AIMS Agriculture and Food, 3(1): 66–84.
DOI: 10.3934/agrfood.2018.1.66
Received: 01 February 2018
Accepted: 05 March 2018
Published: 08 March 2018

http://www.aimspress.com/journal/agriculture

Research article

Microencapsulation properties of wall systems consisting of WHPI and carbohydrates

Jing Zhang[1], Yael Rosenberg[2] and Moshe Rosenberg[2,*]

[1] College of Food Science and Engineering, Northwest A&F University Yangling, Shaanxi, Peoples Republic of China
[2] Department of Food Science and Technology, University of California Davis, Davis, CA, USA

*** Correspondence:** Email: mrosenberg@ucdavis.edu; Tel: +5307524682.

Abstract: Microencapsulation allows entrapment, protection and delivery of sensitive desired nutrients and other food ingredients and compounds. The research has investigated the encapsulation, by spray drying (SD), of a model oil in wall systems consisting of blends of wheat proteins isolate (WHPI) and maltodextrins (MD, DE 5 or 15) or corn syrup solids (CSS, DE 24). Wall solutions contained 2.5–10% (w/w) WHPI and 17.5–10% (w/w) MD or CSS. Oil load in core-in-wall emulsions (CIWE) ranged from 25 to 75% (w/w). Mean particle diameter in CIWE was smaller than 0.5 μm. Surface excess in the CIWE ranged from 1.544 to 6.497 mg/mL and was influenced ($p < 0.05$) by the composition of the CIWE. Microcapsules exhibited structural characteristics that are typical to spray dried microcapsules and a limited extent of surface indentation. In all cases, the protein-coated lipid droplets were embedded throughout the wall matrices and no visible cracks connecting the core domains with the environment could be detected. Core retention during microencapsulation ranged from 77.7 to 97.2% and was governed by a combined influence of the wall composition and wall-to core ratio ($p < 0.05$). Microencapsulation efficiency, MEE, ranged from 11.71 to 97.79% and was significantly ($p < 0.05$) affected by the combined influence of the composition of the wall matrices, the DE value of the COH and by the wall-to-core ratio in the CIWE. Results indicated that wall solutions containing 2.5–10% WHPI and 17.5–10% maltodextrins can offer opportunities for microencapsulation, by spray drying, of high oil load. Results thus open a new horizon for utilization of WHPI as microencapsulating agent in food applications.

Keywords: wheat proteins isolate; microencapsulation; spray drying core retention; microencapsulation efficiency; core-in-wall-emulsion

1. Introduction

Microencapsulation refers, collectively, to technologies and processes that allow entrapping or embedding gas bubbles, liquid droplets or solid particles ("core") in particulate matrices ("wall") that are design to prevent the deterioration of the core's physico-chemical, biological or functional properties and, ultimately release the core at a desired set of conditions [1–7]. In recent decades, microencapsulation has been utilized in food applications to an extent that is second only to pharmaceutical applications for this technology [1]. Microencapsulation has allowed to successfully meeting otherwise unattainable goals pertinent to food processing and to the effective delivery of desired nutrients, sensitive and functional ingredients, microorganisms and biologically active compounds [2,6,8–16]. Microencapsulation provides means for effectively protecting such constituents of food formulations and products against deterioration or loss during food processing and throughout the shelf life of the product. Microencapsulation allows preventing undesired and /or pre-mature interactions among constituents of a given formulation. It provides means to modulate the sensorial, textural, functional and biological quality attributes of food products and allows controlling the mode, rate, conditions and environment at which the core is released from the wall matrices [4,15,17–20]. Different physical and chemical microencapsulation processes have been developed and implemented, to varying extent, by the food and related industries. In recent decades a broad array of microcapsules and microspheres with different geometry, dimensions, structure, texture, physico-chemical, core release and functional properties has been developed [3,4,6,13,21–25]. These accomplishments allow tailoring microcapsules and microsphere to meet different challenges in food systems.

The availability of highly functional and cost effective GRAS wall materials for microencapsulation in food applications has been a challenge and a continuous effort to identify new highly functional GRAS microencapsulating agents exists [16,20,21,26–28]. Proteins exhibit physico-chemical and functional properties that are desired in microencapsulating agents for food applications. Among these characteristics are emulsification and gelation properties, film forming, pH-dependent solubility profile, surface-activity, association properties etc [5,29–32]. By wisely highlighting these properties, applications for some animal- and plant-derived, native or modified, proteins as microencapsulating agents have been developed and demonstrated [5,29–36]. It has been suggested that plant-derived proteins offer some advantages over animal-derived proteins when application as wall material for microencapsulation is considered [32]. Effort to developed such applications has significantly increased during recent years [29,32,35,37,38].

Wheat proteins consist of gluten, that accounts for about 80% of wheat proteins, and non-gluten proteins. The gluten fraction consists of two major components, gliadin and glutenin, that differ in their physico-chemical properties. Gliadin consists of single chain polypeptides (25–100 kDa) linked by intramolecular disulfide bonds. The glutenin fraction consists of gliadin-like subunits, stabilized by intermolecular disulfide bonds, with a molecular weight higher than 105 kDa [34,39]. The physico-chemical properties of gluten and its fractions as well as those of wheat protein isolate have been investigated and the properties that are important to their utilization as microencapsulating agent have been highlighted [40–43]. Overall, the extent to which applications of wheat proteins as microencapsulating agents in food and pharmaceutical applications has been investigated is relatively limited and, in many cases, such applications involved utilization of solvents and chemical cross-linking [44–50].

Recently, we have reported on the functionality of blends consisting of soy proteins isolate (SPI) and carbohydrates as effective wall system for microencapsulation of lipids by spray drying [51]. Using a similar approach, the objective of the research that is reported here was to investigate the microencapsulation, by spray drying, of a model oil in wall systems consisting of wheat protein isolate (WHPI) and selected carbohydrates (COH).

2. Materials and methods

2.1. Wall and core materials

Wheat Protein isolate (WHPI) ProliteTM 100 containing 90% proteins (w/w, N × 6.25) was obtained from Archer Daniels Midland (Keokuk, IA) and served as protein-based wall constituent. Maltodextrins, with a dextrose equivalent (DE) of 5 (MD5) or 15 (MD15) and corn syrup solid (CSS) with a DE value of 24 were purchased from Grain Processing Corporation (Muscatine, LA) and served as carbohydrate-based wall constituents (COH). Soy oil was purchased at a local supermarket and served as a model core material.

2.2. Microencapsulation by spray drying

Preparation of core-in-wall emulsions (CIWE): Wall Solutions (WS) containing 20% (w/w) solids consisting of 2.5, 5.0 or 10.0% (w/w) WHPI and 17.5, 15.0 or 10.0% (w/w) COH, respectively, were prepared in de-ionized water (Millipore, 18.2 MΩ.cm). These WS were designated 2.5/17.5, 5.0/15.0 and 10.0/10.0, respectively. In all cases, an aqueous dispersion of WHPI was prepared at 40 ± 1 °C and after adding 0.02% sodium azide (Fisher Scientific, Pittsburgh, PA) it was slowly stirred for 12 h at 25 ± 1 °C to allow full hydration and swelling of the protein constituents. Then, the COH constituent of the WS was dissolved into the WHPI solution and the mixture was stirred for additional 2 h at 25 ± 1 °C.

Soybean oil was emulsified into the WS at a wall-to-core mass ratio (W:C) of 75:25, 50:50 or 25:75. Emulsification was carried as previously described [51]. In short, a coarse emulsion was prepared by emulsifying 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 at 25 ± 2 °C. The coarse emulsion was then homogenized (at ambient temperature) for four successive homogenization steps at 50 MPa using a model NS1001L2K—PANDA high pressure homogenizer (Niro Soavi S.p.A., Parma, Italy). The resulting CIWEs were designated according to their WHPI and COH content, the type of COH that was included in the WS and the W:C ratio. For example: The CIWE 2.5/17.5/MD5/25:75 was prepared with WS containing 2.5% WHPI and 17.5% MD5, and had a wall-to-core mass ratio of 25:75.

Spray drying: The investigated CIWE were spray dried using an APV Anhydro Laboratory Spray Dryer (APV Anhydro A/S SØborg, Denmark). The 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 ± 2 °C, and 80 ± 2 °C, respectively. Microcapsule powders were collected, placed in hermetically closed glass jars and kept in desiccators pending analyses.

2.3. Analyses

Particle size distribution: The particle size distribution (PSD) properties of the CIWE were determined using a Malvern Mastersizer MS20 (Malvern Instruments, Malvern, England). In all cases, analysis was carried out in quadruplicates using a 2-mW He-Ne laser beam (633 nm) and a 45-mm focus lens. The PSD, mean particle size (volume-size average, $d_{3,2}$ μm) and the specific surface area (SSA m^2/mL) were recorded.

Surface excess: The amount of protein that was adsorbed or directly engaged per unit surface area of the O/W interface in the investigated CIWE (Γ, mg/m^2) was investigated using a procedure that was developed by modifying protocols that had been previously reported [52,53]. The procedure allows recovering protein-coated lipid droplets and removing proteins that are either entrapped or only loosely engaged at the surface of the lipid droplets. Sucrose was added to samples of CIWE to a final concentration of 28.6% (w/w) a 5 mL aliquot of the treated CIWE was then placed under a layer of 25 mL de-ionized water that had been placed in a 50-mL centrifuge tubes, using a 10-mL syringe. Following centrifugation at 10,000 g for 60 min at 20 °C (Marathon 21 K/R centrifuge, Fisher Scientific, Pittsburgh, PA), the aqueous phase of the separated CIWE was removed and the cream layer was re-suspended ("washed") in de-ionized water to the original volume of the treated sample. The centrifugation and the "washing" steps were repeated two additional times. In all cases, triplicate samples of the investigated CIWE were treated as described. Results of a preliminary study (data not provided here) indicated that after three successive "washing and separation" steps, the protein content of the separated "cream" reached a minimum that was not affected by additional washing steps. The protein constituents of such "creams" could thus be considered to be truly adsorbed or strongly engaged at the O/W interface. The "cream" obtained after the third centrifugation was collected and analyzed for total protein and fat content.

Protein content: Total protein content (N × 6.25) of the separated washed cream was determined according to the Macro-Kjeldhal method [54], using a Kjeltec system (Tecator, Hoganas, Sweden).

Oil content: Total oil content of the separated washed cream was determined using a modification of the Roese-Gottlieb method as previously reported [51].

The parameters: SSA (of CIWE), protein and fat content of washed cream, and the density of soy oil at 20 °C (0.915 g/mL) were used to calculate the surface excess (Γ) of the investigated CIWE as previously described [55] according to Eq 1.

$$\Gamma = \frac{P}{\frac{O}{0.916} \times SSA} \tag{1}$$

Where: Γ is surface excess (mg/m^2), P and O are protein and oil content in washed cream (mg/g), respectively, and SSA is specific surface area of CIWE (m^2/mL).

Total core content: Core (lipids) content of the spray-dried (SD) microcapsules (OMC) was determined, in quadruplicates, using a modification of the Roese-Gottlieb method, as previously reported [51,56]. In short, 1 g of dry microcapsules was reconstituted in 9 mL of de-ionized water and the resulted emulsion was treated with 1.25 mL ammonium hydroxide. After adding 10 mL of ethanol, the lipids were extracted (three successive times) with a mixture of ethyl ether and petroleum ether. Analysis was carried out in quadruplicate.

Core retention: The core (lipids) retention during spray drying (CR) was defined as the ratio (expressed in %) of core content included in 100 g of moisture-free SD microcapsules to that in 100 g moisture free CIWE solids [51] and was expressed according to Eq 2.

$$\text{CR (\%)} = \frac{\text{OMC}}{\text{OE}} \times 100 \tag{2}$$

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 [51]. MEE was defined as the proportion (in %) of OMC that was not extracted by petroleum ether from the microcapsules during 15 min of extraction at standard conditions. In short, one gram of SD microcapsule was placed in a 50 mL Quorpak glass bottle (Fisher Scientific, Pittsburgh, PA) and was dispersed in 25 mL of petroleum ether (analytical grade, bp 70 ˚C, Fisher Scientific, Pittsburgh, PA). The bottles were capped with a Teflon-lined closure, were 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 15 min at 25 °C. 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 °C, and the solvent-free extract was dried (45 °C, 6.7 kPa). The dry extract was allowed to reach room temperature in a desiccator and its mass (EO) was then determined gravimetrically. Analysis were carried out in quadruplicate. Microencapsulation efficiency was calculated according to Eq 3

$$\text{MEE (\%)} = \frac{\text{OMC-EO}}{\text{OMC}} \text{ X } 100 \tag{3}$$

Where: EO is the amount of core (oil) that was extracted after 15 min (mg), OMC is the amount of core that is included in 1 g of dry microcapsules.

2.4. Scanning electron microscopy (SEM)

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 attached to 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 analyzed using a Philips XL-FEG scanning electron microscope at 5 keV.

2.5. Statistical analysis

The significance of the results was tested at $p < 0.05$ using the analysis of variance (ANOVA) test procedures included in the SigmaStat software (Jandel Scientific Software, San Rafael, CA).

3. Results and discussion

3.1. Emulsion properties

The formation, stability and functionality of lipids-containing spray-dried microcapsules is significantly affected by the physico-chemical, rheological, stability and structural properties of the CIWE [14,57,58]. The colloidal characteristics and stability of protein-stabilized CIWE as well as the oxidative stability and functionality of microcapsules that are prepared from this CIWE are significantly influenced by the formation, composition, structure and rheological properties of stable protein-based films at the O/W interfaces [51,56,58–60]. The formation of CIWE with a mean particle diameter smaller than 0.5μm is of prime importance to success in microencapsulation of lipids by spray drying [14,56,61]. Results (Figure 1) indicated that in most cases PSD of the investigated CIWE exhibited uni-modal or close to unimodal PSD. An indication for some bi-modal features of the PSD was exhibited only by CIWE containing 2.5% WHPI with a wall-to-core ratio of 25:75 and, to a lower extent, by CIWE containing 5% WHPI with a core-to-wall ration of 25:75. Each of the four successive homogenization steps in the single stage configuration resulted in a significant increase in the total interfacial area of the emulsion and thus challenged the formation of stable protein-based films at the O/W interface, especially when protein content was low and lipid load was high. In these cases, the presence of a significant number of oil droplets with diameter larger than 1 μm could be attributed to some coalescence and formation of homogenization clusters in the CIWE [62]. The extent to which these phenomena are manifested increases with oil load in the emulsion, especially when the concentration of the surfactant is low [63]. It has been established that for given homogenization conditions and for a given wall composition and non-limiting availability of surface active material (protein), increase in $d_{3,2}$ with lipid load can be attributed to some phenomena that occur inside the homogenization valve. Among these are an overall longer particles disruption time, a longer period of time that is needed to complete the adsorption of proteins at the newly formed O/W interface and a very significant decrease in the particles encountering time, that is, the time that is needed for two or more partially protein-coated oil droplets to encounter each other and form a cluster [62]. The bi-modal PSD of CIWE with 2.5% or 5% WHPI and a W:C ratio of 25:75 suggested that, in addition to the latter mechanisms, protein concentration was probably a limiting factor that affected the PSD.

Some of the CIWE exhibited a very small shoulder at the low end of the particles diameter range that indicated the presence of a significant proportion of particles with a very small diameter. Overall, the PSD that were obtained with the investigated CIWE were similar to what had been reported for CIWE containing blends of soy protein isolate (SPI) and COH [51] and were similar or superior to some of those that had been reported for CIWE containing blends of whey protein isolate (WPI) and COH [59,61].

Results (Table 1) indicated that in all cases the $d_{3,2}$ of the investigated CIWE was smaller than 0.5 μm and ranged from 0.270 to 0.485 μm and that the SSA of the investigated CIWE ranged from 12.396 to 22.212 m^2/mL. The $d_{3,2}$ of the CIWE was significantly influenced ($p < 0.05$) by the proportions of WHPI and COH in the CIWE, by the core-to-wall ratio and by the type of COH. In all but one case, for a given wall composition (WHPI/COH) and regardless of the type of COH, $d_{3,2}$ was proportionally related ($p < 0.05$) to the core load in the CIWE (Table 1). This effect could be

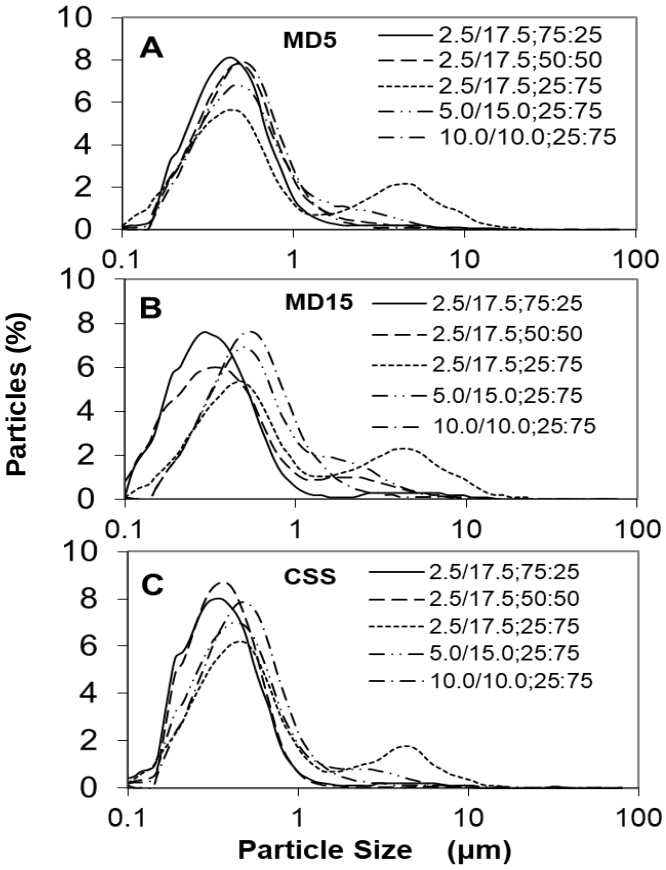

Figure 1. Particles size distribution of CIWE consisting of oil dispersed in wall solutions consisting of WHPI and MD5 (A), MD15 (B) or CSS (C). CIWEs are denoted as described in the methods and materials.

attributed to the influence of lipids load on coalescence and formation of homogenization clusters, as discussed above. It could have been expected that, in general, for a given wall-to-core ratio and type of COH, $d_{3,2}$ would be inversely related to the proportion of WHPI that was included in the wall system. Results (Table 1) indicated that the latter was evident only to a limited extent and that in some cases $d_{3,2}$ was either unaffected by protein content or exhibited some increase with protein content. Results indicated that, regardless of lipid load in the CIWE, the surface active properties of the protein constituents of the WHPI allowed effective formation and stabilization of CIWE at all the investigated lipids loads and in addition to the mere availability of proteins, $d_{3,2}$ was probably also influenced by the effect of protein content on the viscosity of the aqueous phase of the CIWE. It has been reported that at a given set of homogenization conditions and lipids load, the homogenization efficiency is adversely influenced by increase in viscosity [62]. It has been reported that the $d_{3,2}$ of CIWE containing blends of proteins and COH consisting of MD or CSS was inversely related to the DE value of the COH [51,59,61]. Results (Table 1) indicated that in all but one case, for a given wall composition (WHPI/COH) and W:C ratio, $d_{3,2}$ of CIWE that contained MD5 was larger ($p < 0.05$) than that of CIWE containing CSS. In general, in most cases, for a given wall composition

(WHPI/COH) and W:C ratio, $d_{3,2}$ of CIWE that contained MD15 was larger ($p < 0.05$) than that of CIWE containing CSS. In all but two cases, for a given wall composition (WHPI/COH) and W:C ratio, $d_{3,2}$ of CIWE that contained MD5 was larger ($p < 0.05$) than that of CIWE containing MD15. Results were similar to those reported for CIWE with wall solutions containing blends of soy proteins and COH [51]. The effect of DE value on $d_{3,2}$ has been attributed to the DE value-dependent formation of a pseudo-network consisting of HMW oligosaccharides in which proteins can, potentially, become entangled [59,61]. It has been suggested that in such cases, the effective availability of proteins to become engaged at the O/W interface during homogenization is lower that what can be expected based on their overall concentration [61]. In such cases, and especially with CIWE that contain high lipids load and relatively low protein concentration, the formation of homogenization cluster is likely to be enhanced. The extent to which such phenomena manifest itself is likely to be inversely related to the DE value of the COH constituents of the wall solution. The SSA of the CIEW prepared with WS containing 2.5, 5.0 or 10.0% WHPI ranged from 12.396 to 22.338 m^2/mL, from 13.141 to 22.212 m^2/mL and from 12.798 to 19.395 m^2/mL, respectively, and reflected the overall combined influence of wall composition and W:C ratio on the PSD properties of the CIWEs.

Table 1. Mean particle sizes ($d_{3,2}$) and specific surface area (SSA) of core-in-wall emulsions.

WHPI/COH[1]	W:C[2]	d_{32} (μm) / SSA (m^2/mL)		
(%/%)	(%:%)	MD 5	MD 15	CSS
2.5/17.5	75:25	$0.350^{a,C}/17.023^{c,A}$	$0.270^{c,C}/22.338^{a,A}$	$0.290^{b,C}/20.623^{b,A}$
	50:50	$0.390^{a,B}/15.452^{c,B}$	$0.290^{c,B}/21.067^{a,B}$	$0.305^{b,B}/19.698^{b,B}$
	25:75	$0.418^{b,A}/14.379^{a,C}$	$0.485^{a,A}/12.396^{b,C}$	$0.410^{b,A}/14.721^{a,C}$
5.0/15.0	75:25	$0.350^{a,C}/17.087^{c,A}$	$0.295^{b,B}/20.491^{b,A}$	$0.270^{c,C}/22.212^{a,A}$
	50:50	$0.360^{a,B}/16.615^{b,B}$	$0.305^{b,B}/19.630^{a,A}$	$0.310^{b,B}/19.444^{a,B}$
	25:75	$0.400^{b,A}/15.129^{b,C}$	$0.453^{a,A}/13.141^{c,B}$	$0.365^{c,A}/16.434^{a,C}$
10.0/10.0	75:25	$0.335^{a,C}/18.021^{b,A}$	$0.330^{a,C}/17.903^{b,A}$	$0.310^{b,C}/19.395^{a,A}$
	50:50	$0.400^{a,B}/14.928^{b,B}$	$0.390^{a,B}/15.391^{b,B}$	$0.333^{b,B}/18.022^{a,B}$
	25:75	$0.460^{a,A}/12.798^{c,C}$	$0.430^{b,A}/13.827^{b,C}$	$0.390^{c,A}/15.441^{a,C}$

[ABC]For a given wall system, means, of a given measured variable, in a given column followed by different letters are significantly different ($p < 0.05$). [abc]For a given wall system, means, of a given measured variable, in a given row followed by the different letters are significantly different ($p < 0.05$). [1]Proportions (%) of WHPI and carbohydrate (COH) in wall solution of CIWE. [2]Wall-to-core ratio (%:%) in CIWE.

Table 2. Surface excess (mg/m^2) of CIWEs.

WHPI/COH[1]	*W:C*[2]	*surface excess(mg/m^2)*		
(%/%)	*ratio*	MD 5	MD 15	CSS
2.5/17.5	75:25	4.821 $^{a, A}$	3.587 $^{b, A}$	2.735 $^{c, A}$
	50:50	4.368 $^{a, B}$	3.241 $^{b, B}$	2.518 $^{c, B}$
	25:75	1.736 $^{b, C}$	2.117 $^{a, C}$	1.544 $^{c, C}$
5.0/15.0	75:25	5.284 $^{b, A}$	5.373 $^{a, A}$	2.262 $^{c, C}$
	50:50	4.587 $^{b, B}$	4.932 $^{a, B}$	3.015 $^{c, A}$
	25:75	3.133 $^{b, C}$	3.928 $^{a, C}$	2.422 $^{c, B}$
10.0/10.0	75:25	5.976 $^{a, B}$	5.768 $^{b, A}$	4.134 $^{c, A}$
	50:50	6.497 $^{a, A}$	5.528 $^{b, B}$	3.971 $^{c, B}$
	25:75	4.368 $^{b, C}$	5.021 $^{a, C}$	3.768 $^{c, C}$

^{ABC}For a given system, means in a given column, followed by different letters are significantly different ($p < 0.05$). ^{abc}For a given system, means in a given row, followed by different letters are significantly different ($p < 0.05$). [1]Proportions (%) of WHPI and carbohydrate (COH) in wall solution of CIWE. [2]Wall-to-core ratio (%:%) in CIWE.

The formation of well-established continuous and stable protein-based structures (films) at the O/W interface is critically important to the formation, stability and functionality of protein-stabilized CIWEs as well as to the ultimate quality, stability and functionality of microcapsules that are prepared from these CIWEs. Ideally in such cases, it is desired that the entire newly formed interfacial surface that is created during homogenization will become completely covered by protein-based structure before the emulsion leaves the homogenization valve. Success in meeting this objective is affected by the combined influence of homogenization pressure, effective concentration of proteins, surface activity of the proteins, flexibility of the protein molecules, surface hydrophobicity properties of the protein, lipid load in the emulsion, temperature, as well as by process and equipment configuration [62,63]. As has been explained above, the protein content of the washed creams could be considered to represent proteins that were directly adsorbed at the O/W interfaces or proteins that were tightly engaged in interactions with proteins that were adsorbed at the O/W interface. Therefore, effects of the composition of CIWE and their PSD properties on surface excess (Γ) could be assessed. Results (Table 2) indicated that surface excess of the investigated CIWEs was significantly affected by the combined influence of wall composition, lipid load in the CIWE and by the particles size distribution properties of the emulsions (Table 1). In general, surface excess ranged from 1.544 to 6.497 mg/mL (Table 2) and, in all but two cases, for a given WHPI and COH blend, Γ was inversely proportional to lipid load in the CIWE ($p < 0.05$). For CIWE containing 2.5% WHPI, the surface excess of CIWE with W:C ratio of 25:75 was 64.2, 59.1 and 56.5% of that found for CIWE with W:C

ratio of 75:25 that contained MD5, MD15 and CSS, respectively (Table 2). Similarly, surface excess in CIWE that contained 5% WHPI with a W:C ratio of 25:75 was 53.6, and 73.1% of that in CIWI with W:C ratio of 75:25, that contained MD5 and MD15, respectively. Results obtained with CIWEs containing 10% WHPI indicated that surface excess in CIWE with W:C ratio of 25:75 was 73.2, 87.1 and 91.1% of that in CIWE with a W:C ratio of 75:25 that contained MD5, MD15 and CSS, respectively. In all cases, for a given W:C ratio and COH, surface excess increased with WHPI concentration in the WS (Table 2). In light of the very small differences in $d_{3,2}$ among the CIWE (Table 1) this could be probably attributed to post-homogenization protein-protein interactions where proteins that were adsorbed at the O/W interface interacted with unengaged protein molecules from the bulk phase to form advanced structures or "multi-layer films" at the O/W interface [63]. The latter also suggested that the limited manifestation of bi-modality by some of the CIWE with W:C of 25:75 was probably not due to limiting concentration of proteins but rather to particle-particle encounters inside the homogenizer valve, as explained above. Overall, results indicated that in all cases WHPI exhibited effective functionality as surface-active wall constituent that allowed the formation and stabilization of CIWEs.

3.2. Microstructure

All of CIWE that contained 10% WHPI at W:C 25:75 were too viscous to allow atomization during spray drying. In all other cases, CIWE were successfully spay dried to yield dry microcapsule powders. Representative micrographs that depict the outer topography and inner structure of the SD microcapsules are presented in Figure 2. In all cases, spherical microcapsules with a diameter ranging from < 5 μm to about 50 μm were obtained. In all cases, outer surfaces of the microcapsules were free of cracks or visible pores. Evidence for surface indentation was exhibited mainly by small microcapsules and could be attributed to the effect of the COH on viscoelastic and drying rate of the wall system that, collectively resulted in formation of solid crust around the drying microcapsules prior to the completion of the "dent erasing" process [51,59,64,65]. The extent of surface indentation was similar to that reported for microcapsules with wall system consisting of blends of SPI or WPI and COH [51,64]. The inner structure of the investigated microcapsules (Figure 2b,c,d) was similar to what has been previously reported for spray-dried, oil-containing microcapsules with wall systems consisting of proteins and carbohydrates [51,64,66]. In all cases, core domains, in the form of fine droplets, were embedded throughout the wall matrices. Results of structure analysis (Figure 2c,d) reveals the presence of a dense layer around each of the core droplets that could be attributed to the interfacially-adsorbed film of WHPI, as described earlier in this paper. The presence of such dense films is typical to microcapsules prepared by spray drying of protein-stabilized CIWE and represents the results of protein adsorption at the O/W interface of the CIWE during homogenization [51,64,67]. Studying the inner structure of the microcapsules did not reveal the presence of pores or cracks connecting the outer environment with core domains, thus suggesting that the lipid content of the SD microcapsules was physically isolated from the environment.

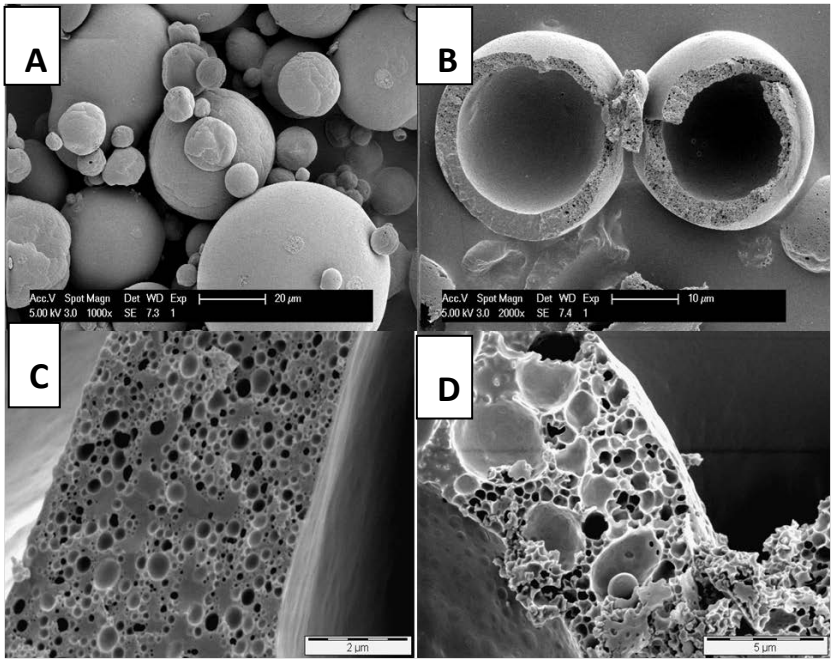

Figure 2. Representative micrographs depicting the outer topography (A) and inner structure of spray-dried microcapsules. Wall solution contained 5.0% WHPI and 15% maltodextrin (MD15). Core load in the CIWE was 75%.

3.3. Core retention

Overall, high core retention during spray drying was obtained with all the investigated CIWEs (Table 3). In only one case core retention was lower than 80 and 50% of the investigated CIWEs resulted in core retention higher than 90% (90.52–98.63%). In all other cases, core retention ranged from 80.15 to 89.56%. The level of core retention during SD that characterized the investigated CIWEs was similar to what has been reported for comparable CIWEs consisting of SPI and COH [51] and, in some cases, slightly lower than that reported for CIWEs consisting of blends of WPI and COH [61]. Retention of non-volatile core (such as lipids) during microencapsulation by SD is governed by the combined influence of physico-chemical properties and composition of the CIWE, drying conditions, atomization conditions and by the drying and film-forming properties of the constituents wall materials [56,61,68,69]. In all cases, CIWEs were atomized at the same atomization conditions and spray dried at the same inlet- and outlet-air temperatures. The among-CIWEs differences in core retention (Table 3) can therefore be attributed to the influence of the inherent properties of the investigated CIWEs. Lipids loss during spray drying of CIWE represents the combined loss of lipid from droplets that reside at the surface of the drying microcapsule, immediately after atomization, and loss from oil droplets that arrive at the outer surface of the drying microcapsule, due to internal mixing, prior to the formation of a dry crust around the drying microcapsule [68]. Core losses during spray drying of CIWEs thus mainly occurs during the period of time that elapses between atomization and the end of the constant rate stage of the drying [51,61,68,69]. It has been reported that in some cases core retention during microencapsulation by spray drying was inversely related to the mean particle diameter of the emulsions [68]. In most of these cases, large among-CIWE differences in mean particle size existed and a mean particle diameter > 1 μm

characterized the reported CIWEs [68]. Results (Tables 1 and 3) indicated that such relationship was not evident in the present study. Theoretically, $d_{3,2}$ of the investigated CIWEs might have influenced core retention during spray drying. However, it can be suggested that the small $d_{3,2}$ (< 0.5 μm) that characterized all of the investigated CIWEs, the relatively small among-CIWE differences in $d_{3,2}$ (Table 1) as well as the overall similar PSDs (Figure 1) affected core retention to an extent that was much smaller than the effect of the compositional variables and physico-chemical properties of the CIWE and their constituents. The relative proportion of lipids (out of the total lipid content of the CIWE) that resides at the outer surface of the atomized droplet of CIWE immediately after atomization decreases with core load and thus it is likely that core retention will be proportionally related to core load. Results indicated that indeed, for a given WHPI:COH ratio, core retention increased significantly ($p < 0.05$) with the core load of the CIWE, in a COH type-dependent manner. For CIWEs containing 2.5, 5.0 or 10.0% WHPI, the increase in core retention with core load was by about 5–9%, 7–18% and 2–14% for CIWEs containing CSS, MD15 and MD5, respectively. Results thus indicated a combined influence of core load and wall composition of core retention. In all cases, for a given WPI/COH and W:C ratio, the lowest and highest ($p < 0.05$) core retention were exhibited by CIWEs containing MD5 and CSS, respectively. The effect of DE on core retention could be attributed to a higher drying rate, and thus a shorter duration of the constant rate stage of the drying that, in turn, resulted in a faster formation of a dry crust at the outer surfaces of the drying capsule. The latter has been reported to be promoted by wall systems containing COH with a high DE value [51,59,61]. For a given W:C ratio, core retention was proportionally related to the concentration of the COH in the CIWE, thus reflecting the effect that COH had on drying rate and the resulting overall faster completion of the constant rate stage of the drying [59,61,68]. Results thus indicated that in the case of wall systems consisting of WHPI and COH, core retention was significantly ($p < 0.05$) enhanced by high initial core load, high DE value of the COH and a high COH concentration in the CIWE. Results indicated that, similar to what have been reported for other wall systems consisting of blends of proteins and carbohydrates, core retention is mainly influenced by the effect of wall composition on the drying rate.

3.4. Microencapsulation efficiency (MEE)

When investigating the microencapsulating properties of a given wall and core system it is important to understand the spatial distribution of the core domains throughout the wall matrix and the extent to which these domains are protected from the outer environment. It is of importance to understand what proportion of the total core content of a given population of microcapsules resides on- or just below the outer surface of the microcapsules and what is the proportion of the core content that is embedded and protected in domains that are relatively far from the outer surface. The accessibility of encapsulated core, such as lipids, to solvents, or, the proportion of core that can be extracted, at standardized conditions, can assist in developing such understanding [66,68,69]. At a given set of extraction conditions, the proportion of lipids that can be extracted from microcapsules consists of: True surface oil (SO), lipids that are extracted from emulsified core droplets that reside at the outer surface of microcapsules, lipids that can be extracted from sub-surface domains of the wall matrices through capillary forces and lipids that can be reached by solvent through empty wall matrix domains left by already extracted core domains [66,69]. It has been established that for microcapsules prepared at a given set of atomization and drying conditions, MEE reflects the overall combined influence of the

microstructural features and composition of the wall matrices, composition and physico-chemical characteristics of the structures that are adsorbed at the dry O/W interfaces, composition of the outer surface of microcapsules, PSD of CIWE after drying, as well as the physico-chemical properties, state and hydrophobicity of wall constituents [51,61,66,68,69]. A standard method for the determination of MEE has not been established yet and different extraction conditions have been used for studying different microcapsules and dry emulsion powders [69]. MEE has been suggested as a potential indicator for the extent to which encapsulated oil is protected against oxidation in SD microcapsules [69]. In order to challenge the extent to which the lipid core was physically protected in the wall matrices of the investigated microcapsules, a relatively long extraction time of 15 min was used in all cases.

Results (Table 4) indicated that MEE ranged from 11.71 to 97.79% and was significantly ($p < 0.05$) affected by the combined influence of the composition of the wall matrices, the DE value of the COH and by the wall-to-core ratio in the CIWE. In 50% of the investigated systems MEE was higher than 90% (92.81–97.79%), in 5 systems MEE was lower than 50% and in 11 of the investigated systems MEE ranged from 58.89 to 86.54%. Regardless of COH type, microcapsules that were prepared with CIWE containing 2.5% WHPI at W:C of 25:75 exhibited the overall lowest MEE (11.71–33.12%). The very low MEE can be probably attributed to the very high proportion of surface oil that reflected that instability of lipid droplets at the outer surface of the drying capsules, enhanced coalescence and flocculation of lipid droplets during early stages of the drying as well as to the relatively low surface excess in these systems [69].

In most cases, for a given WHPI/COH, MEE was proportionally related to the DE value of the COH. The effect of DE value on MEE has been attributed to the enhancement of glass phase formation during spray drying of wall systems that contain relatively high proportion of low molecular weight COH [66]. The proportion of low molecular weight carbohydrates that is included in maltodextrins increases with the DE value of the COH. It has been established that low molecular weights carbohydrates form a glass phase during spray drying. The spatial distribution of this glass phase throughout the dry wall matrices presents a barrier that limits the diffusion of the solvent during extraction, thus leading to a higher MEE [51,58,59,61,66,69]. For a given COH and WHPI/COH, MEE was proportionally related ($p < 0.05$) to the W:C ratio (Table 4). At a given wall composition, higher core content increased the number of core domains that were embedded in a unit volume of wall matrix. The latter and the thinner wall layers that separated these core domains from each other resulted in an overall shorter diffusional path during extraction that, in turn, resulted (at a given set of extraction conditions) in a lower MEE value [51,58,61,64,69]. It has to be noted that the extent to which core load affected MEE was influenced by the DE value of the COH and thus suggested the combined influence of wall composition and W:C ratio on MEE. For example, with WHPI/COH of 2.5/17.5, the differences between MEE that was obtained at a W:C ratio of 25:75 and that at W:C ratio of 75:25 was 64.2, 72.63 and 59.64%, for systems containing MD5, MD15 and CSS, respectively. Similarly, with WHPI/COH of 5.0/15.0, the differences were 45.6, 38.6 and 32.92%, for systems containing MD5, MD15 and CSS, respectively.

Results (Table 4) indicated that regardless of the type of COH and core load in the CIWE, for a given W:C ratio, MEE increased with the proportion of WHPI that was included in the wall system. For example, in the case of system containing MD5 at a W:C ratio of 75:25, MEE of systems containing 2.5, 5.0 and 10% WHPI was 75.93, 88.83 and 95.64%, respectively (Table 4). It can be suggested that the viscosity of the CIWE increased with the proportion of WHPI in the CIWE. It can be assumed that the higher viscosity decreased the extent of internal mixing during the early stages of the SD and thus lowered the overall migration of core droplets to the outer surface of the drying droplet

Table 3. Effects of wall composition and wall-to-core ratio on core retention during spray drying.

WHPI/COH[1] (%/%)	W:C[2] (%:%)	CR (%)		
		MD 5	MD 15	CSS
2.5/17.5	75:25	$87.85^{c,C}$	$89.32^{b,C}$	$94.75^{a,C}$
	50:50	$90.52^{c,B}$	$94.79^{b,B}$	$97.18^{a,B}$
	25:75	$95.32^{c,A}$	$96.89^{b,A}$	$98.63^{a,A}$
5.0/15.0	75:25	$80.10^{c,C}$	$82.58^{b,C}$	$89.56^{a,C}$
	50:50	$89.26^{b,B}$	$90.74^{a,B}$	$91.33^{a,B}$
	25:75	$94.64^{b,A}$	$95.82^{a,A}$	$96.15^{a,A}$
10.0/10.0	75:25	$77.67^{c,B}$	$80.15^{b,B}$	$87.63^{a,B}$
	50:50	$88.43^{b,A}$	$88.92^{a,A}$	$89.51^{a,A}$

[ABC]For a given wall system, means in a given column followed by different letters are significantly different ($p < 0.05$). [abc]For a given wall system, means in a given row followed by different letters are significantly different ($p < 0.05$). [1]Proportions (%) of WHPI and carbohydrate (COH) in wall solution of CIWE. [2]Wall-to-core ratio (%:%) in CIWE.

Table 4. Effects of wall composition and wall-to-core ratio on microencapsulation efficiency (MEE).

WHPI/COH[1] (%/%)	W:C[2] (%:%)	MEE (%)		
		MD 5	MD 15	CSS
2.5/17.5	75:25	$75.93^{b,A}$	$92.99^{a,A}$	$92.81^{a,A}$
	50:50	$34.15^{c,B}$	$76.32^{b,B}$	$86.54^{a,B}$
	25:75	$11.71^{c,C}$	$20.36^{b,C}$	$33.17^{a,C}$
5.0/15.0	75:25	$88.83^{c,A}$	$97.56^{a,A}$	$95.31^{b,A}$
	50:50	$84.69^{b,B}$	$93.40^{a,B}$	$93.13^{a,B}$
	25:75	$43.37^{c,C}$	$58.89^{b,C}$	$62.39^{a,C}$
10.0/10.0	75:25	$95.64^{b,A}$	$97.79^{a,A}$	$96.40^{b,A}$
	50:50	$95.48^{a,A}$	$95.59^{a,B}$	$93.48^{b,B}$

[ABC]For a given wall system, means in a given column followed by different letters are significantly different ($p < 0.05$). [abc]For a given wall system, means in a given row followed by different letters are significantly different ($p < 0.05$). 1Proportions (%) of WHPI and carbohydrate (COH) in wall solution of CIWE. [2]Wall-to-core ratio (%:%) in CIWE.

and to domains just below this surface. The overall result of the latter decreased the proportion of core that was highly accessible to the diffusing solvent during extraction. Results (Table 4) indicated that for microcapsules prepared with CIWEs containing 10% WHPI, DE value of the COH and core load had a very limited effect on MEE that ranged from 93.48 to 97.79%. In addition to the effect on viscosity, it can be suggested that the effect of protein content in the wall system on MEE can be also attributed to

the effect of protein content on surface excess (Table 2). Overall, the increase in surface excess with WHPI load in the CIWE indicated the formation of thicker and probable denser structures at the O/W interfaces. These structures could better protect the emulsified lipid droplets against colloidal deterioration at the outer surface of the drying droplets and thus allowed reducing the proportion of core that was in the form of surface oil after SD. Increasing the WHPI load in the CIWE resulted in the formation of more structurally developed protein-based layer at the O/W interface that, in turn, probably presented an effective barrier to the diffusing solvent, hence, increasing the MEE.

4. Conclusions

Results of the study have indicated that blends of WHPI and selected COH can be utilized effectively for microencapsulation of lipids by spray drying. Results indicated that stable and fine CIWE can be prepared with wall solutions containing even a low concentration of 2.5% WHPI. Results also indicated that the details of PSD properties and surface excess of the CIWE are significantly influence by the concentration of WHPI, the DE value of the COH and by the wall-to-core ratio. The properties of the CIWE affect the formation of microcapsules during SD and influence the functional and structural properties of the dry microcapsules. The investigated microcapsules exhibited high core retention and, in most cases, high MEE that were significantly influenced by the combined effects of the composition of the CIWE, ratio of wall-to-core and by the type of COH that was included in the CIWE. Results of the research have highlighted the importance of developing understanding pertinent to these effects in order to tailor the composition of the CIWE for specific applications. Overall, results indicated that wall systems consisting of blends of WHPI and COH allowed preparing microcapsules with desired properties indicated the potential for utilizing WHPI as effective microencapsulating agent in food applications.

Conflict of interest

The authors report no conflict of interests in this research.

References

1. Dias MI, Ferreira IC, Barreiro MF (2015) Microencapsulation of bioactives for food applications. *Food Funct* 6: 1035–1052.
2. Vandamme TF, Gbassi GK, Nguyen TTL, et al. (2015) Microencapsulating Bioactives for Food. *Benefic Microbes Fermented Funct Foods* 255–271.
3. Zhang C, Li X, Liu YN, et al. (2015) Utilization of Microcapsule Technology in Foods. *J Nanosci Nanotechnol* 15: 9330–9340.
4. Quek S, Chen Q, Shi J (2016) Microencapsulation of Food Ingredients for Functional Foods. *Funct Food Ingredients Nutraceuticals* 13: 267–318.
5. Chen L, Remondetto GE, Subirade M (2006) Food protein-based materials as nutraceutical delivery systems. *Trends Food Sci Technol* 17: 272–283.
6. Santiago LG, Castro GR (2016) Novel technologies for the encapsulation of bioactive food compounds. *Curr Opin Food Sci* 7: 78–85.

7. Castro-Rosas J, Ferreira-Grosso CR, Gómez-Aldapa CA, et al. (2017) Recent advances in microencapsulation of natural sources of antimicrobial compounds used in food—A review. *Food Res Int* 102: 575–587.
8. Nazzaro F, Orlando P, Fratianni F, et al. (2012) Microencapsulation in food science and biotechnology. *Curr Opin Biotechnol* 23: 182–186.
9. Özkan G, Bilek SE (2014) Microencapsulation of natural food colourants. *Int J Nutr Food Sci* 3: 145–156.
10. Desobry S, Debeaufort F (2015) Encapsulation of flavors, nutraceuticals, and antibacterials. *Handb Encapsulation Controlled Release* 801–832.
11. Donsì F, Sessa M, Ferrari G (2015) Encapsulation of Bioactive Compounds. *Handb Encapsulation Controlled Release* 765–799.
12. Edwards-Lévy F, Munin-César A (2015) Encapsulation of Polyphenolics. *Handb Encapsulation Controlled Release* 741–763.
13. Martin MJ, Lara-Villoslada F, Ruiz MA, et al. (2015) Microencapsulation of bacteria: A review of different technologies and their impact on the probiotic effects. *Innovative Food Sci Emerging Technol* 27: 15–25.
14. Bakry AM, Abbas S, Ali B, et al. (2016) Microencapsulation of Oils: A Comprehensive Review of Benefits, Techniques, and Applications. *Compr Rev Food Sci Food Saf* 15: 143–182.
15. Li Y (2015) Nano-Microencapsulation Technology and Applications in Fortified and Functional Foods. *Funct Food Ingredients Nutraceuticals* 13: 319–371.
16. De SSL, Madalena DA, Pinheiro AC, et al. (2017) Micro- and nano bio-based delivery systems for food applications: *In vitro* behavior. *Adv Colloid Interface Sci* 243: 23–45.
17. Sanguansri L, Augustin MA (2016) Microencapsulation and Delivery of Omega-3 Fatty Acids. *Funct Food Ingredients Nutraceuticals* 13: 373–407.
18. Gunasekaran S, Ko S (2014) Rationales of nano- and microencapsulation for food ingredients. *John Wiley & Sons Ltd* 43–64.
19. Ré M, Santana M, dAvila M (2015) Encapsulation Technologies for Modifying Food Performance. *Handb Encapsulation Controlled Release* 643–684.
20. Paulo F, Santos L (2017) Design of experiments for microencapsulation applications: A review. *Mater Sci Eng C Mater Biol Appl* 2017: 1327–1340.
21. Wandrey C, Bartkowiak A, Harding SE, (2010) Materials for Encapsulation, In: Zuidam NJ, Nedovic V, Editors, *Encapsulation Technologies for Active Food Ingredients and Food Processing*, New York: Springer New York 31–100.
22. Đorđević V, Balanč B, Belščak-Cvitanović A, et al. (2015) Trends in Encapsulation Technologies for Delivery of Food Bioactive Compounds. *Food Eng Rev* 7: 452–490.
23. Nedovic V, Kalusevic A, Manojlovic V, et al. (2011) An overview of encapsulation technologies for food applications. *Procedia Food Sci* 1: 1806–1815.
24. Vos PD, Faas MM, Spasojevic M, et al. (2010) Encapsulation for preservation of functionality and targeted delivery of bioactive food components. *Int Dairy J* 20: 292–302.
25. Fang Z, Bhandari B (2012) Encapsulation Techniques for Food Ingredient Systems. *Food Mater Sci Eng* 320–348.
26. Augustin MA, Sanguansri L (2012) Challenges in developing delivery systems for food additives, nutraceuticals and dietary supplements. *Encapsulation Technol Delivery Syst Food Ingredients Nutraceuticals* 2012: 19–48.

27. Mcclements DJ (2012) Requirements for food ingredient and nutraceutical delivery systems. *Encapsulation Technol Delivery Syst Food Ingredients Nutraceuticals* 3–18.
28. Vasisht N (2014) Selection of materials for microencapsulation. *Elsevier Inc* 173–180.
29. Karaca AC, Low NH, Nickerson MT (2015) Potential use of plant proteins in the microencapsulation of lipophilic materials in foods. *Trends Food Sci Technol* 42: 5–12.
30. Meng Y, Cloutier S, (2014) Gelatin and Other Proteins for Microencapsulation, In: *Microencapsulation in the Food Industry,* San Diego: Academic Press, 227–239.
31. Tavares GM, Croguennec T, Carvalho AF, et al. (2014) Milk proteins as encapsulation devices and delivery vehicles: Applications and trends. *Trends Food Sci Technol* 37: 5–20.
32. Sharif HR, Williams PA, Sharif MK, et al. (2017) Current progress in the utilization of native and modified legume proteins as emulsifiers and encapsulants—A review. *Food Hydrocolloids* 76: 2–16.
33. Livney YD (2010) Milk proteins as vehicles for bioactives. *Curr Opin Colloid Interface Sci* 15: 73–83.
34. Nesterenko A, Alric I, Silvestre F, et al. (2013) Vegetable proteins in microencapsulation: A review of recent interventions and their effectiveness. *Ind Crops Prod* 42: 469–479.
35. Nesterenko A, Alric I, Violleau F, et al. (2014) The effect of vegetable protein modifications on the microencapsulation process. *Food Hydrocolloids* 41: 95–102.
36. Augustin MA, Oliver CM, (2014) Use of Milk Proteins for Encapsulation of Food Ingredients, In: *Microencapsulation in the Food Industry,* San Diego: Academic Press, 211–226.
37. Liu F, Chen Z, Tang CH (2014) Microencapsulation properties of protein isolates from three selected Phaseolus legumes in comparison with soy protein isolate. *LWT—Food Sci Technol* 55: 74–82.
38. Karaca AC, Nickerson M, Low NH (2013) Microcapsule production employing chickpea or lentil protein isolates and maltodextrin: Physicochemical properties and oxidative protection of encapsulated flaxseed oil. *Food Chem* 139: 448–457.
39. Veraverbeke WS, Delcour JA (2002) Wheat Protein Composition and Properties of Wheat Glutenin in Relation to Breadmaking Functionality. *Crit Rev Food Sci Nutr* 42: 179–208.
40. Ahmedna M, Prinyawiwatkul W, Rao RM (1999) Solubilized wheat protein isolate: Functional properties and potential food applications. *J Agric Food Chem* 47: 1340–1345.
41. Ducel V, Richard J, Popineau Y, et al. (2005) Rheological Interfacial Properties of Plant Protein—Arabic Gum Coacervates at the Oil-Water Interface. *Biomacromolecules* 6: 790–796.
42. Boire A, Menut P, Morela MH, et al. (2013) Phase behaviour of a wheat protein isolate. *Soft Matter* 9: 11417–11426.
43. O'Sullivan J, Park M, Beevers J (2016) The effect of ultrasound upon the physicochemical and emulsifying properties of wheat and soy protein isolates. *J Cereal Sci* 69: 77–84.
44. Iwami K, Hattori M, Nakatani S, et al. (2006) Spray-Dried Gliadin Powders Inclusive of Linoleic Acid (Microcapsules): Their Preservability, Digestibility and Application to Bread Making. *Agric Biol Chemy* 51: 3301–3307.
45. Ezpeleta I, Irache JM, Stainmesse S, et al. (1996) Gliadin nanoparticles for the controlled release of all-trans-retinoic acid. *Int J Pharm* 131: 191–200.
46. Mauguet MC, Legrand J, Brujes L, et al. (2002) Gliadin matrices for microencapsulation processes by simple coacervation method. *J Microencapsulation* 19: 377–384.

47. Davidov-Pardo G, Joye IJ, Mcclements DJ (2015) Encapsulation of resveratrol in biopolymer particles produced using liquid antisolvent precipitation. Part 1: Preparation and characterization. *Food Hydrocolloids* 45: 309–316.
48. Joye IJ, Davidov-Pardo G, Mcclements DJ (2015) Encapsulation of resveratrol in biopolymer particles produced using liquid antisolvent precipitation. Part 2: Stability and functionality. *Food Hydrocolloids* 49: 127–134.
49. Joye IJ, Nelis VA, Mcclements DJ (2015) Gliadin-based nanoparticles: Fabrication and stability of food-grade colloidal delivery systems. *Food Hydrocolloids* 44: 86–93.
50. Yu JY, Lee WC (1997) Microencapsulation of pyrrolnitrin from Pseudomonas cepacia using gluten and casein. *J Ferment Bioeng* 84: 444–448.
51. Rosenberg M, Rosenberg Y, Frenkel L (2016) Microencapsulation of model oil in wall matrices consisting of SPI and maltodextrins. *AIMS Agric Food* 1: 33–51.
52. Patton S, Huston GE (1986) A method for isolation of milk fat globules. *Lipids* 21: 170–174.
53. Cano-Ruiz ME, Richter RL (1997) Effect of Homogenization Pressure on the Milk Fat Globule Membrane Proteins. *J Dairy Sci* 80: 2732–2739.
54. Hooi R, Barbano DM, Bradley RL, et al. (2004) Chemical and Physical Methods, In: Arnold EA, Editor, *Standard Methods for the Examination of Dairy Products*, American Public Health Association.
55. Sharma R, Singh H, Taylor MW (1996) Composition and Structure of Fat Globule Surface Layers in Recombined Milk. *J Food Sci* 61: 28–32.
56. Young SL, Sarda X, Rosenberg M (1993) Microencapsulating Properties of Whey Proteins. 1. Microencapsulation of Anhydrous Milk Fat. *J Dairy Sci*76: 2868–2877.
57. Gharsallaoui A, Roudaut G, Chambin O, et al. (2007) Applications of spray-drying in microencapsulation of food ingredients: An overview. *Food Res Int* 40: 1107–1121.
58. Jafari SM, Assadpoor E, He Y, et al. (2008) Encapsulation Efficiency of Food Flavours and Oils during Spray Drying. *Drying Technol* 26: 816–835.
59. Sheu TY, Rosenberg M (1995) Microencapsulation by Spray Drying Ethyl Caprylate in Whey Protein and Carbohydrate Wall Systems. *J Food Sci* 60: 98–103.
60. Mcclements DJ, Decker EA, Weiss J (2007) Emulsion-Based Delivery Systems for Lipophilic Bioactive Components. *J Food Sci* 72: R109–R124.
61. Young SL, Sarda X, Rosenberg M (1993) Microencapsulating Properties of Whey Proteins. 2. Combination of Whey Proteins with Carbohydrates. *J Dairy Sci* 76: 2878–2885.
62. Walstra PWJ, Geurts TJ, (2006) Homogenization, In: *Dairy Science and Technology*, 2 Eds., Florida: CRC Press, 279–296.
63. Mcclements DJ (2004) Protein-stabilized emulsions. *Curr Opin Colloid Interface Sci* 9: 305–313.
64. Sheu TY, Rosenberg M (1998) Microstructure of Microcapsules Consisting of Whey Proteins and Carbohydrates. *J Food Sci* 63: 491–494.
65. Rosenberg M, Talmon Y, Kopelman IJ (1988) The Microstructure of Spray-Dried Microcapsules. *Food Microstruct* 7: 15–23.
66. Moreau DL, Rosenberg M, Miller MM, et al. (1993) Microstructure and Fat Extractability in Microcapsules Based on Whey Proteins or Mixtures of Whey Proteins and Lactose. *Food Struct* 12: 457–468.
67. Rosenberg M, Young SL, Brooker BE, et al. (1993) Whey proteins as microencapsulating agents. Microencapsulation of anhydrous milkfat—structure evaluation. *Food Struct* 12: 31–41.

68. Jafari SM, Assadpoor E, He YH, et al. (2008) Encapsulation efficiency of food flavours and oils during spray drying. *Drying Technol* 26: 816–835.
69. Vega C, Roos YH (2006) Invited review: Spray-dried dairy and dairy-like—emulsions compositional considerations. *J Dairy Sci* 89: 383–401.

AIMS Press