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EFFECT OF SPRAY DRYING PARAMETERS ON ROSEMARY AROMA MICROENCAPSULATION

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Key words: microcapsulation, rosemary, spray drying, SPME

Results of rosemary oil microcapsulation process are shown in the presented work. The microcapsulation process has taken place in a spray dryer. The core of the microcapsules was maltodextrin. The two mass concentrations of maltodextrin in emulsion, *i.e.* 25%and 30%, one spray disc speed, inlet air temperature of 200°C and three feed fluid fluxes were used. Efficiency of closing rosemary aroma in the capsule was estimated by the amount of aroma compounds in the initial emulsion, powders and emulsions received from the powder. The best aroma closure was received in the case of 30% carrier concentration and at the medium flux (64.2 mL/min). It was found that higher powders apparent density and lower bed porosity were responsible for quantity increase of the closed aroma compounds.

INTRODUCTION

Spray drying is the most widespread and the oldest method of microcapsulation in the food industry. This method is economic and flexible, devices are available, and the product received has good quality [Dziezak, 1988]. Parameters of spray drying (temperature, disc rotation speed or feed flux, concentration and composition of the carrier) influence the size and the shape of received powders particles as well as determine the final water content in the material and first of all they influence the degree of aroma ingredients preservation in the product [Gouin, 2004; Janiszewska & Witrowa-Rajchert, 2006; Soottitantawat *et al.*, 2005; Yoshii *et al.*, 2001]. While analysing scientific literature of aroma microcapsulation with the method of spray drying it is noticeable that the most of attention is paid on to the kind and composition of carriers which are responsible for adequate aroma retention. However, other parameters of the drying process are more rarely discussed. Therefore, the goal of the presented research was to determine feed flux influence and the carrier concentration influence on the capsulated rosemary aroma amount during spray drying.

MATERIALS AND METHODS

Emulsions for spray drying were prepared by using the carrier low-crystallized DE=8 maltodextin (Barents Sp. z o. o.) with concentration of 25% or 30%, with added 0.5 mL of rosemary oil (ETOL Polska Sp. z o. o.). Solutions were homogenized for 90 sec at 11,000 rpm.

Drying of the obtained solutions was carried out in a spray dryer (Anhydro) at the speed of the spray disc reaching 39,000 rpm and three raw material fluxes: 51.4, 64.2 and 79.5 mL/min. Drying was carried out with co-current method and the inlet air temperature was 200°C. The powder dry matter counted was measured according to Polish Standard [PN-A-79011-3].The apparent powders density and bed bulk density were measured in helium pycnometer (Stereopycnometr of the Quantachrome) [Domian & Bialik, 2006].

Determination of aroma composition in rosemary oil, powder and in reconstructed from microencapsulated powder emulsion, was made with the GC-MS technique using the HS-SPME method. In the case of powder, 1 g of the powder dry matter was stored in 100 mL bottle. In the case of rosemary aroma, 0.0054 g of oil was taken and 40 mL of distilled water was added. Emulsion reconstructed from the powder was prepared at the concentration equal to emulsion concentration for drying, by adding the adequate amount of distilled water. The emulsion (0.2 mL) was separated and 40 mL of distilled water was added.

SPME fiber was inserted for 15 min into bottles with samples and analyses were carried out. The analyses were conducted by means of GC-MS QP-2010 (Shimadzu). Use was made of BPX 90 column (thickens 0,25 μm). MS parameters were: the ionization voltage 0.2 kV, mass to load ratio at the beginning 40 and at the end 500, ionization source temperature 190°C. GC parameters were: initial process temperature 40°C, it was kept for 1 min and then increased 4°C/min to a temperature of 180°C, head pressure 159.7 kPa, carrier gas: helium, injection temperature: 220°C.

The data was analysed using multi-sample comparison. All calculations were carried out with the use of Statgraphics Plus 4.1 software.

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TABLE 1. Characteristics of received powders.

Carrier concentration (%)	Feed flux (mL/min)	Outlet temperature (°C)	Moisture content (%) $\bar{x}\pm SD$	Apparent density (g/cm^3) $\bar{x}\pm SD$	Bulk density (g/cm^3) $\bar{x}\pm SD$	Bed porosity $\bar{x}\pm SD$
25	51.4	98	2.60 ± 0.034^{ab}	0.758 ± 0.0026^{a}	0.214 ± 0.02^{a}	0.718 ± 0.005^{a}
30		99.5	1.92 ± 0.007^{a}	0.808 ± 0.005^{a}	0.249 ± 0.009^{a}	0.691 ± 0.01^{b}
25	64.2	95.5	3.33 ± 0.629^{bc}	0.775 ± 0.015^{a}	0.214 ± 0.015^{a}	0.723 ± 0.023^{a}
30		97.5	2.13 ± 0.101^{a}	0.801 ± 0.017^{a}	0.253 ± 0.004^{a}	0.684 ± 0.012^{b}
25	79.5	93	3.86 ± 0.601^{c}	0.798 ± 0.108^{a}	0.221 ± 0.02^{a}	0.73 ± 0.01^{a}
30		95	2.70 ± 0.630^{ab}	0.821 ± 0.015^{a}	0.254 ± 0.003^{a}	0.681 ± 0.017^{b}

The values in the same column denoted with different letters: a, b, c differ statistically at $p>0.05$

TABLE 2. Characteristics of chosen aroma compounds in powders and emulsion received from the powders (in relation to compounds contained in the oil).

Compound name	Emulsion with 25% carrier concentration						Emulsion with 30% carrier concentration					
	powder (%)			emulsion from powder (%)			powder (%)			emulsion from powder (%)		
	51.4 (mL/min)	64.2 (mL/min)	79.5 (mL/min)	51.4 (mL/min)	64.2 (mL/min)	79.5 (mL/min)	51.4 (mL/min)	64.2 (mL/min)	79.5 (mL/min)	51.4 (mL/min)	64.2 (mL/min)	79.5 (mL/min)
Acetic Acid	0.56^{b}	0.215^{ab}	0.112^{a}	17.115^{B}	22.745^{B}	24.91^{B}	0.03^{a}	0.02^{a}	0.03^{a}	1.142^{A}	0.786^{A}	0.93^{A}
Borneol	0	0	0	3.328^{A}	4.129^{A}	12.98^{A}	0	0	0	5.265^{A}	6.114^{A}	4.854^{A}
Camphor	0	0	0	29.53^{A}	55.715^{A}	38.68^{A}	0.04^{a}	0.03^{a}	0.04^{a}	47.57^{A}	57.1^{A}	47.24^{A}
Benzene, 1-methyl-4-(1-methylethyl)	0	0.021^{a}	0.004^{a}	0.352^{A}	1.195^{A}	0.647^{A}	1.17^{c}	0.7^{b}	0.46^{b}	0.99^{A}	2.69^{A}	5.22^{A}
Bicyclo[3,1,1] hept-2-ene, 2,6,6-trimethyl	0	0	0	5.252^{A}	8.341^{A}	3.155^{A}	0.01^{a}	0.01^{a}	0.01^{a}	2.302^{A}	3.669^{A}	3.151^{A}
Bicyclo[2,2,1] heptane, 2,2-dimethyl-3-methylene	0	0	0	5.417^{A}	9.135^{A}	3.324^{A}	0	0	0	2.473^{A}	4.476^{A}	4.252^{A}
Bicyclo[3,1,1] heptane, 6,6-dimethyl-2-methylene	0	0	0	3.785^{A}	0	2.809^{A}	0	0	0	0.997^{B}	2.53^{A}	2.819^{A}
Cyclohexanol 1-methyl-4-(1methylethenyl)	0	0	0	0	0	0	0	0	0	17.44^{A}	16.64^{A}	18.46^{A}
Alpha,-Caryophyllene	0	0	0	7.045^{A}	7.881^{A}	0	0	0	0	7.07^{A}	14.44^{B}	14^{B}

The values in the same verse denoted with different letters: a, A, b, B, c differ statistically at $p>0.05$

RESULTS AND DISCUSSION

An increase in the feed flux resulted in the change in evaporating intensity, which led to lowering the outlet air temperature and the increase of the water content in powders (Table 1). Simultaneously, the increase in the feed flux during spraying coused that bigger drops came out from spray disc, which also caused an increase of powders particle size and a negligible increase of their apparent density. Maltodextrin concentration increase caused a decrease of water content, the increase of powders apparent density and the statistically significant decrease of the powder bed porosity. The performed statistical analysis demonstrated that powders apparent density changes were not significant in statistical content (Table 1).

As basic aromatic substances, there were detected bicyclo [3,1,1] hept-2-ene 2,6,6-trimethyl (19,05%), bicyclo[2,2,1] heptane 2,2-dimethyl-3-methylene (10,5%), bicyclo[3,1,1] heptane 6,6-dimethyl-2-methylene (2,51%), camphor (0,1%), borneol (0,28%), acetic acid (1,31%), benzene, 1-methyl-4-(1-methylethyl) (9,68%) and cyclohexanol 1-methyl-4-1methylethenyl (48%), (Table 2). These results were confirmed by Zawirska-Wojtasiak [2006].

Analysis of the aromatic compounds content in emulsions received from the powders demonstrated that volatile compounds were kept better by microcapsules for which the higher carrier concentration was used (Figure 1). Rosemary oil was best capsulated at the flux of 64.2 mL/min.

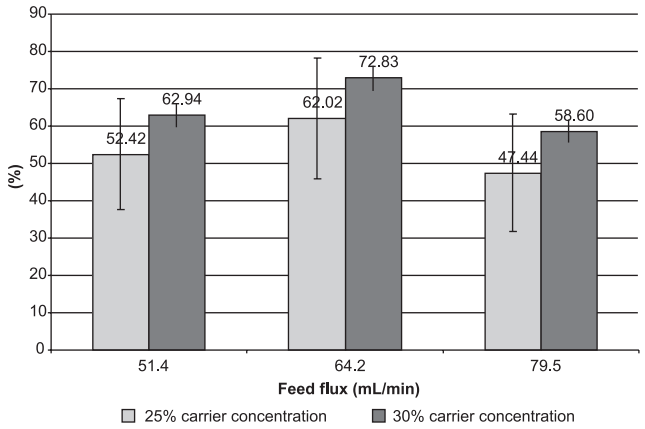

FIGURE 1. Influence of feed flux and carrier concentration on the amount of capsulated aroma compounds.

The observed tendencies were not confirmed in the statistical analysis, which failed to demonstrate any significant differences between experiments. The amount of capsulated aromatic compounds ranged from 47 to 72% (in relation to compounds contained in the oil).

While analysing aromatic substances above the surface of powders it was observed the existence of only few of them. They were in the amount lower than 1% of their content in the starting oil (Table 1). It proves good maintenance of aromatic substances inside the capsules.

The amount of capsulated single aromatic compounds received from the powder emulsion (Table 1) coincides with the results shown in Figure 1. For the majority of compounds the best microcapsulation was received when applying the average feed flux and with maltodextrin concentration of 30%. Those compounds which are to be found in the oil in minor quantity (*e.g.* camphor) are the best for capsulation. And those which are in majority in oil remain in the powder in lower quantity.

Comparing the received results of closing volatile compounds with physical properties of powder a tendency can be observed that higher apparent density and lower bed porosity support the aroma microcapsulation.

CONCLUSIONS

The best results of closing aromas were received with the use of 30% emulsion concentration at the average emulsion flux, *i.e.* 64.2 mL/min.

Analyses of chromatograms received after laboratory tests which were taken from the surface of powders demonstrated good microcapsulation inside the capsules.

Better closing of the volatile compounds was obtained for powders with higher powder apparent density and lower bed porosity.

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WPŁYW PARAMETRÓW SUSZENIA ROZPYŁOWEGO NA PROCES MIKROKAPSULACJI OLEJKU ROZMARYNOWEGO

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W prezentowanej pracy przedstawiono wyniki badań zamykania olejku rozmarynowego w trakcie suszenia rozpyłowego w mikrokapsułce z maltodekstryny. Zastosowano dwa stężenia maltodekstryny w emulsji 25 oraz 30%, jedną prędkość dysku rozpyłowego, jedną temperaturę powietrza wlotowego 200°C oraz trzy strumienie zasilania surowca. Efektywność zamknięcia aromatu w kapsułce oceniano na podstawie ilości związków aromatycznych będących w olejku wyjściowym, otrzymanym po suszeniu proszku oraz emulsji powstałej po odtworzeniu z proszku. Najlepsze zamknięcie aromatów uzyskano dla 30% stężeń nośnika i przy średnim strumieniu surowca (64,2 mL/min). Stwierdzono, że wyższa gęstość pozorna proszków i niższa porowatość odpowiadały za zwiększenie ilości zakapsułkowanych związków.