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MICROENCAPSULATION OF RAPESEED OIL BASED ON THE SPRAY DRYING METHOD

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The aim of this work was to examine the influence of three levels of various amounts of particular ingredients, which form emulsion exposed to drying, on the microencapsulation efficiency and selected physical properties of powdered encapsulated oil. Spray-drying of rapeseed oil onto a maltodextrin carrier with the addition of acacia gum enabled obtaining a powdered product with complete water reconstitution and very poor wettability and flowability. The greatest susceptibility to quantitative changes in particular components of the emulsions was shown for traits linked with surface oil content and powder particles density. The microencapsulation efficiency, ranging from 79 to 95%, was affected to the greatest extent by the solid content of emulsion subjected to spray drying.

INTRODUCTION

In the microencapsulation process, reactive, sensitive or volatile food additives may be transformed into stable food components, which affects the improvement of their action efficiency and extends the spectrum of their applications. The food industry uses encapsulated powders to preserve delicate ingredients in the powdered form. Coating is applied to a variety of materials, including aroma, mineral and acidifying substances, vitamins, oils and fats, microorganisms, enzymes and dyes [Jankowski, 1995; Korus *et al.*, 1997]. Many sensitive ingredients are lipid-based compounds, which exist in a liquid form at room temperature. Emulsions, which have been stabilized by homogenization in the presence of emulsifier, can be spray-dried to produce microencapsulated fat powders [Turchiuli *et al.*, 2005]. A degree of active substance entrapment into microcapsules is affected by homogeneity and stability of emulsion, composition of a coating material, and spray drying conditions [Vega & Ross, 2003; Dalgleish, 2006]. The quality of the microcapsules is often estimated from the fraction of non-encapsulated lipid corresponding to the oil exposed at the surface [Imagi *et al.*, 1992].

A carrier or matrix of capsules may be various synthetic and natural compounds, *e.g.*: hydrocolloids, gums, modified starches, maltodextrins, cyclodextrins, proteins of milk, soybean and wheat, various sugars [Korus *et al.*, 1997]. Those substances differ between one another in terms of emulsifying capacity, permeability towards core substances, and degree of preventing oxygen penetration into microcapsule interior [Leahy *et al.*, 1983]. An ideal wall material used for microencapsulation should have bland flavor, high solubility, and possess the necessary emulsification, film-forming, and drying properties. In addition, a concentrated solution should

have low viscosity to facilitate the spraying step [Rosenberg & Young, 1993]. In practice, materials of the matrix are a composition of carriers combining their advantages [McNamee *et al.*, 2001]. Many researches have extensively studied microencapsulation by spray-drying using dairy ingredients [Vega & Ross, 2003]. There have been a number of reports on the use of sodium caseinate and whey proteins as encapsulants and sugar or hydrolyzed starch as fillers [Fäldt & Bergenståhl, 1996; Imagi *et al.*, 1992; Hogan *et al.*, 2001]. Flavorings and citrus oils have been encapsulated in food gums and modified starches that behave like gums [Reineccius, 1991; Bhandari *et al.*, 1992]. The main advantage of maltodextrin is the fact that it well protects contents of a capsule against oxidation [Lewandowicz *et al.*, 2005]. Oxygen absorption is decreasing along with an increase in a glucose equivalent DE, hence products coated with maltodextrin with DE above 36 exhibit high stability and few-year shelf life without oxidative changes [Reineccius, 1991]. Maltodextrin has no emulsifying capacity [Lewandowicz *et al.*, 2005]. Acacia gum, a low viscosity hydrocolloid, unlike maltodextrin, has a very good emulsifying capacity and provides high retention of volatile components in the process of spray drying, but it does not prevent oxidation of encapsulated substances [Krishnan *et al.*, 2005; Turchiuli *et al.*, 2005].

Besides providing a good protection of the active ingredient, the encapsulated powder must also have a good stability during storage (no wall collapse, sticking) ensured by a low water content and a low water activity [Yoshii *et al.*, 2003]. It must be able to reform the original emulsion by reconstitution, in water for example [Hogan *et al.*, 2001]. The ease of using the powder during dosage, mixing with other powders, *etc.* is another critical index of its quality [Vega & Ross, 2003]. For a dispersed system of solid particles, such properties as

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bulk density, flowability, ease of dosage, avoidance of components segregation or dust generation are of key significance [Domian, 2005b]. Encapsulation, however, leads to demonstrable difficulties in handling, owing to changes in bulk properties of the powders [Konstance *et al.*, 1994]. Spray drying yields a fine powder with generally poor handling properties. The composition of the powder surface dictates the behavior of the bulk in terms of wettability, flowability, and stability. [Schubert, 1987; Schubert *et al.*, 2003]. Analyses of surface of milk powders and dehydrated emulsions showed that powder surface was covered mainly by fat, even when the fat content is very low [Fäldt & Bergenståhl, 1996].

Constantly increasing popularity of the microencapsulation of lipid-based compounds such as anti-oxidants, nutraceuticals or flavours prompted us to undertake a study on microencapsulation by means of spray drying in a model system of vegetable oil with maltodextrin and acacia gum as a coating material.

The aim of the study was to analyse the effect of changes in the amounts of particular components forming emulsion subjected to the drying process on the oil microencapsulation efficiency and on selected physical properties of powder.

MATERIAL AND METHODS

Raw materials used in the study included: refined rapeseed oil (ZT "Kruszwica" S.A., Poland), maltodextrin Glucidex DE19 (Roquette, France), and acacia gum (type 4639A, Hortimex).

The research on oil microencapsulation with the method of spray drying was conducted based on a factorial experiment that included 11 runs $(=2^k+3)$, where $k=3$ – number of variables). Experimental design was collated in Table 1. The following microencapsulation variables were chosen: solid content of carrier (maltodextrin and acacia gum)

TABLE 1. Microencapsulation variables and experimental design levels.

Levels of coded	Values of original variables							
variables	DM $(\%)$	AG (g/100 g)	OL (g/100 g)					
$+1$	40	50	15					
$\overline{0}$	35	40	10					
-1	30	30	5					
Run number	Experimental design							
1	-1	-1	-1					
2	-1	-1	1					
3	-1	1	1					
4	-1	1	-1					
5	1	1	1					
6	1	1	-1					
7	1	-1	1					
8	1	-1	-1					
9	Ω	Ω	Ω					
10	Ω	θ	θ					
11	Ω	θ	0					

in the emulsion *DM*, acacia gum amount in the solid content of the emulsion *AG* and amount of oil in relation to carrier of emulsion *OL.* Selected properties were estimated: microencapsulation yield *MY* and microencapsulation efficiency *ME*, water content *w* and activity a_w , particles density ρ , loose ρ_1 and tapped bulk density ρ _P, loose ε _L and tapped porosity ε _P, flowability expressed as pouring time I_s and Hausner ratio I_{HR} , wettability *Z* and solubility index *R*.

A relationship between each property and microencapsulation variables was expressed by a regression equation:

$$
Y_i = B_o + B_i X_i + B_i X_2 + B_i X_3
$$

In that equation, values of physical properties were substituted for Y_i , whereas coded microencapsulation variables X_1, X_2, X_3 attained values of -1 , 0 or $+1$ (Table 1). Numerical data were subjected to a statistical analysis at a significance level of $p=0.05$ for verification of the hypothesis: H_0 : $B_1 = B_2 =$ $B_3 = 0$, assuming that levels of variables have no significant effect on the microencapsulation efficiency nor on the physical properties of the powder.

For each run, emulsion was prepared in the amount that provided obtaining *ca*. 300 g of ready powder after drying. Emulsions of oil in an aqueous solution of maltodextrin and acacia gum, prepared using an ULTRA TURRAX T25/IKA Labortechnic homogenizer (6 min, 11,000 rpm). Before use, emulsions were stored at 5° C. Emulsions were spray-dried in an APV Anhydrous Laboratory Spray Drying (APV Anhydro A/S, Søborg, Denmark) at a constant temperature of inlet and outlet air: $200 \pm 2^{\circ}$ C and $100 \pm 2^{\circ}$ C, respectively, and at speed of a spray disk reaching 34,000 rpm.

The total oil content T_c was determined through oil extraction from 5 g of powder reconstituted in 10 mL of water with the use of a 90-mL methanol/chloroform mixture (1/2). The surface oil content T_P was assayed through extraction of oil from 10 g of powder using 50 mL of petroleum benzine as a solvent [Soerensen *et al.*, 1978; Turchiuli *et al.*, 2005]. The microencapsulation yield *MY* was calculated as a ratio of total oil content in powder product to the initial oil content in feed emulsion (on the dried solid basis). The microencapsulation efficiency *ME* was calculated based on the total and surface oil content according to the following equation: $ME = (T_c - T_p)/T_c \cdot 100\%$.

Particle density *ρ* was determined with the use of a helium pycnometer (Stereopycnometer/Quantachrome Instruments). Loose bulk density ρ_L (bulk density of loosely poured material) and tapped bulk density ρ_T (bulk density of material packed with 1250 standard taps) were determined using a shaking volumeter STAV 2003/Engelsmann AG, Germany. Values of ρ , ρ _L and ρ _T densities enabled calculating porosity of powder bed: both the loose $\varepsilon_{\text{L}} = (1 - \rho_{\text{L}} / \rho)$ and the tapped one $\varepsilon_{\rm T} = (1 - \rho_{\rm T}/\rho)$.

Flowability was expressed with a Hausner ratio I_{H} , as the following ratio $I_H = \rho_T / \rho_L$ and with a flowability coefficient I_s , expressed as time necessary for pouring 25 cm³ of powder through gaps of a rotating vessel [Santomaso *et al.*, 2003; Soerensen *et al.*, 1978].

Wettability *Z* in water at a temperature 20°C was assayed as time necessary to wet all powder particles contained in 10 g mass [Domian, 2005b]. Solubility *R* was determined as a volume of precipitate in mL after reconstitution of 15 g of powder in 100 mL of water at a temperature of 20°C [Soerensen *et al.*, 1978].

Water activity was assayed with the use of a Rotronic apparatus model Hygroskop DT at a temperature of 25°C.

Microstructure of powders was analysed based on microscope pictures taken with the use of a scanning electron microscope FEI, Quanta 200.

RESULTS AND DISCUSSION

Physical properties of microencapsulated powdered oil obtained in particular runs of microencapsulation with the method of spray drying are presented in Tables 2 and 3. The observed variability of the examined physical properties of microencapsulated oil powder depends on contents of particular components forming the emulsion for the drying process. All regression coefficients between the analysed parameters and coded values of independent variables are presented in Table 4. After verification of statistically insignificant terms and their elimination as well as after restoration of original variables, relations are presented in Table 5.

The capsules were characterised by regular and spherical shape, smooth surface with visible cavities, and constituted powder particles with sizes ranging from ca . 10 to 90 μ m (Figure 1). The total oil content in powders obtained in particular runs was lower than that of the initial oil content in emulsions. Oil doses of 5, 10 and 15 g/100 g carrier were introduced into the emulsion. In the obtained powders, the total oil content accounted for 4.17-4.69 g, 8.61-9.23 g and 10.58- -12.10 g oil/100 g carrier, respectively. Oil microencapsulation yield in the powders was at a level from 71 to 94%. Depending on the experimental run, surface oil content ranged from 0.21 to 2.38 g/100 g powder. The surface oil content should be as low as possible. Its amount on the surface may affect flowability and also contribute to the caking of powders. Oil located outside the capsule is exposed to changes upon the atmospheric influence, which leads to its decreased shelf life and stability during storage. Contents of surface oil and total oil in the powders enabled determining the degree of oil

TABLE 2. Oil microencapsulation yield and efficiency in powders.

entrapment in capsules. Powders obtained in the study were characterised by microencapsulation efficiency at a level from 79 to 95% (Table 3). It is known that solvent extraction also accounts for some fat coming from the interior of the particles [Fäldt & Bergenståhl, 1996]. The solvent can reach the interior through cracks and pores. Surface fat does not necessarily correlate to solvent-extractable free fat because the latter also includes some near-surface fat.

The degree of oil entrapment inside the capsules was mainly determined by oil and carrier content (maltodextrin and acacia gum) in the emulsion. With oil content *OL* increasing in the emulsion from 5 to 15 g/100 g carrier, both microencapsulation yield *MY* and microencapsulation efficiency *ME* were decreased (Tables 4 and 5). It has been shown by various workers [Rosenberg & Young, 1993; Fäldt & Bergenståhl, 1996], that as the emulsion fat content increases, the median globule diameter of the emulsion increases. The content of free fat and surface fat coverage the powder particles will also increase. An increase in the total content of carrier in the emulsion *DM* from 30 to 40% contributed to a diminished content of oil surface and to an increase in the microencapsulation efficiency (Tables 4 and 5). These results showed similar trends as those reported by others [Reineccius, 1988; Bhandari *et al.*, 1992], although the latter group reported that increasing total solids concentration had little effect on microencapsulation efficiency of powders [Hogan *et al.*, 2001]. Increasing the content of acacia gum *AG* from 30 to 50 g/100 g carrier did not change the microencapsulation efficiency. It had a statistically significant effect on an increase in microencapsulation yield *MY* and, consequently, on a reduction in losses of oil during spray drying (Tables 4 and 5). These results suggested that oil to gum ratios of 0.1 to 0.5 were needed to obtain similar microencapsulation efficiency. Probably the most interesting property of acacia gum, one that brings it close to being the ideal encapsulant for emulsion spray drying, is that it can act as an emulsifier [Vega & Roos, 2006]. The content of protein components is relatively small, and the concentration of acacia gum needed to obtain long-term emulsion stability is rather high (12% for a 20% orange oil emulsion; Randall *et al.* [1988]). Mc-Namee *et al.* [2001] analysed in detail the emulsification and

FIGURE 1. Scanning electron micrographs of powders obtained in particular runs (bar = 20μ m).

encapsulation properties of acacia gum. Emulsions of acacia gum and soybean oil at an oil to gum ratio of 0.25 to 5.0 were prepared to produce spray-dried powders with an oil content ranging from 20 to 82% (w/w). They found that at oil to gum ratio between 1.0 to 2.0, the quantity of acacia gum available to act as an emulsifier was reduced, which resulted in the production of larger oil droplets and the decrease of the encapsulation efficiency in spray-dried powders. Microencapsulation efficiency decreased from 100 to 48% when the oil to gum ratio was increased from 0.25 to 5.0, respectively. Similar results were found by Soottitantawat *et al.* [2005] after encapsulating D-limonene at the same oil to gum ratio of 0.25 to 1. Maltodextrins are frequently used as coencapsulating agents in emulsion spray drying. In the case of flavour encapsulation by spray drying, it has been shown that flavour retention depends on the ratio between maltodextrin and acacia gum with an optimal retention for weight ratios between 2/3 and 3/2 [Bhandari *et al.,* 1992]. Yoshii *et al.* [2001], by using various concentrations of maltodextrin and acacia gum in microencapsulation of an odorant (ethyl butyrate) with the method of spray drying, proved that an increasing concentration of maltodextrin (10-30%) was accompanied by an increasing efficiency of the microencapsulation process. Carbohydrate with a DE of \sim 20 was chosen for subsequent formulations due to its high microencapsulation efficiency and low sweetness and hygroscopicity. Hogan *et al.* [2001] analysed the ef-

TABLE 3. Physical properties of powdered encapsulated oil.

Run	W	a_{w}	ρ_L	$\rho_{\scriptscriptstyle T}$	ρ	ε_L	ε_{τ}	I_{S}	I_{HR}	Z	\mathbb{R}
	$(\%)$	$(-)$	(kg/m ³)	(kg/m ³)	(kg/m ³)	$(-)$	$(-)$	(s)	$(-)$	(s)	(mL)
1	2.53	0.043	487	716	1288	0.62	0.44	>180	1.47	>300	$\overline{0}$
	± 0.00	± 0.005	±4	±5	± 1	± 0.01	± 0.01	± 0	± 0.00	± 0	± 0
$\overline{2}$	3.10	0.074	430	695	1220	0.65	0.43	>180	1.62	>300	Ω
	± 0.03	± 0.010	±18	±4	± 0	± 0.00	± 0.02	± 0	± 0.08	± 0	± 0
\mathfrak{Z}	2.65	0.046	460	704	1218	0.62	0.42	>180	1.53	>300	$\overline{0}$
	± 0.06	± 0.006	±3	±13	± 0	± 0.01	± 0.00	± 0	± 0.02	± 0	± 0
$\overline{4}$	3.35	0.064	462	698	1264	0.63	0.45	>180	1.51	>300	$\overline{0}$
	± 0.02	± 0.007	±5	±1	± 0	± 0.00	± 0.00	± 0	± 0.02	± 0	± 0
5	2.85	0.053	480	765	1184	0.60	0.35	>180	1.59	>300	$\overline{0}$
	± 0.10	± 0.007	± 1	±5	± 1	± 0.00	± 0.00	± 0	± 0.01	± 0	± 0
6	4.79	0.147	484	694	1249	0.61	0.44	>180	1.43	>300	$\overline{0}$
	± 0.21	± 0.014	±14	±4	± 0	± 0.00	± 0.00	± 0	± 0.05	± 0	± 0
7	2.35	0.037	487	702	1256	0.61	0.44	>180	1.44	>300	$\overline{0}$
	± 0.14	± 0.005	± 10	± 1	± 0	± 0.01	± 0.01	± 0	± 0.03	± 0	± 0
8	2.51	0.046	479	708	1231	0.61	0.42	>180	1.47	>300	$\overline{0}$
	± 0.14	± 0.005	± 1	±5	± 0	± 0.00	± 0.00	± 0	± 0.01	± 0	± 0
9	3.51	0.091	470	703	1205	0.61	0.42	>180	1.49	>300	θ
	± 0.05	± 0.009	±7	± 1	± 0	± 0.00	± 0.00	± 0	± 0.02	± 0	± 0
10	2.93	0.053	488	718	1244	0.61	0.42	>180	1.47	>300	Ω
	± 0.11	± 0.006	±13	±20	± 0	± 0.00	± 0.00	± 0	± 0.00	± 0	± 0
11	2.96	0.048	489	736	1240	0.61	0.41	>180	1.50	>300	Ω
	± 0.22	± 0.006	±9	±16	± 0	± 0.00	± 0.00	± 0	± 0.06	± 0	± 0

TABLE 4. Coefficients in regression equation $Y_i = B_0 + B_1 X_1 + B_2 X_2 + B_3 X_3$ for different physical properties of powdered encapsulated oil (using coded values of independent variables).

 Y_i b₀ b₁ b₂ b₃ *Tc* 1.64 - 0.71 *T_p* 2.22 -0.07 - 0.12 *MY* 99.23 - 0.34 -1.13 *ME* 67.81 - 0.73 - -4.00 *w* 1.14 - 0.04 *a_w* 0.02 - 0.001 *ρ*_{*L*} 410.7 2.29 - -1.79 *ρ*_{*T*} 626.2 - - - - - *ρ* 1387.6 -1.72 -1.01 -5.12 *ε^L* 0.715 -0.002 - *ε^T* 0.566 -0.002 - -0.004 I_{HR} 1.539 - - 0.009 I_{S} >180 - - - - -*Z* >300 - - - -*R* 0 - - - -

*significant at a probability level of $p = 0.05$

fect of maltodextrin DE on emulsion stability, drying, and reconstitution of soybean oil emulsions stabilized by sodium caseinate. The microencapsulation efficiency increased from 0 to 88.4% with increasing DE from 0 to 28. The positive relationship between microencapsulation efficiency values and DE was attributed to the smaller oligosaccharides in high DE powders forming less porous, more uniform matrices on drying, which were more impervious to the solvent than those formed by low DE preparations.

Directly after drying, the powders demonstrated low water activity, *i.e.* a_w 0.04-0.15, at moisture content *w* of 2.5-4.8%. Loose and tapped bulk density as well as a porosity of loosely poured and packed bed reached: ρ _L 430-489 kg/m³ and ρ _T 695- -765 kg/m^3 as well as ε_L 0.60-0.65 and ε_T 0.35-0.45, respectively (Table 3). Particles of microencapsulated oil powder were characterised by density ρ ranging from 1184 to 1288 kg/m³. Physical properties such as fat content, moisture, and product density affect powder flow [Peleg, 1978]. Particle size also has a major influence on powder flowability. A powder may be considered as having a particle size of $\langle 200 \mu m \rangle$, and as the size is reduced below this value, flowability is progressively impaired. This reduction in flowability is due to the increased surface area per unit mass of powder. More surface is available for cohesive forces, in particular, frictional forces to resist flow [Schubert, 1987]. The higher the ratio between tapped and loose density values (Hausner ratio), the more cohesive, or the less able to flow is the powder [Domian 2005a]. Spray drying yields a fine powder with generally poor handling properties. Flowability indices attained values typical of sparingly flowing powders, *i.e.* Hausner ratio I_{HR} > 1.4 [Santomaso *et al.*, 2003] and pouring time $I_s \gg 60s$ [Soerensen *et al.*, 1978] (Table 3). Greater total content of the carrier *DM* in the emulsion resulted in an increase in the loose bulk density and in a decrease in particle density and bed porosity (Tables 4 and 5). Higher oil content *OL* of the emulsion decreased loose bulk and particle density, porosity of tapped bed, which resulted in reduced flowability (Tables 4 and 5). Powders with higher levels of unencapsulated fat on the surface (therefore, a greater amount of extractable fat) tended to stick together and form lumps, which impeded the flow [Konstance *et al.*, 1995]. Addition of acacia gum in the carrier *AG* contributed to an increase in the content and activity of water as well as a decrease in particle density (Table 4 and 5). Particle density decreasing with an increase of *AG* and *DM* in the emulsion may indicate an effect of the carrier, especially of acacia gum, on reducing the number of open pores in capsules and on increasing tightness.

A major property of encapsulated powders manufactured for consumer use is their ease of reconstitution. Irrespective of emulsion composition, the powders belonged to sparingly reconstitutable in water, and were characterised by complete solubility $(R=0)$, but very poor wettability in water $(Z>300s)$ (Table 3). The reconstitution process in water can be divided in 4 steps: wetting, submersion, dispersion, and dissolving [Schubert *et al.*, 2003]. Wettability is understood as the ability of a bulk powder to imbibe a liquid under to influence of capillary forces. Generally, it depends on powder particle size, density, porosity, surface charge, surface area, and the presence of amphipathic substances. Fast wetting is also favoured by large particles of high porosity [Domian, 2005b]. Changing the content of components in the emulsion, in the range examined, did not affected flowability nor reconstitution of the powders in water (Tables 4 and 5). The microencapsulated oil, irrespective of changes in the factors discussed, was a sparingly flowing, completely soluble powder, yet requiring constant stirring for reconstitution in water.

CONCLUSIONS

Spray-drying microencapsulation of rapeseed oil onto a maltodextrin carrier with the addition of acacia gum, carried out in the applied range of process variables, enabled obtaining a powdered product with complete water reconstitution and very poor wettability and flowability. A changeable number of particular components forming emulsions subjected to drying had a complex effect on the microencapsulation efficiency as well as on physical and functional properties of the powder. Multiple regression analysis of the effect of the assumed levels of microencapsulation factors demonstrated the existence of significant correlations in most of those variables. The greatest susceptibility to quantitative changes in particular components of the emulsions was shown for traits linked with surface oil content and powder particles density. The microencapsulation efficiency, ranging from 79 to 95%, was affected to the greatest extent by the solid content of carrier in the material subjected to spray drying.

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