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FULL PAPER

Microencapsulation of corn mint (*Mentha arvensis* L.) essential oil using spray-drying technology

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Abstract

The objective of this study was to investigate the factors affecting the microencapsulation process of corn mint (*Mentha arvensis* L.) essential oil using the spray drying method. Different impact factors were evaluated including concentration of maltodextrin (20-30%), concentration of essential oils (0.5-2.0%), homogenization method, inlet temperature (130-150°C), and feed flow rate (4 -10 mL/min). The suitable conditions were the wall material concentration of 25% (w/w), essential oil concentration of 1.5% (w/w), using rotor-stator blend, the temperature of 140°C, and feed flow rate of 8 mL/min. The microencapsulation efficiency, the microencapsulation yield and spray-drying yield were 98.9%, 68.6%, and 84.8%, respectively.

1. Introduction

Mentha arvensis L. (MaL) is a perennial grass that belongs to the *Lamiaceae* family and includes 42 species, 15 hybrids, and hundreds of subspecies, varieties, and cultivars. The genus Mentha includes 25 to 30 species of growth in temperate regions of Eurasia, Australia and South Africa (Johnson *et al.,* 2011). MaL has a body from 10 to 60-70 cm tall, can be up to 1 m tall, hairy on the body. Leaves opposite, ovate or oblong, margins serrated. The flowers were ring-shaped in the interstitium, the petals are purple or pale pink, sometimes white. MaL essential oil (EO) commercially known as corn mint essential oil (CEO), contains main Menthol (>70%). In addition, it also contains menthone, methyl acetate and piperitone. Menthol was very important because of its applications in various areas of the pharmaceutical industry (Makkar *et al.,* 2018). CEO is widely used in fields such as cough medicine (helping to improve irritable bowel symptoms), and cosmetic products including fragrance, shampoo, toothpaste (Verma *et al.,* 2009). EO has some disadvantages such as evaporation, degradation, and oxidation (Cowan, 1999). To overcome the above disadvantages, the CEO was encapsulated for the preservation.

The encapsulation could be defined as a process of encapsulating one substance in another. Encapsulation can be classified into two types based on the size of the microencapsulated particles those were microencapsulation and nano-encapsulation. There are different methods of encapsulation such as spray-drying, spray cooling, extrusion, coacervation, lyophilization and emulsification (Silva *et al.,* 2014). Spray drying is a commonly used technique because of its outstanding advantages. Spray drying can be described as a simple process - an active-stage drying process. Spray drying fluid can be a solution, emulsion or suspension containing core and wall material after the drying process which produces microcapsules filled with aroma or flavour oils. More than that, the equipment is simple, effective, has a low moisture content, uniform product size. Therefore, spray-drying is used to encapsulate flavours, lipids and pigments (Martins *et al.,* 2014).

The recent research presents that the effect of different commercial starch materials on the flavour retention of the EO of peppermint (*Mentha piperita* L.) during spray-drying and storage was evaluated. The obtained results revealed that the emulsification and encapsulation efficiencies of peppermint EO were higher for all *n*-octenyl succinic anhydride (OSAN)-modified starches as compared to those of hydrolyzed starches (dextrins) (Baranauskienė *et al.,* 2007). In one other study, microencapsulation of L-menthol by spray drying, using gum arabic (GA) and modified starch (CAPSUL, HI-CAP 100) making capsule material. The results showed higher L-menthol retention as the initial solid concentration increased. HI-CAP 100, showed higher

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retention than other capsule materials. Furthermore, the release properties of L-menthol were also investigated (Soottitantawat *et al.,* 2005). In general, the above two studies had not identified the impact factors on the spray-drying process such as inlet temperature, and feed flow rate of the spray dryer. In addition, other parameters to evaluate the encapsulated powder have not been studied specifically.

The objective of this study was to find out the suitable conditions for the CEO spray-drying process. The characterisation of microencapsulated essential oil powder will be also determined including microencapsulation efficiency, microencapsulation efficiency and the yield of spray drying.

2. Materials and methods

2.1 Materials

Corn mint (*Mentha arvensis* L.) essential oil, purchased from Phuc Thinh Essential Oil Co., Ltd in Ho Chi Minh City, Viet Nam which was used as the core material. Maltodextrin from corn starch with DE 12 obtained from Qinhuangdao lihua starch co.,ltd was used as wall materials. Tween 80 (99%, Xilong, Shantou, China) was used as an emulsifier. *n*-pentane (99%, Duksan, Gyeonggi, Korea) was applied to regulate the microencapsulation efficiency.

2.2 Microencapsulation of essential oil by using spray-drying

Firstly, wall material consisting of maltodextrin at a concentration of 20- 30% (w/w) was mixed with 300 mL of distilled water under stirring. The mixture was stabilised at room temperature overnight. Secondly, the CEO as core material stated ratio 0.5-2% was mixed with wall material solution with a varying concentration between 20% and 25% and 30%. Subsequently, Tween 80 with an amount equal to 0.5% weight of essential oil under stirring at 6000 rpm for 20 mins, using a rotor-stator blender to form an emulsion. Thirdly, 450 mL of the solution is spray-dried. The solution was spray-dried with variable input temperatures of 130°C, 140°C and 150°C, feed flow rate of 4 mL/min, 6 mL/min, 8 mL/min and 10 mL/min. Finally, the essential oil powder was kept in the sealed glass bottle at room temperature for further analysis.

2.3 Characterisation of the powder

2.3.1 Determination of moisture content

The moisture content of the micro-powder was determined by an oven and dried at 105°C to constant weight. The moisture content of powder was determined by the method of Nhan *et al.* (2020) with modifications.

2.3.2 Determination of drying yield (DY)

The DY, or powder recovery, was defined as the mass of the obtained spray-dried product over the quantity of feed solution, calculated on a dry basis (Equation 1).

$$\mathrm{DY}\ (\%) = \frac{m_2 * (1-\omega_B)}{m_1 * X} \times 100 \qquad (1)$$

where m_1 is the mass of the feed emulsion (g), m_2 is the mass of the powder (g), X is the total solid content (g), and is ω_B the moisture content of the powder (%)

2.3.3 Determination of microencapsulation yields (MEY)

The microencapsulation yield (MEY), or oil retention, is defined as the quantity of essential oil existing in the obtained sprayed-drying product over the quantity of essential oil existing in the feed solution (Equation 2)

$$\mathrm{MEY}\ (\%) = \frac{m_P}{m_F} \times 100 \qquad (2)$$

Where M_P: the quantity of essential oil in the sprayed-drying product (calculated on the dry basis) (g) and m_F: the quantity of essential oil in the feed solution (calculated on the dry basis) (g)

2.3.4 Determination of microencapsulation efficiency (MEE) and surface oil (SO)

Microencapsulation efficiency (MEE) was determined as the quantity of encapsulated essential oil over the total essential oil existing in the obtained spray-dried product, calculated on the dry basis. The procedure for the determination of the mass of encapsulated essential oils is as in Equation 3.

$$\mathrm{MEE} = \frac{m_E}{m_P} \times 100 \qquad (3)$$

Where m_E is the quantity of encapsulated essential oil (calculated on the dry basis) (g) and m_P is the quantity of essential oil in the sprayed-drying product (calculated on the dry basis) (g)

The surface oil (SO) was determined as SO = (1 - MEE) x 100%.

2.3.5 Gas chromatography-mass spectrometry

A Gas Chromatography-Mass Spectrometry (GC-MS) was applied to investigate the composition of the essential oil samples prior to and after encapsulation. An amount of 25 µL of the sample of essential oil was mixed with 1.0 mL of n-hexane. The equipment used was GC Agilent 6890N coupled with MS 5973 inert with an HP5-MS column. The head column pressure was set to 9.3 psi. The GC-MS system operates under the following conditions: carrier gas was He; flow rate of 1.0 mL/min; the split ratio of 1:100; injection volume of 1.0 µL; injection temperature at 250°C; oven temperature progress included an initial hold at 50°C for 2 min, then increased by 2°C/min to 80°C, and increased by 5°C/min to 150°C, risen to 200°C at 10°C/min and finally risen to 300°C at 20°C/min for 5 mins (Nhan *et al.,* 2020).

3. Results and discussion

3.1 The chemical composition of the corn mint essential oil

The chemical composition of the CEO before and after spray-drying was determined by gas chromatography-mass spectrometry (GC-MS). The GC-MS spectrum of essential oil samples is shown in Figure 1.

Figure 1 illustrates the chemical composition of the CEO sample before microencapsulation. The results show that there are 12 main components accounting for about 88.3% of the total essential oil: Menthol (52.1%), Menthone (18.5%), Menthyl acetate (7.1%), Isomenthone (5.3%), Caryophyllene (1.8%), Eucalyptol (0.9%), *α*-Terpineol (0.8%), *δ*-Cadinene (0.3%), Neoisomenthol (0.3%), Pulegone (0.3%), *β*-Bourbonene (0.3%), Nerolidol (0.2%). Two peaks located at 18.9 mins and 20.04 mins were found to have the greatest intensities. Mass spectrometry proximates the molecular formula and weight of the two substances as m/z = 154 and m/z = 156, respectively. The study of Kalemba and Synowiec (2019) showed similar results when isolating from CEO, showing the main constituents of Menthol (above 60%), and Menthone (4-18%).

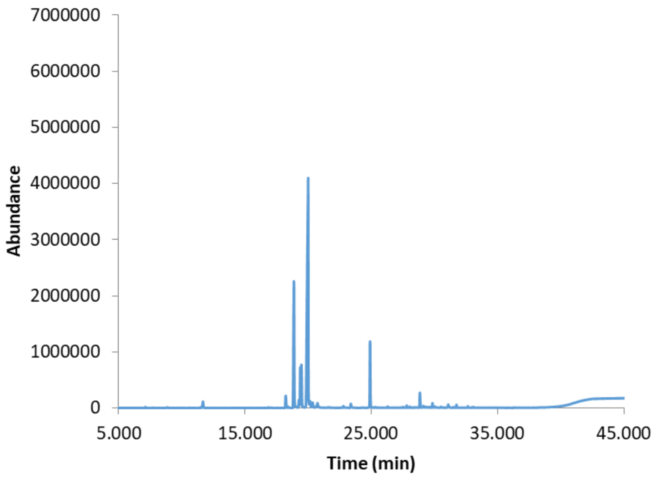

Figure 1. The spectrum of CEO samples

3.2 Effects wall material concentration

Figure 2 shows the effect of maltodextrin concentration on product specifications (MEY, MEE, SO). When increasing the maltodextrin concentration from 20% to 30%, MEY increased from 61% to 86.9%. Also, the MEE increased when maltodextrin concentration increased from 20 to 25% and decreased at 30%. The MEE reached the highest value of 95.4% at 25% of maltodextrin.

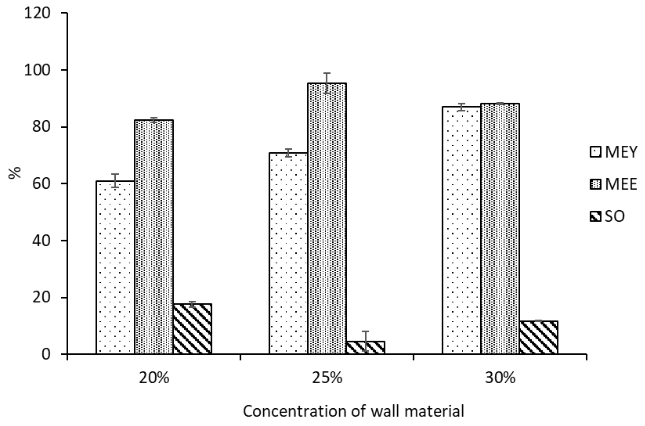

Figure 2. Effect wall material concentration on MEY, MEE, SO

ANOVA analysis's results showed that the concentration of wall material had a significant effect on MEY, MEE and SO values at 95% confidence. The least significant difference (LSD) classification test showed that there was a significant difference in maltodextrin concentration on MEY, MEE, and SO values. LSD of MEE value indicated that there was a difference between the 25% and 30% concentrations of maltodextrin compared to that of 20%, while there was no difference between the above concentrations (25% and 30%). This can be explained by the influence of the surface-active carbohydrates of maltodextrin, which are able to bind with other compounds of volatile matter during microencapsulation (Bae and Lee, 2008). Increasing the dry matter concentration of maltodextrin leads to an increase in the content of surface-active carbohydrates and volatile compounds in the product. Results of Xu *et al.* show the MEE value increased from 76.45% to 88.80% when maltodextrin concentration increased from 0% to 30% (Xu *et al*., 2020). Besides, the study by Sanchez-Reynoso presents that the MEE increased by 90.3% to 96.3% when the maltodextrin concentration was changed from 20% to 30% (Sanchez-Reynoso *et al*., 2017).

3.3 Effects of corn mint essential oil (CEO) concentration

Figure 3 shows the influence of CEO concentration on MEY, MEE, and SO values. Based on Figure 3, when increased the concentration of essential oil, the MEY value decreased from 66.8% to 54.2%. In contrast, the MEE tended to increase when increasing the CEO concentration. The MEE reached the highest value of 96.9% at the CEO concentration of 1.5%.

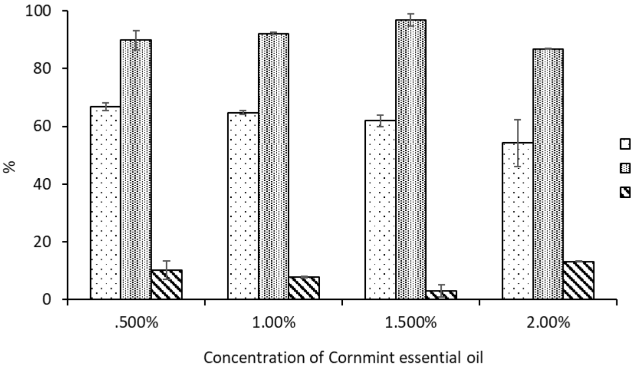

Figure 3. Effects of cornmint essential oil concentration on MEY, MEE, SO

The ANOVA analysis showed that the concentration of essential oil significantly affected the product parameters with 95% confidence. Further testing with LSD multiple tests showed that the MEE values were different at different CEO concentrations. LSD of MEE value indicated that there was a difference at the CEO concentrations of 0.5% and 2% compared with the 1.5%, while there was no difference at the above concentration (0.5% and 2%). The suitable concentration of CEO và maltodextrin for the microencapsulation process is 1.5% and 25%, respectively.

This is explained by the displacement of the hydroxyl groups (-OH) of the hydrophobic compounds out of the linkage network of maltodextrin due to the limitation in the concentration as well as the number of bonds that the coating forms. The results of Hogan *et al.* (2001) showed that the MEE value decreased from 89.2% to 18.8% when the ratio of soybean oil/sodium caseinate (wall material) increased from 0.25 to 0.3%. Besides, the results of Mohideen *et al.* (2015) showed that MEE decreased from 93.68% to 89.28% when the core material increased from 5% to 10%.

3.4 Effects of homogenization method

Figure 4 shows the effect of the homogenization method on the product's characterisations (MEY, MEE, SO). Based on Figure 4, when using the rotor-stator blend, MEY and MEE values were higher than using the ultrasonic homogenizers. The MEE reached the highest value of 98.9%, the MEY reached the highest value of 66.5% when using the rotor-stator blend. ANOVA analysis's result showed a significant difference between the two methods of homogenization at 95% confidence. Many previous studies also used a rotor-stator blender in the spray-drying process (Frascareli *et al.,* 2011; Fernandes *et al.,* 2013). One of the important steps in the encapsulation of essential oils by spray drying is the emulsion preparation including total solid concentration, oil content, viscosity, stability, droplet size and emulsification method. The encapsulation efficiency can be maximized with a higher stability of emulsion and other operating conditions of spray drying including inlet and outlet air temperatures, emulsion temperature, atomization conditions, drying air flow rate and humidity (Jafari *et al.,* 2008)

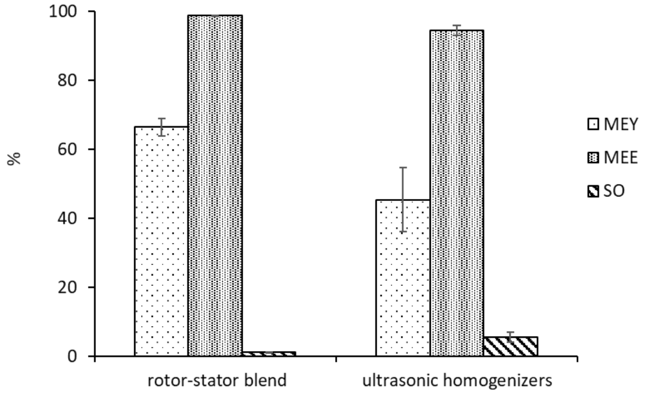

Figure 4. Effects of homogenization method on MEY, MEE, SO

3.5 Effects of inlet spray- drying temperature

Figure 5 shows the effect of inlet temperature on product characterisations (MEY, MEE, SO). Based on Figure 5, when increased the temperature, the microencapsulation yields increased from 56.2% to 62.9%. In contrast, the microencapsulation efficiency decreased when using a higher temperature. The maximum value of microencapsulation efficiency was 98.2% at 140°C.

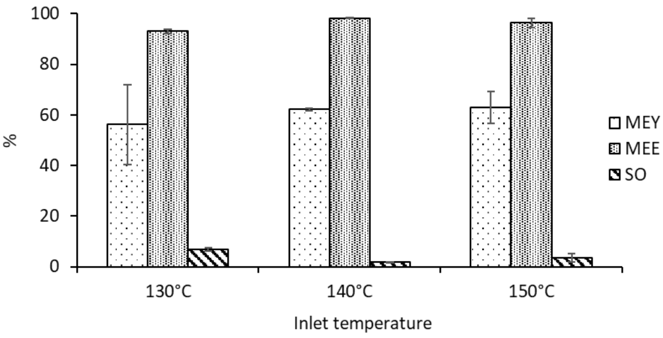

Figure 5. Effects of inlet drying temperature on MEY, MEE, SO

ANOVA analysis showed that temperature had a significant effect on product parameters with 95% confidence. The LSD classifier test showed that there was a difference in the MEE index between temperatures, and the MEY index did not. The inlet temperature of spray drying affects the moisture content and physicochemical properties of the powder. A significant increase in the hygroscopicity when the inlet drying temperature increases. The inlet temperature of 140°C was chosen for this experiment. This result is similar to that of Ferrari *et al.* (2012) that showed the optimal inlet air temperature was 140-150°C. The higher the drying temperature, the higher the water evaporation, which accelerates the formation of the outer shell, creating a strong film around the particles, effectively preventing oil loss. However, when the temperature is too high, it disrupts the balance between the evaporation rate of water and the film-forming process, leading to the formation of cracks that break the surrounding membrane. At the same time, the high temperature denatures the product during the drying process.

3.6 Effects of feeding flow rate of spray-drier

Figure 6 presents the effect of feeding flow rate on product parameters (MEY, MEE, SO). Based on Figure 6, when the input flow increased, the microencapsulation yields were changed. The highest MEY were 73.1% at 6 mL/min. In contrast, the MEE tended to increase from 91.3% to 98.9% when the feeding flow rate increased. The MEE reached the highest value of 98.9% at 8 mL/min.

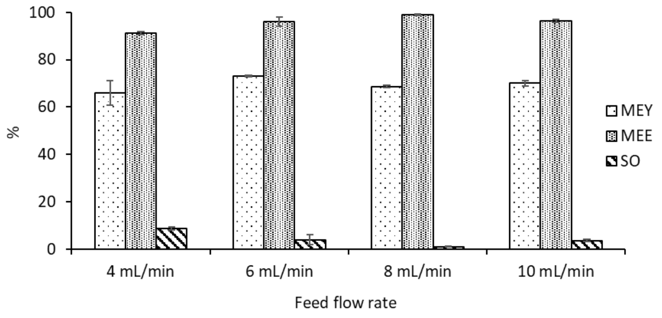

Figure 6. Effects of feeding flow rate on MEY, MEE, SO

The ANOVA analysis showed that the feed flow rate had a significant effect on the MEY, MEE and SO with 95% confidence. The LSD test showed that there was a difference in the MEE value between the feed flow rate. LSD of MEE value indicated that there was a difference between the feed flow rate of 6 mL/min and 8 mL/min compared with the 4 mL/min, while there was no difference between the above feed flow rate (6 mL/min and 8 mL/min).

The feed flow rate at 8 mL/min can help the emulsion process stably, save time, and stabilize moisture content stabilization. The inlet temperature of 140°C and the feed flow rate of 8 mL/min was also applied in the previous study on spray drying of Peppermint (*Mentha piperita* L.) essential oils by Cam *et al.* (2020).

3.7 Chemical composition of essential oil powder

Figure 7 and Table 1 present the composition of the CEO before and after the microencapsulation process. The result showed that there is a decrease in menthone content (from 18.5% to 14.1%) and an increase in menthol content (from 52.1% to 62.7%). The decrease in menthone concentration could be due to the volatility of monoterpenes. The disappearance of neoisomenthol, pulegone, *β*-Bourbonene and nerolidol which, after spray-drying, may cause by a high temperature of drying. Due to the reduction of the above components, the amount of menthol in the essential oil after spray drying increased significantly.

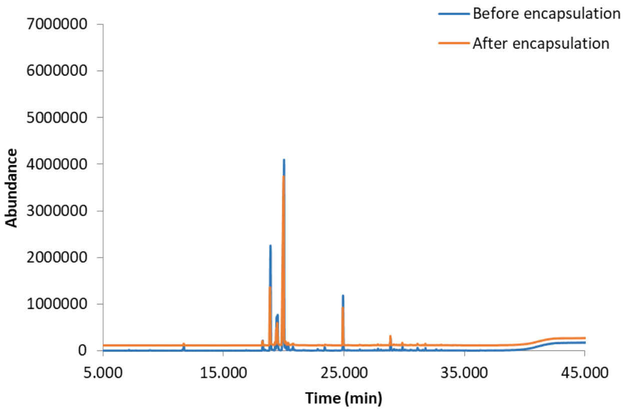

Figure 7. The spectrum of CEO samples before and after spray-drying

4. Conclusion

In this study, different impact factors on the spray-drying process of corn mint essential oil had been exploited including the concentration of wall material,

Table 1. Composition of CEO before and after microencapsulation

No.	Compounds	Before Encapsulation (%)	After Encapsulation (%)
1	Menthol	52.1	62.7
2	Menthone	18.5	14.1
3	Menthyl acetate	7.1	6.6
4	Isomenthone	5.3	3.5
5	Caryophyllene	1.8	1.7
6	Eucalyptol	0.9	0.4
7	*α*-Terpineol	0.8	0.7
8	*δ*-Cadinene	0.3	0.3
9	Neoisomenthol	0.3	-
10	Pulegone	0.3	-
11	*β*-Bourbonene	0.2	-
12	Nerolidol	0.2	-
13	Total	88.3	90.0

the concentration of essential oil, the homogenization method, the inlet temperature and feed flow rate. The suitable conditions for the microencapsulation process of corn mint essential oil using the spray drying technique was the concentration of wall material of 25% (w/w), the essential oil concentration of 1.5% (w/v) using the homogenization method, the inlet spray-drying temperature of 140°C and feed flow rate of 8 mL/min. The highest microencapsulation efficiency and microencapsulation yields were 98.9 % and 68.6%, respectively.

Conflict of interest

The authors declare no conflict of interest.

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References

Bae, E.K. and Lee, S.J. (2008). Microencapsulation of avocado oil by spray drying using whey protein and maltodextrin. *Journal of Microencapsulation*, 25(8), 549–560 https://doi.org/10.1080/02652040802075682

Baranauskienė, R., Bylaitė, E., Žukauskaitė, J. and Venskutonis, R.P. (2007). Flavor retention of peppermint (*Mentha piperita* L.) essential oil spray-dried in modified starches during encapsulation and storage. *Journal of Agricultural and Food Chemistry,* 55(8), 3027-3036. https://doi.org/10.1021/jf062508c

Cam, M., Basyigit, B., Alasalvar, H., Yilmaztekin, M., Ahhmed, A., Sagdic, O., Konca, Y. and Telci, I. (2020). Bioactive properties of powdered peppermint and spearmint extracts: Inhibition of key enzymes linked to hypertension and type 2 diabetes. *Food Bioscience,* 35(1), 100577. https://doi.org/10.1016/j.fbio.2020.100577

Cowan, M.M. (1999). Plant products as antimicrobial agents. *Clinical Microbiology Reviews*, 12(4), 564–582. https://doi.org/10.1128/CMR.12.4.564

Fernandes, R.V.B., Borges, S.V., Botrel, D.A, Silva, E.K., Costa, J.M.G. and Queiroz, F. (2013). Microencapsulation of rosemary essential oil: Characterization of particles. *Drying Technology,* 31 (11), 1245-1254. https://doi.org/10.1080/07373937.2013.785432

Ferrari, C.C., Germer, S.P.M. and Aguirre, J.M. (2012). Effects of spray-drying conditions on the physicochemical properties of blackberry powder. *Drying Technology,* 30(2), 154-163. https://doi.org/10.1080/07373937.2011.628429

Frascareli, E.C., Silva, V.M., Tonon R.V. and Hubinger, M.D. (2012). Effect of process conditions on the microencapsulation of coffee oil by spray drying. *Food and Bioproducts Processing,* 90(3), 413-424. https://doi.org/10.1016/j.fbp.2011.12.002

Hogan, S.A., McNamee, B.F., O'Riordan, E.D. and O'Sullivan, M. (2001). Emulsification and microencapsulation properties of sodium caseinate/carbohydrate blends. *International Dairy Journal*, 11 (3), 137-144. https://doi.org/10.1016/S0958-6946(01)00091-7

Jafari, S.M., Assadpoor, E., He, Y. and Bhandari, B. (2008). Encapsulation efficiency of food flavors and oils during spray drying. *Dry Technology,* 26(7), 816–835. https://doi.org/10.1080/07373930802135972

Johnson, M., Wesely, E.G., Kavitha, M.S. and Uma, V. (2011). Antibacterial activity of leaves and inter-nodal callus extracts of *Mentha arvensis* L. *Asian Pacific Journal of Tropical Medicine*, 4(3), 196-200. https://doi.org/10.1016/S1995-7645(11)60068-0

Kalemba, D. and Synowiec, A. (2019). Agrobiological interactions of essential oils of two menthol mints: *Mentha piperita* and *Mentha arvensis*. *Molecules*, 25 (1), 59. https://doi.org/10.3390/molecules25010059

Makkar, M.K., Sharma, S. and Kaur, H. (2018). Evaluation of *Mentha arvensis* essential oil and its major constituents for fungitoxicity. *Journal of Food Science and Technology,* 55(9), 3840-3844. https://doi.org/10.1007/s13197-018-3291-y

Martins, I.M., Barreiro, M.F., Coelho, M. and Rodrigues, A.E. (2014). Microencapsulation of essential oils with biodegradable polymeric carriers for cosmetic applications. *Chemical Engineering Journal,* 245(1), 191-200. https://doi.org/10.1016/j.cej.2014.02.024

Mohideen, F.W., Peter, J.S., Kevin Mis Solval, B. and David, J.B. (2015). Effects of Blueberry (*Vaccinium Corymbosum*) Juice on Lipid Oxidation during Spray Drying of Microencapsulated Menhaden Oil. *International Journal of Food Properties*, 18(5), 1139–1153. https://doi.org/10.1080/10942912.2013.877025

Nhan, N.P.T., Thanh, V.T, Cang, M.H, Lam, T.D., Huong, N.C. and Bach, L.G. (2020). Microencapsulation of lemongrass (*Cymbopogon citratus*) essential oil via spray drying: Effects of feed emulsion parameters. *Processes,* 8(1), 40. https://doi.org/10.3390/pr8010040

Sanchez-Reinoso, Z., Osori C. and Herrera, A. (2017). Effect of microencapsulation by spray drying on cocoa aroma compounds and physicochemical characterisation of microencapsulates. *Powder Technology*, 318(1), 110-119. https://doi.org/10.1016/j.powtec.2017.05.040

Silva, P.T., Fries, L.L.M., Menezes, C.R., Holkem, A.T., Schwan, C.L., Wigmann, E.F., Bastos, J.O. and Silva, C.B. (2014). Microencapsulation: Concepts, mechanisms, methods and some applications in food technology. *Ciência Rural*, 44(7), 1304-1311. https://doi.org/10.1590/0103-8478cr20130971

Verma, K., Pandeya, S.N., Singh, U.K., Gupta, S. and Prashant, P., Anurag and Bhardwaj, G. (2009). Synthesis and pharmacological activity of some substituted menthone semicarbazon e and thiosemicarbazone derivatives. *International Journal of Pharmaceutical Sciences and Nanotechnology,* 1 (4), 357-362. https://doi.org/10.37285/ijpsn.2008.1.4.7

Xu, S., Tang, Z., Liu, H., Wang, M., Sun, J., Song, Z., Cui, C., Sun, C., Liu, S., Wang, Z. and Yu, J. (2020). Microencapsulation of sea buckthorn (*Hippophae rhamnoides* L.) pulp oil by spray drying. *Food Science and Nutrition*, 8(11), 5785–5797. https://doi.org/10.1002/fsn3.1828