Effect of drying methods on the chemical composition and colour of peppermint (\textit{Mentha × piperita} \textit{L.}) leaves

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Abstract

The study was done with the two peppermint (\textit{Mentha × piperita} \textit{L.}) cultivars – ‘Krasnodarskaja’ and ‘Peppermint’, whose leaves were dried using different methods. Investigation of fresh and dried herbs was carried out according to the biochemistry and technology methods. Essential oil was extracted using the hydro distillation method, the amount of chlorophyll \texttext{a} and \texttext{b} was measured spectrophotometrically according to the Vernon method. The highest content of essential oil (0.77\%) and chlorophyll (1.69\%) was found in the cv. ‘Peppermint’. Chlorophyll \texttext{a} to \texttext{b} ratio was different in the fresh peppermint leaves: in the cv. ‘Peppermint’ it was 1.35 and in the cv. ‘Krasnodarskaja’ – 1.44. Peppermint leaves were dried using active ventilation, convection, infrared, vacuum, microwave, and sublimation methods. The quality of dried herbs depended on the properties of plants and drying techniques. The highest content of essential oil (0.64–0.68\% of dry mass) was found in the variously dried peppermint leaves of the cv. ‘Krasnodarskaja’. The lowest content of essential oil (0.08\% and 0.065\% of dry mass) was determined in microwave dried herbs. The highest content of chlorophyll was found in the lyophilized peppermint leaves (715.0 mg 100 g\(^{-1}\) of dry mass) of the cv. ‘Peppermint’. Regardless of the drying method, significant differences between the ratio of chlorophyll \texttext{a} to \texttext{b} were observed in the dried herbs of the cv. ‘Krasnodarskaja’. The fresh and dried peppermint samples of the cv. ‘Peppermint’ had the lowest brightness L\* value (from 22.61 to 35.24) and the lowest yellowness b\* values (from 9.35 to 17.00). The biggest changes in greenness a\* value were in microwave dried peppermint leaves.

Key words: chlorophyll \texttext{a}, chlorophyll \texttext{b}, drying methods, essential oil, \textit{Mentha × piperita}.

Introduction

Aromatic plants are becoming increasingly popular with consumers. Apart from the flavouring use, people are also interested in medicinal and anti-inflammatory properties (Risch, 1997). In the market, most herbs and spices are provided dried, because, due to the high water content in the fresh state, they undergo severe deterioration after microbial growth and biochemical reactions. Drying is one of the most antique processes to preserve quality of aromatic and medicinal plants. It consists of water removal from the raw material up to a level at which microbial spoilage and deterioration reactions are highly minimized (Rocha et al., 2011). Many researches on the mathematical modelling and experimental studies have been conducted on the drying processes of various products, such as mint (\textit{Mentha viridis} \textit{L.}) (Kouhila et al., 2001), aromatic and medicinal plants (Kavak Akpinar, 2006). Among the drying methods, the hot air drying is the most used method, but it can lead to thermal damage and can severely alter the volatile composition of herbs as well as the colour (Antal et al., 2011). Ambient temperatures and temperatures below 30°C are the best to retain volatile compounds (Rocha et al., 2011). However, as determined, in hot air drying the loss of phenolic compounds and antioxidant activity reached up to 60\% compared to freeze drying. The evaluation of other drying methods that remove moisture content at low temperatures with the use of irradiation or vacuum stands a challenging prospective (Harbourne et al., 2009; Orphanides et al., 2013). In recent years, microwave drying has been widely used as an alternative drying method for a wide variety of herbs and spices, and its effect on the quality of the dried product has been evaluated. Several food products have been successfully dried by microwave, hot air application and/or by combined microwave-convective drying (Shaw et al., 2007; Karaaslan et al., 2013).

The changes in biologically active constituents depend not only on drying process, but can be attributed to the specific compound and species (Stanisavljevic et al., 2012). The results showed that drying method had a significant effect on oil content and composition of aromatic plants. The literature indicates that most of the essential oil components decline at temperatures over 30°C (Yadegari et al., 2013).
Mint plants are one of the most interesting research plants; they are between medicinal and aromatic plants (Mekonnen, 2011). Peppermint oil is one of the most important essential oils; it is used in pharmaceuticals, cosmetics and flavouring all over the world (Edris et al., 2003). The mint and its tea extract are rich in the essential minerals such as Na, Mg, K, Ca, Cr, Fe, Co, Cu, Zn and Se (Padmini et al., 2010). The spearmint oil has a potent antimicrobial activity. Previous studies have shown antibacterial, antifungal (Sulieman et al., 2011) and antioxidant activities (Kizil et al., 2010) of the water, ethanol and methanol herbal extracts.

The objectives of this study were to determine the effect of drying methods on biochemical compounds and changes in physical properties in dried peppermint.

Materials and methods

The object of this investigation was peppermint plants of cvs. ‘Krasnodarska’ and ‘Peppermint’. Analyses were performed at the Institute of Horticulture, Lithuanian Research Centre for Agriculture and Forestry in 2012–2013.

Drying methods. Uncrushed peppermint leaves were dried using natural, convection, infrared, vacuum, microwave and sublimation drying methods: 1) active ventilation (A) drying was performed at a temperature of 22 ± 2°C and an air-flow rate of 1.5 m s⁻¹; 2) convective (C) drying was performed in a UDS-150/1 hot-air laboratory dryer (“Utenos krosnys”, Lithuania) at a temperature of 39 ± 1°C and an air-flow rate of 1.5 m s⁻¹; 3) infrared (I) drying was performed with a moisture analyser HA310 IR (Precisa Gravimetrics AG, Switzerland) at 39 ± 1°C, wavelength range 1.2–6.0 µm; 4) microwave/ high-frequency electromagnetic waves (M) drying was performed in a microwave oven AFMW290 (AFTRON, Europe, The Netherlands) at a frequency of 2450 MHz and an intermediate power of 500 W; 5) sublimation/lyophilisation (S) was performed in a sublimator 3 × 4 × 5 (ZIRBUS Technology GmbH, Germany), the condenser temperature was −85°C, and the vacuum was 2 × 10⁻⁶ mPa; 6) vacuum (V) drying was performed in a sublimator 3 × 4 × 5 (ZIRBUS Technology GmbH, Germany) at −6°C, the condenser temperature was 22 ± 2°C and an air-flow rate of 1.5 m s⁻¹; 7) freeze drying was performed in a UDS-150/1 hot-air laboratory dryer (“Utenos krosnys”, Lithuania) at a temperature of 30 ± 1°C for 30 minutes. The vacuum was then reduced to −6 mPa and MgCO₃. The solution was filtered through a glass filter through a vacuum pump into the filter flask. The mortar was rinsed with acetone, until the liquid became colourless. Lastly, the extract was carried for the optical density measurement. Absorbance was measured at two wavelengths 665 and 649 nm.

Colour measurement. Colour was measured with a spectrophotometer MiniScan XE Plus (Hunter Associates Laboratory Inc., USA). CIEX*a*b* colour parameters were recorded as L* (lightness), a* (+ redness) and b* (+ yellowness). The chroma (C* = (a*² + b*²)¹/²) and hue angle (h° = arctan (b*/a*)) were also calculated (McGuire, 1992).

Statistical analysis. The analyses of chemical composition were carried out in triplicate and colour measurements were carried out in five replicates. The results were statistically processed using MS Excel and software package SELEKCIJA (Tarakanovas, Ragonius, 2003) and are presented as the average of three measurements with the standard errors of the mean. Data of chemical compounds on fresh peppermint was evaluated statistically using one-way, colour characteristics on dry peppermint using two-way and factorial ANOVA analysis. Fisher’s LSD test and the determination coefficient were calculated at a probability level of P ≤ 0.05.

Results and discussion

The chemical composition of fresh peppermint leaves was determined after harvesting. The results of analyses are presented in Table 1. The amount of dry soluble solids, ascorbic acid, nitrates and carotenoids was similar in both investigated peppermint cultivars or slightly differed. However, the amount of total sugar and chlorophyll substantially differed (P ≤ 0.05). Between the examined peppermint cultivars, ‘Krasnodarska’ was characterized by a significantly higher content of total sugar, while ‘Peppermint’ was characterized by slightly higher total chlorophyll (Table 1). The content of total chlorophyll, ascorbic acid and dry soluble solids observed in the study was similar to that reported by Grzeszczyk and Jadczyk (2009).

Essential oil content in the species Mentha piperita (L.) determined by other authors also varied. Adaszyńska et al. (2013) determined peppermint oil in
the tested peppermint ‘Asia’ at the level of 2.1%. The highest content (0.77%) of essential oil was determined in the fresh leaves of the cv. ‘Peppermint’. In the leaves of cv. ‘Krasnodarskaja’, essential oil accounted for 0.70% of fresh mass. The results obtained in this study are comparable to those determined by other researchers. The mint leaves from conventional farming have mint oil content at the level 0.75–1.89% (Newerli-Guz, Kobyłanska, 2013). The results of essential oil content in the dried herbs are given in Figure 1. The drying method affected the essential oil content of dried peppermint leaves. However, the changes between the investigated peppermint cultivars were different. Larger quantities of essential oil in dry mass remained in both types of peppermint leaves, which had been dried by active ventilation, vacuum, convection and sublimation. The lowest essential oil content remained when leaves were dried with high-frequency waves – 0.08% d.m. (cv. ‘Peppermint’).

Table 1. The quantitative chemical composition of fresh peppermint leaves

<table>
<thead>
<tr>
<th>Fresh leaves</th>
<th>Total sugar</th>
<th>Dry soluble solids</th>
<th>Ascorbic acid mg 100 g⁻¹</th>
<th>Carotenoids mg 100 g⁻¹</th>
<th>Chlorophyll mg 100 g⁻¹</th>
<th>Nitrates mg 100 g⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>‘Peppermint’</td>
<td>2.06</td>
<td>10.6</td>
<td>22.43</td>
<td>5.7</td>
<td>169.0*</td>
<td>83.0</td>
</tr>
<tr>
<td>‘Krasnodarskaja’</td>
<td>2.26*</td>
<td>10.6</td>
<td>21.53</td>
<td>5.8</td>
<td>139.0</td>
<td>82.8</td>
</tr>
<tr>
<td>LSDₐ</td>
<td>0.029</td>
<td>0.069</td>
<td>1.185</td>
<td>0.143</td>
<td>1.50</td>
<td>2.364</td>
</tr>
</tbody>
</table>

Note. * – values are significantly different (P ≤ 0.05) by Fisher’s LSD test.

Chlorophyll is a group of compounds responsible for colour (such as green) intensity of plants. In the case of degradation, there is also the deterioration of colour, flavour and nutritional value. The results show that drying has a significant impact on chlorophyll degradation; however, the level of its losses is different for each herb species (Śledź, Witrowa-Rajchert, 2012). Plant properties and drying methods had influence on the stability of chlorophyll a and b in peppermint leaves. Larger quantities of pigments remained in dried leaves of the cv. ‘Peppermint’ – 302.0–715.0 mg 100 g⁻¹ – and most of them are found in sublimation/lyophilisation dried leaves (Fig. 2). Chemical composition of chlorophyll a and b is very similar, but they differ in colour. Chlorophyll a is bluish-green, chlorophyll b – yellow-green. Chlorophyll a is more widespread and important, because without it there is no photosynthesis. Plants have less chlorophyll b than chlorophyll a, and the latter varies more in mints (Grzeszczuk, Jadczak, 2009). As noted before, the stability of chlorophyll a and b in peppermint leaves depends on drying methods. Depending on the drying method, greater chlorophyll differences between peppermints were in sublimation dried leaves. In the peppermint cultivar from Poland, chlorophyll a amount was 419.0 mg 100 g⁻¹, while cv. ‘Krasnodarskaja’ leaves had twice as low amount – 189.0 mg 100 g⁻¹. The results of chlorophyll b amount also differ – in the leaves of Polish peppermint there was 2.3 times more chlorophyll b than in those of cv. ‘Krasnodarskaja’. When peppermint cultivar from Poland was dried by other methods, chlorophyll a averaged 238.0 mg 100 g⁻¹, chlorophyll b – 164.0 mg 100 g⁻¹. Meanwhile in dried leaves of cv. ‘Krasnodarskaja’ chlorophyll a averaged 172.0 mg 100 g⁻¹, chlorophyll b – 117.0 mg 100 g⁻¹.

The ratio between chlorophyll a molecules and chlorophyll b molecule is 3:1 (Grzeszczuk, Jadczak, 2009), sometimes this ratio can be greater. In investigated fresh peppermint leaves, chlorophyll a and b ratio was 1.44 (cv. ‘Krasnodarskaja’) and 1.35 (cv. ‘Peppermint’). In dried peppermint, this rate varied in all samples (Fig. 3). The greatest chlorophyll a to b ratio (1.57) was found in cv. ‘Krasnodarskaja’ leaves that were dried in vacuum at 30 ± 1.0°C temperature. In comparison with fresh leaves, in dried leaves of cv. ‘Krasnodarskaja’ chlorophyll a and b ratio was approximately 2.6%
smaller. However, dried leaves of cv. ‘Peppermint’ had higher changes in chlorophyll a and b. That was seen by the ratio changes in both chlorophylls, the ratio changed from 1.41 to 1.50 and in comparison with fresh leaves, depending on drying method, it varied from 4.6% (active ventilation) to 11.1% (microwave). Similar chlorophyll a and b differences were in both investigated peppermint cultivars, which were dried naturally (20 ± 2.0°C) and by convection (40 ± 2.0°C). Higher changes between ratios were in sublimated peppermint leaves (Fig. 3).

Results from colour analysis of fresh and dried material are presented in Table 2. The dried cv. ‘Peppermint’ leaves, in which chlorophyll retention was the highest, were characterised by the greatest colour stability. Fresh and dried leaves of cv. ‘Peppermint’ had the lowest L* value (from 22.61 to 35.24), particularly when dried in sublimation, and were significantly (P ≤ 0.05) different from the dried cv. ‘Krasnodarska’ samples (Table 2). Dried samples of cv. ‘Krasnodarska’ peppermint had the lowest brightness L* value of 31.97, when leaves were dried using convection method. It was found that lightness was not significantly affected by the drying method (Krokida, Maroulis, 1999).

The change of green colour was largely related to the loss of chlorophyll. It was found that greater retention of chlorophyll a was accompanied by lesser change of green colour in the direction of red (Śledź, Witrowa-Rajchert, 2012). The biggest changes in greenness a* value were in both peppermint samples, that had been dried by microwave (P ≤ 0.05). The positive value of greenness a* in the cv. ‘Peppermint’ (1.92) shows that the samples dried by a microwave had a reddish tint.

The yellowness b* values differed for dried and fresh samples. Active ventilation, vacuum and sublimation dried samples were statistically different from convective, infrared and microwave dried samples. Other authors also obtained similar results of colour changes for peppermint (Arslan et al., 2010). Fresh and dried leaves of the cv. ‘Peppermint’ had the lowest yellowness b* values (from 9.35 to 17.00) and were significantly different from other drying methods.

<table>
<thead>
<tr>
<th>Drying method</th>
<th>Peppermint leaves</th>
<th>lightness L*</th>
<th>red/green a*</th>
<th>yellow/blue b*</th>
<th>chroma C*</th>
<th>hue angle h°</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fresh leaves</td>
<td>‘Krasnodarska’</td>
<td>35.98 ± 1.75</td>
<td>-7.92 ± 0.77</td>
<td>15.54 ± 3.14</td>
<td>17.47 ± 3.01</td>
<td>117.4 ± 3.99</td>
</tr>
<tr>
<td></td>
<td>‘Peppermint’</td>
<td>31.02 ± 2.41</td>
<td>-5.96 ± 0.58</td>
<td>9.35 ± 0.57</td>
<td>11.09 ± 0.77</td>
<td>122.5 ± 1.35</td>
</tr>
<tr>
<td>Active ventilation (A)</td>
<td>‘Krasnodarska’</td>
<td>40.60 ± 4.54</td>
<td>-4.29 ± 2.94</td>
<td>19.84 ± 4.61</td>
<td>20.40 ± 4.68</td>
<td>100.8 ± 7.57</td>
</tr>
<tr>
<td></td>
<td>‘Peppermint’</td>
<td>33.41 ± 3.18</td>
<td>-3.78 ± 1.10</td>
<td>14.32 ± 1.82</td>
<td>14.83 ± 2.02</td>
<td>104.6 ± 2.61</td>
</tr>
<tr>
<td>Convection (C)</td>
<td>‘Krasnodarska’</td>
<td>31.97 ± 0.96</td>
<td>-0.22 ± 1.59</td>
<td>12.97 ± 2.36</td>
<td>13.04 ± 2.42</td>
<td>90.35 ± 6.44</td>
</tr>
<tr>
<td></td>
<td>‘Peppermint’</td>
<td>29.53 ± 3.38</td>
<td>-1.23 ± 0.26</td>
<td>11.20 ± 0.99</td>
<td>11.27 ± 0.99</td>
<td>96.3 ± 1.39</td>
</tr>
<tr>
<td>Infrared (I)</td>
<td>‘Krasnodarska’</td>
<td>34.34 ± 1.23</td>
<td>-2.76 ± 2.49</td>
<td>15.30 ± 2.81</td>
<td>15.66 ± 3.14</td>
<td>98.99 ± 7.97</td>
</tr>
<tr>
<td></td>
<td>‘Peppermint’</td>
<td>31.89 ± 1.92</td>
<td>-0.67 ± 1.28</td>
<td>13.50 ± 1.88</td>
<td>13.55 ± 1.96</td>
<td>92.43 ± 4.66</td>
</tr>
<tr>
<td>Microwave (M)</td>
<td>‘Krasnodarska’</td>
<td>37.90 ± 5.77</td>
<td>-1.64 ± 1.24</td>
<td>17.42 ± 2.25</td>
<td>17.52 ± 2.31</td>
<td>95.23 ± 3.57</td>
</tr>
<tr>
<td></td>
<td>‘Peppermint’</td>
<td>22.61 ± 4.78</td>
<td>1.92 ± 1.44</td>
<td>10.78 ± 3.76</td>
<td>11.09 ± 3.52</td>
<td>77.42 ± 10.0</td>
</tr>
<tr>
<td>Sublimation (S)</td>
<td>‘Krasnodarska’</td>
<td>39.80 ± 5.31</td>
<td>-6.41 ± 0.94</td>
<td>18.08 ± 1.14</td>
<td>19.19 ± 1.37</td>
<td>109.4 ± 1.63</td>
</tr>
<tr>
<td></td>
<td>‘Peppermint’</td>
<td>35.24 ± 2.65</td>
<td>-5.94 ± 0.73</td>
<td>17.0 ± 1.28</td>
<td>18.02 ± 1.30</td>
<td>109.3 ± 2.24</td>
</tr>
<tr>
<td>Vacuum (V)</td>
<td>‘Krasnodarska’</td>
<td>38.76 ± 2.77</td>
<td>-7.73 ± 0.41</td>
<td>21.86 ± 3.48</td>
<td>23.20 ± 3.38</td>
<td>109.8 ± 2.32</td>
</tr>
<tr>
<td></td>
<td>‘Peppermint’</td>
<td>35.02 ± 3.15</td>
<td>-5.85 ± 0.61</td>
<td>15.99 ± 1.56</td>
<td>17.03 ± 1.63</td>
<td>110.1 ± 1.35</td>
</tr>
</tbody>
</table>

Note. Means ± SD are given in the Table; significantly (P < 0.05) outstanding parameters are shown in bold.
Convective dried samples had the lowest yellowness $b^*$ value changes as compared with fresh peppermint samples. Significantly biggest yellowness $b^*$ and chroma $C^*$ value changes were observed in sublimation and vacuum dried samples. Chroma values of the microwave dried peppermint leaves did not decrease. In turn, good retention of colour also was observed in nettle (Alibas, 2010) and coriander (Sarimeseli, 2011). The colour saturation or chroma ($C^*$) parameter permits the determination of the strength of response to the hue of a colour in a qualitative manner, through interpretation of its intensity and depth (Gozdecka, 2006). Among dried and fresh peppermint, higher chroma $C^*$ values were in cv. ‘Krasnodarskaja’ samples. Between both investigated fresh peppermint plant samples hue angle $h^\circ$ did not differ significantly, but drying method had an impact on the change. In comparison with fresh leaves, significantly large hue angle ($h^\circ$) changes were in infrared, convective and microwave dried samples, mostly in the cv. ‘Peppermint’ leaves.

Conclusions

1. The investigated fresh leaves of peppermint ($Mentha \times piperita$ L.) plants mostly differed by the amount of total sugar, chlorophyll and essential oil.

2. Among the tested plants, the highest essential oil retention was in variously dried cv. ‘Krasnodarskaja’ peppermint leaves. Larger amount of pigments remained in the dried leaves of the cv. ‘Peppermint’, but it had bigger chlorophyll $a$ to $b$ ratio changes.

3. More essential oil remained in the leaves of both peppermint cultivars, which had been dried by active ventilation at a temperature of $22 \pm 2°C$, in vacuum ($30 \pm 2°C$), in convective dryer ($40 \pm 2°C$) and by sublimation/lyophilisation ($-85°C$). Dried by sublimation/lyophilisation and in vacuum leaves of the cvs. ‘Peppermint’ and ‘Krasnodarskaja’ had higher chlorophyll content.

4. The changes of colour characteristics of dried leaves depended on peppermint properties and drying techniques. The results show that dried leaves of the cv. ‘Peppermint’ had the lowest brightness $L^*$ value and the lowest yellowness $b^*$ values. The smallest change in colour characteristics was observed in sublimation and vacuum dried leaves in the investigated cultivars.

5. The microwave drying method ensured retention of chlorophyll and colour, but in dry peppermint leaves left a minimum content of essential oil. Minimum essential oil content, chlorophyll $a$ and $b$ content, and the smallest change in colour were observed in peppermint leaves that had been dried by sublimation, convection and vacuum.

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Santrauka

Tirtos dvi pipirmėtės veislės: ‘Krasnodarskaia’ ir ‘Peppermint’. Pipirmėtės (Mentha × piperita L.) augalai buvo džiovinti įvairiais būdais. Džiovinintų ir šviežių pipirmėčių tyrimai atlikti taikant biochemijos ir technologijos metodus. Eterniniai aliejai išgauti naudojant garų distiliaciją, chlorofilas a ir b nustatytas spektrofotometriškai pagal Vernono metodiką. Nustatyta, jog daugiausia eterninių aliejų (0,77 %) ir chlorofilų (169,0 mg 100 g⁻¹) turėjo veislės ‘Peppermint’ pipirmėtės. Chlorofilų a ir b santykius buvo nevienodos šviežiuose augaluose ir siekė 1,35 veislės ‘Peppermint’ bei 1,44 veislės ‘Krasnodarskaia’ pipirmėtėse. Pipirmėčių lapai džiovinti taikant aktyviąją ventiliaciją, konvektinį, infraraudonųjų spindulių, vakuuminių, mikrobanų ir sublimacinį džiovinimą būdą. Džiovinintų augalų kokybė priklauso nuo augalo savybės, ir nuo džiovinimo būdo. Didžiausias kiekis (0,64–0,68 % sausos masės) eterninių aliejų nustatytas įvairiai džiovinant veislės ‘Krasnodarskaia’ pipirmėčių lapus. Mažiausias kiekis (0,08 ir 0,065% sausos masės) eterninių aliejų nustatytas augalus džiovinant mikrobangomis. Didžiausias kiekis (715,0 mg 100 g⁻¹ sausos masės) chlorofilų buvo sublimuotose veislėse ‘Peppermint’ lapuose. Nėpapaisant džiovinimo būdo, didžiausiai chlorofilų a ir b santykio skirtumai nustatyti džiovinuose veislėse ‘Krasnodarskaia’ pipirmėčių lapuose. Švieži ir išdžiovinęs eterniniai aliejai pipirmėčių pipirmėčių lapuose. Reikšmingiai žodžiai: chlorofilas a, chlorofilas b, džiovinimo būdai, eterninis aliejus, pipirmėtė.