wiley 9 mass spectral library and retention indices (RI) with the National Institute of Standards and Technology standard reference database. The RIs were calculated by using a series of n-alkanes (C8-C26) (Sigma-Aldrich, USA) under the same conditions in the GC-MS. VCs were semi-quantified by multiplying the concentration of IS by the ratio of the peak area of each VC to that of the IS (μg kg^{-1}).

2.10. Statistical analysis

All experiments were performed in triplicate and the data were expressed in terms of means ± standard deviation. The differences between the means were tested via One-Way analysis of variance (ANOVA) followed by Duncan's multi comparison test ($p<0.05$). In addition, the data of VC were tested by principal component analysis (PCA) to visualize the relationships between samples and VC. All statistical analyses were performed using the SPSS software package (version 16.0, IBM Inc., Chicago, IL, U.S.A.).

3. Results and Discussion

3.1. Total phenolic and total flavonoid content (TFC)

The TPC and TFC in spearmint samples are given in Figure 2. The TPC in ODS, SDS and PDS were 136.88±1.95, 139.85±1.37 and 166.79±2.05 mg GAE g^{-1}, respectively (Figure 2a). The level of TFC in ODS, SDS and PDS were 75.50±1.81, 78.24±1.54 and 101.76±2.81 mg CE g^{-1} DW (Figure 2b). As can be seen, the level of both TPC and TFC in the spearmint dried by the PVT dryer were higher than that in shade and oven dried samples ($p<0.5$). However, the difference between both TPC and TFC in ODS and SDS was not significant ($p>0.05$), most likely due to the relationship between their drying temperatures and drying times. The amount of bioactive compounds in mint leaves can decrease as a result of both longer drying time and higher drying temperature (Mokhtarian et al. 2020) The lower TPC and TFC in SDS compared to those in PDS may be caused by the longer drying time (5.55-fold) for SDS (Samoticha et al. 2016) vacuum (VD, while the lower levels of these contens in ODS could be explained by the higher drying temperature (Arslan et al. 2010). Phenolic compounds in plants may degrade because of thermal and enzymatic oxidation during drying processes. Mokhtarian et al. (2020) reported a higher (by 25%) TPC in peppermint dried by a solar dryer than that of shade dried, supporting the relationship between drying time and temperature with TPC (Mokhtarian et al. 2020).

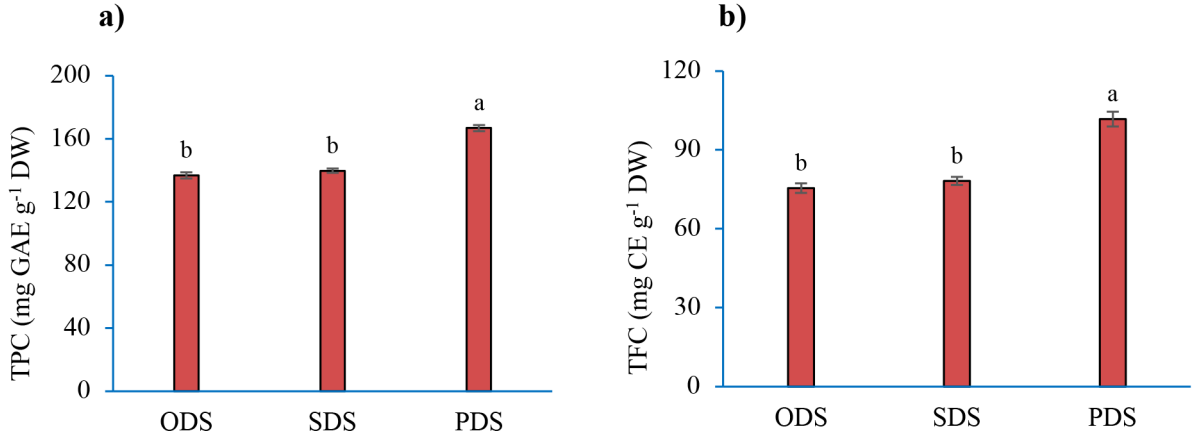

Figure 2- Total phenol (a) and total flavonoid (b) content in ODS, SDS and PDS
ODS: Oven dried spearmint, SDS: Shade dried spearmint, PDS: Photovoltaic dried spearmint, a-b Different lowercase letters were significantly different among samples ($p<0.05$)

TPC and TFC in dried *Mentha* species vary depending on breeding conditions (Lv et al. 2012), extraction methods (Koşar et al. 2005; Jeong et al. 2018; Mahendran & Rahman 2020), drying methods (Arslan et al. 2010; Uribe et al. 2016) and variety (Hinneburg et al. 2006). Uribe et al. (2016) demonstrated that TPC and TFC in a cultivar of peppermint (*Mentha piperita*) dried at different temperatures (50-90 °C) ranged between 11.56-27.12 mg GAE g^{-1} DW and 29.24-53.17 mg CE g^{-1} DW, respectively (Uribe et al. 2016). Cirlini et al. (2016) reported a TPC of 262.97 mg g^{-1} in a dry spearmint (*Mentha spicata*) (Cirlini et al. 2016). Another study reported that TPC and TFC in dried peppermint leaves accounted for 19-23% and 12% of its total weight, respectively (Jeong et al. 2018), as a similar result was observed for ODS, SDS and PDS. In a recent study (Said et al. 2022), it is reported that the TPC in leaves of a *Pelargonium graveolens* dried by shade was higher than that dried in an oven at 60 °C, similarly observed for SDS and ODS.

3.2. Antioxidant capacity

The AC values of the samples are illustrated in Figure 3. PDS exhibited the highest (133.56±2.95 mg TE g^{-1} DW) AC ($p<0.05$), while AC of ODS (93.31±2.04 mg TE g^{-1} DW) and SDS (92.72±2.80 mg TE g^{-1} DW) were not statistically different from each other ($p>0.05$). Similarly, Mokhtarian et al. (2020) found that the AC of peppermint dried in a solar dryer had 26% higher AC compared to peppermint dried by shade (Mokhtarian et al. 2020) Jeong et al. (2018) reported that the DPPH scavenging activity of dried peppermints obtained from different origins were ranged from 63.10 to 93.50 mg TE g^{-1} (Jeong et al. 2018). Lv et al. (2012) found the DPPH scavenging capacity as 233.57 and 394.17 mg TE g^{-1} for conventional and organic dry peppermint, respectively (Lv et al. 2012).

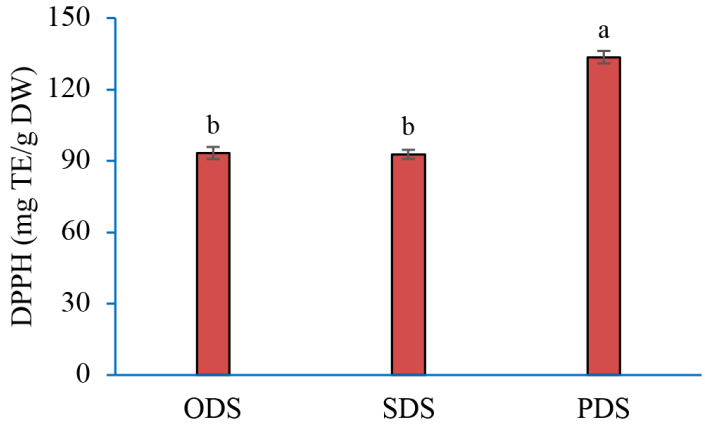

Figure 3- Antioxidant capacity of ODS, SDS and PDS
ODS: Oven dried spearmint, SDS: Shade dried spearmint, PDS: Photovoltaic dried spearmint
a,b Different lowercase letters were significantly different among samples ($p<0.05$)

It was clearly seen that the relationship between the AC values of the dried spearmints exhibited similarity with that of their TPC and TFC values. Polyphenolic compounds are one of the main components responsible for the antioxidant activities of *Mentha* plants (Riachi & De Maria 2015). In fact, there was a high correlation between the AC and both the TPC (Pearson's coefficient=990, $p<0.01$) and TFC (Pearson's coefficient=0.992, $p<0.01$). Uribe et al. (2016) also reported positive correlations between DPPH radical-scavenging capacity and the TPC (r^2= 0.97, $p<0.05$) and TFC (r^2= 0.62, $p<0.05$) of peppermint (Uribe et al. 2016). Moreover, Jeong et al. (2018) also found high correlations for both TPC (Pearson's correlation=0.785, $p<0.5$) and TFC (Pearson's correlation=0.745, $p<0.5$) in a dried peppermint genotype, although these values were lower than those observed for ODS, SDS and PDS.

3.3. Chlorophylls contents

The Chl contents in samples are depicted in Figure 4. The amount of Chl *a*, Chl *b* and the total chlorophyll (Chl *a* + Chl *b*) were significantly affected by the drying method ($p<0.05$). The PDS had the highest level of both Chl *a* (5.94±0.00 mg g^{-1} DW) and Chl *b* (3.93±0.01 mg g^{-1} DW), whereas the ODS had the lowest amount in both Chl *a* (5.01±0.01 mg g^{-1} DW) and Chl *b* (3.52±0.01 mg g^{-1} DW). The lower Chl contents in the oven dried spearmint can be explained by the higher drying temperature, causing more losses in these pigments (Uribe et al. 2016). A similar result was also observed by Yilmaz and Alibas (2022) for rosemary leaves. They found that the total Chl content in the shade-dried rosemary leaves was higher than that in the convective dried at 50 °C. In contrast, Mokhtarian et al. (2020) found that the total Chl content in peppermint dried by a solar dryer was higher than in shade dried peppermints (Mokhtarian et al. 2020) They attributed this difference to shorter drying time (2.5 h) when using solar drying compared to shade (5 h) drying.

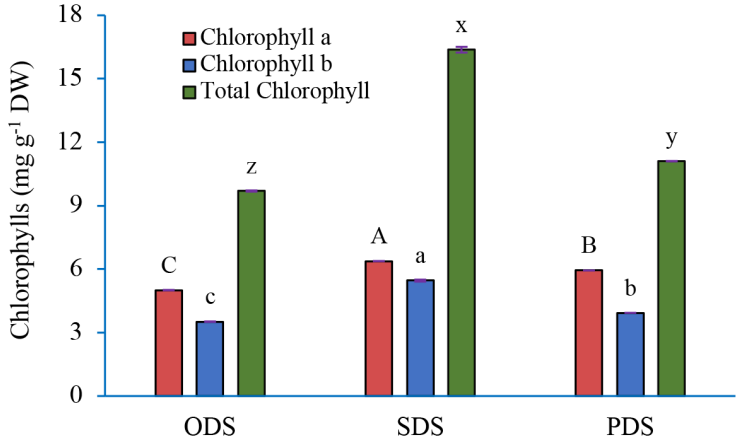

Figure 4- Chlorophylls content in ODS, SDS and PDS
ODS: Oven dried spearmint, SDS: Shade dried spearmint, PDS: Photovoltaic dried spearmint,
A-C, a-c, x-z: Different lowercase, uppercase, and Latin letters were significantly different among samples for Chl a, Chl b, and total chlorophylls contents, respectively ($p<0.05$)

The total Chl amount in dried leaves of *Mentha* types differs particularly depending on variety and the employed drying methods. For instance, Kannan et al. (2021) reported this amount in dried *Mentha arvensis* to range between 88.73-96.80 mg g^{-1} a higher range than the levels in ODS, SDS and PDS. However, Uribe et al. (2016) determined a range of 0.76-0.89 mg g^{-1} DW for dried *Mentha piperita* by vacuum drying at different temperatures (50-90 °C), a lower range than the amount in the three sample types. Another study (Nalawade et al. 2019) reported the total Chl content in a cultivar of fresh spearmint (*Mentha spicata*) leaves to be 4.09 mg g^{-1} DW and that the percentages of retention of this content in samples following different drying methods ranged between 38.97-61.12%.

3.4. Volatile compounds

The identified VC and their relative quantity in the samples are listed in Table 3. In total, 87 VC in the three spearmints were identified and grouped as monoterpenoids (Kannan et al. 2021), sesquiterpenoids (16), triterpenoid (1), aldehydes (9), alcohols (8), ketones (2), esters (9) and miscellaneous (6). The lowest total VC content was obtained from ODS (3056.48±253.45 µg kg^{-1} DW) ($p<0.05$). However, there was no statistically significant difference between the total VC content in SDS (5320.14±299.06 µg kg^{-1} DW) and in PDS (4760.46±240.67 µg kg^{-1} DW) ($p>0.05$).

Terpenoids were observed to be the most abundant group in the samples, both in terms of number (53) and quantity. The total amount of this group was found as 82.59%, 87.76% and 86.45% of the total VC concentration in the ODS, SDS and PDS, respectively. The most of the individual contents of terpenoids were the lowest in ODS ($p<0.05$). This could be due to its higher drying temperature that causes a greater decrease in monoterpenes by oxidation or evaporation (Chua et al. 2019). There were no significant differences between the contents of many compounds of terpenoids in SDS and in PDS ($p>0.05$). In general, the profile of terpenoids (oxigenated monoterpenes, monoterpenes and sesquiterpenes) in the samples of spearmints was similar to that found in previous studies on the common *Mentha* species (Silva & Câmara 2013; Verma et al. 2011).

Carvone, an oxygenated monoterpene, constituted the majority of the total amount of VC in samples, accounting for 47.68%, 45.04% and 39.24% of the total VC content in ODS, SDS and PDS, respectively. SDS contained the highest level of carvone (2.396,17±140.04 µg kg^{-1} DW) ($p<0.05$). This compound is the most responsible for the typical aroma (minty) of the cultivars of spearmint. Carvone has also been identified as the main VC in dried forms and essential oils (Díaz-Maroto et al. 2003) of spearmints (Mokhtarikhah et al. 2020; Nalawade et al. 2019). Conversely, Cirlini et al. (2016) reported that the amount of carvone in an extract of a dry spearmint was lower than that of several other VC. Additionally, Silva and Câmara (2013) have not found carvone in fresh spearmint leaves but did find it in peppermint leaves as the major VC. The other predominant compounds in samples were D-limonene (citrus) and eucalyptol (1,8-cineole) as found by Díaz-Maroto et al. (2003), Da Porto and Decorti (2009). D-Germacrene and (E)-β-bourbonene (woody) were the predominant sesquiterpenes (Da Porto & Decorti 2009; Díaz-Maroto et al. 2003). These compounds also had the highest content of SDS.

In addition to the aforementioned differences in concentrations, the samples varied based on the presence or absence of some terpenoids. For example, monoterpenes such as (D)-α-pinene (minty), menth-2-en-1-ol (herbal) and 4-terpineol (cooling-mentholic) were present only in PDS, while cis-dihydrocarvone (minty), camphol (woody-camphor) and hedycaryol were detected only in SDS.

The majority of aldehydes and alchols detected in samples were lipid derivatives such as (E)-2-hexenal, octanal, nonanal, 1-hexanol, 3-hexenol, 3-octanol, 1-octanol, 3-nonanol and 1-octanol. These compounds are produced from certain polyunsaturated fatty acides by enzymatic pathway (Silva & Câmara 2013). Overall, the content of these derivatives in both SDS and PDS were greater than in ODS ($p<0.05$), most likely due to their lower drying temepratures which were in a suitable range for enzymatic activities. The presence of fatty acides products enhanced the 'green' and 'fresh-herbal' aroma in many spices.

PDS contained a greater amount of esters than the two other samples ($p<0.05$). Carvyl acetate (minty) was detected as the major ester in all samples, but its conents in samples were close to one another ($p>0.05$). Esters of acetic acid such as dihydrocarvyl acetat (floral), 1-ethylhexyl acetate (green) and 1-pentylallyl acetate (green) were found only in PDS, while 2-methylbutyl isovalerate (fruity) was present only in SDS. Miscellaneous volatiles including anethol (licorice), dimethyl sulfide (cabbage-like), heneicosane, (E,Z)-1,3,5-undecatriene showed their highest content in SDS ($p<0.05$). As in the terpenoids, the highest VC in all others groups identified in the samples have also been reported by previous studies on different Mentha species (Chen et al. 2011; Cordero et al. 2012).

Table 3- Volatile compounds (µg kg^{-1} dry weight) in ODS, SDS and PDS

	Compound	*RI*[*]	*Samples*		
			ODS	*SDS*	*PDS*
	Monoterpenoids		2524.49±223.86^{b}	4509.72±250.84^{a}	4115.07±192.44^{a}
1	α-Pinene	1061	20.63±1.03^{b}	54.75±3.97^{a}	55.69±2.07^{a}
2	α-Thujene	1067	1.68±0.10^{b}	4.3±0.22^{a}	4.34±0.34^{a}
3	Camphene	1099	nd	1.07±0.66^{a}	nd
4	2-β-Pinene	1134	35.71±2.00^{c}	77.7±3.62^{b}	95.43±4.57^{a}
5	Sabinene	1147	28.81±1.14^{b}	70.33±4.34^{a}	76.96±8.89^{a}
6	β-Pinene	1188	48.03±7.43^{b}	117.28±5.95^{a}	122.43±3.56^{a}
7	1,8-p-Menthadiene	1191	1.8±0.23^{a}	nd	nd
8	α-Terpinene	1201	0.85±0.05^{b}	2.2±0.06^{b}	6.07±0.90^{a}
9	D-Limonene	1224	415.27±30.98^{b}	900.2±44.91^{a}	934.43±62.45^{a}
10	Eucalyptol	1230	158.48±42.6^{b}	354.44±13.43^{a}	410.63±8.69^{a}
11	1,5,8-p-menthatriene	1237	3.7±0.42^{b}	10.93±1.32^{a}	10.28±1.28^{a}
12	(E)-Ocimene	1255	45.61±4.57^{b}	102.95±9.14^{a}	nd
13	(D)-α-Pinene	1256	nd	nd	106.41±6.16^{a}
14	γ-Terpinen	1264	2.7±0.35^{b}	5.35±0.28^{a}	5.28±0.18^{a}
15	(Z)- β-Ocimene	1271	13.59±0.97^{c}	31.06±1.2^{b}	40.87±2.79^{a}
16	α-Terpinolene	1298	4.28±0.48^{c}	10.49±2.2^{b}	12.93±1.86^{a}
17	α-Pinene epoxide	1372	9.52±1.00^{b}	20.86±3.1^{a}	26.44±0.89^{a}
18	(Z)-Alloocimene	1384	1.73±0.37^{b}	4.78±0.95^{a}	5.08±1.19^{a}
19	Cosmene	1445	nd	6.78±0.69^{a}	nd
20	p-Cymenene	1450	nd	6.8±0.80^{a}	5.94±1.02^{a}
21	Limonene epoxide	1461	7.09±0.28^{a}	13.11±1.05^{a}	12.44±2.39^{a}
22	(E)-Sabinene hydrate	1473	142.06±6.56^{a}	125.11±4.88^{a}	71.1±2.15^{b}
23	β-Linalool	1552	14.62±1.00^{b}	22.57±2.15^{a}	16.22±0.91^{ab}
24	Menth-2-en-1-ol	1555	nd	nd	7.14±0.39^{a}
25	4-Terpineol	1613	nd	nd	11.83±1.04^{a}
26	cis-Dihydrocarvone	1626	nd	40.48±2.97^{a}	nd
27	trans-Dihydrocarvone	1627	18.37±3.37^{b}	nd	57.01±5.44^{a}
28	(E)-p-2,8-Menthadien-1-ol	1636	1.76±0.14^{b}	3.11±0.11^{a}	2.76±0.24^{a}

Table 3- Continued

	Compound	*RI*[*]	*Samples*		
			ODS	*SDS*	*PDS*
29	Pulegone	1666	33.01±2.98[b]	50.11±4.9[ab]	67.48±4.78[a]
30	α-Terpineol	1709	22.81±0.55[b]	33.58±2.07[a]	39.29±1.04[a]
31	Camphol	1714	nd	6.63±0.64[a]	nd
32	Isopinocarveol	1724	nd	3.95±0.47[a]	nd
33	Carvone	1767	1457.36±114.95[b]	2396.17±140.04[a]	1868.44±74.8[b]
34	6-Hydroxycarvone	1820	3.76±0.23[a]	5.43±0.42[a]	5.03±0.51[a]
35	Carveol	1873	23.5±2.2[b]	23.75±0.9[b]	30.11±1.11[a]
36	Piperitenone	1947	2.75±0.13[b]	3.55±0.41[ab]	4.14±0.14[a]
	Sesquiterpenoids		345.19±15.58[b]	519.37±31.78[a]	370.63±21.79[b]
37	α-Copaene	1507	6.33±0.34[b]	10.67±0.91[a]	10.43±0.42[a]
38	(E)-β-Bourbonene	1537	93.17±3.72[b]	131.41±10.15[a]	100.67±6.58[ab]
39	α-Gurjunene	1548	1.77±0.24[b]	3.46±0.14[a]	2.46±0.06[b]
40	β-Ylangene	1591	16.85±2.10[b]	25.68±1.58[a]	nd
41	β-Elemene	1609	2.96±0.17[b]	nd	12.48±1.51[a]
42	Caryophyllene	1619	20.22±1.62[b]	34.85±5.65[a]	15.93±0.81[b]
43	β-Gurjunene	1657	2.46±0.36[a]	nd	nd
44	Alloaromadendrene	1671	2.38±0.21[b]	3.85±0.42[a]	nd
45	Hedycaryol	1680	nd	36.97±3.98[a]	nd
46	Elemol	1681	27.1±2.91[a]	nd	39.26±3.94[a]
47	γ-Muurolene	1691	4.7±0.69[b]	9.72±0.60[a]	nd
48	Bicyclosesquiphellandrene	1699	26.5±2.07[b]	44.52±3.27[a]	28.08±3.03[b]
49	D-Germacrene	1743	127.09±5.38[b]	190.85±8.01[a]	144.28±3.86[b]
50	β-Cadinene	1777	nd	6.98±1.43[a]	nd
51	Calamenene	1855	13.67±2.37[b]	19.40±0.80[a]	14.06±1.06[b]
52	Viridiflorol	2102	nd	nd	2.986±0.55[a]
	Triterpenoids		16.17±2.32[a]	nd	5.25±0.49[b]
53	Squalene	2148	16.17±2.32[a]	nd	5.25±.49[b]
	Aldehydes		38.52±4.84[b]	77.64±4.18[a]	49.40±2.59[b]
54	Acetaldehyde	710	1.97±0.07[b]	3.134±0.02[a]	1.28±0.08[c]
55	2-Methylpropanal	898	2.24±0.10[b]	5.31±0.02[a]	1.84±0.09[b]
56	2-Methylbutanal	930	3.99±0.10[b]	7.23±0.22[a]	2.35±0.25[c]
57	3-Methylbutanal	945	4.77±1.06[b]	8.46±0.42[a]	3.21±0.20[b]
58	(E)-2-Hexenal	1241	10.61±1.48[c]	32.44±2.11[a]	18.14±1.19[b]
59	Octanal	1305	1.85±0.08[c]	2.53±0.03[b]	5.50±0.05[a]
60	Nonanal	1406	1.39±0.31[b]	nd	4.53±0.43[a]
61	Phenylmethanal	1541	3.17±0.62[b]	5.69±0.05[a]	2.13±0.05[b]
62	Perilla aldehyde	1810	8.351±1.11[b]	12.85±1.15[a]	10.44±0.45[ab]
	Alcohols		58.62±1.72[b]	91.87±5.14[ab]	109.46±13.32[a]
63	1-Penten-3-ol	1184	0.63±0.03[a]	nd	nd
64	3-methylbutanol	1226	nd	2.52±0.07[a]	nd
65	1-Hexanol	1362	0.95±0.05[b]	2.41±0.15[a]	2.26±0.16[a]
66	3-Hexenol	1392	1.91±0.03[b]	2.99±0.18[a]	nd
67	3-Octanol	1399	37.14±1.09[b]	48.25±4.65[b]	80.10±10.92[a]

Table 3- Continued

	Compound	*RI*[*]	*Samples*		
			ODS	*SDS*	*PDS*
68	3-Octenol	1457	13.55±0.41[c]	28.30±0.31[a]	19.56±1.70[b]
69	3-Nonanol	1496	0.96±0.05[c]	2.28±0.13[b]	3.46±0.33[a]
70	1-Octanol	1560	3.48±0.15[b]	5.17±0.13[a]	4.09±0.21[b]
	Ketones		3.26±0.74[b]	5.51±0.45[a]	5.62±0.47[ab]
71	3-Octanone	1273	nd	1.00±0.03[b]	1.99±0.09[a]
72	Jasmone	1962	3.26±0.74[b]	6.50±0.45[a]	3.63±0.37[b]
	Esters		40.78±2.08[b]	40.06±3.10[b]	68.34±7.28[a]
73	2-Methylbutyl 2-methylbutyrate	1295	0.89±0.10[b]	nd	2.14±0.03[a]
74	Isopentyl isovalerate	1296	nd	nd	2.74±0.46[a]
75	2-Methylbutyl isovalerate	1313	nd	1.55±0.41[a]	nd
76	1-Ethylhexyl acetate	1352	nd	nd	10.43±1.57[a]
77	3-Octanyl acetate	1352	4.31±0.08[b]	7.24±0.65[a]	nd
78	1-Pentylallyl acetate	1392	nd	nd	3.77±0.08[a]
79	(Z)-3-Hexenyl valerate	1498	3.31±0.16[b]	nd	7.89±0.75[a]
80	Dihydrocarvyl acetate	1692	nd	nd	11.11±1.45[a]
81	Carvyl acetate	1786	32.27±1.96[a]	31.27±2.05[a]	30.25±3.05[a]
	Miscellaneous		29.47±2.30[b]	74.97±3.57[a]	36.70±2.26[b]
82	Dimethyl sulfide	765	5.03±0.19[b]	10.04±1.01[a]	3.43±0.05[b]
83	Heneicosane	1260	nd	10.63±0.83[a]	5.67±0.64[b]
84	Phytane	1308	0.99±0.08[b]	1.87±0.09[a]	nd
85	(E, Z)-1,3,5-Undecatriene	1403	7.93±0.51[b]	14.46±1.06[a]	10.59±0.36[b]
86	Estragole	1685	nd	3.71±1.20[a]	nd
87	Anethol	1843	15.52±1.97[b]	34.66±3.20[a]	17.00±1.20[b]
	Total		3056.48±253.45[b]	5320.14±299.0[6]a	4760.46±240.6[7]a

[*]Calculated retention indices on DB-HeavyWax column. ODS: Oven dried spearmint, SDS: Shade dried spearmint, PDS: Photovoltaic dried spearmint, nd: not detected, [a-c]Different lowercase letters in the same row indicate significant difference among samples

3.5. Principal component analysis

The differences and similarities between samples were also evaluated by PCA regarding their VC contents (Figure 5.). 97.34% of the total variance in the data set listed in Table 1 can be explained by the two first principal components. The first component (PC1) accounted for 51.77% of the total variance, while the second component (PC2) accounted for 45.57%. As depicted in Figure 5a, ODS was characterized by 1,8-p-menthadiene, β-gurjunene, squalene and 1-penten-3-ol, while PDS was primarily characterized by (D)-α-pinene, viridiflorol, (Z)- β-ocimene, trans-dihydrocarvone, carveol, 4-terpineol, 3-octanol, 3-nonanol, octanal, 2-methylbutyl 2-methylbutyrate. On the other hand, SDS was associated with greater diversity in VC, some of which were carvone, D-germacrene, cis-dihydrocarvone, cosmene, caryophyllene, 2-methylbutanal, 3-methylbutanal 3-octenol, (E)-ocimene, α-gurjunene, phenylmethanal, dimethyl sulfide and camphol. As shown in the score plot (Figure 5b), SDS was separated from the other samples by PC1, whereas PDS was separated from ODS by PC2. PCA revealed an unequivocal discrimination between the profiles of VC in the spearmints obtained using the different drying methods.

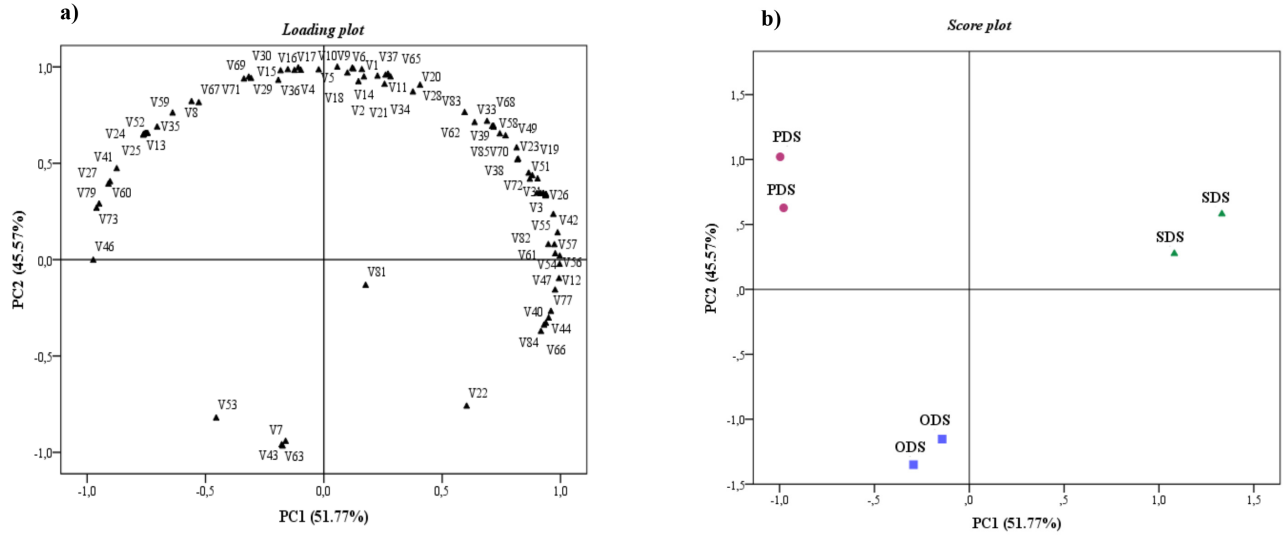

Figure 5- Loading plot (a) and score plot (b) in rotated space of the two first components of PCA for ODS, SDS and PDS samples. V: Volatile compounds codes were numbered from 1 to 87 as given in Table 3; ODS: Oven dried spearmint, SDS: Shade dried spearmint, PDS: Photovoltaic dried spearmint

4. Conclusions

In this study, a new type of dryer was developed. The mints dried in the developed dryer were compared with the products dried by the shade and oven drying methods. The products were compared in terms of consumed energy and food quality. Due to the usage of PVTD in the experiments conducted in outdoor weather conditions, the drying time was reduced by a factor of 6 compared to shade drying. The electrical (halogen lamp and fan) and thermal (heating of the drying air) energy required for the system were obtained from PVT. The sustainability of the system is ensured by storing excess energy.

The spearmint dried by PVTD had the highest TPC, TFC and AC, whereas the differences between these features of the shade-dried and oven-dried spearmints were not significant. Additionally, it was determined that the Chl content as a color agent was the highest in the shade-dried spearmint.

The VC compositions of all three samples were determined to be different from each other. The results show that the concentration of VCs representing the characteristic aroma in the shade-dried spearmint were greater compared to those in others. It was also found that the amounts of these typical VCs in the spearmint dried by shade were the lowest.

This study showed the PVTD can preserve the spice quality of spearmint.

Future studies should focus on the properties of other foods dried by PVTD in a variety of climatic conditions.

Data availability: Data are available on request due to privacy or other restrictions.

Authorship Contributions: Concept: E.A., M.K., Design: E.A., Data Collection or Processing: E.A., Analysis or Interpretation: A.K., Literature Search: A.K., M.K., Writing: A.K., E.A., M.K.

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