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Mixtures of Modified Starch and Rice and Pea Protein Concentrate as Wall Material in the Microencapsulation of Flaxseed Oil

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Abstract: Flaxseed oil is rich in polyunsaturated fatty acids, and its incorporation into food formulations is limited due to its hydrophobic nature and susceptibility to oxidation. The aim of this work was to analyze the effect of wall material mixtures (modified starch Capsul® and rice and pea protein concentrate) on the efficiency of flaxseed oil encapsulation by freeze-drying, physical characterization, and determining oxidative stability. For the preparation of powders, four emulsions with an oil–wall material ratio of 1:3 were produced and characterized. The mass ratio between rice and pea proteins was fixed at 50–50%. The mass ratio of the protein-Capsul® mixtures was varied by 0–100%, 10–90%, 20–80%, and 30–70%. Based on the creaming index results, all emulsions showed good stability after 24 h of analysis. The powders showed low moisture content (<3.23%), bulk density (<0.2659 kg/kg), and packed bulk density (<0.4389 kg/kg). Encapsulation efficiency decreased with increasing protein content, ranging from 93.40% (protein-Capsul® ratio of 0–100%) to 18.26% (protein-Capsul® ratio of 30–70%). However, the best oxidative stability results (smaller increases in the peroxide index values at the end of the stability experiments) were obtained for the powders containing the highest levels of vegetable proteins (protein-Capsul® ratio of 20–80% and 30–70%, respectively).

Keywords: microencapsulation; vegetable oil; lipid oxidation; modified starch; rice protein; pea protein

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1. Introduction

The flaxseed (*Linum usitatissimum*) is an oilseed rich in polyunsaturated fatty acids (PUFAs), phenolic compounds, and dietary fiber and has been gaining scientific attention due to its high nutritional and functional value [1]. Flaxseed oil is characterized as a polyunsaturated oil rich in α-linolenic acid ($C_{18}H_{30}O_2$) and linoleic acid ($C_{18}H_{32}O_2$), essential fatty acids of the omega-3 and omega-6 groups, respectively [1,2]. These compounds have beneficial health effects, especially concerning the prevention of cardiovascular and neurological diseases [3,4].

However, the use of flaxseed oil in food products is not trivial due to its hydrophobicity and high susceptibility to oxidation [5,6]. The polyunsaturated fatty acids present in flaxseed oil are prone to oxidative and hydrolytic cleavage of their double bonds [7]. Oxidative reactions result in the rancidity of the oil with the formation of undesirable aromas, the formation of free radicals, and a marked loss of product quality.

Processing and handling can worsen the oxidation of flaxseed oil, reducing the shelf life of the product [2]. An alternative to expanding the application of products susceptible to oxidation is the use of microencapsulation. Most oil microencapsulation methods involve a first step of oil-in-water emulsion production, and the microencapsulation efficiency and the oxidative stability of products can be affected by the properties of these emulsions [8,9]. Emulsifying agents, which have the property of reducing the interfacial tension between oil and water, are fundamental in the stabilization of these emulsions [10].

The microencapsulation process can increase shelf life and promote the diversification of applications for emulsified products, resulting in the formation of a solid particulate product [11]. Among the techniques used for microencapsulation, freeze-drying can be considered a valuable technique when it comes to the conservation of biological compounds since it involves two reliable methods of conservation: freezing and dehydration [12]. One of the main factors to be analyzed in the microencapsulation process is the type of wall material. Usually, wall materials are hydrophilic protectors or belong to hydrophobic groups, creating a protection network for the compound of interest, whose selection is related to the material to be protected and the desired characteristics of the microcapsules [13,14].

The interest in rice and pea proteins has increased due to the growing choice for vegetarian, vegan, and flexitarian lifestyles. These proteins are also hypoallergenic and can be consumed by people with gluten and dairy intolerances [15]. Pea protein has in its composition high levels of lysine but low levels of methionine. On the other hand, rice protein has in its composition high levels of methionine and low levels of lysine [16,17]. Thus, mixing these proteins in correct proportions ensures the amount recommended by the Food and Agriculture Organization (2011) of all essential amino acids in the human diet [18].

Starches are inexpensive ingredients widely used in the food industry that can be modified to improve their functional properties, increase the consistency and softness of food pastes, and stabilize products during freezing and thawing processes and storage. Furthermore, some modified starches are used as emulsifiers and as wall materials in microencapsulation processes [2,19–22]. Capsul® is a waxy maize starch modified with octenyl succinate, a lipophilic component, which increases the stability of emulsions in formulations. This modified starch is a polymer widely used in controlled-release systems since it contributes to obtaining less porous materials. Capsul® is also used for better retention of bioactive compounds in film-forming matrices and microcapsules [23].

Few works in the literature have employed vegetable proteins as wall material in the process of oil microencapsulation [5,24–26]. Noguera et al. [25] analyzed the production of rice bran oil by spray drying using rice flour and rice protein (co-products of the rice production chain) to improve emulsions stability and the properties of the powders. It was observed that particles containing the co-products were the most appropriate to decelerate the autooxidation process. Overall, rice flour and rice protein were able to improve the emulsion characteristics, powder properties, and stability of the encapsulated oil. Perrechil et al. [5] evaluated the flaxseed oil microencapsulation process using mixtures of rice protein concentrate and modified starch as wall materials. The authors observed that increasing the concentration of modified starch resulted in a considerable increase in encapsulation efficiency. However, the oxidative stability of the encapsulated oil was not analyzed. Gomes and Kurozawa [26] analyzed the production of flaxseed oil by spray drying using isolated rice protein hydrolysates as emulsifiers and wall materials. The emulsions were produced employing maltodextrin with 10 DE and rice protein hydrolysates (obtained by Alcalase or Flavourzyme). The authors concluded that the use of rice protein hydrolysates resulted in higher encapsulation efficiency and oxidative stability when compared to rice protein without treatment.

Keeping the above information as background, the mandate of the study was to analyze the influence of wall material mixtures (modified starch Capsul®, rice protein concentrate, and pea protein concentrate) in the process of microencapsulation of flaxseed oil by freeze-drying, carrying out a physical characterization of the particles, and determining the oxidative stability of microencapsulated oil. It is worth highlighting that this research focused on the use of proteins of vegetable origin as wall materials and the use of emulsions to produce microparticles containing encapsulated oil. Moreover, no published work has reported on the use of a combination of modified starch with rice and pea proteins by freeze-drying to produce microcapsules from flaxseed oil.

2. Materials and Methods

The ingredients used in the preparation of the emulsions and production of the powders were flaxseed oil (acquired in the local market, São Paulo, Brazil), rice protein concentrate (RPC) (Growth Supplements, Brazil), pea protein concentrate (PPC) (Growth Supplements, Brazil), modified starch with octenyl succinic anhydride (OSA-modified starch) Capsul® (Ingredion Ingredients, Brazil), and distilled water.

2.1. Fatty Acids Profile

Fatty acid composition analysis was carried out by gas chromatography with a capillary column after esterification, employing the method of Hartman and Lago [27]. The preparation of fatty acid methyl esters was done according to the AOCS Ce 2-66 method [28] using an Agilent DB-23 column (50% cyanopropyl-methylpolysiloxane) with dimensions of 60 m, internal diameter of 0.25 mm, and 0.25 μm film.

The temperature conditions of the oven were 110 °C-5 min, 110 °C–215 °C (5 °C/min), and 215 °C-24 min; detector temperature of 280 °C; injector temperature of 250 °C. Helium was employed as carrier gas at split ratio 1:50, and injected volume of 1.0 μL. The identification of fatty acids was conducted by comparing the retention times of the peaks with those of the respective fatty acid standards.

2.2. Emulsions Production and Characterization

Formulations were prepared by adding the wall materials (rice protein concentrate, pea protein concentrate, and OSA-modified starch) in water followed by homogenization at 5000 rpm for 5 min using an Ultra Turrax T25 (IKA, Staufen, Germany). Subsequently, the oil was added and homogenized at 14,000 rpm for 5 min using an Ultra Turrax T25 (IKA, Germany).

The four emulsions were produced using an oil-wall material ratio of 1:3, based on the results published by Perrechil et al. [5]. The mass ratio between RPC and PPC was 50–50%. The vegetable proteins–Capsul® mixtures ratios, in mass percentage, were: 0–100% (formulation A); 10–90% (formulation B); 20–80% (formulation C); and 30–70% (formulation D). Immediately after preparation, part of all emulsions was frozen at −18 °C and then freeze-dried. The composition of each formulation is shown in Table 1.

Table 1. Composition of water, oil, rice protein concentrate (RPC), pea protein concentrate (PPC), and Capsul® in each formulation.

Formulation	Water (g)	Oil (g)	RPC + PPC (g)	Capsul® (g)
A	60	10	0	30
B	60	10	3	27
C	60	10	6	24
D	60	10	9	21

All emulsions were characterized by solids concentration, pH, rheology, droplet size distribution, and stability. The solids concentration of the emulsions (C_s) was determined by drying 3 g of emulsion in an oven at 105 ± 2 °C, until constant mass. The pH of the emulsions was measured in a previously calibrated pH meter.

The rheological trials were performed on a compact modular rheometer MCR92 (Anton Paar, Graz, Austria) using a parallel plate geometry with 60 mm diameter and gap of 1 mm. Flow curves were obtained from a multi-step program at 25 °C. The shear rate ranged from 0 to 300 s^{-1} in 3 sweeps to eliminate thixotropy. The experimental data were fitted according to the Power Law rheological model, Equation (1).

$$\sigma = k \cdot \dot{\gamma}^{n} \quad (1)$$

where: σ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), k is the consistency index ($Pa \cdot s^{n}$), and n is the flow behavior index (-).

The droplet size distribution was determined by light scattering using the equipment Cilas Particle Size Analyzer (model 1190, Orléans, France), employing distilled water as a dispersion medium. The stability of the emulsions was determined based on the creaming index (ICr) using the method described in Carneiro et al. [2]. All analyses were performed in triplicate.

2.3. Freeze-Drying and Encapsulation Efficiency

The emulsions were stored at −18 °C for 120 h and submitted to the freeze-drying process in a bench-scale freeze-drier at a pressure of 0.5 mmHg and a temperature of −30 °C, for 168 h (L101 LIOTOP, São Carlos, Brazil). The powders were crushed using a porcelain spatula, sieved using a 1 mm opening sieve, and stored in glass containers at a temperature of −18 °C.

Encapsulation efficiency was defined as the ratio between the amount of encapsulated oil and the amount of total oil. The amount of unencapsulated oil (superficial oil content) was determined using the methodology presented by Bae and Lee [29] with modifications. For this, 15 mL of hexane was added to 1.5 g of powdered sample in a falcon tube, and the mixture was manually stirred for 2 min at room temperature to extract the free oil. Subsequently, the particle-solvent mixture was filtered through a No. 1 Whatman paper filter, and the material retained on the filter was washed two more times with 20 mL of hexane. The solvent was evaporated at 25 °C and then at 60 °C, until constant mass. The amount of unencapsulated oil was determined from the mass difference before and after extraction. The encapsulation efficiency was determined by Equation (2).

$$EE = \frac{Ot - On}{Ot} \cdot 100 \quad (2)$$

where: *EE* is the encapsulation efficiency (%); *On* is the superficial oil content (g); and *Ot* is the total oil content (g).

2.4. Powders Characterization

The physical characterization of the powders was performed in terms of moisture content, bulk density, packed bulk density, Carr Index, particle size distribution and De Brouckere average diameter D[4,3], and sorption isotherms. The moisture content of the powders (U) was determined by drying 1 g samples in an oven at 105 ± 2 °C until constant mass, in triplicate.

Bulk density (ρ_{bulk}) and packed bulk density (ρ^{C}_{bulk}) were determined following the methodologies described in Tonon et al. [30]. The analyses were repeated ten times for each powder formulation. The flowability of the powders was evaluated based on Carr Index (CI) determined by Equation (3).

$$CI(\%) = \left[1 - \frac{\rho_{bulk}}{\rho^{C}_{bulk}}\right] \cdot 100 \quad (3)$$

The particle size distribution of the powders was analyzed by laser diffraction using the Cilas Particle Size Analyzer (model 1190, Orléans, France) with ethanol as the dispersion medium. The particle size was expressed as De Brouckere mean diameter D[4,3], that is, the diameter of a sphere with the same volume of the particle.

Powder sorption isotherms were measured according to the static gravimetric method [31]. Thus, seven different saturated saline solutions were prepared to obtain different relative humidity (RH) values: LiCl solution, 11.2%; CH_3COOK solution, 22.6%; $MgCl_2$ solution, 32.8%; NaBr solution, 57.7%; NaCl solution, 75.3%; KCl solution, 84.3%; and $BaCl_2$ solution,

90.2%. Modified Brunauer-Emmett-Teller (BET) model (Equation (4)), and the Guggenheim-Anderson-de-Boer (GAB) model, Equation (5), were fitted to the experimental data.

$$X_{eq} = -\frac{(X_m \cdot C_{BET} \cdot a_w) \cdot \left(1 - (N+1) \cdot (a_w)^N + N \cdot (a_w)^{N+1}\right)}{(1 - a_w) \cdot \left((1 - C_{BET}) \cdot a_w - C_{BET} \cdot (a_w)^{N+1}\right)} \quad (4)$$

$$X_{eq} = -\frac{(X_m \cdot C_{GAB} \cdot K_{GAB} \cdot a_w)}{(1 - K_{GAB} \cdot a_w) \cdot (1 - K_{GAB} \cdot a_w + C_{GAB} \cdot K_{GAB} \cdot a_w)} \quad (5)$$

where: X_{eq} is the equilibrium moisture content (kg water/kg dry powder); X_m is the monolayer moisture content (kg water/kg dry powder); C_{BET}, C_{GAB}, and K_{GAB} are the constants; a_w is the water activity; and N is the number of adsorbed layers.

The parameters of the BET and GAB models were obtained by non-linear regression using Microsoft Excel software. The performance of the models was analyzed by the correlation coefficient R^2 and average deviation (AD).

2.5. Oxidative Stability

The peroxide index method (standard method IDF 74A:1991) was employed to determine the oxidative stability of the oil. Samples of oil and powders from each formulation were stored in containers at a temperature of 45 °C to accelerate the oxidation process [2]. The extraction of the oils present in the powders was carried out according to the methodology described by Partanen et al. [32], with some modifications. Approximately 0.5 g of powder was placed in a test tube and then 5 mL of water was added. The test tube was shaken for 30 min to dissolve the sample powder. Subsequently, a volume of 300 µL was withdrawn and stirred three times for 10 s with 1.5 mL of an iso-octane/isopropanol (2:1) solution to extract the oil. The phases were separated by centrifugation at 4000 rpm for 4 min. The extraction process was performed in triplicate.

Approximately 0.12 g of oil or a 200 µL aliquot of the extraction medium was added to 10 mL of a chloroform/methanol (7:3) solution. Afterward, 50 µL of an iron (II) chloride solution and 50 µL of an ammonium thiocyanate solution were added to the samples. The samples were shaken and kept at rest in the dark for a period of 5 min, and then the absorbance was measured at 500 nm in a spectrophotometer. All analyses were performed in triplicate.

A standard curve of Fe^{+3} concentration versus absorbance was constructed to determine the peroxide index [33]. The peroxide index was calculated by Equation (6).

$$PI = \frac{(A_A - A_B)}{55.84 \cdot m_A \cdot 2 \cdot \alpha} \quad (6)$$

where: PI = peroxide index (mEq of peroxide/kg of oil); A_A = sample absorbance; A_B = blank absorbance; α = slope of the standard curve; m_A = mass of oil (g); 55.84 = atomic mass of iron; and 2 = factor for the PI value to be expressed in milliequivalents of peroxide instead of milliequivalents of oxygen.

2.6. Statistical Analysis

The results of the emulsions and powders characterizations were submitted to the Tukey Test, using the Minitab® software (PA, State College, USA), for a confidence level of 95% ($p \leq 0.05$). The average deviation (AD) was calculated by Equation (7).

$$AD = \frac{\sum |V_i - V_m|}{n} \quad (7)$$

where: V_i = observed value; V_m = mean value; n = number of analyses.

3. Results and Discussion

3.1. Fatty Acids Profile

Table 2 shows the results of the flaxseed oil fatty acids composition. The oil presented 15.58% of saturated fatty acids, 23.73% of monounsaturated fatty acids, and 60.68% of polyunsaturated fatty acids. In addition, it contained 23.26% of oleic fatty acid (omega 9 group), 39.87% of linoleic acid (omega 6 group), and 20.48% of linolenic fatty acid (omega 3 group).

Table 2. Flaxseed oil fatty acid composition.

Fatty Acid		% (*w*/*w*)
C12:0	Lauric	0.02
C14:0	Myristic	0.08
C15:0	Pentadecylic	0.01
C16:0	Palmitic	9.49
C16:1	Palmitoleic	0.09
C17:0	Margaric	0.10
C18:0	Stearic	4.99
C18:1	Oleic	23.26
C18:2	*t-Linoleic	0.09
C18:2	Linoleic	39.87
C18:3	*t-Linolenic	0.24
C18:3	Linolenic	20.48
C20:0	Arachidic	0.37
C20:1	Gondoic	0.25
C22:0	Behenic	0.40
C22:1	Erucic	0.09
C24:0	Lignoceric	0.12

*t = trans.

According to the literature, flaxseed oil has an average value of 73% of polyunsaturated fatty acids, 18% of monounsaturated fatty acids, and 9% of saturated fatty acids. The average percentage of linoleic fatty acid is 16% and α-linolenic fatty acid is 57% [34]. In addition, Mikołajczak and Tańska [35] analyzed the composition of 30 samples of flaxseed oil obtained by cold pressing. The authors reported maximum and minimum values of palmitic fatty acid of 4.83% and 6.23%, stearic fatty acid of 2.55% and 4.72%, oleic fatty acid of 15.16% and 26.08%, linoleic fatty acid of 11.72% and 21.87%, and linolenic fatty acid of 48.68% and 62.76%, respectively.

The variations in the composition of linoleic and linolenic fatty acids in the oil used in this work may be related to the addition of other vegetable oils to flaxseed oil. Soybean oil, corn oil, rapeseed oil, and sunflower oil are frequently used in oil adulteration for economic reasons since these oils are inexpensive [36].

3.2. Emulsions Characterization

The physical characterization of the emulsions was performed in terms of pH, solids concentration (C_s), creaming index (ICr), droplet diameter (d_p), average droplet diameter (Dp), and rheological parameters, as shown in Table 3. The increase in the Capsul® content led to a statistically significant decrease in the pH value. Such behavior was also reported by Perrechil et al. [5] for emulsions containing Capsul®, RPC, and flaxseed oil. The values of solids concentration did not show significant differences among the different formulations ($p \leq 0.05$). This result was already expected since the oil-wall material mass ratio remained constant at 1:3 for all formulations evaluated.

Table 3. Characterization of emulsions A, B, C, and D: pH, solids concentration (C_s), emulsion stability (ICr), droplet diameter (d_p), consistency index (k); behavior index (n), and apparent viscosity at 100 s^{-1} ($\mu_{100s}{}^{-1}$).

Analyse	Formulation A	Formulation B	Formulation C	Formulation D
pH	2.82 ^{a} ± 0.01	4.09 ^{b} ± 0.01	4.64 ^{c} ± 0.02	5.08 ^{d} ± 0.01
C_s (kg/kg)	0.3787 ^{a} ± 0.0003	0.3636 ^{a} ± 0.0099	0.3818 ^{a} ± 0.0017	0.3786 ^{a} ± 0.0003
ICr (%)	0.0 ^{b}	2.7 ^{a} ± 0.9	0.0 ^{b}	0.0 ^{b}
d_p (μm) 10%	0.91 ^{b} ± 0.07	0.87 ^{b} ± 0.06	1.11 ^{a} ± 0.04	1.12 ^{a} ± 0.03
d_p (μm) 50%	1.64 ^{b} ± 0.01	1.64 ^{b} ± 0.16	2.19 ^{b} ± 0.18	3.13 ^{a} ± 0.42
d_p (μm) 90%	2.84 ^{b} ± 0.16	5.89 ^{b} ± 4.55	23.16 ^{a,b} ± 10.61	43.24 ^{a} ± 3.39
Dp (μm)	1.78 ^{b} ± 0.04	2.57 ^{b} ± 1.29	6.84 ^{b} ± 2.68	13.89 ^{a} ± 1.57
k ($Pa{\cdot}s^{n}$)	0.1708 ^{a} ± 0.0035	0.1693 ^{a} ± 0.0059	0.1035 ^{b} ± 0.0127	0.1251 ^{b} ± 0.0041
n	0.894 ^{b} ± 0.003	0.904 ^{a,b} ± 0.013	0.941 ^{a} ± 0.009	0.927 ^{a,b} ± 0.005
$\mu_{100s}{}^{-1}$ (mPa·s)	104.09 ^{a} ± 0.38	108.97 ^{a} ± 2.38	78.88 ^{b} ± 6.27	89.82 ^{a,b} ± 0.86

Different letters in the same column indicate a significant difference between samples at $p < 0.05$.

The stability of the emulsions was evaluated based on creaming index results. No phase separation was observed for emulsions A, C, and D after 24 h of homogenization. Exceptionally, a small separation layer was observed for emulsion B, which resulted in a creaming index value of only 2.7 (%).

Regarding the results of the rheological tests, it was observed that all formulations exhibited a shear thinning behavior based on the results of the flow behavior index ($n < 1$). This result was already expected due to the presence of biopolymers in the emulsions, such as amylose, amylopectin, and rice and pea proteins [37,38]. The consistency index (k) and apparent viscosity at 100 s^{-1} increased significantly with the increase in OSA-modified starch content for formulations A (100% Capsul®) and B (90% Capsul®) when compared to the values obtained for formulations C (80% Capsul®) and D (70% Capsul®). No statistically significant differences were observed between formulations A and B and between formulations C and D. Apparent viscosity values of the emulsions ranged from 111.35 to 72.61 mPa.s.

The droplet diameter values for all emulsions remained below 1.15 μm (considering 10% of the droplets) and below 3.55 μm (considering 50% of the droplets). Analyzing the results for a percentage of 90% of the droplets, the increase in the concentration of vegetable proteins led to an increase in the droplet diameter and the average deviation, mainly for formulations B and C.

Based on the results for the average diameter (Dp), it was observed that formulation D (containing 30% of vegetable proteins and 70% of OSA-modified starch) presented a statistically significant variation when compared to the other formulations, with a larger mean diameter. The presence of small droplets dispersed in the continuous phase was attributed to the increase in the concentration of Capsul®, since this modified starch has good emulsifying properties [39].

Figure 1 shows the droplet size distribution for all emulsions studied. Considering the emulsion A (containing 100% Capsul®), the droplet diameter ranged from approximately 0.3 μm to 4 μm, with a modal distribution of only one peak. Analyzing the emulsions B, C, and D (containing mixtures between Capsul® and vegetable proteins), the droplet diameter varied from approximately 0.2 μm to 90 μm, with a modal distribution of three peaks, indicating the occurrence of three dominant droplet diameters (around 2 μm, 10 μm, and 30–40 μm, respectively). The first peak is related to the small oil droplets, whereas the other two peaks, in bigger diameters, probably represent flocculated droplets and insoluble fractions of proteins [5].

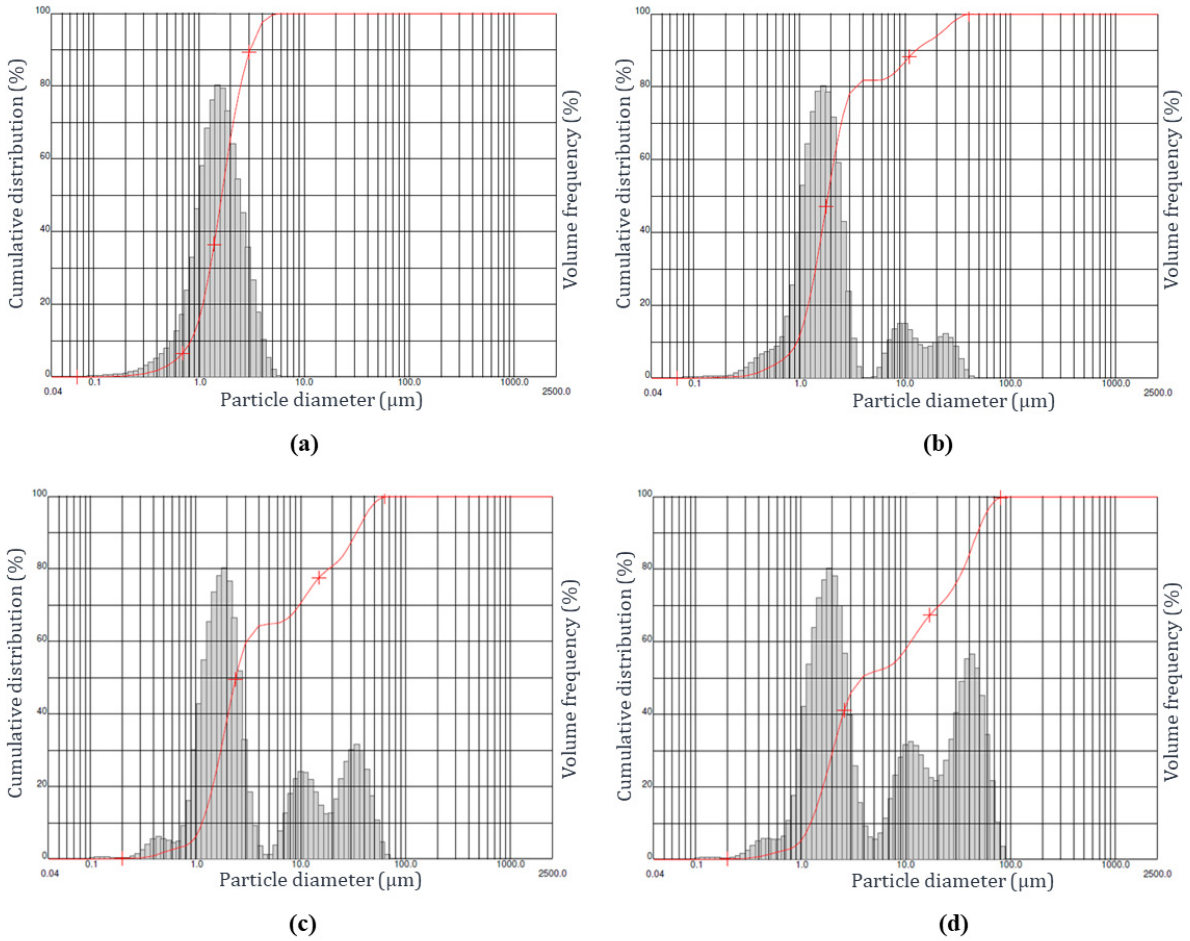

Figure 1. Droplet size distribution of emulsions: (**a**) 100% Capsul®, (**b**) 90% Capsul®-10% vegetable proteins, (**c**) 80% Capsul®-20% vegetable proteins, and (**d**) 70% Capsul®-30% vegetable proteins.

3.3. Encapsulation Efficiency and Powder Characterization

The physical characterization of the powders was performed in terms of encapsulation efficiency (EE), moisture content (U), bulk density (ρ_{bulk}), packed bulk density (ρ^{c}_{bulk}), Carr index (CI), particle diameter (d_p), and De Brouckere mean diameter D[4,3], as shown in Table 4.

Table 4. Characterization of powders A, B, C, and D: encapsulation efficiency (EE), moisture content (U), bulk density (ρ_{bulk}), packed bulk density (ρ^{c}_{bulk}), Carr index (CI), particle diameter (d_p), and De Brouckere mean diameter D[4,3].

Analyse	Powder A	Powder B	Powder C	Powder D
EE (%)	93.40	71.45	58.47	18.26
U (kg/kg)	$0.0313^{a} \pm 0.0009$	$0.0265^{b} \pm 0.0008$	$0.0094^{c} \pm 0.0009$	$0.0102^{c} \pm 0.0010$
ρ_{bulk} (g/cm^3)	$0.2568^{a} \pm 0.0060$	$0.2622^{a} \pm 0.0036$	$0.2440^{b} \pm 0.0041$	$0.2208^{c} \pm 0.3578$
ρ^{c}_{bulk} (g/cm^3)	$0.4321^{a} \pm 0.0067$	$0.3915^{b} \pm 0.0094$	$0.3678^{c} \pm 0.0025$	$0.3578^{c} \pm 0.0063$
CI	40.56	33.03	33.64	38.31
d_p (µm) 10%	$11.19^{a} \pm 0.02$	$9.22^{b} \pm 0.02$	$12.66^{a} \pm 1.11$	$11.87^{a} \pm 0.12$
d_p (µm) 50%	$43.01^{b} \pm 1.65$	$37.22^{b} \pm 0.94$	$54.73^{a} \pm 3.95$	$52.32^{a} \pm 1.79$
d_p (µm) 90%	$76.81^{a,b} \pm 5.49$	$68.46^{b} \pm 3.66$	$110.06^{a} \pm 17.44$	$109.14^{a} \pm 11.31$
D[3,4] (µm)	$44.04^{a,b} \pm 2.33$	$38.26^{b} \pm 1.51$	$59.15^{a} \pm 8.09$	$58.20^{a} \pm 5.01$

Different letters in the same column indicate a significant difference between samples at $p < 0.05$.

Encapsulation efficiency decreased significantly with the increase in RPC and PPC content. Analyzing the results for powder A (100% Capsul®) and powder D (30% vegetable

proteins and 70% Capsul®), a decrease of 80.44% in encapsulation efficiency was observed. The results were consistent with those reported by Perrechil et al. [5], who observed EE of 90.58% for flaxseed oil microencapsulated with 100% of Capsul® and EE of 12.93% for flaxseed oil microencapsulated with a mixture of wall materials (66.6% Capsul® and 33.4% RPC, *w*/*w*). The authors concluded that the lower EE values could be related to the poor emulsifying properties of the rice protein concentrate (a low RPC emulsifying activity index value of 1.15 ± 0.03 m^2/g was reported).

The powders obtained from all emulsion formulations showed low moisture content values (below 3.23%). It is important to highlight that a low moisture content results in a smaller amount of water being available for the occurrence of chemical reactions and the development of microorganisms, directly impacting the stability and quality of the product [40].

Bulk and packaged bulk densities are important properties for powdered products since they determine the conditions of packaging, transport, and storage of a product [38]. Analyzing Table 4, it was found that all powders showed low bulk density values. Powders obtained from formulations A and B showed higher values of bulk density when compared to formulations C and D. Analyzing the results of packaged bulk density, it was observed that the formulation containing 100% Capsul® showed the highest value. In addition, density values showed a tendency to decrease as the concentration of vegetable proteins increased.

Perrechil et al. [5] reported an opposite tendency to increase the bulk density as the protein concentration was increased. Bulk density is a property affected by the size, shape, and surface characteristics of particles [41]. Thus, the differences in particle size distribution due to the composition of powders and different processes of grinding and sieving could lead to this distinct tendency. In the previous work [5], only rice protein was employed in the formulations and in a very high proportion in relation to modified starch.

Carr Index (CI) can be related to the flow properties of the powders. It was observed that all formulations presented high CI values (above 32), indicating that the powders presented poor-quality flows [42]. The powders obtained from all formulations showed particle diameter values below 13.78 μm (considering 10% of the particles), below 58.69 μm (considering 50% of the particles), and below 127.6 μm (considering 90% of the particles). The average De Brouckere mean diameter of the powders was 49.91 μm, and no statistical differences were observed for powders obtained from formulations A, C, and D. Formulation B showed the smallest mean diameter. The particle size is an important property of powders because this can affect other physical properties of samples and their further application in food products [41].

Figure 2 presents the particle size distribution for all powder formulations studied. All formulations showed a modal distribution of one peak, indicating the occurrence of only one dominant powder diameter. Powder A (containing 100% Capsul®) presented particle diameters ranging from approximately 0.45 μm to 180 μm. Powder B (containing 90% Capsul® and 10% vegetable proteins) showed smaller particle diameters ranging from approximately 0.20 μm to 140 μm. Powder C (containing 80% Capsul® and 20% vegetable proteins) and powder D (containing 70% Capsul® and 30% vegetable proteins) presented a broader particle size distribution, with particles ranging from approximately 0.20 μm to 400 μm, indicating the lesser homogeneity of these samples.

The equilibrium moisture content (X_{eq}) as a function of water activity (a_w) for A and B powders formulations is shown in Figure 3. According to the IUPAC (International Union of Pure and Applied Chemistry) classification, curves from powders A and B showed typical behavior of type II isotherms, which are sigmoidal sorption isotherms. Regarding the curves of powders C and D, it was not possible to determine the equilibrium moisture content for any water activity value. Since low encapsulation efficiency values were obtained for powders C and D, part of the oil remained on the surface of the particles [5]. Therefore, due to hydrophobic interactions, mass transfer was compromised, and no mass variations of the samples were observed.

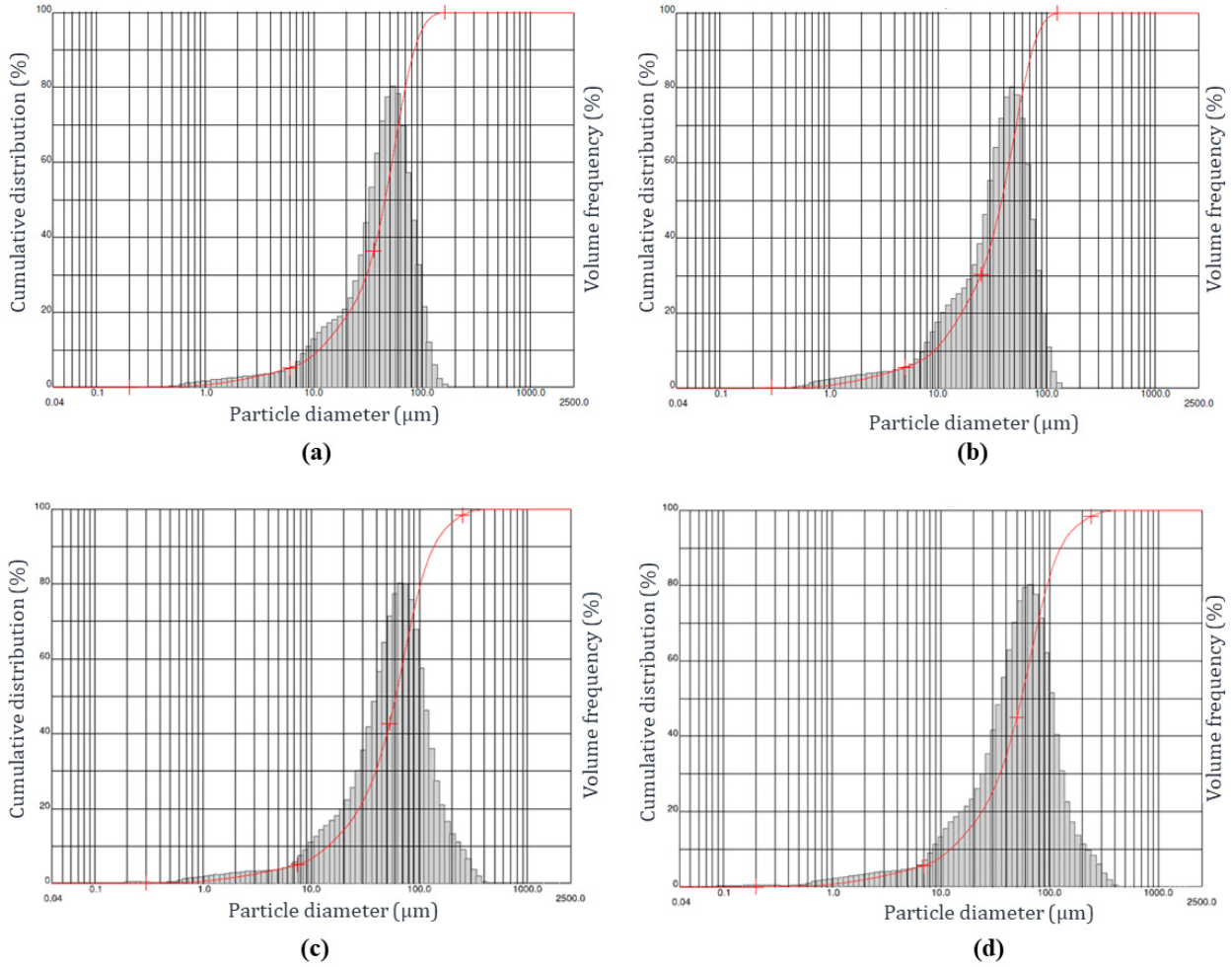

Figure 2. Particle size distribution of powder formulations: (**a**) 100% Capsul®, (**b**) 90% Capsul®-10% vegetable proteins, (**c**) 80% Capsul®-20% vegetable proteins, and (**d**) 70% Capsul®-30% vegetable proteins.

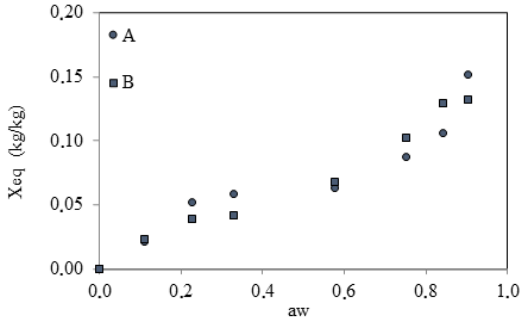

Figure 3. Sorption isotherms at 45 °C of the powders produced from emulsions A (100% Capsul®) and B (90% Capsul®-10% vegetable proteins).

BET and GAB models were fitted to the experimental data of powders A and B, and the fitted parameters are shown in Table 5. It is important to mention that both models present the monolayer moisture content (X_m) parameter, which is considered an optimal value to maintain the stability of the powdered food [38,43]. Analyzing the results of powder A (100% Capsul®) and B (90% Capsul®-10% rice and pea protein concentrate), it was observed that the monolayer moisture content remained low for both formulations.

Table 5. Estimated BET and GAB parameters for powder.

Model	Parameters	A	B
GAB	X_m	0.0345	0.0337
	C_{GAB}	52.15	16,110,367.11
	K_{GAB}	0.8410	0.8464
	R^2	0.9364	0.9585
	AD (%)	17.88	15.32
BET	X_m	0.0273	0.0350
	C_{BET}	38.66	30.44
	N	1.23	0.39
	R^2	0.9195	0.9973
	AD (%)	18.22	4.13

3.4. Oxidative Stability

Figure 4 illustrates the results of the oxidative stability of bulk oil and powders from each formulation studied. Considering the bulk oil, the initial and final peroxide index (PI) was 4.35 mEq peroxide/kg of oil (DM = 0.21) and 17.12 mEq peroxide/kg of oil (DM = 0.80), respectively, representing a 3.93-fold increase in the peroxide index at the end of the analysis.

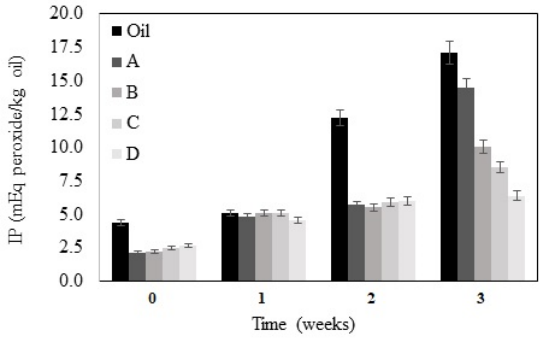

Figure 4. Oxidative stability of bulk oil and powders A, B, C, and D. Data represent the average peroxide index (PI) values (n = 3).

Analyzing Figure 4, it was observed that at time zero, all powders showed low PI values (ranging from 2.11 to 2.68 mEq peroxide/kg of oil). After 7 days of storage, the powders presented PI values between 4.54 and 5.09 mEq peroxide/kg of oil (non-statistically significant difference, $p \leq 0.05$). After 14 days of storage, powders presented PI values between 5.53 and 6.00 mEq peroxide/kg of oil (non-significant difference, $p \leq 0.05$).

At the end of the analysis period, statistically significant differences were observed in the PI values among all formulations. Powder A (containing only modified starch as a wall material) showed the highest PI value among all four formulations, resulting in a 6.84-fold increase in the peroxide index at the end of the study.

Powder B (10% proteins and 90% Capsul®) showed a 4.59-fold increase in the peroxide index at the end of the study. Powder C (20% proteins and 80% Capsul®) showed a 3.38-fold increase in the peroxide index at the end of the study. Powder D (30% protein and 70% Capsul®) showed a 2.38-fold increase in the peroxide index at the end of the experiment. Despite presenting lower values of encapsulation efficiency, it was found that the formulations of powders C (EE = 58.47%) and D (EE = 18.26%) were the most suitable to increase the oxidative stability of the oil, based on peroxide formation analysis.

Several works in the literature have evaluated the stability of flaxseed oil against different forms of the product and storage conditions. Mikołajczak and Tańska [35] analyzed the initial state of 30 samples of flaxseed oils (cold pressed), and the oxidative stability and formation of oxidation products during a storage period of one month at room temperature

and with exposure to light. The authors reported PI values of fresh oils (time 0) from 0.00 to 0.83 mEq O_2/kg of oil and after the storage period, PI values from 9.46 to 37.45 mEq O_2/kg.

Carneiro et al. [2] evaluated the effect of combining maltodextrin with different materials to maximize the encapsulation efficiency and the oxidative stability of the flaxseed oil. The experiments were carried out in a spray dryer. Therefore, maltodextrin was mixed with gum Arabic, modified starch Hi-Cap 100TM, whey protein concentrate, and OSA-modified starch Capsul® TA, in a proportion of 25:75, to be used as wall materials. Stability tests were conducted at 45 °C, based on the peroxide index. The authors observed that at time zero, all powders showed low peroxide index values (ranging from 6.12 to 8.77 mEq peroxide/kg of oil). After four weeks, the powders obtained from the mixtures of gum Arabic/maltodextrin and Capsul® TA/maltodextrin showed the highest PI values (approximately 140 mEq peroxide/kg of oil). Although the whey protein concentrate/maltodextrin mixture resulted in the lowest encapsulation efficiency value (EE = 62.3%), this combination resulted in the lowest PI value (approximately 110 mEq peroxide/kg oil).

Kaushik et al. [24] analyzed the microencapsulation process of flaxseed oil in a matrix formed by complex coacervation between flaxseed protein isolate and flaxseed gum. The matrix was cross-linked with glutaraldehyde. Liquid microcapsules were obtained employing three oil-wall material ratios: 1:2; 1:3; and 1:4. Afterward, the microcapsules were subjected to freeze-drying and spray-drying processes. The oxidative stability of bulk flaxseed oil and powders was analyzed during storage at 4 °C for 30 days, and the first PI analysis was performed after five days of storage. Considering the bulk oil, the initial and final PI values ranged from 3.18 to 8.72 mEq O_2/kg of oil. The powders obtained by freeze-drying showed initial and final PI values ranging from 3.25 to 8.72 mEq O_2/kg of oil. The powders obtained via spray drying showed initial and final PI values ranging from 2.85 to 5.52 mEq O_2/kg of oil. The authors concluded that freeze-drying was not able to provide better oxidative stability results compared to bulk flaxseed oil. However, the oxidative stability of the oil was significantly improved when the spray-dryer technique was employed. Finally, the powders obtained using the spray-dryer and in oil-wall material proportions of 1:3 and 1:4 remained stable until the end of the experiments.

Elik, Yanik, and Göğüş [44] evaluated the influence of the addition of carotenoids (236.35 μg β-carotene/g flaxseed oil) on the oxidative stability of powders obtained by spray-freeze-dryer and spray-dryer. As wall material, a mixture (ratio of 6:1) of maltodextrin 6 DE + (low methoxylation pectin and sunflower wax, in a fixed ratio of 2:1, w/w) was used. Stability tests were conducted at 25 °C for 45 days. The authors reported values of PI of flaxseed oil (initial and final, without the addition of carotenoids) microencapsulated via spray-freeze-dryer of 0.81 ± 0.07 and 3.25 ± 0.21 mEq O_2/kg of oil, resulting in a 4.01-fold increase in PI at the end of storage. Moreover, PI values of flaxseed oil (initial and final, without the addition of carotenoids) microencapsulated via spray-dryer were 6.39 ± 0.11 and 50.18 ± 0.42 mEq O_2/kg, with a 7.85-fold increase in PI at the end of the experiment. The authors related the higher oxidation rate of the powder obtained via spray-dryer to the high initial PI value, passing the oxidation induction period quickly.

4. Conclusions

Based on the results of the physical characterizations of the emulsions, it was found that the increase in the OSA-modified starch content resulted in a decrease in pH values. All formulations showed a behavior of the pseudoplastic fluid. The formulation containing 30% of vegetable proteins and 70% of OSA-modified starch presented an average droplet diameter value statistically superior to the other formulations. Based on the creaming index results, it was found that all emulsions showed good stability after 24 h of analysis.

The powders showed low values of moisture content, bulk density, and packed bulk density. An inverse relationship between the vegetable protein content and the encapsulation efficiency was observed. Considering the formulation containing 100%

Capsul®, an EE of 93.40% was obtained. On other hand, the formulation containing 70% of Capsul® and 30% of vegetable proteins as wall materials showed an EE value of 18.26%.

The bulk oil presented a 3.93-fold increase in the peroxide index value at the end of the accelerated stability test. The best stability results were obtained for powders C (20% of proteins and 80% of OSA-modified starch) and D (30% of proteins and 70% of OSA-modified starch). Thus, powders C and D showed only 3.38-fold and 2.38-fold increases in peroxide index at the end of the storage period, respectively. Despite presenting lower values of encapsulation efficiency, it was found that powders C (EE = 58.47%) and D (EE = 18.26%) were the most suitable to protect the oil against peroxide formation.

We highlight the possible applications of powders in bakery products and meat products. For a complete analysis of the powders, we suggest an investigation of oil oxidation by other methods reported in the literature and the influence of powder content on the physical and physical-chemical properties of the products.

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