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Research article

Microencapsulation of model oil in wall matrices consisting of SPI and maltodextrins

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Abstract: Microencapsulation can provide means to entrap, protect and deliver nutritional lipids and related compounds that are susceptible to deterioration. The encapsulation of high lipid loads represents a challenge. The research has investigated the encapsulation by spray drying of a model oil, at a core load of 25–60%, in wall systems consisting of 2.5–10% SPI and 17.5–10% maltodextrin. In general, core-in-wall-emulsions exhibited unimodal PSD and a mean particle diameter < 0.5 μm. Dry microcapsules ranged in diameter from about 5 to less than 50 μm and exhibited only a limited extent of surface indentation. Core domains, in the form of protein-coated droplets, were embedded throughout the wall matrices and no visible cracks connecting these domains with the environment could be detected. Core retention ranged from 72.2 to 95.9% and was significantly affected ($p < 0.05$) by a combined influence of wall composition and initial core load. Microencapsulation efficiency, MEE, ranged from 25.4 to 91.6% and from 12.4 to 91.4% after 5 and 30 min of extraction, respectively ($p < 0.05$). MEE was significantly influenced by wall composition, extraction time, initial core load and DE value of the maltodextrins. Results indicated that wall solutions containing as low as 2.5% SPI and 17.5% maltodextrin were very effective as microencapsulating agents for high oil load. Results highlighted the functionality of SPI as microencapsulating agent in food applications and indicated the importance of carefully designing the composition of core-in-wall-emulsions.

Keywords: microencapsulation; spray drying; soy proteins; maltodextrins; core-in-wall-emulsions; microstructure

1. Introduction

A broad array of different applications for microencapsulation in food formulation and processing has been developed during recent decades [1-6]. Results of numerous studies have allowed overcoming a multitude of challenges and successfully meeting otherwise unattainable goals pertinent to food processing and effective delivery of desired nutrients, functional ingredients and biologically active compounds [1-4,6-12]. Information about the health promoting properties of specific dietary lipids, phytochemicals and different natural biologically-active compounds calls for enhancing their consumption through food [13,14]. In light of their sensitivity to conditions prevailing in food processing and storage, such nutrients and ingredients have to be protected throughout food processing and pending consumption [1,3,8]. Microencapsulation has been shown to allow effective and safe delivery of such sensitive nutrients and compounds through foods [1,3,4,6,12,14-17]. Among the challenges for microencapsulation in food applications is the availability of highly functional, GRAS, cost-effective microencapsulating agents, or, as there are referred to, wall materials [4,18-20]. The microencapsulating properties of different animal- and plant-derived proteins have been investigated and results of these studies have been reviewed [15,21-24]. It has been established that by wisely highlighting and exploiting specific physico-chemical properties of different proteins they can be effectively utilized for the entrapment and delivery of desired nutrients and functional ingredients, collectively referred to as "core materials" through foods [15,21,24,25]. Soy protein isolate (SPI) with a protein content of at least 90% is a highly functional value added product of soybean processing. It is obtained by separating the protein constituents of the soybean from the water-soluble and water-insoluble constituents of the bean in a process consisting of aqueous extraction followed by isoelectric precipitation of the protein constituents [26]. SPI has been suggested as a functional ingredient in food applications [26,27] and is utilized in a variety of food applications where the physico-chemical and functional properties of its main protein constituents, 7S (glycinin) and 11S (β-conglycinin) constituents are highlighted [27-30]. Surface activity and emulsification properties of microencapsulating agents are critical to effective microencapsulation of lipids and similar core systems [14]. Soy proteins exhibit good emulsification properties [31-37] and SPI has thus been suggested as a microencapsulating agent in food applications [24,38-52]. This, and the manifestation of desired film forming properties [24,53,54] have made SPI an attractive potential GRAS wall material for microencapsulation by spray drying. Microencapsulation by spray drying of lipids, essential oils, aroma and other core compounds in wall systems consisting of SPI or blends of SPI with other microencapsulating agents has been studied to a certain extent [24,39,41-44,55-58,60,61].

The application of wall systems consisting of whey proteins isolate (WPI) and different carbohydrates (COH) has been previously reported by us and results of these studies have highlighted the functionality of such blends as effective wall systems for encapsulation of lipids and volatiles by spray drying [62-66]. The effective encapsulation of lipids at a high core-to-wall ratio is a challenge and the encapsulation of high lipids load in wall systems consisting of SPI and COH has not been reported yet. The objectives of the present study were to investigate the formation and some properties of core-in-wall-emulsions (CIWE) containing 25–60% model oil and wall systems consisting of SPI and different maltodextrins (MD) and to study the properties of spray-dried (SD) microcapsules prepared with these CIWE.

2. Materials and Methods

2.1. Wall and core materials

Soy protein isolate (SPI, Supro 670) containing 92% (w/w) proteins (N × 6.25) was obtained from Protein Technologies International (St. Louis, MO). Maltodextrins (MD) with dextrose equivalent (DE) of 7.5 or 17.5 were obtained from Cerestar USA Inc. (Hammond, Indiana). Soy oil, as a model core, was purchased at a local supermarket and served as a model core material.

2.2. Microencapsulation by spray drying

Wall Solutions (WS) containing 20% (w/w) solids consisting of 2.5, 5.0 or 10.0% (w/w) SPI and 17.5, 15.0 or 10.0% (w/w) MD, respectively, were prepared in de-ionized water (Millipore, 18.2 MΩ.cm) and were designated A, B, and C, respectively. These WS were also designated according to the DE value of the MD constituent, H or L for DE 17.5 and DE 7.5, respectively. In all cases, SPI was dispersed in water (40 °C) and after adding 0.02% sodium azide (Fisher Scientific, Pittsburgh, PA) the dispersion was slowly stirred overnight (25 °C) to allow full hydration and swelling of the protein constituents. Then, the MD component of the WS was dissolved into the SPI solution and the mixture was stirred for additional 2 h at 23–25 °C. In all cases, pH of WS was adjusted to 6.90 ± 0.1.

Soybean oil at 23–25 °C was emulsified into the WS to a final core (oil) load of 25, 50 or 60% (w/w). Emulsification was carried out as previously reported [63]. In short, a coarse emulsion was prepared by emulsifying (at room temperature) the oil into the WS using an Ultra-Turrax T25 high shear homogenizer (IKA Works, Cincinnati, OH) operated at 13,000 rpm for 45 s. The coarse emulsion was then homogenized (at room temperature) for four successive homogenization steps at 50 MPa using a model NS1001L2K–PANDA high pressure homogenizer (Niro Soavi S.p.A., Parma, Italy). CIWE prepared in this way were designated according to their WS composition and core content. For example, CIWE A25L was prepared with WS containing 2.5% (w/w) SPI and 17.5% (w/w) MD with a DE value of 7.5 and contained 25% oil (w/w). The composition and designated codes of the investigated CIWE are provided in Table 1. Attempts to prepare CIWE containing 60% oil using WS containing 5 or 10% SPI resulted in a very viscous CIWE that were not suitable for atomization and spray drying. Similarly, B type WS containing L type MD yielded "lumpy" CIWE and thus could not be spray dried.

Spray Drying: The investigated CIWE were spray dried using an APV Anhydro Laboratory Spray Dryer (APV Anhydro A/S SØborg, Denmark). The dryer had an evaporation rate of 7.5 kg/h and a chamber diameter of 1 m. In all cases, CIWE were atomized using the centrifugal atomizer of the dryer operated at 50,000 rpm and drying, in the co-current configuration, was carried out at an inlet and outlet air temperature of 160 and 80 °C, respectively. Dry microcapsules were collected at the bottom of the dryer's cyclone, placed in hermetically closed glass jars and kept in desiccators pending analyses. In all cases, duplicate CIWE were spray dried.

2.3. Analyses

Particle Size Distribution (PSD) properties of CIWE were determined using a Malvern Mastersizer MS20 (Malvern Instruments, Malvern, England). In all cases, analysis was carried out using a 2-mW He-Ne laser beam (633 nm) and a 45-mm focus lens. Analysis was carried out in quadruplicate and the PSD and mean particle size (volume-size average, d_{32} μm) were recorded.

Moisture content of SD microcapsules was determined gravimetrically in quadruplicates, after 12 h of Vacuum drying (65 °C, 6.7 kPa), as previously described [62,63].

Core (lipids) content of the SD microcapsules was determined, in quadruplicates, using a modification of the Roese-Gottlieb method, as previously reported [62,63]. In short, 1 g of capsules is reconstituted in 9 mL of water and the resulted emulsion is treated with 1.25 mL ammonium hydroxide. After adding 10 mL of ethanol, the lipids are extracted (three successive times) with an ethyl ether and petroleum ether.

Core retention was defined as the ratio (expressed in %) of core content included in 100 g of moisture-free SD microcapsules to that in 100g moisture free CIWE solids and was expressed according to Eq. 1

$$CR\ (\%) = \frac{OMC}{OE} \times 100 \qquad \text{Eq. 1}$$

where: CR is core retention, OMC and OE are core (oil) content per unit mass of moisture free SD microcapsules and CIWE solids, respectively.

Microencapsulation efficiency (MEE) was determined as previously reported [63]. MEE was defined as the proportion (in %) of OMC that was not extracted by petroleum ether from the microcapsules during a given extraction time at a given set of conditions. One gram of microcapsule powder was weighed into a 50 mL Quorpak glass bottle (Fisher Scientific, Pittsburgh, PA), 25 mL of petroleum ether (analytical grade, bp 70 ℃, Fisher Scientific, Pittsburgh, PA) were added and the bottle was capped with a Teflon-lined closure. The extraction systems were then placed on a Model 360 Garver shaker (Garver Mfg., Union City, IN) and the extraction, at a gentle shaking condition to avoid breaking microcapsules, was carried out for 5, 15 or 30 min at 25 ℃. Following the extraction, the mixture was filtered through a 0.45 μm, 47 mm diameter GN-6 filter (Gelman Science, Ann Arbor, MI), the solvent was evaporated using a water bath at 70 ℃, and the solvent-free extract was dried (45 ℃, 6.7 kPa). The dry extract was allowed to reach room temperature in a desiccator and its mass was then determined gravimetrically.

Microencapsulation efficiency after a given extraction time ($MEE_{(t)}$) was calculated according to Eq. 2

$$MEE_{(t)}\ (\%) = \frac{OMC - EO_{(t)}}{OMC} \times 100 \qquad \text{Eq. 2}$$

where: $EO_{(t)}$ is the extractable core at time t (mg). MEE values that were obtained after 5, 15 and 30 min of extraction were designated $MEE_{(5)}$, $MEE_{(15)}$ and $MEE_{(30)}$, respectively.

2.4. Electron microscopy

The outer topography and inner structure of the microcapsules were investigated by scanning electron microscopy (SEM). A layer of dry microcapsules was attached to a doubled sided adhesive tape (Ted Pella, Redding, CA) that had been placed on a specimen holder. In order to study the inner structure, microcapsules were fractured by moving a razor blade perpendicularly through a layer of microcapsules attached to the specimen holder. In all cases, the specimens were coated with gold using a Polaron sputter coater (model E-50050; Bio Rad, San Jose, CA) and studied using a Philips XL-FEG scanning electron microscope at 5 keV. The particle size distribution of the investigated microcapsules was determined by analyzing 7 micrographs of each of the investigated populations of microcapsules, using the AnalSYS image analysis software (Soft Imaging Systems, Lakewood, CO).

In all cases, more than 500 capsules per batch were quantified.

Table 1. Composition of the investigated Core-In-Wall-Emulsions (CIWE).

CIWE	SPI (%)[1]	Maltodextrin (%)[2]		Soy oil (%)[4]
		D.E.[3] 7.5	D.E. 17.5	
A25L	2.5	17.5		25
A25H	2.5		17.5	25
A50L	2.5	17.5		50
A50H	2.5		17.5	50
A60L	2.5	17.5		60
A60H	2.5		17.5	60
B25H	5.0		15.0	25
B50H	5.0		15.0	50
C25L	10.0	10.0		25
C25H	10.0		10.0	25
C50L	10.0	10.0		50
C50H	10.0		10.0	50

[1]Soy protein isolate (% w/w) in wall solution

[2]Maltodextrin in the wall solution (%, w/w)

[3]Dextrose equivalent of the maltodextrin

[4]Core, (soy oil) load in CIWE (%, w/w)

2.5. Statistical analysis

In all cases, replicate microencapsulation batches were prepared and analyses were carried out in quadruplicates (N × n = 8). The significance of the results was tested by ANOVA, using the SigmaStat software (San Raphael, CA). In all cases, the significance at $p < 0.05$ was determined.

3. Results and Discussion

3.1. Properties of CIWE

A key to success in microencapsulation of lipids is the formation of fine and stable CIWE [4,6,43,60,62,63]. Emulsion characteristics have been reported to be of critical importance to the quality, stability and functionality of microencapsulated systems [4,6,17,43,55,60,62,63]. In cases of protein-stabilized CIWE, the formation of protein-based films at the O/W interface is of critical importance to the colloidal stability of the CIWE and to the oxidative stability of the dry microcapsules [6,17,43]. The formation of CIWE with small mean particle size is of importance to success in microencapsulation by spray drying [6,43]. It has been suggested that in the case of lipids encapsulation, CIWE with a mean particle size smaller than 0.5 μm is desired [62,63] and accomplishing the latter dependents on homogenization conditions, composition of the CIWE and the inherent physico-chemical properties of the wall constituents [62,63].

In all cases CIWE exhibited a unimodal PSD similar to that presented in Figure 1 and, except for one case, $d_{3,2}$ of these emulsions was smaller than 0.5 μm (Table 2). A small mean particle size in CIWE has been shown to limit the proportion of surface fat after spray drying and to enhance lipids

retentions [6,43]. Soy proteins have been reported to manifest effective emulsification properties [31,34,52,54] and, in agreement with what has been previously described [43,55,56], the $d_{3,2}$ of CIWE ranged from 0.310 to 0.563 μm (Table 2). Maltodextrins have no surface activity and thus did not become involved at the O/W interface. Success in forming CIWE with desired PSD properties could thus be solely attributed to the surface-active properties and emulsification characteristics of the protein constituents included in the investigated SPI. Results thus indicated that this surface activity was effective even at a low SPI concentration of 2.5% and a high core load of 60%.

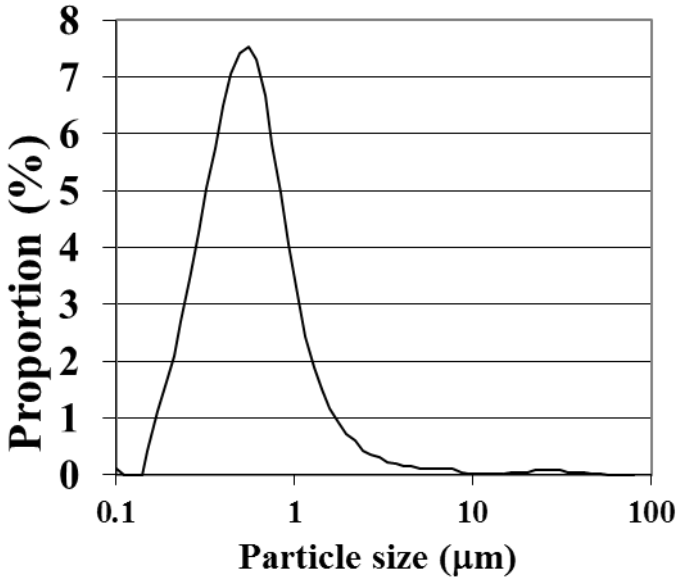

Figure 1. A representative particle size distribution (PSD) of CIWE A50H (see Table 1 for details).

Results (Table 2) indicated some relationships between the mean particle size and the composition of CIWE. In most cases, $d_{3,2}$ of CIWE prepared with a given WS was proportionally related to core load ($p < 0.05$). At given homogenization conditions and for a given wall composition and non-limiting availability of surface active material, increase in $d_{3,2}$ with lipid load reflects influence of the latter on phenomena inside the homogenization valve. These include a longer particles disruption time, a longer time that is needed to complete the adsorption of proteins at the newly formed O/W interface and a sharp decrease in the time needed for two or more partially protein-coated oil droplets to encounter each other and form a cluster [72].

Results obtained with "A" wall systems at a given core load (Table 2) indicated that $d_{3,2}$ was inversely proportional to the DE value of the MD constituent of the WS. Although, in most cases, the observed effect was small yet significant ($p < 0.05$), it probably reflected some DE value-dependent physical interactions between proteins and MD-based pseudo network consisting of high molecular weight oligosaccharides in the bulk phase of the emulsion [65]. It has been suggested that the relatively high proportion of high MW oligosaccharide molecules included in maltodextrin with low DE value adversely affect the availability or ease with which protein molecules can approach the O/W during homogenization. The latter was attributed to physical entanglement of protein molecules in the pseudo-network consisting of high MW oligosaccharides [65]. Such interference could be

expected to result in some coalescence, once the emulsion leaves the homogenization valve, as well in the formation of a larger number of homogenization clusters consisting of partially coated lipid droplets [65,72]. Results (Table 2) suggested that this effect was not evident at a higher concentration of SPI, probably reflecting increase in protein availability at the O/W interface as well as probably a more significant influence of the higher viscosity of the bulk phase of emulsion containing a higher SPI content on homogenization efficiency [72]. Overall, results indicated that wall solutions containing blends of SPI and maltodextrins allowed preparing CIWE with properties that are desired for microencapsulation of lipids by spray drying, even at a low proteins concentration of 2.5%. Results indicated that the micrometric properties of these CIWE were governed by a combined influence of the respective concentration of SPI and MD, DE value of the MD and core content. In all cases, the $d_{3,2}$ of CIWE in the present research was smaller than that reported by Nesterenko et al. [57] for CIWE consisting of α-tocopherol emulsified in wall solutions containing SPI or chemically-modified SPI and for CIWE containing different oils emulsified in SPI solutions [36].

Table 2. Mean particle size of CIWE.

Wall system	Core (%, w/w)	$d_{3,2}$ (μm)
A25L	25	0.328^{Abm}
A50L	50	0.438^{Aal}
A60L	60	0.475^{Aa}
A25H	25	0.310^{Bcm}
A50H	50	0.405^{Bbl}
A60H	60	0.435^{Ba}
B25H	25	0.375^{bl}
B50H	50	0.433^{am}
C25L	25	0.430^{Aal}
C50L	50	0.428^{Bal}
C25H	25	0.408^{Abm}
C50H	50	0.563^{Aam}

^{abc}For a given wall system, means followed by different letters differ significantly ($p < 0.05$)

^{AB}For a given SPI concentration and core load, means followed by different letters differ significantly ($p < 0.05$)

^{lm}For a given core load and DE value, means followed by different letters differ significantly ($p < 0.05$)

3.2. Size and microstructure of spray-dried microcapsules

In all cases (Table 3) the diameter of spray-dried microcapsules ranged from about 5 to less than 50 μm. All of the investigated CIWE were atomized and dried at the same conditions and thus among-batches differences in size and structure of microcapsules could be attributed to influence of the among-CIWE differences in composition. Results (Table 3) indicated that the proportion of microcapsules with $d \leq 20$ μm ranged from 54.7 to 98.4% and in most cases was higher than 70%. In general, results (Table 3) indicated that populations of microcapsules that were prepared with CIWE containing H type MD was much higher than that in powders prepared with L type MD. It was interesting to note that 93.2–98.4% of microcapsules prepared with CIWE B25H, B50H and C25H were smaller than 20 μm and that 90.2% of microcapsules prepared with CIWE B25H were smaller than 10 μm. The relationship between DE value and the diameter of spray dried microcapsules could be attributed to a faster drying rate and, consequently, lower extent of expansion (ballooning) during

spray drying that was promoted by the higher proportion of low molecular weight oligosaccharides in H type MD in comparison to that in L type MD [62,63,65]. Results (Table 3), indicated that the size distribution of microcapsules was affected by a combined influence of wall composition and core load, thus probably reflecting the effect of the latter on the visco-elastic, drying and film-forming properties of the CIWE. At a given atomization and drying conditions, these properties of the CIWE govern the formation and size distribution of droplets during atomization and later their drying properties. The ultimate result of these effects determines the ultimate size distribution of spray dried microcapsules [62,63,65].

Table 3. Size distribution of spray dried microcapsules.

CIWE	A ($d < 9.9$ μm) %	B ($10 \leq d \leq 19.9$ μm) %	C ($20 \leq d \leq 29.9$ μm) %	D ($d \geq 30$ μm) %
A25L	26.7	46.2	21.7	5.4
A50L	18.6	36.1	25.1	20.2
A60L	18.7	37.2	26.9	17.2
A25H	47.6	21.5	16.9	14.4
A50H	49.9	35.7	11.7	2.7
A60H	41.2	39.3	14.3	5.2
B25H	90.2	8.2	1.1	0.4
B50H	75.7	18.5	4.5	1.4
C25L	38.5	43.0	12.0	6.6
C50L	32.3	36.0	19.1	12.7
C25H	73.6	21.3	4.7	0.4
C50H	58.0	30.5	9.5	2.2

The typical outer topography and inner structure of microcapsules prepared with different CIWEs are presented in Figures 2–4. In all cases, spherical microcapsules ranging in diameter from less than 5 μm to about 50 μm were obtained. Microcapsules exhibited excellent physical integrity and outer surfaces of the microcapsules were free of visible cracks (Figure 2). Only a very few microcapsules exhibited some surface pores (Figure 2) that could probably be attributed to a severe extent of "ballooning" that these capsules had experienced during spray drying [66]. The outer surface of the microcapsules exhibited only a limited extent of surface indentation. Polysaccharide-based spray-dried particles are known to exhibit a significant extent of surface indentation [66,67]. The extent of surface indentation that was exhibited by microcapsules in the present research was significantly lower than that reported for SPI-based, oil-containing spray dried microcapsules [54,55]. Results thus suggested that the protein constituents of SPI influenced the mechanical properties of the wall system and allowed formation of smooth and spherical capsules. These results were in agreement with what has been reported for wall systems consisting of whey proteins and carbohydrates [62-64,73]. Analysis of the inner structure of microcapsules (Figures 3 and 4) revealed a typical structure of oil-containing spray-dried microcapsules. In all cases, the presence of a central void was evident and the encapsulated core was embedded, in the form of fine droplets, throughout the wall matrix. In all cases, the core droplets were individually coated with a very dense layer of proteins (Figure 4), similar to what has been reported for microcapsules prepared with wall systems containing whey proteins or blends of whey proteins and carbohydrates [62-64,73].

The dense protein films at the surface of the core domains represent the result of soy proteins adsorption at the O/W interface during the homogenization process [62-64,72,73]. In all cases, the core droplets were well isolated from the environment and no pores or channels connecting core domain with the outer surface of the capsules could be detected.

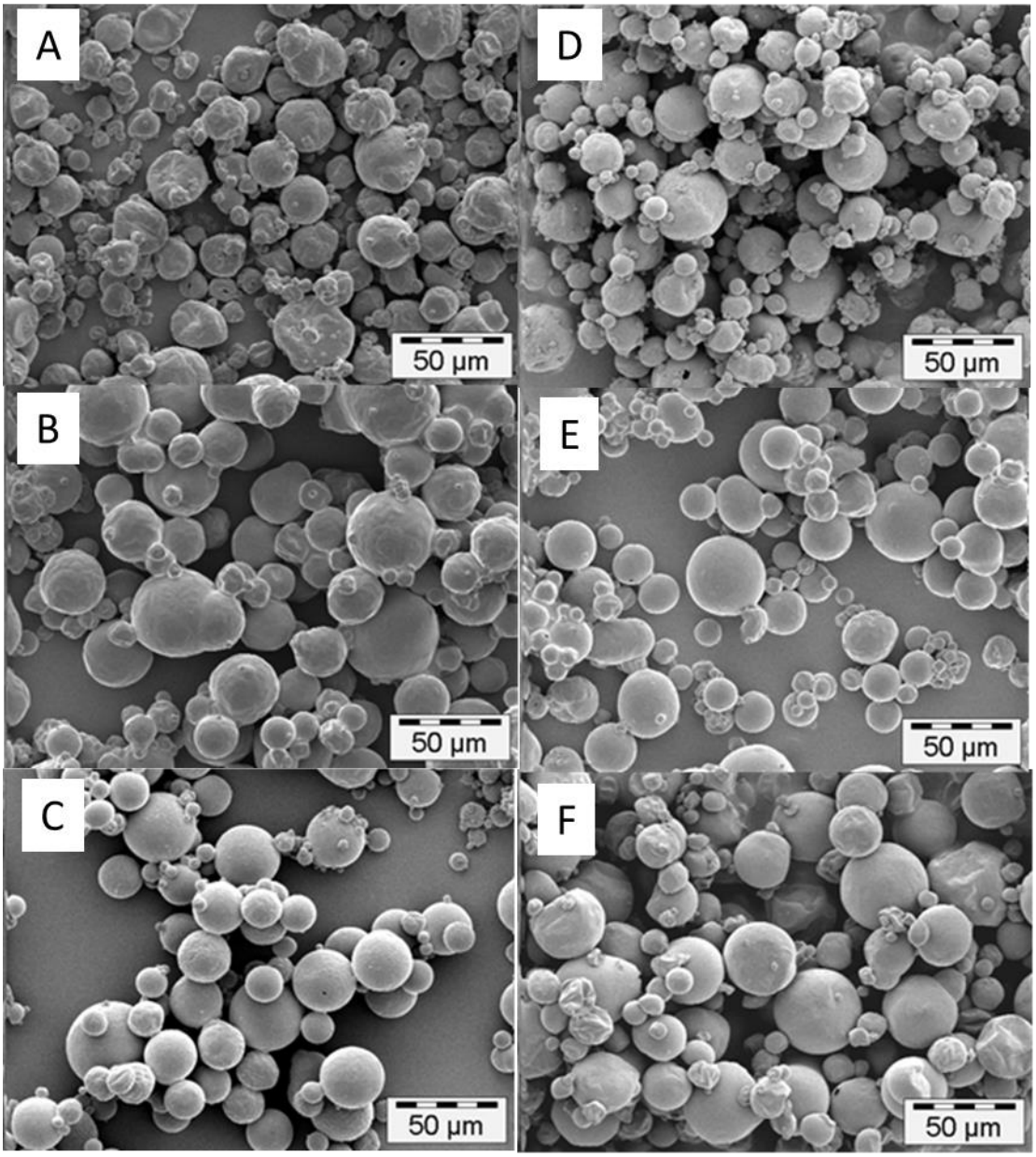

Figure 2. Outer topography of spray dried microcapsules prepared with CIWE A25L (A, B); A50H (C); B50H (D); A60H (E) and C50L (F). For detailed composition of CIWE see Table 1.

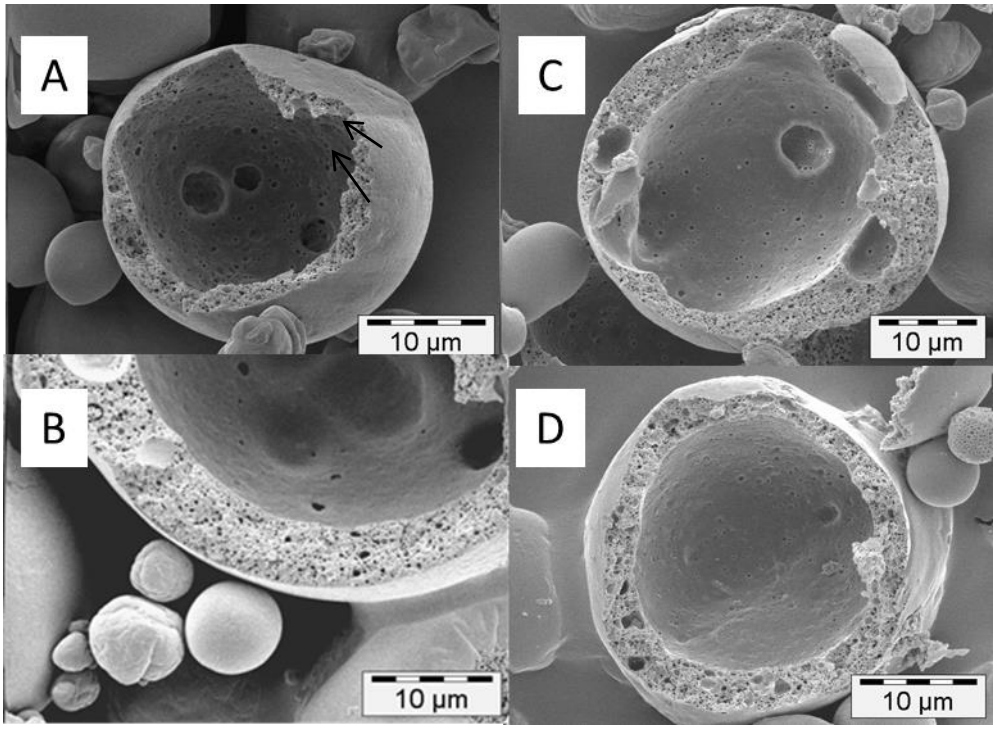

Figure 3. Typical inner structure of spray-dried microcapsules prepared with CIWE A50L (A), B50H (B), C25H (C) and C50H (D). For detailed composition of CIWE see Table 1. Arrows— "Footprints of core droplets".

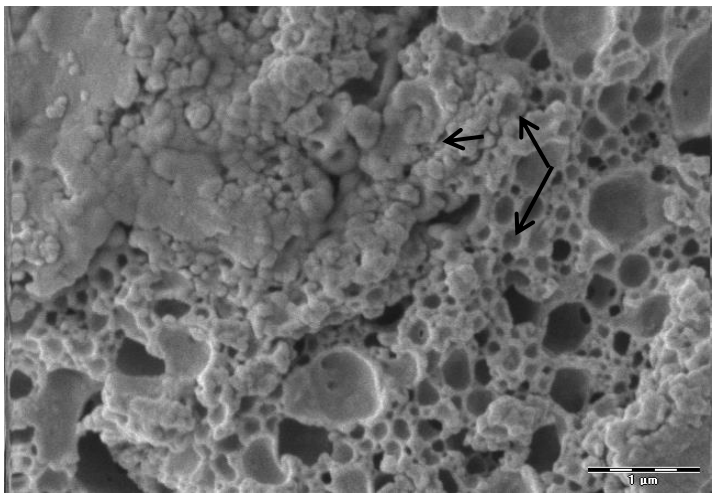

Figure 4. High resolution micrograph of a fracture plan through the wall matrix of spray-dried microcapsule prepared with CIWE B50H revealing the inner structure. Arrows—empty core domains surrounded by dense films of SPI.

3.3. Moisture content

Moisture content of powders varied among powders prepared with different CIWE (Table 4) and ranged from 0.89 to 1.648%, from 1.12 to 1.36% and from 0.23 to 2.11%, for microcapsules prepared with CIWE "A", "B" and "C", respectively. The among-powders differences in moisture

content reflected the influence of the CIWE composition on atomization and drying rate during spray drying. For example, at a given SPI concentration and core load, moisture content of "A" powders was proportionally related to the DE value of their MD constituent. Given that all CIWE were dried at the same atomization and drying conditions, this could be attributed to the influence of the between-maltodextrins differences in molecular weight distribution of their oligosaccharide constituents on drying rate [62,63,65]. Although not quantified in this research, the investigated CIWEs differed in their viscosity as a function of wall composition and core load. In light of the effect of viscosity, at a given atomization conditions, on the size of the atomized droplets and, consequently, on drying rate, the among-powders differences in moisture content could be attributed to influence of both proportion and type of MD and the viscosity of the CIWE.

3.4. Core retention and microencapsulation efficiency

Core content of spray dried microcapsules (Table 4) exhibited an appreciable among-powder differences that could be attributed to the combined influence of compositional aspects that affected core retention during spray drying, as explained below. Core content of microcapsules prepared with CIWE containing 25, 50 and 60% (w/w) oil ranged, from 18.7 to 22.9% (w/w), from 41.2 to 47.9% (w/w) and from 52.2 to 53.2% (w/w), respectively.

Core retention during microencapsulation by spray drying is affected (among other things) by the properties and composition of the CIWE, and by the influence of atomization and drying conditions [6,62,63,69]. Results (Table 5) indicated that core retention ranged from 72.2 to 95.9% and was significantly ($p < 0.05$) affected by the composition of the CIWE. Core retention in this study was higher than that reported by Tang and Li [55] for encapsulation by spray drying of oil in SPI-based wall matrices. Core retention was mainly influenced by a combined influence of the relative proportions of SPI and MD that were included in the wall solution, and by the core-to-wall ratio in the CIWE. It has been established that at a given atomization and drying conditions, losses of lipid type core during microencapsulation by spray drying requires core droplets to reach the outer surface of the drying particles from where they can be removed by the turbulent air flow around the particles [6,62,63]. Core losses are thus influenced by the proportion of core that is present at the surface of the drying particles as they leave the atomizer and by the migration of core droplets to the surface from interior parts of the drying particles, due to the internal mixing that exists during the constant rate phase of the drying, prior to the formation of dry crust [6,62,63,69]. Results of structural details of outer surface of the dry microcapsules revealed the presence of "foot prints" of core droplets that had been swept off the surface during the drying process (Figure 3A). Results of the present study agreed with those previously reported for lipid-containing, whey-protein-based microcapsules [63,66]. Core retention during spray drying of CIWE is influenced by compositional aspects that govern droplets formation during atomization, drying rate and, consequently, the time elapses between atomization and the formation of dry crust around the drying droplets [6,62,63,66]. Results (Table 5) indicated that core retention obtained with wall solutions AL, CL and CH was proportionally related ($p < 0.05$) to the initial core content in the CIWE while that obtained with wall solutions AH and BH was not influenced by the initial core load. At SPI concentration of 10%, core retention was proportionally related to the initial core load ($p < 0.05$), regardless of DE value of the carbohydrate. Results (Table 5) indicated that the overall lowest core retention was obtained with CIWE C25L (74.9%) and C25H (72.2%) while very high retention was obtained with CIWE C50L (94.2%) and C50H (94.3%). Although Results (Table 3) did not indicate a high extent of ballooning

in powders obtained with CIWE C25L and C25H, the relatively low MD content (10%) could, potentially, contributed to a relatively slower drying rate than that with higher MD content that allowed an extended period of time prior to crust formation around the drying droplets [63]. It can be suggested that the latter and the initial relative low core content in CIWE (25%) could have led to a relatively high core loss from the surface of the drying droplets. Results presented in Table 5 suggested that core retention was affected by a combined influence of the proportion of SPI and MD in the wall solutions as well as by the DE value of MD. It has been indicated that at a given total solids of CIWE with WS consisting of proteins and carbohydrates, core retention is promoted by the proportion of carbohydrates included in the wall system [56,61-63,66]. The latter was attributed to a faster drying rate that promoted a rapid formation of crust around the drying particle and thus limited the period of time over which significant core losses could occur [6,62,63]. The extent to which these effects influenced core retention varied among the investigated CIWE (Table higher than that obtained with C25L and C25H, respectively. However, core retention obtained with CIWE with 50% lipids and H type MD was in the order C50H = B50H > A50H and core retention obtained with CIWE containing 50% lipids and L type MD was not affected by the proportion of SPI included in CIWE ($p > 0.05$). Results of the study thus indicated that core retention reflected the overall balance of the extents to which each of the wall constituents affected the physico-chemical properties of the drying droplets that, in turn, governed core losses. It has been reported that for MD-containing CIWE, core retention during microencapsulation by SD was proportionally related to the DE value of the MD [6,63]. Results presented in Table 5 indicated only a limited effect of the DE value of the MD on core retention. Core retention obtained with CIWE A50L was significantly higher than that obtained with CIWE A50H ($p < 0.05$). In all other cases, core retention obtained at a given composition of CIWE was not affected ($p > 0.05$) by the DE value of the COH.

A discussion about the effects of compositional variables on MEE has to recognize the physical meaning of core extractability and MEE. The proportion of core that can be extracted from microcapsules at a given set extraction conditions consists of the true surface oil, the outer layer core in the surface layer of the particle, core that can be extracted by the solvent from sub-surface domains of the wall matrices through capillary forces and core that can be reached by solvent through empty wall matrix domains left by already extracted core [68,69]. For microcapsules prepared at a given set of atomization and drying conditions, core extractability (and thus MEE) is governed by the combined influence of composition and microstructure of the wall matrices, composition and properties of the structures adsorbed at the surface of core droplets, composition of the outer surface of microcapsules, PSD of CIWE and changes in the latter during drying as well as by the physico-chemical properties, state and hydrophobicity of wall constituents [62-65,68,69].

The proportion of lipid-type core that resides on the surface of spray dried microcapsules, true surface core, is of significant important to the reconstitution characteristics, flow properties and oxidative stability of the microcapsules [68-71]. The latter and the proportion of core that cannot be extracted, at a given set of conditions, from such microcapsules (MEE) can allow assessing the extent to which the lipid core is partitioned throughout the wall matrix as well as the extent to which this core is physically "protected" in the wall system [68-71]. A "standard method" for determination of MEE has not been established yet and different analytical approaches have been suggested [69]. In the present research, MEE was investigated at both relatively short, 5 min ($MEE_{(5)}$, and long, 30 min, $MEE_{(30)}$, extraction times and thus allowed establishing some understanding and information about surface core and core that could be extracted from interior parts of the microcapsules [69]. Results

(Table 5) indicated that the investigated microcapsules exhibited a very broad range of MEE values and suggested a combined influence of extraction time and composition on core extractability ($p < 0.05$). The overall highest and lowest MEE were exhibited by microcapsules prepared with CIWE B25H (91.6–91.4%) and A60H (25.4–12.4%). The very high proportion of small microcapsules that was included in the powder prepared with CIWE B25H could have been expected to lead to a low MEE [68] and yet it exhibited the overall highest MEE. The latter suggested that in this powder, the effect of wall composition and initial core load in CIWE on MEE was more significant than that of the ratio of surface area to volume. Results indicated that extraction time had an insignificant effect on MEE (Table 5). It can thus be assumed that the less than 10% of core that was extracted from this powder represented true surface oil and that the rest of the core was well protected by wall matrices that were impervious to the extracting solvent. The proportion of core that resides at the surface of the SD microcapsules has been shown to be proportionally related to the mean particle size in CIWE [6]. Results (Table 2) indicated some small, yet significant, among-batches differences in $d_{3,2}$ of CIWE however, the small magnitude of these differences probably did not have a very dramatic effect on MEE.

In most cases, for a given wall system, MEE of microcapsules prepared with CIWE containing 25% lipids was not significantly affected by extraction time ($p > 0.05$). However, in most other cases, MEE of microcapsules prepared with a higher initial core load was inversely proportional to extraction time ($p < 0.05$). Results could be attributed to effect of number of core domain per unit volume of wall matrix and their spatial distribution on core extractability [63,64,68,69]. For a given wall system and regardless of extraction time, MEE decreased with the initial core content in CIWE ($p < 0.05$). For example, $MEE_{(5)}$ and $MEE_{(30)}$ of microcapsules prepared with CIWE A60H was 3.5 and 7.8 times lower than that obtained with microcapsules prepared with CIWE A25H, respectively (Table 5). At a given wall composition, increasing core content resulted in thinner wall layers separating core droplets from each other. The thinner matrix layer represented a shorter diffusion path and thus, at a given extraction time, the overall amount of core that could be extracted increased and lead to a lower MEE [6,63,64,69]. In most cases (Table 5), for a given type of MD and regardless of extraction time, MEE of microcapsules prepared at a given lipid load was inversely proportional to SPI content ($p < 0.05$). For example, $MEE_{(5)}$ of microcapsules prepared with CIWE A25L was 19.5% higher than that obtained with microcapsules prepared with CIWE C25L and $MEE_{(5)}$ of microcapsules prepared with A50L was 55.1% higher than that of microcapsules prepared with CIWE C50L (Table 5). The effect of SPI content on MEE could be attributed to the overall increase, with SPI content, in the number of hydrophobic domains in wall matrices. Core extraction by the non-polar solvent is governed by a leaching process [62,63,69] and it is thus clear that the permeation and diffusion of the extracting solvent in the wall matrix was promoted by SPI content [62-64]. Regardless of core load and SPI content in CIWE, MEE was proportionally related to DE value of the MD constituents included in the wall system ($p < 0.05$). At a given SPI and core contents, MEE obtained with H type MD (DE value 17.5) was significantly higher (Table 5) than that obtained with L type MD with DE value of 7.5 ($p < 0.05$). For example, $MEE_{(5)}$ and $MEE_{(30)}$ of microcapsules prepared with CIWE containing 25 or 50% core and L type MD ranged from 32.3 to 87.6% and from 22.7 to 89.4%, respectively. However, similarly, $MEE_{(5)}$ and $MEE_{(30)}$ of microcapsules prepared with CIWE containing H type MD ranged from 57.9 to 90.2% and from 47.9 to 90.3%, respectively (Table 5). The effect of DE value had on MEE could be attributed to the increase, with DE value, in the proportion of low molecular weight oligosaccharides in the MD. The

latter has been shown to allow a better formation of a less porous matrix during spray drying. This, and the formation of a hydrophilic amorphous phase, consisting of the dry oligosaccharides, that filled spaces between the protein constituents of the wall, limited the diffusion of the extracting solvent through the matrix and thus enhanced MEE [63,65,69,74].

Table 4. Moisture and oil (core) content of spray-dried microcapsules.

CIWE[1]	Moisture (%, w/w)	Oil (%, w/w)
A25L	0.89 (0.03)[2]	20.53 (2.51)
A25H	1.16 (0.21)	20.58 (2.34)
A50L	0.90 (0.07)	47.93 (0.79)
A50H	1.65 (0.07)	41.25 (1.32)
A60L	1.10 (0.11)	53.28 (2.57)
A60H	0.94 (0.11)	52.28 (1.37)
B25H	1.12 (0.07)	22.88 (0.68)
B50H	1.36 (0.14)	47.14 (0.64)
C25L	0.23 (0.051)	18.72 (1.50)
C25H	0.41 (0.14)	18.05 (0.84)
C50L	2.11 (0.19)	47.12 (0.69)
C50H	1.53 (0.29)	47.15 (2.07)

[1]See Table 1 for composition of CIWE

[2]x(y)—mean value and standard deviation

Table 5. Core retention and microencapsulation efficiency in spay-dried microcapsules.

CIWE	Core retention (%)	$MEE_{(5)}$[1] (%)	$MEE_{(15)}$ (%)	$MEE_{(30)}$ (%)
A25L	82.1^{Abl}	87.6^{cACm}	89.7^{cACl}	89.4^{cACl}
A50L	95.9^{Aal}	50.1^{cADm}	40.7^{dADm}	33.1^{fADm}
A60L	88.8^{abl}	NA	NA	NA
A25H	82.3^{Aal}	90.2^{cACl}	90.2^{cACl}	90.3^{cACl}
A50H	82.5^{Bam}	57.9^{cBDl}	52.9^{dBDl}	47.9^{fBDl}
A60H	87.1^{al}	25.4^{cF}	16.6^{dF}	12.4^{fF}
B25H	91.5^{Aa}	91.6^{cAC}	91.3^{cAC}	91.4^{cAC}
B50H	94.3^{Aa}	76.7^{cAD}	73.2^{dAD}	70.5^{fAD}
C25L	74.9^{Bbl}	73.8^{cBCm}	70.2^{dBCm}	68.3^{fCm}
C50L	94.2^{Aal}	32.3^{cBDm}	26.0^{dBDm}	22.7^{fBDm}
C25H	72.2^{Bbl}	86.1^{cBCl}	84.9^{cBCl}	82.3^{cBCl}
C50H	94.3^{Aal}	59.5^{cBDl}	52.9^{dBDl}	48.3^{fBDl}

[1]$MEE_{(x)}$ Microencapsulation efficiency obtained after 5, 15 or 30 min of extraction

[ab]For a given wall system, means of core retention or followed by different letters differ significantly ($p < 0.05$)

[AB]For CIWE containing a given core load (25 or 50%) and maltodextrin of a given DE value (L or H, see Table 1), means of core retention or means of MEE in a given column followed by different letters differ significantly ($p < 0.05$)

[lm]For CIWE containing a given SPI concentration (A B or C, see Table 1) and a given core load (25, 50 or 60%), means of core retention or means of MEE in a given column followed by different letters significantly different ($p < 0.05$)

[cdf]For a given CIWE, means in a given row followed by different letters are significantly different ($p < 0.05$)

[CDF]For a given wall system, means of MEE in a given column followed by different letters are significantly different ($p < 0.05$)

4. Conclusions

Results of the present study indicated that wall solutions consisting of SPI and maltodextrin allowed, even at SPI concentration of 2.5%, effective formation of CIWE, containing up to 60% model oil, that exhibited desired PSD properties. Results clearly indicated that although all of the investigated CIWE could be spray dried into microcapsules with desired microstructural properties, and, in most cases, high core retention was attained, the MEE of the capsules varied significantly. MEE can be used as an indicator for the protection provided by the wall matrices to the core. Additionally, MEE can provide, in an extraction-time-dependent manner, information about the spatial distribution of core, both on the outer surfaces and in the interior parts of the microcapsule. In light of the importance of the latter to the stability and functionality of lipids-containing spray dried microcapsules, results of the study clearly highlighted the need to carefully assess and optimize the mass ratio between protein- and carbohydrate-based wall constituents as well as the molecular weights distribution of the carbohydrates. Results add to the volume of information that has already established pertinent to applications for SPI in microencapsulation and provides a new insight into the opportunities and constrains that govern this application

Conflict of Interest

The authors report no conflict of interests in this research.

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