

Aroma retention and flavour release of peppermint essential oil encapsulated by spray-drying into food starch based matrices

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Introduction

The encapsulation of active components in powders has become a very attractive process in the last decades. Many sensitive ingredients such as flavours, anti-oxidants or nutraceuticals are lipid-based compounds, which exist in liquid form at room temperature. For these lipophilic substances the simplest means of encapsulation consists in emulsifying/dispersing the component in an aqueous solution. For use in a conventional tablet or capsule form for oral absorption for example, the emulsion may be incorporated into a solid form, i.e. powder. This can be performed by drying the aqueous emulsion such that, when solidifying the wall materials contained in the aqueous phase entrap dispersed oil drops (Turchiuli et al., 2005). The main purposes of microencapsulation is entrap sensitive ingredients, such as hydrophobic flavours, in solid carriers to increase their protection against oxygen, water and/or light, promote easier handling, reduce evaporation, control their release; controlled release can improve the effectiveness of food additives, broaden the application range and ensure optimal dosage (Pegg et al., 1999; Gouin, 2004; Reineccius, 2006).

This study was aimed to investigate the properties of several commercial food starch based matrices for the coating of the essential oil (EO) of peppermint (*Mentha piperita* L.) by spray-drying. The following objectives were raised in this study: (i) to determine the emulsification efficiency, retention of volatile flavour compounds and evaluate the efficiency of microencapsulation; (ii) to determine particle size distribution, moisture content and water activities (a_w) of spray-dried encapsulated products; (iii) to assess the changes in the composition of peppermint flavours taking place during processing; (iv) to measure the release of volatiles from different starches matrixes by dynamic headspace gas chromatography analysis; and (v) to determine the aroma release from microencapsulated peppermint oil powder products during storages at different water activities by static headspace gas chromatography.

Material and methods

Materials. Peppermint (*Mentha piperita* L.) EO (TŪB “Mėta”, Vilnius, Lithuania) was selected as core material. The following modified food starches matrices were used as encapsulating agents: HI-CAP 100 (chemically modified food starch refined from waxy maize), N-LOK (chemically modified food starch with corn syrup added), CAPSUL (chemically modified food starch derived from waxy maize), ENCAPSUL 855 (dextrin refined from tapioca and maize starch) and CRYSTAL TEX 627 (dextrine refined from tapioca starch) were kindly gifted by National Starch Group. CIEmCap 12633 (stabilized and acid-thinned instant waxy maize starch, N-OSA), CIEmCap 12634 (spray-dried waxy maize starch ester; starch sodium octenyl succinate) and CIEmCap 12635 (stabilized and acid-thinned instant waxy maize starch, N-OSA) – were supplied by Cerestar.

Preparation of microencapsulated flavours. The solutions of coating matrices (30% w/w) were prepared by dispersing dried powders in 40°C deionized water; after cooling were mixed overnight to enhance hydration. EO (15.25% w/w of matrix solids) was emulsified into the hydrated coating material. Homogenization was accomplished by Ultra Turrax Ika 25 homogenizer (Janke & Kunkel GmbH&Co, Germany) operating at 13500 rpm for 7 min. Emulsions were spray-dried in a Büchi 190 Mini Spray Dryer (Donau, Switzerland) under the following parameters: spray nozzle (inlet) temperature 200±10°C, outlet air temperature 120±10°C, pressure – 400 mm/H₂O.

Total and surface oil determination. Total oil content of the liquid emulsions and spray-dried microencapsulated products were determined in duplicate by distilling 10 g of liquid emulsion or encapsulated powder for 3 h in a Clevenger-type apparatus (AOAC). The volume of peppermint EO collected in the trap was multiplied by a density factor 0.8969 g/mL to calculate the weight of oil recovered from the sample. Surface oil was being washed from 10 g of spray-dried encapsulated powder for 4 h in a Soxhlet extraction apparatus by using pentane. One mL of pentane containing IS, undecane 0.3% (v/v) was added prior extraction; each extract was concentrated to ~10 mL at room temperature by Büchi rotary evaporator and then to a final volume of ~2 mL at room temperature under a stream of nitrogen. The compositions of pure oil, total oil, isolated from emulsions and processed products and surface oil (non entrapped into matrices) were analysed by GC and GC-MS.

Release of volatiles by dynamic headspace gas chromatography (DHS-ATD-GC-FID). 0.5 g of each encapsulated powder with surface oil removed was placed into 125 mL sample flask. The flask was placed in water bath of 25 °C temperature. The glass tops were connected to the nitrogen flow of 120 mL min⁻¹. One minute preflush was used to remove the volatiles accumulated in the headspace above the encapsulated product. Then sample was purged with timed intervals with nitrogen in order to trap released volatiles on Tenax traps (Perkin Elmer, CN, USA) packed with 225 mg Tenax GC (Tenax Chrompack, Bergen op Zoom, The Netherlands). Tenax tubes were changed every 2 min, and the total purge time was 30 min. Three replicates were carried out for each sample. 1 µL of IS, 0.3% (v/v) undecane in pentane, was injected on Tenax prior desorption. Trapped volatiles were desorbed from Tenax by thermal desorption device *Perkin Elmer ATD 400* under following parameters: desorption time 3 min, desorption temperature 250 °C, cold trap -30 °C, trap heating temperature 250 °C, hold for 1 min. After desorption, volatiles were directed through a heated transfer line and analyzed by GC.

Release of volatiles by static headspace analysis (SHS-GC-FID) at different water activities. 0.05 g of spray-dried and pentane washed encapsulated peppermint oil product (four replicates) was weighted into 2 mL HPLC bottle, which was placed into headspace 22,3 mL-vial over 2 mL of saturated salt solution to yield water activity $a_w = 0.43$ (K₂CO₃) and 0.75 (NaCl) at 25 °C, tightly capped and left for timed intervals of 0, 5, 30, 60, 240, 1440, 2880, 4320 and 6060 min at the 25 °C in order to recover volatiles released. After proper time period vials were transferred to automated headspace autosampler *Perkin Elmer HS 40 XL* and analysed by GC.

Gas chromatography (GC). Diluted in pentane pure EO, samples of retained after spray-drying total and surface oils (10 µL in 1 mL) were analysed on a *Fisons 8000* series GC (Fisons Instruments Inc., Rodano MI, Italy) equipped with a flame ionization detector (FID) and a DB-5 fused silica capillary column (polydimethylsiloxane, 5% phenyl, 50 m length, 0.32 mm i.d., 0.25 µm film thickness, J&W Scientific, Folsom, CA). The carrier gas was helium at a linear flow velocity of 43 cm s⁻¹ at 50 °C; the detector's temperature was 320 °C, the oven temperature was programmed from 50 °C (2 min) to 280 °C (hold 10 min) at the ratio of 5 °C min⁻¹. A split/splitless injector was used at 260 °C in split mode at a ratio of 1:5, the injection volume was 1 µL. The content of eluted compounds was expressed as a GC peak area percent; mean values were calculated from quadruplicate injections. The coefficient of variation is defined as the ratio of corresponding standard deviation (%RSD) to the average value from 4 replicate injections.

The peppermint volatiles trapped on Tenax tubes (DHS analysis) and then desorbed by thermal desorption device were directed through a heated transfer line to injection port of a *Hewlett Packard 5890* gas chromatograph (Hewlett-Packard, Wilmington, DE, USA), equipped with a flame ionization detector (FID) and a DB-5 fused silica capillary column (polydimethylsiloxane, 5% phenyl, 30 m length, 0.25 mm i.d., 1 µm film thickness). The carrier gas was helium at a flow rate of 1.1 mL min⁻¹ which was equivalent to 39 cm s⁻¹ volumetric flow at 60 °C; the detector's temperature was 300 °C, the oven temperature was programmed from 60 °C, without holding, to 295 °C (10 min hold) at the ratio of 5 °C min⁻¹.

One mL of headspace gas phase above sample (SHS analysis) was automatically withdrawn using a *Perkin Elmer HS 40 XL* autosampler and injected into *Perkin Elmer XL* gas chromatograph using injection times of 0.06 s. Autosampler was set up with following parameters: oven temperature 60 °C, time in oven 0 min, needle temperature 65 °C, transfer line temperature 75 °C, pressurization time 3 min, withdrawal time 0.50 s and GC cycle 23 min. GC was equipped with a DB-5 (60 m length \times 0.25 mm i.d.; 1.0 μ m film thickness) and FID heated at 300 °C. Oven temperature was programmed from 50 °C (1 min hold) to 270 °C (1 min hold) increasing at 20 °C min⁻¹. The concentration of aroma in the gas phase was determined from the calibration curves. Calibration curves were determined by injecting 1% (v/v) peppermint EO solution diluted in pentane (1, 4, 8 and 10 μ L) into the headspace vials and analyzing after approximately 24 h under the analogous GC conditions.

Gas chromatography-mass spectrometry (GC-MS). GC-MS analyses were performed using a *Perkin Elmer Clarus 500* GC coupled to a *Perkin Elmer Clarus 500* series mass selective detector (Perkin Elmer Instruments, Shelton, USA) in the electron impact ionization mode at 70eV, the mass range was m/z 29-550. Volatile compounds were separated using an Elite – 5 MS capillary column (dimethylpolysiloxane, 5% diphenyl, 30 m length, 0.25 mm i.d., 0.25 μ m film thickness, Perkin Elmer Instruments, Shelton, USA). The oven temperature was programmed from 50 °C (for 2 min) to 280 °C (hold 10 min) at the ratio of 5 °C min⁻¹. Carrier gas, helium, adjusted to a linear velocity of 36.2 cm s⁻¹ at 50 °C. Split mode was used at ratio of 1:20 and an injector temperature of 250 °C. The components were identified by comparison their Kovats retention indexes (KI) related to C₅-C₁₈ *n*-alkanes obtained on nonpolar column with those provided in the literature (Adams, 2001) and by comparison of their mass spectra with the data provided by the NIST (vers. 1.7) mass spectral library.

Determination of water activity, moisture content and particle size distribution. Water activity of spray-dried powders was determined in triplicate using *AquaLab* Water Activity Meter (Decagon, Washington, USA). Moisture was determined by distillation with toluene method. A laser diffraction-based Malvern particle size analyzer *Mastersizer 2000* was used for determination of powder particle diameter.

Statistical analysis. Data were statistically handled by one-way analysis of variance (ANOVA, vers. 2.2., 1999). Duncan's multiple-range test was applied for the calculation of the significant differences among the microencapsulated into different wall materials peppermint EO products, at the probability level $P=0.05$.

Results and discussion

Properties of liquid emulsion and spray-dried microencapsulation products. Total oil content in liquid homogenized peppermint EO emulsions was obtained from 13.5 g 100 g⁻¹ for ENCAPSUL 855-emulsified peppermint oil product to 15.2 g 100 g⁻¹ for CIEmCap 12633-emulsified product (Table 1). Emulsification efficiency of peppermint EO in the liquid emulsions varied from 88.8 % to 99.7 % for ENCAPSUL 855- and CIEmCap 12633-emulsified peppermint oil products. It mostly depends to the emulsification properties of matrix and its tendency to form films at the interfaces between the emulsion phases; this is in agreement that maltodextrins usually lack the emulsification efficiency (Pegg et al., 1999). Some loss of oil could also be attributed to the effect of emulsion droplet size (Soottitawat et al., 2005a; Re, 1998).

The total oil contents of the spray-dried microencapsulated peppermint EO products were statistically different at $P=0.05$ and ranged from 6.1 g 100 g⁻¹ for ENCAPSUL 855 microencapsulated peppermint EO product to 14.9 g 100 g⁻¹ for CIEmCap 12633 microencapsulation product (Table 1). It is clear that the type of solids used effect the retention of volatiles. The lowest ability to retain peppermint EO was obtained for both dextrins matrices in our

study. The literature show that maltodextrins do not perform excellent in volatiles retention. It was reported that maltodextrins retain aroma compounds well that are water soluble or soluble at their use level; the poor retention of insoluble aroma compounds by maltodextrins relates to their lack of emulsification properties (Reineccius et al., 2003).

Table 1. Properties of liquid emulsion and spray-dried microencapsulated peppermint EO products

	HI-CAP 100	N-LOK	CAPSUL	ENC 855	CR TEX 627	CIEmCa p 12633	CIEmCa p 12634	CIEmCa p 12635
	liquid microencapsulated product							
total oil content, g 100 g ⁻¹	15.03 ^{cd}	14.44 ^{bcd}	14.73 ^{bcd}	13.54 ^a	14.14 ^{ab}	15.21 ^d	14.44 ^{bcd}	14.81 ^{bcd}
emulsification efficiency, %	98.56 ^{cd}	94.69 ^{bcd}	96.59 ^{bcd}	88.78 ^a	92.72 ^{ab}	99.74 ^d	94.69 ^{bcd}	97.11 ^{bcd}
	spray-dried microencapsulated product							
total oil content, g 100 g ⁻¹	13.49 ^b	12.15 ^c	13.96 ^c	6.10 ^a	9.41 ^d	14.94 ^g	13.67 ^{bc}	14.48 ^f
surface oil content, g 100 g ⁻¹	0.20 ^{cd}	0.18 ^{bc}	0.25 ^{def}	0.13 ^{ab}	0.23 ^{cdef}	0.08 ^a	0.17 ^{bc}	0.29 ^f
encapsulation efficiency, %	87.15 ^b	78.49 ^e	89.90 ^c	39.15 ^a	60.19 ^d	97.44 ^g	88.52 ^{bc}	93.05 ^f
moisture, mL 100 g ⁻¹	1.83 ^a	2.08 ^{ab}	3.34 ^{cd}	1.49 ^a	3.39 ^{cd}	2.35 ^{ab}	2.85 ^{bcd}	3.75 ^d
water activity a _w	0.39 ^{ab}	0.43 ^{abc}	0.35 ^a	0.45 ^{bc}	0.44 ^{bc}	0.44 ^{bc}	0.49 ^{cde}	0.55 ^e
particle size diameter D ₄₃ , μm	27.19 ^d	34.99 ^f	30.50 ^e	11.9 ^a	23.52 ^b	137.63 ^h	56.88 ^g	24.82 ^c
specific surface area, m ² g ⁻¹	17.7 ^e	16.6 ^d	16.8 ^d	0.7 ^a	10.5 ^a	4.3 ^b	19.4 ^f	17.4 ^e

* Computed on the basis of a theoretical oil content of 15.25% of the solids. ^{a-h} Values within rows followed by the same letter do not differ statistically at $P=0.05$.

The content of oil remained on the surface of the spray-dried powder is important factor for storage stability, because surface oil can be easily oxidized and form unacceptable off-flavour compounds. The content of surface oil on the spray-dried microencapsulated peppermint EO products powder particles varied from 0.1 g 100 g⁻¹ for CIEmCap 12633 and to 0.3 g 100 g⁻¹ for CIEmCap 12635 encapsulated flavour products.

The moisture content of spray-dried microencapsulated peppermint EO products ranged from 1.5 mL 100 g⁻¹ (ENCAPSUL 855) to 3.8 mL 100 g⁻¹ (CIEmCap 12635). The water activity of spray-dried encapsulated peppermint oil products was in ranges from 0.54 for HI-CAP 100 to 0.76 for ENCAPSUL 855 encapsulated products. When surface oil was removed a decrease in a_w was observed and ranged from 0.35 for CAPSUL to 0.55 for CIEmCap 12635 microencapsulated peppermint EO product (Table 1).

The efficiency of microencapsulation of peppermint EO via spray-drying into different carbohydrate-based matrices varied from 39.2% (ENCAPSUL 855) to 97.4% (CIEmCap 12633), and there were statistical differences at $P=0.05$ between different encapsulated peppermint EO products. Maltodextrins do not perform well in volatile retention, mostly to the lack of their emulsification properties (Pegg et al., 1999; Reineccius et al., 2003). Encapsulating modified starches involve the addition of lipophilic groups. It was reported that both of CAPSUL and HI-CAP 100, which were derived from waxy maize base, were modified with n-octenyl succinic anhydride (OSA) for using in the flavour encapsulation process; HI-CAP 100 is blended with high dextrose equivalent (DE) corn syrup solids with the final of 32-37 DE and designed for the high load encapsulation agent (Sootitawat et al., 2005b).

Menthol (47.5%), menthone (19.8%), *iso*-menthone + *neo*-menthol (1.1:1) (15.9%), menthyl acetate (4.2%) and pulegone (2.2%) were the main volatile compounds in pure peppermint EO used as initial material for encapsulation. The compositions of pure, emulsified and encapsulated in different starches based matrices peppermint EO were quite similar, however some changes in the percentages of some individual compounds were observed. More remarkable differences in the compositions of surface oils from various encapsulation products were determined. In general, this phenomenon could be explained by the losses of more volatile hydrophobic compounds not entrapped in the capsules and consequently not protected from evaporation.

Flavour Release of peppermint EO volatiles by DHS-ATD-GC-FID. The recovery of peppermint EO volatiles that were released into headspace of each of the spray-dried microencapsulated products (surface oil removed) was determined as a function of nitrogen purge time up to 30 min. The obtained results indicate that components were released by different rates by each of microencapsulated products (Fig. 1). Total amount of peppermint volatiles was released during 2 min of purge treatment at concentrations ranging from 12.0 $\mu\text{g g}^{-1}$ by CIEmCap 12633 spray-dried microencapsulated peppermint oil product to 97.9 $\mu\text{g g}^{-1}$ for ENCAPSUL 855 microencapsulated product. The major compound menthol was released during the 2 min of purge treatment at concentrations ranging from 4.4 $\mu\text{g g}^{-1}$ by CIEmCap 12633 microencapsulated product to 36.6 $\mu\text{g g}^{-1}$ for ENCAPSUL 855 microencapsulated spray-dried product. The range of concentrations oil volatiles during 30 min purge time was from 58.9 $\mu\text{g g}^{-1}$ for CIEmCap 12633 to 524.8 $\mu\text{g g}^{-1}$ CIEmCap 12635, of which amount of released menthol constituted from 34.4 $\mu\text{g g}^{-1}$ to 264.0 $\mu\text{g g}^{-1}$, for CIEmCap 12633 and CIEmCap 12635 microencapsulated peppermint oil products, respectively. Menthone, as the second major component, during timed purge treatment was released from 2.0 $\mu\text{g g}^{-1}$ for CIEmCap 12633 to 69.2 $\mu\text{g g}^{-1}$ for CIEmCap 12634 microencapsulated products.

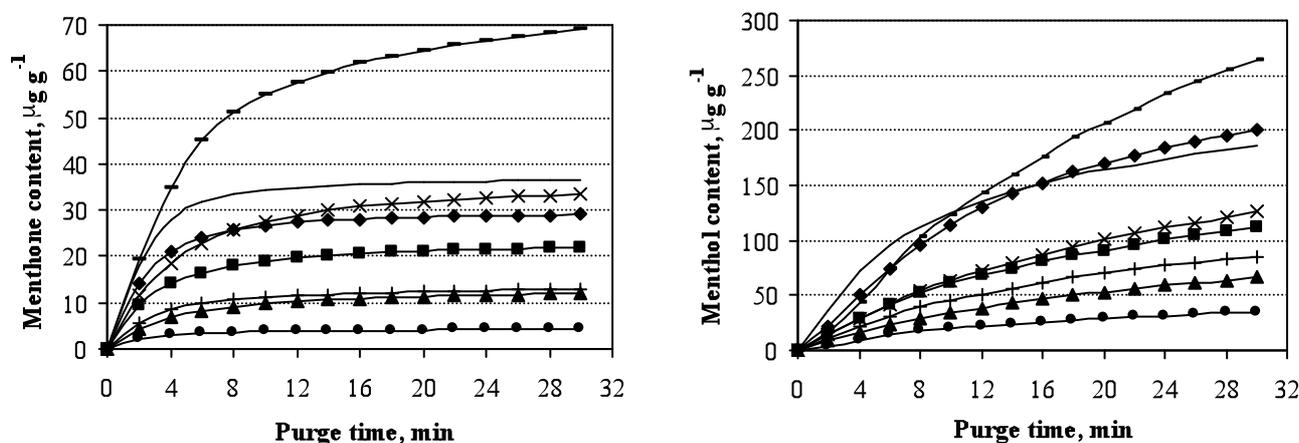


Fig.1. Concentrations of major volatiles released from microencapsulated peppermint oil products by DHS analysis; \blacklozenge HI-CAP 100; \blacksquare N-LOK; \blacktriangle CAPSUL; \times CRYSTAL TEX 627; — ENCAPSUL 855; \bullet CIEmCap 12633; + CIEmCap 12634; = CIEmCap 12635

Flavour release of peppermint EO volatiles by SHS-GC-FID at different water activities. The release of flavour through the capsule matrix, as well, as the oxidation of encapsulated flavour, is also used as an index of the encapsulated flavour stability. The release time-course of peppermint EO volatiles in spray-dried microencapsulated powder products was measured at 25 °C and $a_w = 0.43$ and 0.75. The effect of 0.43 a_w on the kinetics of peppermint volatiles released is provided in Fig. 2. At 0.43 a_w peppermint volatiles released from various microencapsulated products ranged from 4.6 ng g^{-1} powder for N-LOK microencapsulated product to 212.9 ng g^{-1} for HI-CAP 100 powder product. The permeability of CIEmCap 12633 was the lowest during storage at 0.43 a_w , and the total amount of peppermint volatiles released varied from 5.2 to 26.2 ng g^{-1} (Fig. 2). The highest amount of peppermint volatiles was observed above HI-CAP 100 wall system, and varied from 12.9 to 212.9 ng g^{-1} . The most intensive aroma release was observed during first 60 min of storage. Menthol was released from $4.0 \cdot 10^{-3}$ to 1.3 ng g^{-1} during storage. The smallest amounts of menthol released into the headspace was observed above CIEmCap 12633 microencapsulated peppermint oil product and ranged from $4.0 \cdot 10^{-3}$ to 0.1 ng g^{-1} . Significantly higher amounts of menthol, compared with all modified starch-based matrices analysed, were emitted from HI-CAP 100 microencapsulated product. Menthone was released from $2.0 \cdot 10^{-4}$ ng g^{-1} for CIEmCap 12633 and

CIEmCap 12634 microencapsulated products up to 0.4 ng g^{-1} for HI-CAP 100 encapsulated product. The least permeable wall matrix in case of menthone was CIEmCap 12634, the released amounts varied from $2.0 \cdot 10^{-4} \text{ ng g}^{-1}$ to $1.2 \cdot 10^{-2} \text{ ng g}^{-1}$; while, again the highest leakage of this compound was observed in the headspace above HI-CAP 100 microencapsulated peppermint EO powder product.

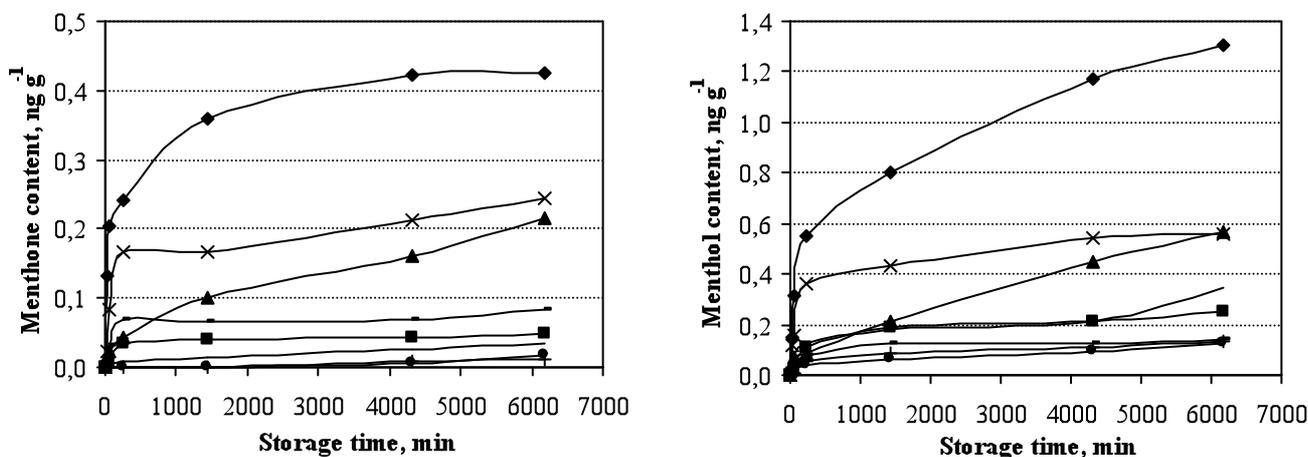


Fig.2. Release kinetics of volatiles from microencapsulated peppermint EO products by SHS analysis at $a_w=0.43$; \blacklozenge - HI-CAP 100; \blacksquare - N-LOK; \blacktriangle - CAPSUL; \times - CRYSTAL TEX 627; — ENCAPSUL 855; \bullet - CIEmCap 12633; + - CIEmCap 12634; = - CIEmCap 12635

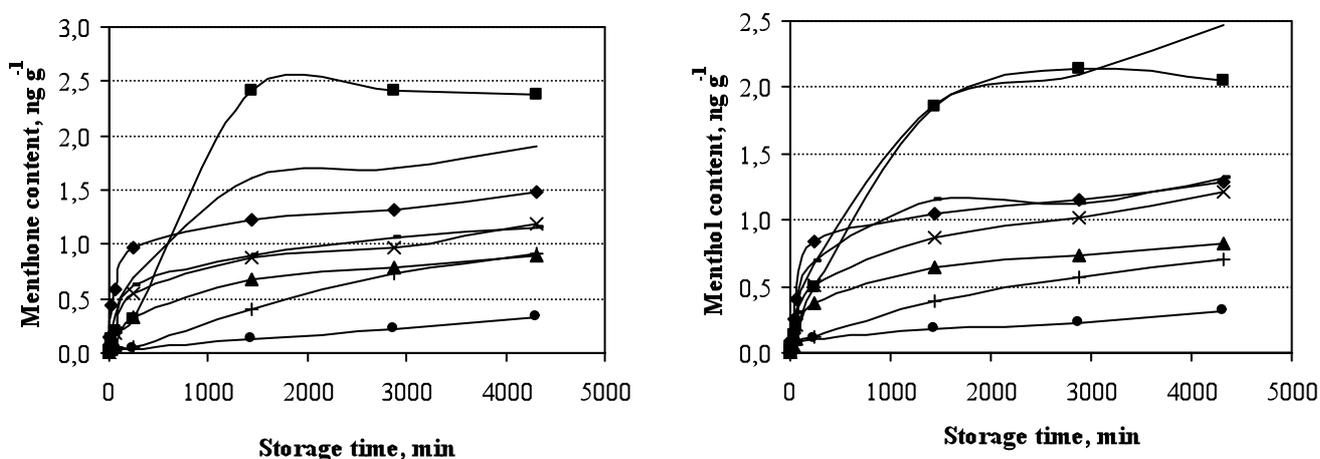


Fig.3. Release kinetics of volatiles from microencapsulated peppermint EO products by SHS analysis at $a_w=0.75$; \blacklozenge - HI-CAP 100; \blacksquare - N-LOK; \blacktriangle - CAPSUL; \times - CRYSTAL TEX 627; — ENCAPSUL 855; \bullet - CIEmCap 12633; + - CIEmCap 12634; = - CIEmCap 12635

The results on release kinetics of volatiles from microencapsulated peppermint EO products by SHS at $a_w=0.75$ are provided in Fig. 3. It is clear that relative humidity (RH) ($a_w = \text{RH}/100$) shows a pronounced effect on the leakage rates of peppermint EO volatiles from different encapsulated powder products, which was greatly accelerated by an increase in a_w . The amounts of menthol released at $0.75 a_w$, compared to that of $0.43 a_w$, increased from 1.3 to 6.7 times from HI-CAP 100, 4.4-16.8 from N-LOK, 1.5-4.4 from CAPSUL, 3.3-11.6 from ENCAPSUL 855, 1.4-2.2 from CRYSTAL TEX 627, 2.5-13.5 from CIEmCap 12633, 2.4-145 from CIEmCap 12634 and 1.9-10.2 from CIEmCap 12635 encapsulation peppermint EO products, respectively. The concentrations of

menthone at 0.75 a_w increased from 2.2-96.7 for CRYSTAL TEX 627 encapsulation product up to 62.5-410 for CIEmCap 12634 spray-dried peppermint flavour product compared to that at 0.43 a_w (Fig. 2 and 3). Our results are in agreement with other studies and can be explained by the change of matrix structure and that the water uptake at higher RH destroyed the capsule structure (Rosenberg et al., 1990; Yoshii et al., 2001). Also, it could be suggested that the release of partially water soluble flavours is controlled by the diffusion mechanism through the wall of particles, and the ultimate retention is determined by the combined effects of molecular diffusion and core droplet mobility (Soottitantawat et al., 2005b).

Conclusions

In general, all modified starches matrices used in our study retained peppermint oil very efficiently. The CIEmCap 12633 food starch modified was considered to be the least emitting coating material in our study; CAPSUL and CIEmCap 12634 were also very effective to retain peppermint EO volatiles and were enough stable during storage at different a_w conditions, as well, and could be concluded as the next least leaking matrices. Both dextrin matrices used ENCAPSUL 855 and CRYSTAL TEX 627 showed the poorest emulsification efficiency and retention of peppermint volatiles and quite high leakage of volatiles into the headspace from spray-dried encapsulated powdered products. The modified starch HI-CAP 100 retained enough high amounts of peppermint oil volatiles after spray-drying (87.2%), however it was not enough stable during storage, especially at 0.43 a_w , compared to other matrixes used.

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