

DRYING EFFECT ON VOLATILE OILS ON MINTS

Lenuța Iuliana EPURE, Doru Gabriel EPURE, Nicoleta Alina UDROIU

University of Agronomic Sciences and Veterinary Medicine of Bucharest, 59 Mărăști Blvd,
District 1, 011464, Bucharest, Romania

Corresponding author email: doru.epure@gmail.com

Abstract

The mint is aromatic plant with utilisation in medicine, aromatic and cosmetic industry as well as in human consumption. The research aimed to present the influence of drying on chemical composition of different species of genus *Mentha*. Seven varieties of Mint have been analysed in this experiment: *M. spicata* L., *M. aquatica* L., *M. arvensis* L., *M. longifolia* L., *M. suaveolens* Ehrh., *M. pulegium* L., and *M. piperita* L. The drying experiment have been conducted using a Thermo-Cabinet model Hohenheim with recirculation of drying air. The drying experiments have been conducted at the 40°C, 50°C and 60°C constant temperature. Have been describe the drying behaviour plotted as wet base as well as dry base as well as drying rate for all species of mints analysed. The drying time decrease with increasing of drying temperature from 19.29 h to 04.46 h. The drying time as well as drying behaviour is strongly dependent to species of mint analysed. The chemical composition data have been processed for fresh mint, dried mint at 40°C, 50°C and 60°C. A strong correlation between variety and drying temperature on chemical composition have been found and described.

Key words: drying, mints, essential oil content, thermos cabinet.

INTRODUCTION

The genus *Mentha* L. (*Lamiaceae*) is distributed all over the world and can be found in many environments. *Mentha* species, one of the world's oldest and most popular herbs, are widely used in cooking, in cosmetics, and as alternative or complementary therapy, mainly for the treatment of gastrointestinal disorders like flatulence, indigestion, nausea, vomiting, anorexia, and ulcerative colitis. Furthermore, it is well documented that the essential oil and extracts of *Mentha* species possess antimicrobial, fungicidal, antiviral, insecticidal, and antioxidant properties (Brahmi et al., 2017). Mint (*Mentha* spp.), a genus of aromatic perennial herbs, is included in the family *Lamiaceae*. The genus *Mentha* comprises more than 25 species which found mostly in temperate and sub-temperate areas of the world (Bhat et al., 2002). Several mint species are distributed all across the globe for cultivation as industrial crops (Park et al., 2016). Numerous researches have been investigated to analyse the constituents of different species from Genus *Mentha* including flavonoids, phenolic acids, terpenoids, and other volatile compounds from. Anyhow the volumes is

strongly different from one species to another, and a high amount is losing during drying process (Figueiredo et al, 2008). Genus *Mentha* L. belong to the Family *Lamiaceae*, Order *Lamiales*, Subclass *Asteridae*, Class *Magnoliopsida*, Division *Magnoliophyta*, Superdivision *Spermatophyta*, Subkingdom *Tracheobionta*, Kingdom *Plantae* (Jedrzejczyk & Rewers, 2018; Roman et al., 2008). Determination of species in the genus *Mentha* L. is difficult because the genus has a complex taxonomy. Most *Mentha* species can produce hybrids (Andro et al., 2011). The genus *Mentha* includes 25 to 30 species that grow in the temperate regions of Europe, Australia, Africa, Asia, and North America (Dorman et al., 2003; de Sousa Guides et al., 2016). The *Mentha* section includes 7 basic species: *Mentha spicata* L. (Spearmint), *Mentha aquatica* L. (Water mint), *Mentha arvensis* L. (Wild mint), *Mentha longifolia* L./Huds (Horse mint), *Mentha suaveolens* Ehrh (Apple mint), *Mentha pulegium* L. (Pennyroyal mint) and *Mentha piperita* L. (Peppermint). It has been suggested that the frequent interspecific hybridization between these species has given rise to 11 naturally occurring hybrids (Mamadalieva et al., 2020). The yield and

composition of *Mentha* essential oils are related to climatic conditions (Brahmi et al., 2022). The heat stress strongly affects essential oil yield, chemical composition, and antibacterial activities in *M. piperita* and *M. arvensis* L. (Heydari et al., 2018). Average rainfall, temperature, and altitude and pH of soil strongly affect the antioxidant activity and phytochemical composition of mints (Mollaei et al., 2020). For mints plants it is use only *herba*. The harvest shall be done at full flowering time (EC 55 phenophase). A harvesting to early has as result a bad quality of essential oil, and a delay of harvesting has as results in a loss of content of essential oils, for all species from genera *Mentha*. (Salehi et al., 2018; Aflatuni et al., 2006; Roman et al., 2008). *Mentha* can be used as a fresh harvested *herba* or most often as a dried plant. The fresh material is usually used for culinary dishes or for chemical extraction of essential oils (Roman et al., 2008; Muntean et al., 2003). The major components found in the essential oils of *Mentha* spp. are linalool, pulegone, menthone, carvone, menthol, and piperitenone (Brahmi et al., 2017). Drying is the most common method of mint preservation, reduce the moisture content and water activity. Different drying approaches like microwave drying, hot air drying, solar drying, open sun drying, shadow drying, freeze drying, drum drying and vacuum drying have been used for dehydrating mint leaves (Kannan et al., 2021; Venkatachalam et al., 2020; Doymaz, 2006; Muntean et al., 2003). The objective of drying mints is to extend the shelf life and conserving the fresh characteristics. This is achieved by reducing the water activity (a_w) of the product to a value which will inhibit the growth and development of pathogenic and spoilage microorganisms, significantly reducing enzyme activity and the rate at which undesirable chemical reactions occur. The removal of most of the water from the product reduces the weight to be carried per unit product value. This can lead to substantial savings in the cost of handling and transporting the dried product as compared with the fresh material. A reduction in volume of the dried material, as compared with the fresh, can lead to savings in the cost of storage and transport. During drying occurs changes in *Mentha herba*. The size and shape of leaves and stems

change during drying. Color changes may also occur due to the removal of water or as a result of exposure to high temperatures during drying. But many changes could occurs in flavour contents, due to the loss of essential oils, biotherpens and polyphenolic compounds. As higher the drying temperatures is as higher the losses in essential oils is. Also at higher temperature drying occurs the change of colours of product (*herba*). The most appropriate method for drying medicinal herbs are by using thermos-cabinet model Hohenheim, with recirculation of drying air and full control of relative humidity and drying temperature of air. Such conditions should be minimized by careful selection of the drying method and conditions and good control of the drying operation (Epure et al., 2003).

MATERIALS AND METHODS

For this research 7 species from genus *Mentha* have been used: *Mentha spicata* L. (Spearmint), *Mentha aquatica* L. (Water mint), *Mentha arvensis* L. (Wild mint), *Mentha longifolia* L./Huds (Horse mint), and *Mentha suaveolens* Ehrh (Apple mint). *Mentha pulegium* L. (Pennyroyal mint) and *Mentha piperita* L. (Peppermint). The material (*herba*) have been harvested just before flowering from Botanical Garden fields. Apart have been distilled for chemical compound analyses for fresh material and a part have been dried using thermos-cabinet dryer model Hohenheim (Figure 1). The dried material have been also analysed for observe variation of chemical composition. The chemical analyzis have been carried using Gas Chromatography - Mass Spectrometry (GS-MS) international method ISO 7609:1985 (Deutsches Arzneibuch, 1986). The variable taken into consideration was the air temperature. The drying experiments were carried out at two different temperatures 40 and 50°C, with intervals of 10 K. The effect of temperature, drying rate and drying time were conducted at constant air velocity of 1 m/s, with a dew point temperature of 12°C characteristic for Romanian climatic and normal atmospheric pressure. For determination of moisture content of mints was used Karl-Fischer method. After drying of product, that was stored 24 hours in

refrigeration in hermetic containers for homogenisation. Regardless of the method used, there are possibilities of measurement errors in determining the moisture content. To reduce the possibilities of errors, two replicates were used.

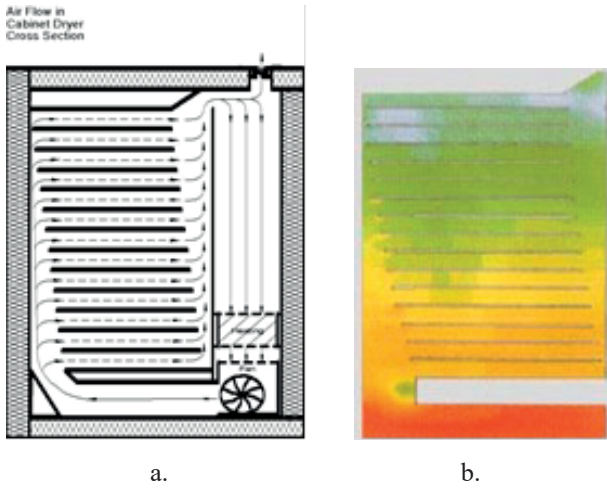


Figure 1. Thermo cabinet model Hohenheim with data acquisition system (a - air flow distribution; b - pressure distribution)

The wet base moisture content of mints is calculated using formula (1):

$$M(t) = \frac{(m(t) - m_D)}{m(t)} * 100 \quad [\%] \quad (1)$$

The dry base moisture content is computed using the following mathematical formula (2) based on the instantaneous weight changes of mints herba during the drying process.

$$X(t) = \frac{m(t) - m_D}{m_D} \quad [\text{kg/kg}] \quad (2)$$

The drying rate was analysed by plotting the dry base moisture content against the drying time using formula (3):

$$\frac{dX}{dt} = \frac{X(t) - X(t + \Delta t)}{\Delta t} \quad [\text{kg/kg h}] \quad (3)$$

where:

- $M(t)$ is Moisture content (wet base);
- $m(t)$ represent total mass;
- m_D represent dry mass;
- $X(t)$ is moisture content (dry base);
- t is time;
- Δ represent difference.

A typical curve for drying of aromatically herbs as mints plants is presented in Figure 2. The preheating period, characterized by non-linear curve, represent increasing of water temperature into the plant to the evaporation point, which require less energy for drying and occurs at the very beginning of drying process. After preheating begins the constant drying rate period. The period is characterized by a straight section if the drying conditions are stable, represent evaporation of free water which fill among the cellular capillarity, which require less energy for drying. At point K1 the straight section ends, and the curve tends asymptotically to the equilibrium moisture content. In that section from K1 to K2 the linked water from inside of cells are removed (Kowalski, 2003; Molnar, 2007).

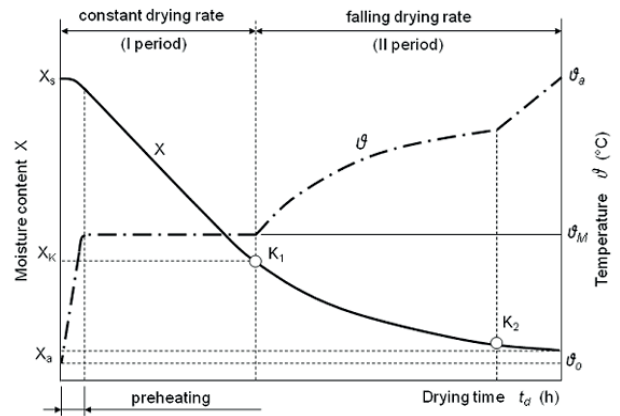


Figure 2. Drying curve for a colloidal capillary-porous substances (Kowalski, 2003)

RESULTS AND DISCUSSIONS

The drying characteristics of mint to reach the final moisture content is significantly influenced by the drying temperature such that the drying is faster with increasing temperature, as illustrated in Figure 2. The increase of temperature to accelerate the drying process is limited by the heat sensitivity of the product. In that way, the temperature above 50°C have to be avoided in order to prevent colour changes induced by high temperature (Nalawade et al., 2019). There were significant differences in the drying time as a function of air temperatures between 40 and 60°C. These differences were found to less in the temperature range above 50°C, as indicated in Figure 3. High drying temperatures shall be avoided due to loss in

essential oils contents of dried mints High temperatures lead to a decrease in volatile compounds (Koller, 1987), due to the acceleration of evaporation, decomposition of essential oils components, and due to destruction of the cellular walls (Mokhtarihah et al., 2020).

As is presented in Table 1, after drying up to 10% moisture content (wet base), the longest drying time have found for drying temperature of 40°C, and registered by *Mentha pulegium* and *Mentha aquatica*, at 19.29 h. The shortest drying time have been registered for drying at 60°C for *M. aquatica*, and has value of 4.46 h. For drying at 40°C, the shortest time for drying

have been reported by *M. pulegium* at 18.33 h, followed by *M. spicata* and *M. longifolia* with 18.54 h and *M. piperita* with 19.15 h, *M. arvensis* with 19.22 h. For drying at 50°C, the fastest drying process have been registered by *M. arvensis* at 07.42 h, followed by *M. pulegium* with 8 h, *M. spicata* and *M. longifolia* with 8.09 h, *M. piperita* with 8.18 h and *M. aquatica* and *M. suaveolens* with 8.24 h. For drying at air temperature of 60°C, *M. piperita* registered the longest drying time of 5.02 h, followed by *M. suaveolens* with 5.00 h, *M. spicata* with 4.54 h, *M. pulegium* with 4.52 h, *M. arvensis* and *M. longifolia* with 4.48 h (Figure 3).

Table 1. Drying time of different species of Mint, dried at different temperatures

Variety Drying temperature	<i>M. spicata</i>	<i>M. aquatica</i>	<i>M. arvensis</i>	<i>M. longifolia</i>	<i>M. suaveolens</i>	<i>M. piperita</i>	<i>M. pulegium</i>
40°C	18.54	19.29	19.22	18.54	19.29	19.15	18.33
50°C	08.09	08.24	07.42	08.09	08.24	08.18	08.00
60°C	4.54	04.46	04.48	04.48	05.00	05.02	04.52

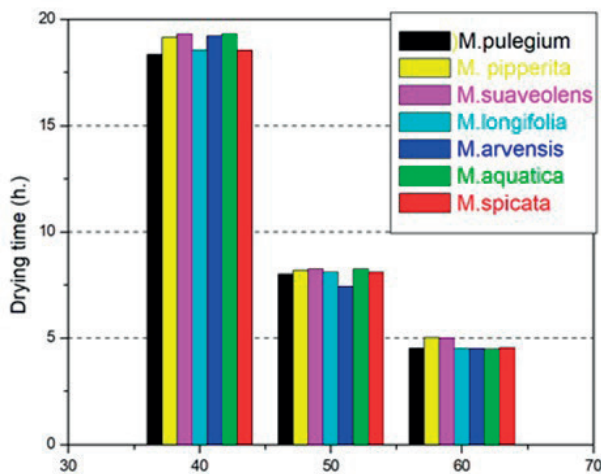


Figure 3. Drying time of different species of Mints at different drying temperature

The drying behaviour of different species of Mint at 40°C have been analysed at wet base (Figure 4) and dry base (Figure 5). The initial moisture content is different for every species. In presented experiments the initial moisture content taking into calculation have been founded with followed values: 84.99% for Spearmint, 86.36% for Water mint, 83.64% for Wild mint, 82.39% for Horse mint, 86.99% for Apple mint, 84.99% for Peppermint and 84.74% for Pennyroyal mint.

Using formula (1), and plotting Moisture content vs drying time have been presented drying behaviour of mints (wet base) (Figure 4), and plotting moisture content using formula (2) have been presented drying behaviour for mints (dry base) (Figure 5).

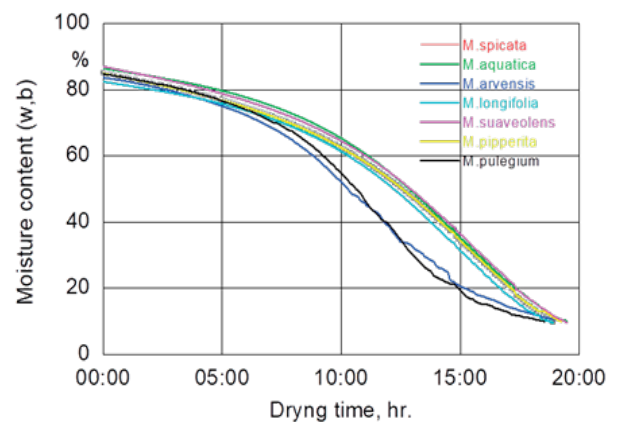


Figure 4. Drying behaviour of different species of Mints at 40°C, wet base

Different initial moisture content (dry base) have been founded in fresh material, with values among (Figures 5, 7, 9).

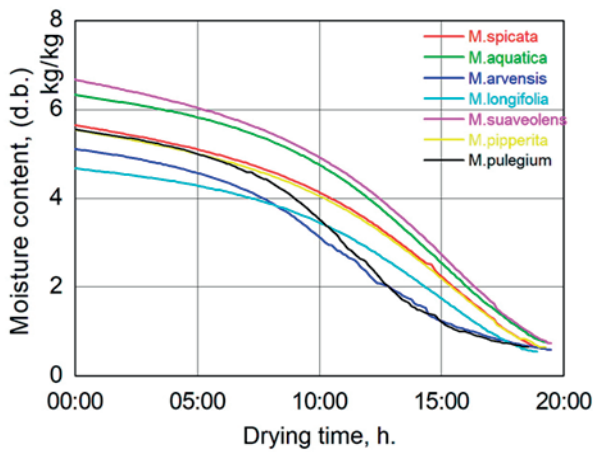


Figure 5. Drying behaviour of different species of Mints at 40°C dry base

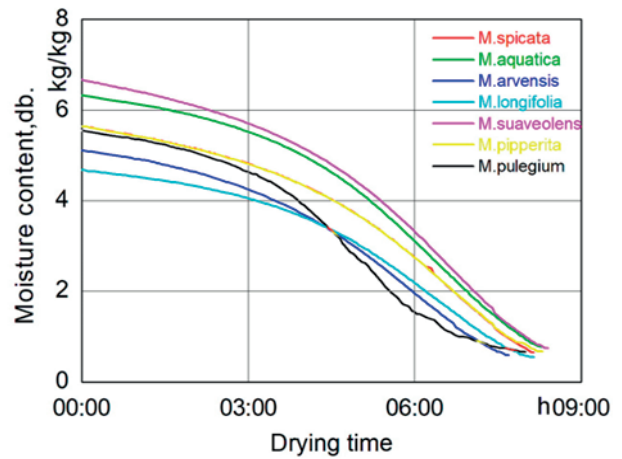


Figure 7. Drying behaviour of different species of Mints at 50°C dry base

Due to different water bound into plants (free water, intercellular and intracellular water) different drying behaviours have been founded. For drying the mints varieties at 50°C, the different behaviours have been founded (Figures 6, 7).

Due to rise of drying temperature different phenomena occur into the plant (e.g. degradation of Menthol, evaporation of Eucalyptol, etc.).

That phenomena change the behaviours of drying kinetics for each drying temperature.

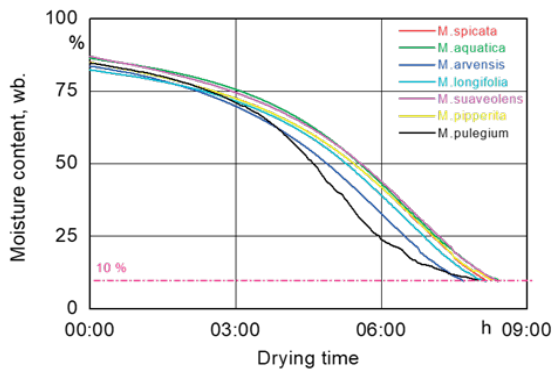


Figure 6. Drying behaviour of different species of Mints at 50°C, wet base

The behaviours of species have been different at drying at 50°C. If drying time of Pennyroyal mint have been shorter with 49 minutes than drying time of Wild mint at drying at 40°C, at drying at 50°C the time is shorter for drying Wild mint with 18 minutes than drying time of Pennyroyal mint.

Increasing drying temperature with 20 K from 40°C to 60°C has as result not only shortening of drying time (Figure 3), but also the behaviours for each variety analysed (Figures 8, 9). Water mint even is having the highest amount of moisture content in fresh material, at 60°C, has registered the shortest drying time, but for 40°C and 50°C has registered one of the longest drying time.

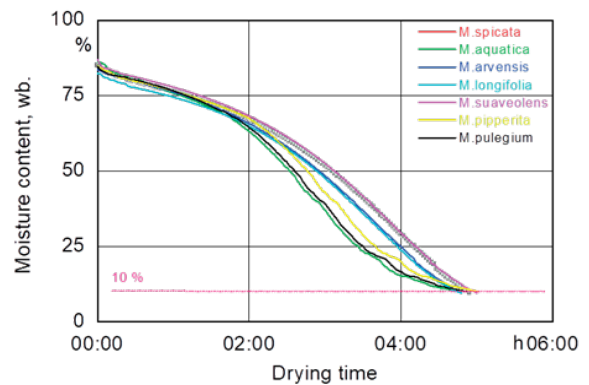


Figure 8. Drying behaviour of different species of Mints at 60°C, wet base

The drying rate have been calculated using formula (3) and plotting again drying time for each of drying temperatures tacked into account (Figures 10, 11, 12). The drying rates have been founded typically from second degree accordingly with Kowalski (2003), with a preheating period which differ with variety and temperature, a constant drying rate (when is eliminated the free water) and falling drying rate (when is eliminated linked water).

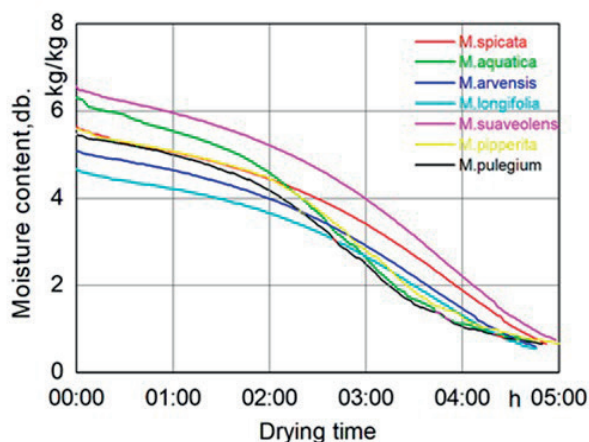


Figure 9. Drying behaviour of different species of Mints at 60 °C dry base

For drying at 40°C, the maximal drying rate has ranged among 2.1 kg/kg h, for *M. longifolia* and 3.5 kg/kg h for *M. pulegium*. Anyhow these values have been reached after preheating time which differ from one variety to another from 7 min for *M. aquatica* to 42 min for *M. suaveolens* (Figure 10).

For rising a drying temperature from 40°C at 50°C, the drying rates change the maximal values as well as the kinetics. The maximum drying rate reported has been at 3.5 kg/kg h after 8 min of drying for Pennyroyal mint, and lowest maximal value of 2.45 kg/kg h after 20 min for Water mint.

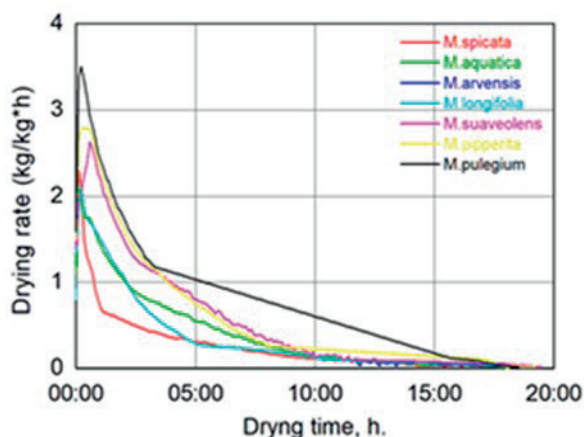


Figure 10. Drying rate of different species of Mints at drying temperature of 40°C

For Spearmint the maximum value has 2.54 kg/kg h after 7 min of starting drying process, for Wild mint 2.63 kg/kg h after 20 min, for Horse mint 2.9 kg/kg h after 12 min, for Apple mint 2.63 kg/kg h after 20 min and for Peppermint 2.9 kg/kg h after 8 min.

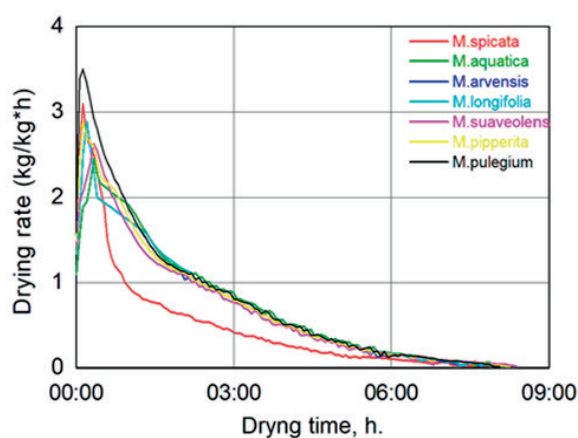


Figure 11. Drying rate of different species of Mints at drying temperature of 50°C

At drying temperature of 60°C, the preheating time is reducing for each variety of mints from 4 to 8 min. The highest preheating time have been reported to *M. longifolia* and *M. suaveolens*. The drying rate values has increased for every mint variety, and maximal values have been ranged among 4,2 kg/kg h for *M. piperita* and 5,4 kg/kg h for *M. aquatica*. The other values are 5.23 kg/kg h for *M. Pulegium*, 5.1 kg/kg h for *M. suaveolens*, 5.06 kg/kg h for *M. spicata*, 4.7 kg/kg h for *M. arvensis* and 4.6 kg/kg h for *M. longifolia* (Figure 12).

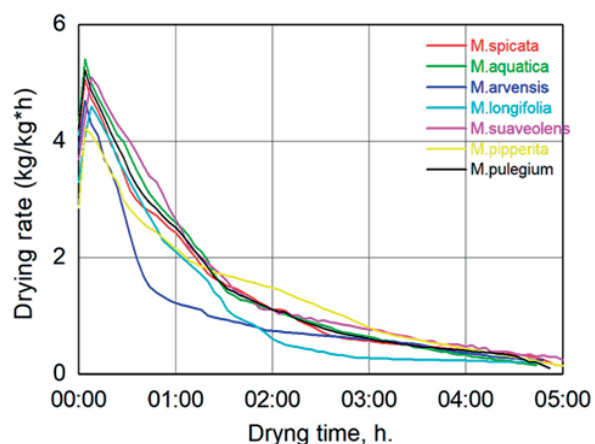


Figure 12. Drying rate of different species of Mints at drying temperature of 60°C

A chemical composition has been analysed for all variants tacked into experiment. Have been analysed from fresh material for each species as well as after drying at 40°C, 50°C and 60°C. Not all compounds have been tacked into account, only the main compound for each variant. Anyhow, the chemical composition is

dependent of variety, but could vary also with growing condition, weather, harvest time as well as transport and manipulation. The initial moisture content for fresh material is dependent with variety, but final dried material have been 10% moisture content for each variety, and drying temperature. Some compounds could be found in most of variants (e.g.: Menthole, Menthone, Caryophyllene, etc.), and other compounds are specific to one variety (e.g.: Carveone to Spearmint) (Table 2).

For Peppermint the main essential oil compounds analysed were: Menthole, Menthone, Cineole, Eucalyptole, γ -Terpinene and Caryophyllene (Figure 13). The total content of oil have been increased after drying, but the chemical composition have been modified. The Menthole content have reduced from 40.47% in fresh mater to 40.12% in mint dried at 40°C and 30.95% in mint dried at 50°C and 33.93% in mint dried at 60°C.

Table 2. The main volatile compound found in different varieties of *Mentha* spp. (%)

Essential oil - Variety	Menthole	Menthone	Iso-menthole	Methofuran	Pulegone	Cineole	Carveone	Eucalyptol	γ -Terpinene	Cubenol	Caryophyllene	Limolene	Piperitone
<i>Mentha piperita</i>	40.47	26.58	ND	ND	ND	8.70	ND	6.20	5.09	1.14	5.50	ND	ND
<i>Mentha spicata</i>	40.47	3.2	ND	ND	ND	ND	32.84	6.25	1.34	0.7	5.47	ND	ND
<i>Mentha aquatica</i>	2.02	1.01	ND	61.47	ND	ND	ND	11.39	0.18	0.92	8.61	9.01	ND
<i>Mentha suaveolens</i>	ND	29.00	9.30	ND	32.30	ND	ND	9.49	5.25	ND	2.92	5.94	ND
<i>Mentha longifolia</i>	ND	ND	ND	ND	ND	ND	ND	9.35	2.15	0.58	0.48	4.30	43.90
<i>Mentha pulegium</i>	3.28	3.09	1.56	2.15	6.45	1.39	ND	0.40	0.34	0.84	1.36	1.2	21.18
<i>Mentha arvensis</i>	21.35	29.39	10.79	ND	ND	ND	ND	6.92	ND	ND	ND	1.48	ND

Determined by Gas Chromatography- Mass Spectrometry (GS-MS) international method ISO 7609:1985. ND = not detectable.

Menthone content have been increase in content from 26.58% in fresh matter to 32.21% in dried product at 40°C, 38.45 at 50°C and 42.47% at 60°C. That behaviour is due to oxidative property of Menthole resulting Menthone. Could observe an relative equilibrium between Menthone and Menthole in product dried at 50°C. In fresh and dried at 40°C the majority is represent by Menthole and at 60°C by Menthone.

The most volatile compounds Cineole, γ -Terpinene and Eucalyptol decrease drastically after 50°C, Cineole form 8.7% in fresh mater to 3.84 at 40°C, 2.36 at 50°C and 1.63% at 60°C, Eucalyptol from 6.2% in fresh mater to 2.76 at 40, 1.67 at 50 and 1.17 at 60°C. γ -Terpinene content has decrease from 5.09% in fresh mater to 2.46 at 40, 1.53 at 50 and 1.11 at 60°C dried product. The Caryophyllene content is relative constant in Peppermint during drying process with a relative content of 5.5% in fresh mater, 5.42 in product dried at 40°C, 5.24 at 50°C, and 5.46% at 60°C (Figure 13).

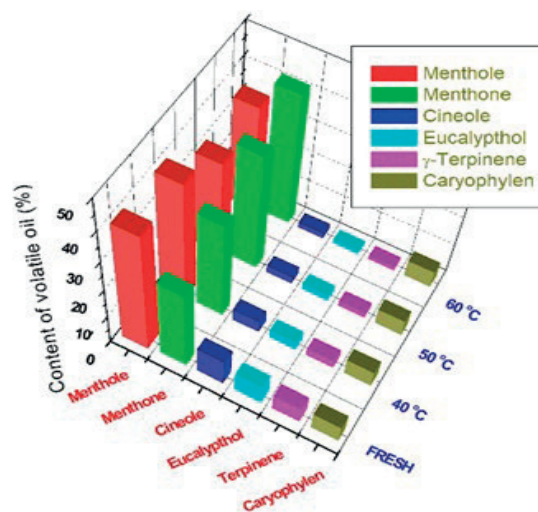


Figure 13. The main volatile compound found in *Mentha piperita* fresh and dried product

For Spearmint the most important compound is Menthole, followed by Carveone which is characteristic for Spearmint (Figure 14). The content of Menthole decrease from 40.47% in fresh product to 34.96 at 40, 27.51 at 50 and 20.07 at product dried at 60°C.

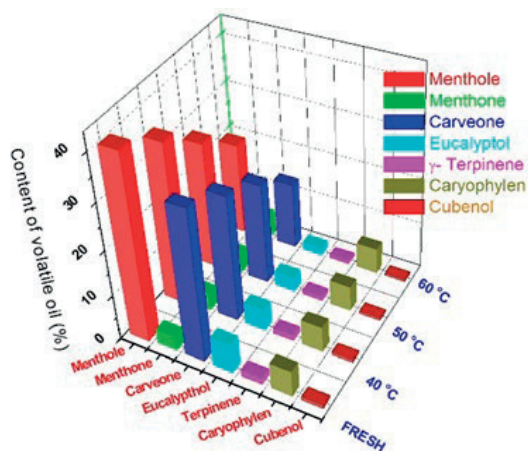


Figure 14. The main volatile compound found in *Mentha spicata* fresh and dried product

The Menthone content is lowest than in Peppermint but has also an increasing rate with increasing of drying temperature from 3.2% in fresh product to 3.8 in dried product at 40, 4.5 at 50 and 5.2% in dried product at 60°C. The Carveone content has also decrease from fresh material to dried product and decrease with drying temperature from 32.84% in fresh product to 27.36 at 40, 21.89 at 50 and 14.43% at dried product at 60°C. Eucalyptol and γ -Terpinene have the same trend as in Peppermint (Figure 14). Eucalyptol content decrease from 6.25% in fresh product to 4.69% in dried product at 40°C, 3.13% in dried product at 50°C and 1.56% in dried product at 60 °C. γ -Terpinene content decrease from 1.34% in fresh product to 1.12% in dried product at 40°C, 0.89% in dried product at 50°C and 0.68% in dried product at 60°C. Caryophyllen and Cubenol content is not influenced by drying temperature, but some modification in content occurs during drying process. Values found for Caryophyllen content are 5.47% in fresh product, 5.41% in dried product at 30°C, 5.37% in dried product at 50°C, 5.43% in dried product at 60°C. Cubenol content is reducing from 0.7% in fresh product to 0.62% in dried product at 40°C, 0.56% in dried product at 50°C, and 0.47% in dried product at 60°C (Figure 14). In Water mint the main compounds of essential oil are represented by Methafuran, Eucalyptole, Limolene and Caryophyllen (Figure 15). The Menthole and Menthone are present but in neglected amount. The Menthafuran is strongly volatile and content strongly decrease by heating of fresh product. The content decrease

from 61.47% in fresh product to 46.1% in dried product at 40°C, to 30.74% in dried product at 50°C, and 15.36% in dried product at 60°C.

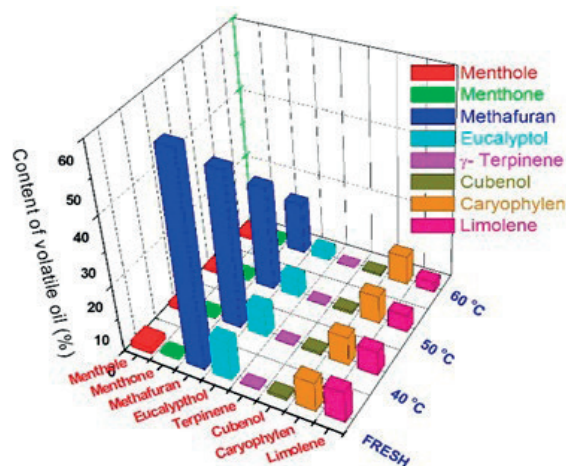


Figure 15. The main volatile compound found in *Mentha aquatica* fresh and dried product

The Eucalyptol content decrease from 11.39% in fresh product to 8.54% in dried product at 40°C, 5.71% in dried product at 50°C and 2.86% in dried product at 60°C. Limolene content in fresh product have values of 9.01%, in dried product at 40°C 7.02%, in dried product at 50°C 5.01% and in dried product at 60°C 3.11%. Menthole content decrease from 2.02% in fresh product to 1.62% in dried product at 40°C, 1.31% in dried product at 50°C and 0.99% in dried product at 60°C. The Menthone content increase with increasing of drying temperature from 1.01% in fresh product to 1.2% in dried product at 40°C, 1.39% in dried product at 50°C and 1.56% in dried product at 60°C. γ -Terpinene is low in content of essential oils and decrease by volatilisation during drying from 0.18% in fresh mint to 0.15 in dried mint at 40°C, 0.11% in dried mint at 50°C and 0.07% in dried mint at 60°C. The Cubenol and Caryophyllen content are less influenced by drying process, being relative stable. The Cubenol content values are: 0.92% in fresh mint, 0.84 in dried mint at 40°C, 0.75% in dried mint at 50°C and 0.66% for dried mint at 60°C. Caryophyllen values are about 8%: 8.61% in fresh mint, 8.46% in dried mint at 40°C, 8.12% in dried mint at 50°C and 8.47% in dried mint at 60°C (Figure 15). In main essential oil compound in fresh Apple mint is Pulegone, but in dried mint is Menthone (Figure 16). During drying process content of Menthone and Isomenthole increase due to

isomerisation of other product during drying process. The Menthone content increase from 29% in fresh mint to 33.14% in mint dried at 40°C, 37.29% in mint dried at 50°C, and 42.8% in mint dried at 60°C. Isomenthole content increase from 9.3% in fresh mint to 11.07% in mint dried at 40°C, 13.72% in mint dried at 50°C and 16.82% in mint dried at 60°C.

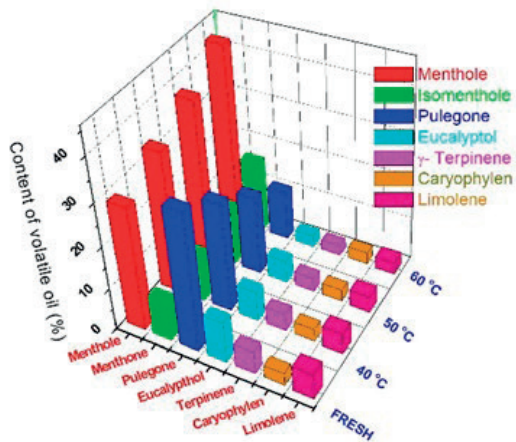


Figure 16. The main volatile compound found in *Mentha suaveolens* fresh and dried product

Pulegone content decrease by drying from 32.3% in fresh mint to 25.38% in dried mint at 40°C, 18.45% in dried mint at 50°C and 11.52% in dried mint at 60°C. Eucalyptol content decrease from 9.49% in fresh mint to 6.91% in mint dried at 40°C, 5.17% in mint dried at 50°C and 2.59% in dried mint at 60°C. γ -Terpinene content decrease from 5.25% in fresh mint at 4.09, 3.21, 2.33 during drying process at 40, 50 and 60°C respectively. Caryophyllen content vary from 2.92% in fresh mint to 2.87, 2.72 and 2.76 in dried mint at 40, 50 and respectively 60°C. Limolene content decrease during drying process from 5.94 % in fresh product to 4.77% in dried product at 40°C, 3.81% in dried product at 50°C and 2.81% in dried product at 60°C (Figure 16). The main compound in Horse mint is Piperitone. Could be found also Eucalyptol, γ -Terpinene, Caryophyllen, Cubenol and Limolene (Figure 17). The Piperitone content increase with increasing of drying temperature from 43.9% in fresh mint to 50.3% in dried mint at 40°C, 55.7% in dried mint at 50°C, 63.03% in dried mint at 60°C. Eucalyptol content decrease with increasing of drying temperature from 9.35% in fresh mint to 7.27% in dried mint at 40°C, 5.21% in dried mint at

50°C, 3.12% in dried mint at 60°C. γ -Terpinene decrease in Horse mint from 2.15% in fresh mint to 1.94% in mint dried at 40°C, 1.37% in mint dried at 50°C and 0.97% in mint dried at 60°C.

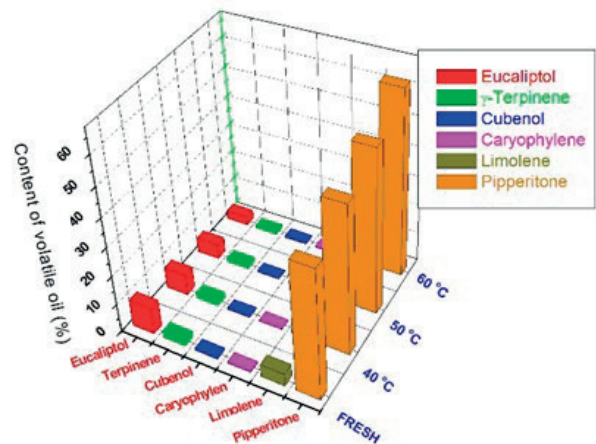


Figure 17. The main volatile compound found in *Mentha longifolia* fresh and dried product

Cubenol content is relative stable during drying process. The found values in Horse mint are: 0.58, 054, 049 and 0.55% for fresh mint and dried mint at 40, 50 and 60°C, respectively. Caryophyllen content decrease from 0.48% in fresh mint to 0.36% in dried mint at 40°C, 0.38% in dried mint at 50°C and 0.33% in dried mint at 60°C. Limolene content decrease from 4.3% in fresh product to 3.45% after drying at 40°C, 2.72% after drying at 50°C and 1.95% after drying at 60°C (Figure 17). The most complex compound of essential oil among the analysed variants is gave by Pennyroyal mint. The main compound as volume is represented by Pulegone and Piperitone (Figure 18). The content of Piperitone is increasing from 21.18% in fresh mint to 24.11% in dried mint at 40°C, 25.32% in dried mint at 50°C and 27.77% in dried mint at 60°C. The Pulegone content decrease with increasing of drying temperature due to thermic instability properties from 35.45% in fresh mint to 31.92% in dried mint at 40°C, 27.98% in dried mint at 50°C and 22.01% in dried mint at 60°C. The Menthone content decrease almost linear with increasing of drying temperature from 3.28% in fresh product, 3.01% in dried product at 40°C, 2.21% in dried product at 50°C and 1.11% in dried product at 60°C. The Menthone content has an opus behaviour increasing almost linear, in value, with increasing of

drying temperatures values, from 1.56% in fresh mint, 1.67% in dried mint at 40°C, 1.78% in dried mint at 50°C, and 1.86% in dried mint at 60°C. Isomenthole also has increase the content with increase of drying temperature. Registered values are: 1.56% for fresh mint, 1.67% for dried mint at 40°C, 1.78% for dried product at 50°C, and 1.86% for dried product at 60°C. Methofuran content decrease in Pennyroyal mint during drying from 2.15% in fresh mint to 1.54% in dried mint at 40°C, 1.07% in dried mint at 50°C and 0.61% in dried mint at 60°C. Cineole content decrease in Pennyroyal mint with increasing of drying temperature from 1.39% in fresh mint to 0.91% in dried mint at 40°C, 0.63% in dried mint at 50°C and 0.32% in dried mint at 60°C. Eucalyptol content also decreases from fresh mint (0.4%) to dried mint at 40°C (0.31%), dried mint at 50°C (0.19%) and dried mint at 60°C (0.09%).

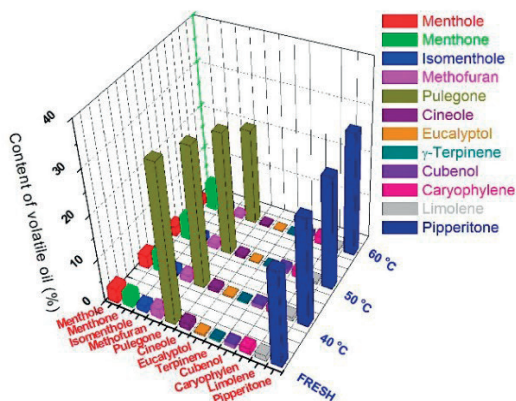


Figure 18. The main volatile compound found in *Mentha pulegium* fresh and dried product

γ -Terpinene level in Pennyroyal mint decrease with increasing of drying temperature from 0.34% in fresh mint to 0.24% in dried mint at 40°C, 0.15% in dried mint at 50°C and 0.09% in dried mint at 60°C. In Pennyroyal mint content of Cubenol is increasing with increasing of drying temperature, perhaps due to isomerisation of some compounds during drying time. The relative values are 0.84% in fresh mint, 0.92% in dried mint at 40°C, 1.01% in dried mint at 50°C and 1.12% in dried mint at 60°C. In Pennyroyal mint the content of Caryophyllen is also increasing from 1.36% in fresh mint to 1.49% in dried mint at 40°C, 1.63% in dried mint at 50°C and 1.79% in dried mint at 60°C. The content of Limolene is

strongly decreasing with increasing of drying temperatures from 1.2% in fresh product to 0.8% in dried product at 40°C, 0.5% in dried product at 50°C and 0.31% in dried product at 60°C (Figure 18). The main essential oils of Wild mint are represented by Menthole, Menthone, Isomenthole, Eucalyptol and Limolene (Figure 19). The main compound is Menthone with values increasing with increasing of drying temperature from 29.39% in fresh mint to 33.96% in mint dried at 40°C, 39.84% in mint dried at 50°C and 43.67% in mint dried at 60°C. The second compound, Menthole is decreasing in content from 21.35% in fresh mint to 17.55% in dried mint at 40°C, 13.77% in dried mint at 50°C and 10.11% in dried mint at 60°C. Isomenthole content is increasing with increasing of drying temperature from 10.79% in fresh mint to 12.33% in dried mint at 40°C, 14.66% in dried mint at 50°C and 17.21% in dried mint at 60°C.

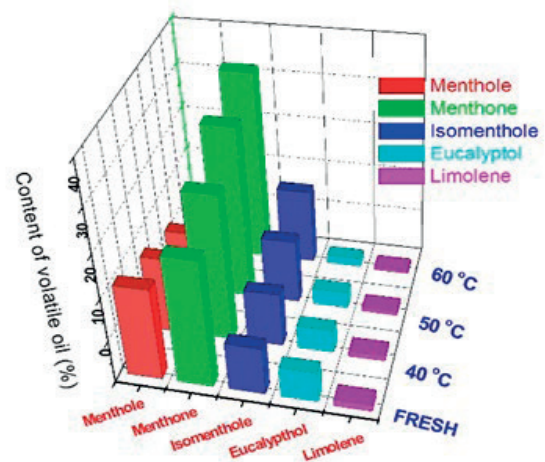


Figure 19. The main volatile compound found in *Mentha arvensis* fresh and dried product

In the same time the content in Eucalyptol is decreasing with increasing of drying temperature from 6.92% in fresh mint to 5.29% in dried mint at 40°C, 3.67% in dried mint at 50°C and 1.93% in dried mint at 60°C.

The content of Limolene have the same trend descendent with increasing of drying temperatures from 1.48% in fresh mint to 1.17% in dried mint at 40°C, 0.86% in dried mint at 50°C and 0.56% in dried mint at 60°C.

CONCLUSIONS

The drying time is dependent of Mint species and drying temperature. The drying is reducing

for each species analysed with increasing of temperature. The longest drying time have been found for drying at 40°C, 19.29 h for *M. aquatica* and *M. suaveolens* and the shortest drying time have been found for drying at 60°C, 04.46 h for *M. aquatica*. At 50°C the fastest drying has been reach by *M. arvensis* at 7.42 h and longest time have been reach by *M. aquatica* and *M. suaveolens* at 8.24 h. At 60°C, the shortest drying time have been found for drying of *M. piperita*. The drying behaviour for each species at any analysed temperature is different, probably due to different chemical composition and due to initial moisture content. Drying rate have been found higher at beginning of drying process for any values of drying temperatures, due to higher content of free water in fresh product. Drying rate for any of mint species fit to Kowalski model of porous material (Kowalski, 2003). The chemical composition of main essential oils have been analysed and plotted again drying temperature. Not all chemical compounds have been found in each variety of mint analysed. Some of chemical compound have been lost during drying, by volatilisation, oxidative process or isomerisation. The other compound increase in value due to isomerisation of other product, due to resilience on evaporation process as well as stability on thermal process. The processes are strongly linked to species. Menthole value have been decreased in any drying variant with drying temperature, as well as Menthone and Isomenthole increase in any variant with increasing of drying temperature. Cubenol, Limenolene and Pulegone content decrease with increasing of drying temperature. Eucalyptol, γ -Terpinene, Cineole and Methofuran content always are strongly decreasing by increasing of drying temperatures. Piperitone content have been found to increase with increasing of drying temperature. The Cubenol and Caryophyllene content have been more stable during drying process with increasing with drying temperatures, but content have been varying with mint species. The recommended drying temperature depend on purpose of the dried compound. For Eucalyptol, γ -Terpinene, Cineole and Methofuran interest drying shall be conducted at 40°C. For utilisation of Piperitone, Menthone and Isomentole, drying

process is recommended to be carried at 60°C. For utilisation of Cubenol, Caryophyllene, Limolene, Carveone and Pulegone is recommended to dry the mints at 50°C. The drying process have to be conduct up to reach the 10% final moisture content.

ACKNOWLEDGEMENTS

This research work was carried out with the support of DAAD Foundation. The authors acknowledge to University of Coimbra, University Hohenheim and Kassel University for their support during conducting of experiments.

REFERENCES

- Aflatuni, A., Sari, E. K., Uusitalo, J., Hohtola, A. (2006). Optimum harvesting time of four *Mentha* species in Northern Finland. *J. Essent Oil Res.*, 18(2):134–8. doi: 10.1080/10412905.2006.9699043
- Andro, A. R., Boz, I., Padurariu, C., Atofani, D., Coisin, M., & Zamfirachie, M. M. (2011). Comparative biochemical and physiological research on taxa of *Mentha* L. genus. *Journal of Plant Development*, 18, 41–45.
- Bhat, S., Maheshwari, P., Kumar, S., Kumar, A. (2002). *Mentha* species: *In vitro* Regeneration and Genetic Transformation. *Molecular Biology Today*, 3(1), 11–23.
- Brahmi, F., Lounis, N., Mebarakou, S., Guendouze, N., Yalaoui Guellal, D., Madani, K. (2022). Impact of growth sites on the phenolic contents and antioxidant activities of three Algerian mentha species (*M. pulegium* L., *M. rotundifolia* (L.) Huds., and *M. spicata* L.). *Front Pharmacol.*, 13: 886337. doi:10.3389/fphar.2022.886337.
- Brahmi, F., Khodir, M., Chibane, M., & Duez, P. (2017). Chemical composition and biological activities of *Mentha* species, aromatic and medicinal plants. In H. A. El-Shemy (Ed.), *Back to nature*. IntechOpen. <https://doi.org/10.5772/67291>
- Dorman, H. J. D., Kosar, M., Kahlos, K., Holm, Y., & Hiltunen, R. (2003). Antioxidant properties and composition of aqueous extracts from *Mentha* species, hybrids, varieties, and cultivars. *Journal of Agricultural and Food Chemistry*, 51, 4563–4569.
- Doymaz, I. (2006). Thin- Layer Drying Behaviour of Mint Leaves (*Mentha spicata* L.). *Journal of Food Engineering*, 74(3), 370–375.
- Deutsches Arzneibuch, D. K. (1986). Wissenschaftliche Verlagsgesellschaft mbh. Stuttgart. ISO 7609:1985
- de Sousa Guedes, J. P., da Costa Medeiros, J. A., de Souza E Silva, R. S., de Sousa, J. M., da Conceição, M. L., de Souza E. L. (2016). The efficacy of *Mentha arvensis* L. and *M. piperita* L. essential oils in reducing pathogenic bacteria and maintaining quality characteristics in cashew, guava, mango, and

- Pineapple juices. *International Journal of Food Microbiology*, 238, 183–192.
- Epure, D. G., Mitroi, A., Muehlbauer, W., Udriou, A. N. (2003). Influence of thermophysical parameters on drying process of tomatoes. *Scientific Papers USAMVB, Seria B, Horticulture, Vol. XLV*, 292–295.
- Figueiredo, A. C., Barroso, J. G., Pedro, L. G., Scheffer, J. J. (2008). Factors affecting secondary metabolite production in plants: volatile components and essential oils. *Journal Flavour and Frag*, 23:213–226.
- Heydari, M., Zanfardino, A., Taleei, A., Shahnejat Bushehri, A. A., Hadian, J., Maresca, V. (2018). Effect of heat stress on yield, monoterpene content and antibacterial activity of essential oils of *Mentha x piperita* var. Mitcham and *Mentha arvensis* var. piperascens. *Molecules*, 23(8):1903. doi: 10.3390/molecules23081903
- Jedrzejczyk, I., Rewers, M. (2018). Genome size and ISSR markers for *Mentha* L. (*Lamiaceae*) genetic diversity assessment and species identification. *Ind. Crops Prod.*, 120: 171–9. doi:10.1016/j.indcrop.2018.04.062
- Kannan, V. S., Arjunan, T., Vijayan, S. (2021). Drying characteristics of mint leaves (*Mentha arvensis*) dried in a solid desiccant dehumidifier system. *J. Food Sci. Technol.*, 58(2):777–86. doi: 10.1007/s13197-020-04595-z
- Koller, D. (1987). Problems with the flavour of herbs and spices. In *Proceeding of the fifth International Flavour conference*, Porto Karras, Chalkidiki, Greece, 123–132.
- Kowalski, S. (2003). *Thermomechanics of drying processes*. Berlin: Springer-Verlag.
- Mamadalieva, N. Z., Hussain, H., Xiao, J. (2020). Recent advances in genus *Mentha*: Phytochemistry, antimicrobial effects, and food applications. *Food Frontiers*, 1:435–58. <https://doi.org/10.1002/fft2.53>
- Mokhtarikhah, G., Ebadi, M. T., Ayyari, M. (2020). Qualitative changes of spearmint essential oil as affected by drying methods. *Ind. Crops Prod.*, 153, 112492.
- Mollaie, S., Ebadi, M., Hazrati, S., Habibi, B., Gholami, F., Mahmoodi, M. (2020). Essential oil variation and antioxidant capacity of *Mentha pulegium* populations and their relation to ecological factors. *Biochem. Syst. Ecol.*, 91:104084. doi: 10.1016/j.bse.2020.104084
- Molnar, K. (2007). *Experimental techniques in drying*. Aruns Mujumdar. Handbook of industrial drying. Boca raton: Taylor & Francis.
- Muntean, L. S. (2003). *Mic tratat de Fitotehnie, vol. II, tutunul, hameiul, plante medicinale și aromatice*. Cluj-Napoca, RO: Risoprint Publishing House.
- Nalawade, S. A., Ghiwari, G. K., Hebbar, H. U. (2019). Process efficiency of electromagnetic radiation (EMR)-assisted hybrid drying in spearmint (*Mentha spicata* L.). *J. Food Process. Preserv.*, 43, e14190.
- Park, Y. J., Baskar, T. B., Yeo, S. K. (2016). Composition of volatile compounds and *in vitro* antimicrobial activity of nine *Mentha* spp. *Springer Plus*, 5, 1628. <https://doi.org/10.1186/s40064-016-3283-1>
- Roman, Gh. V., Toader, M., Epure, L. I., Ion, V., Bășa, A. Gh. (2008). *Cultura plantelor medicinale și aromatice în sistem ecologic*. Bucharest, RO: Ceres Publishing House.
- Salehi, B., Stojanović-Radić, Z., Matejić, J., Sharopov, F., Antolak, H., Kręgiel, D. (2018). Plants of genus *Mentha*: From farm to food factory. *Plants*, 7(3):70. doi: 10.3390/plants7030070
- Venkatachalam, S. K., Thottipalayam Vellingri, A., Selvaraj, V. (2020). Low-temperature drying characteristics of mint leaves in a continuous-dehumidified air drying system. *J. Food Process Eng.*, 43(4):e13384. doi: 10.1111/jfpe.13384