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EFFECT OF THE PEPPERMINT (*MENTHA PIPERITA* L.) COMPACTION PROCESS ON THE CONTENT OF ESSENTIAL OILS*

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ABSTRACT

Herbs have been of great interest in recent years. Peppermint is one of the most popular species in Poland used in the form of herbal tea. The objective of the research was to determine and compare the content of essential oils obtained from peppermint in a loose state and after producing agglomerate therefrom. The scope of the research covered compression of grated peppermint and fractions separated from it. Fractions were separated with the use of a sieve with 2.5 mm square meshes and compressed with a hydraulic press with a closed matrix under the pressure of 193.86 MPa. Before and after the agglomeration process, density of the investigated material and the content of essential oils were determined according to the pharmaceutical requirements by a distillation method on Clevenger's apparatuses. The lowest content of essential oils was in case of the fraction of grated peppermint which did not go through the sieve with 2.5 square meshes. Compression of this material to the level of $1.038 \text{ g}\cdot\text{cm}^{-3}$ resulted in the reduction of oil content by approximately 9%. In case of the remaining fractions, no losses of oil as a result of compression were reported.

Introduction

According to the World Health Organization, almost 80% of people around the globe use herbal products for pro-health purposes (Khan and Smillie, 2012). Poland is a country with great potential regarding both herbs production and processing. Presently, approximately 70 species of herbs and peppermint covers the greatest acreage, namely approximately 4 thousand ha. In our country, 65% of dried herbal raw material is used for production of tea (Seidler-Łożykowska, 2009). Peppermint (*Mentha piperita* L.) is a very popular and valued herb in Poland and in many regions of the world. It is widely used both in the food and cosmetic industry as well as in pharmacy. It is used, inter alia, for indigestion, bloating, enteritis, gastritis, intestinal colic, cramps, stimulation of bile production (McKay and Blumberg, 2006). Peppermint is one of the most popular herbal teas. Herbal teas which

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are available on the market usually take the loose form (leaves) or the portioned one in bags – a fix type. A part of the obtained plant raw materials used later in the pharmaceutical and food industry may be subjected to the granulating process after harvesting and drying. The use of this process is popular in the Chinese medicine. However, there is no explicit literature data comparing the efficiency of granules to traditional herbal teas (Luo et al., 2012). Also dried and ground herbs may be agglomerated through tablet compression (Weathers and Towler, 2014). In the granulation process, material is subjected to mechanical and thermal factors as well as pressure, which in case of such raw material as herbs, may have a negative impact on the content of active substances. As the research show, volatile components of essential oils (Argyropoulos and Müller, 2014; Rohloff et al., 2005), which in peppermint decide on their therapeutic properties are the most sensitive to a thermal factor (Farmakopea, 2011). Absence of information in this scope in the available literature was an inspiration to undertake own research.

The objective of the research was to determine and compare the content of essential oils obtained from grated peppermint in a loose state and after producing agglomerate therefrom.

Research methodology

Research material was in the form of grated peppermint (*Mentha piperita* L.) according to PN-85/R-87017. Its moisture was determined with an oven-dry method and then divided into two fractions with the use of a sieve with a 2.5 mm square mesh. For the grated herb and fractions separated therefrom, an initial density of material in a bulk state was determined according to PN-ISO 7971-2:1998 with the use of grain densimeter RP T 01 77 with the volume of 1 dm³. The research was carried out in three iterations for each combination. Then, also in three iterations for each fraction, the content of essential oils was determined with a two-hour hydrodistillation method on Clevenger type apparatuses according to the guidelines of Farmakopea Polska (2011). Further, separated fractions of raw material were compressed with the use of a hydraulic press to the moment it reached the power of 78.46 kN which was equal to obtaining the pressure of 193.86 MPa. A closed matrix with a diameter of a chamber of 22.1 mm was used, pouring there each time the same previously weighted portion of raw material. For the obtained agglomerate its density was determined after 48 hours of storing from the following formula:

$$\rho = \frac{m}{V} \quad (1)$$

where:

- ρ – density,
- m – mass, (g)
- V – volume, (cm³)

A diameter and length of the produced agglomerate was measured with the use of a caliper with a precision to 0.1 mm.

Moreover, a degree of compression of the agglomerate according to the methodology presented by Skonecki et al., (2013) was defined acc. to the following formula:

$$S_{za} = \rho_{al} \cdot \rho_n^{-1} \quad (2)$$

where:

- S_{za} – compression degree of agglomerate
- ρ_{al} – density of agglomerate after 48 hours of storing, ($\text{g}\cdot\text{cm}^{-3}$)
- ρ_n – initial density of material in the compression chamber (density in a bulk state), ($\text{g}\cdot\text{cm}^{-3}$)

After the agglomeration process, using the compressed material of particular fractions, the content of essential oils was investigated, according to pharmaceutical guidelines, acting analogically as in case of loose raw material.

A statistical analysis of the obtained results was carried out with the use of Statistica 9 software; analysis of variance was made and after showing differences between the considered variables, Duncan test was carried out at the level of significance of $p=0.05$.

Research results and their analysis

All tests were performed on raw material with the same moisture of 11%, which is a top standard, admissible in pharmacy for this species (Farmakopea Polska, 2011). Results of the research of the content of essential oils which occur in loose herbs and extracted fractions before and after the compression process were placed in table 1. Average amounts of oils were within the range of $2.08\text{-}2.62 \text{ ml}\cdot 100\text{g}^{-1}$. In each case the amount of essential oils was within pharmaceutical norms designed for this species (Farmakopea Polska, 2011). The lowest amount of oils was separated from the fraction of raw material exceeding 2.5 mm. And only in this case, its slight decrease after the compression process was determined, at the average of $0.21 \text{ ml}\cdot 100\text{g}^{-1}$. It should be emphasised that in this combination, the highest degree of compression of the agglomerate was reported (Table 2). Most probably in this case, under the influence of great impact, the structure of secreting tissues, which collects oils, was damaged and small amount of oil was lost. In peppermint, oil is stored in special glandular hairs which are located on the surface of leaves (Shanker, 1999). In case of raw material with a lower granulation degree, this phenomenon occurred earlier during grinding. Essential oils are mixtures of volatile substances (Chen et al., 2012). Thus, losses of oil as an effect of e.g. high temperatures of drying are common. Sarosi et al. (2013) in the research carried out with various options of drying thyme, observed its losses exceeding 50% in the temperature of 50°C in comparison to the combination lower by 10°C . In the presented results of own research, no statistically significant differences were reported between the remaining, obtained values of essential oils; they were included into a common homogeneous group.

Table 1
Average content of essential oils in peppermint and separated fractions (ml) before and after the compression process per 100 g of raw material

| Fraction | Content of essential oils preceding the agglomeration process (SD) | Content of essential oils after the agglomeration process (SD) |
|--------------------------------|--|--|
| Grated herb without separation | 2.55± 0.087 a* | 2.62± 0.076 a* |
| > 2.5 mm | 2.29± 0.063 b* | 2.08± 0.043 c* |
| < 2.5 mm | 2.75± 0.087 a* | 2.55± 0.229 a* |

*a, b, c – homogeneous groups acc. to Duncan test; SD – standard deviation

Results of measurements of bulk density of loose herbs and those subjected to the compression process with the use of a hydraulic press presented in table 2. Average bulk density of peppermint and fractions obtained therefrom was within 0.082-0.168 g·cm⁻³. The lowest one reported for the fraction above 2.5 mm, whereas the highest for the fraction below 2.5 mm. Bulk density of grated peppermint assumed mean values, which seems reasonable since it was composed of both bigger and finer fragments of peppermint leaves and stalks. Considerably higher densities were reported for the agglomerate produced from peppermint. The highest values of this coefficient were reported for the fraction below 2.5 mm and non-separated herb, respectively 1.096 and 1.09 g·cm⁻³; they were included into a common uniform group. A high participation of overground raw material affected high values of bulk density of non-separated herbs. Carone et al. (2010) when investigating the compression process of olive tree waste found out that the density of the obtained granulate increases when the raw material fraction decreases. Slightly lower values of this coefficient, statistically proved, were determined for the agglomerate produced from the fraction above 2.5 mm and it was at the average 1.038 g·cm⁻³.

Table 2
Selected physical parameters of herbs and separated fractions from peppermint before and after the compression process

| Fraction | Bulk density (g·cm ⁻³) SD | Density of agglomerate (g·cm ⁻³) SD | Degree of agglomerate compression |
|--------------------------------|---------------------------------------|---|-----------------------------------|
| Grated herb without separation | 0.120 ± 0.00 b* | 1.090 ± 0.009 A* | 9.08 |
| > 2.5 mm | 0.082 ± 0.003 c* | 1.038 ± 0.003 B* | 12.66 |
| < 2.5 mm | 0.168 ± 0.003 a* | 1.096 ± 0.005 A* | 6.52 |

*a, b, c – homogeneous groups for bulk density acc. to Duncan test, *A, B, C – homogeneous groups for the produced agglomerate acc. to Duncan test, SD – standard deviation

Results presented in this paper should be treated as initial; they are first in the entire planned cycle concerning the problem of agglomeration of herbal raw material.

Conclusions

Based on the research, which was carried out and based on the obtained results, the following conclusions can be drawn:

1. The lowest content of essential oils was in case of the fraction of grated peppermint which did not go through the sieve with 2.5 mm square meshes. Compression of this material to the level of $1.038 \text{ g}\cdot\text{cm}^{-3}$ resulted in the reduction of oil content by approximately 9%. In case of the remaining fractions no losses of oil as a result of compression were reported.
2. The highest density in the bulk state was reported for the fraction below 2.5 mm. It was characterized also with the highest density of agglomerate after taking out from the compression chamber, similarly to non-fractionated grated herb.

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WPLYW PROCESU ZAGĘSZCZANIA ZIELA MIĘTY PIEPRZOWEJ (*MENTHA PIPERITA* L.) NA ZAWARTOŚĆ OLEJKÓW ETERYCZNYCH

Streszczenie. W ostatnich latach obserwuje się duże zainteresowanie roślinami zielarskimi. Jednym z popularniejszych gatunków w Polsce wykorzystywanych w postaci naparów jest mięta pieprzowa. Celem prowadzonych badań było określenie i porównanie zawartości olejków eterycznych pozyskiwanych z ziela mięty pieprzowej w stanie luźnym i po wytworzeniu z niego aglomeratu. Zakres prowadzonych badań obejmował zagęszczanie otartego ziela mięty oraz wydzielonych z niego frakcji. Frakcje wydzielono za pomocą sita o boku oczka kwadratowego 2,5 mm i zagęszczano za pomocą prasy hydraulicznej o matrycy zamkniętej pod ciśnieniem 193,86 MPa. Przed procesem aglomeracji, a także po jego zakończeniu ustalono gęstość badanego materiału oraz zawartość olejków eterycznych, zgodnie z wymaganiami farmaceutycznymi, metodą destylacji na aparatach Clevengera. Najmniejszą zawartością olejków eterycznych charakteryzowała się frakcja otartego ziela mięty pieprzowej nieprzesiewającego się przez sito o boku oczka kwadratowego 2,5 mm. Zagęszczenie tego materiału do poziomu $1,038 \text{ g}\cdot\text{cm}^{-3}$ spowodowało obniżenie zawartości olejku o około 9%. W przypadku pozostałych frakcji nie stwierdzono strat olejku w wyniku zagęszczania.

Słowa kluczowe: olejki eteryczne, mięta pieprzowa, aglomeracja