Effect of different combination of wall materials on the encapsulation efficiency of flaxseed oil microencapsulated by spray drying

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ABSTRACT

This study aimed to evaluate the effect of the combination of maltodextrin with different wall materials (WPC/whey protein concentrate and Hi-Cap/modified starch) at different concentrations, on the microencapsulation of flaxseed oil by spray drying, in order to maximize encapsulation efficiency. The stability of emulsions was affected by the type and proportion of the wall materials, and the emulsions prepared with Hi-Cap and maltodextrin were highly stable in all proportions used. For the maltodextrin and Hi-Cap combinations studied, oil droplet size increased with the increase on maltodextrin proportion, but for the maltodextrin and WPC combination the opposite was observed. For both combinations of wall material studied, emulsions viscosity decreased with the increase on maltodextrin content. The type of wall material had significant influence on the encapsulation efficiency of flaxseed oil, since the emulsions prepared with Hi-Cap resulted in higher encapsulation efficiency than those prepared with WPC. The increase on maltodextrin content, when combined to Hi-Cap, led to lower encapsulation efficiency. However, when combined to WPC, higher maltodextrin concentrations led to better results. This work showed that the type and proportion of wall materials have significant influence on the emulsion properties and on the encapsulation efficiency of flaxseed oil.

Keywords: microencapsulation, spray drying, flaxseed oil, encapsulation efficiency, wall material.

INTRODUCTION

The increasing demand for nutritive and healthy foods has led the food industry to focus their research to find products of this nature. Flaxseed oil is a polyunsaturated oil extracted from the flax plant (*Linum usitatissimim*) and rich in α-linolenic acid (ALA), the essential fatty acid Omega (ω)-3 which represents about 57% of total fatty acids from flax [1]. Its nutritional characteristics allow the attribution of functional food, which means that besides the nutritional functions, its consumption may have beneficial effects on health.

With technological development, a lot of systems that carry active compounds have been studied for decades. Microencapsulation by spray drying is one of these systems. It is a process in which small particles of a material are trapped in a protective shell (polymer thin films or covers). The material to be encapsulated is known as “core material” or “active material”, while the material forming the coating is called “wall material” [2].

Maltodextrin is a hydrolyzed starch commonly used as a wall material in microencapsulation of food ingredients. The starch hydrolysates offer advantages such as relatively low cost, neutral aroma and taste and low viscosity at high solids concentrations. They also offer good protection against oxidation. However, the biggest problem of this wall material is its low emulsifying capacity [3]. Therefore, it is common to use mixtures of maltodextrin with modified starches or proteins, which are materials that exhibit good emulsifying capacity and can therefore compensate the lack of this property, helping in the encapsulation of the ingredient.

The emulsion properties such as total solids content, viscosity, stability and droplet size, directly affect the encapsulation efficiency of oils [4], being of great importance in the microencapsulation by spray drying. A successful microencapsulation must result in a powder with minimum surface oil and maximum retention of the active material.

The objective of this study was to evaluate the effect of combinations of maltodextrin with whey protein concentrate and modified starch, at different concentrations, on the microencapsulation of flaxseed oil by spray drying.

MATERIALS & METHODS

Material

Flaxseed oil (Lino Oil, Paranambi, Brazil) was used as core material with the following mean fatty acid composition: 5.77% C16:0, 4.57% C18:0, 20.36 C18:1, 14.21% C18:2 and 53.12% C18:3.

The wall materials used were: Maltodextrin MOR-REX® 1910 (Corn Products, Mogi Guaçu, Brazil), whey protein concentrated WPC 80 (Alibra, Campinas, Brazil) and the modified starch Hi-Cap 100 (National Starch, São Paulo, Brazil). Tests were performed for six combinations of wall materials, listed in Table 1.

Table 1. Different formulations used in the microencapsulation of flaxseed oil.

Formulation	% Maltodextrin	% Hi-Cap	% Whey Protein Concentrate
1	25	75	0
2	50	50	0
3	75	25	0
4	25	0	75
5	50	0	50
6	75	0	25

Methods

Emulsion preparation

For the preparation of emulsions, the wall materials were dissolved in water at 25 ° C and the mixture was stirred until completely dissolved. The total solid concentration was fixed at 30%. Flaxseed oil was then added to the wall material and hydrated at a concentration of 20% with respect to total solids [10] [4] [5]; and the emulsion was formed using an Ultra-Turrax homogenizer T8basic (Ika, Wilmington, USA), operating at a speed of 18,000 rpm for 5 minutes.

Emulsions characterization

Emulsion stability

Immediately after the emulsion preparation, 25 mL aliquots of each sample were transferred to graduated cylinders, 25 mL, sealed, stored at room temperature for one day, and the volume of the aqueous phase measured after 24 hours. The stability was measured by % of separation, expressed as:

$$\%Separation = \left(\frac{H_1}{H_0}\right) \times 100 \tag{1}$$

Where: H_o represents the emulsion initial height and H_1 is the upper phase height.

Emulsion viscosity

The measurement of viscosity was performed by determining flow curves. The tests were performed on a Physica MCR301 Rheometer (Anton Paar, Graz, Austria). Measurements were made in triplicate, in parallel plate geometry of 75 mm in diameter, with temperature controlled at 25ºC by a Peltier system and Gap of 0.2 mm. Rheograms were evaluated according to empirical models and the apparent viscosity of emulsions was calculated as the ratio between shear stress and shear rate.

Emulsion droplet size

Droplet mean diameter was measured using a laser light diffraction instrument, Mastersizer S (malvern Instruments, Malvern, UK). A small sample was suspended in water using magnetic agitation, and the droplet size distribution was monitored during each measurement until successive readings became constant. The emulsion droplet size was expressed as D_{32}, the surface weighted mean diameter.

Microencapsulation by spray drying

Microencapsulation was performed in a laboratory scale spray dryer Lab Plant SD-05 (Huddersfield, UK), with a nozzle atomization system with 1.5 mm nozzle diameter, air flow of 73 m^3/h and pressure of 0.6 bar. The emulsion was fed into the main chamber through a peristaltic pump, feed flow rate was 12 g/min, inlet air temperature was 180±2 °C and outlet temperature was 110±2°C.

Particles characterization

Microencapsulation efficiency

Encapsulation efficiency (EE) was determined according to the method described by Bae & Lee [7]. Fifteen milliliters of hexane were added to 1.5 g of powder in a glass jar with a lid, which was shaken by hand for the extraction of free oil, for two minutes, at room temperature. The solvent mixture was filtered through a Whatman filter paper nº 1. The dust collected on the filter was "washed" twice with 20 mL of hexane. Then, solvent was left to evaporate at room temperature and then at 60ºC, until constant weight. The non-encapsulated oil was determined by mass difference before and after extraction with hexane and microencapsulation efficiency was calculated from Equation (2).

$$EE = \left(\frac{TO - SO}{TO}\right) \times 100 \qquad (2)$$

Where: *TO* is the total oil content and *SO* is the surface oil content.

Moisture content

The moisture content of samples was determined gravimetrically in an oven with air circulation at 105ºC for 24 hours (AOAC, 1997).

Bulk density

Particles bulk density was determined by measuring the volume occupied by 2 g of powdered sample into a 25 mL graduated cylinder [8].

Particle size distribution

Particle mean diameter was measured using a laser light diffraction instrument, Mastersizer S (malvern Instruments, Malvern, UK). A small sample was suspended in ethyl alcohol (99,9%) using magnetic agitation, and the particle size distribution was monitored during each measurement until successive readings became constant. The particle size was expressed as D_{43}, the volume weighted mean diameter.

RESULTS & DISCUSSION

Emulsion characterization

The % of separation, droplets mean diameter (D_{32}) and viscosity of the emulsions prepared with different wall materials, in different proportions, are presented in Table 2.

Table 2. Characterization of emulsions prepared with different wall materials, in different concentrations.

Formulation	% Separation	Viscosity (mPa.s)	D_{32} (μm)
25%MD / 75% HC	0 ± 0 A	14.097 ± 0.783 A	2.113 ± 0.010 A
50%MD / 50% HC	0 ± 0 A	13.226 ± 0.714 AB	2.163 ± 0.004 B
75%MD / 25% HC	0 ± 0 A	11.696 ± 0.466 B	2.163 ± 0.008 B
25%MD / 75% WPC	16.8 ± 0.009 B	17.869 ± 0.320 C	2.099 ± 0.024 A
50%MD / 50% WPC	16.1 ± 0.003 B	14.488 ± 0.139 A	2.108 ± 0.040 A
75%MD / 25% WPC	2.0 ± 0 C	13.774 ± 1.016 A	1.975 ± 0.001 C

Different letters indicate significant difference at $p \leq 0.05$.
MD = Maltodextrin; HC = Hi-Cap; WPC = Whey protein concentrate

During the stability study, it was found that some emulsions were kinetically unstable, with a small region of phase separation. All emulsions prepared with combinations of maltodextrin and Hi-Cap showed 100% stability for 24 hours, with no phase separation. However, the emulsions prepared with WPC and maltodextrin had slight formation of a small separation layer. Moreover, a foam phase was observed 24 hours after emulsion homogenization, being the volume of foam higher for the combinations containing higher amounts of protein. The oil separation was higher in the emulsion prepared with a combination of maltodextrin/WPC in 25/75 and 50/50 proportions, and the lowest oil separation was observed for the 75/25 proportion. Then for higher protein content in the formulation, this foaming was more visible. There are some studies in the literature about the ability of proteins to foam, and can be related to the incorporation of air during homogenization with the rotor-stator homogenizer [6].

The rheological behavior of emulsions was evaluated by determination of flow curves at 25ºC and the experimental data were better adjusted by the Newtonian model, according to which viscosity is constant with shear rate. Emulsions viscosity decreased with increasing proportion of maltodextrin in both wall

material systems. This result may be related to the size of proteins and modified starch molecules, since maltodextrin molecules are relatively smaller. Emulsions produced with whey protein were slighly more viscous than those prepared with Hi-Cap.
The droplet mean diameter of maltodextrin/Hi-Cap emulsions was smaller in samples prepared with higher concentrations of Hi-Cap, since the modified starch has excellent emulsifying properties, while maltodextrin does not hold this property. On the other hand, an opposite result was observed for the maltodextrin/WPC combinations, where the smallest diameter was obtained from the mixing with the highest concentration of maltodextrin concentration.

Encapsulation efficiency

Encapsulation efficiency of the samples obtained from emulsions produced with different wall materials in different proportions are shown in Figure 1.

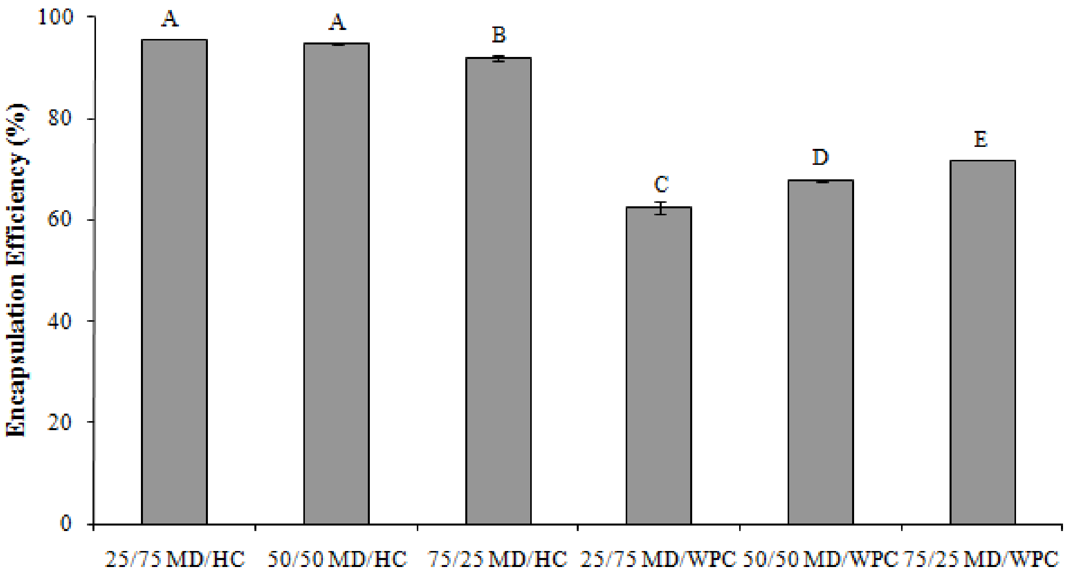

Figure 1. Encapsulation efficiency of powders produced with different wall materials in different proportions. Different letters indicate significant difference at $p \leq 0.05$ (MD = Maltodextrin; HC = Hi-Cap; WPC = Whey protein concentrate).

The type of wall material had significant influence on the encapsulation efficiency of flaxseed oil, since the emulsions prepared with Hi-Cap resulted in considerably higher encapsulation efficiency than those prepared with WPC. Values obtained for the microencapsulation efficiency ranged from 62% to 96%, approximately. The best results were obtained for samples encapsulated with maltodextrin and Hi-Cap. Charve & Reineccius [10] studied the performance of protein and traditional materials in the drying of flavor and obtained a similar result in studying the oil retention in capsules. They observed that the microencapsulated particles with modified starch showed higher oil retention when compared to the particles encapsulated with whey protein. Comparing the encapsulation efficiency values of the microcapsules produced with Hi-Cap and maltodextrin in the present study, it was observed that increasing the maltodextrin concentration resulted in a slight decrease in encapsulation efficiency. This phenomenon is straightly related to the low emulsifying capacity of maltodextrin, which resulted in lower viscosity and higher mean diameters, as previously discussed. The lower the emulsion viscosity, the easier is the oil droplets diffusion inside atomized emulsion, facilitating the oil migration to particle surface. Moreover, the higher surface oil in the particles produced from emulsions with larger droplets can be attributed to the droplets breakdown during atomization [4]. On the other hand, an opposite behavior was observed for the samples produced with maltodextrin and WPC. Higher maltodextrin concentration led to an increase on the microencapsulation efficiency, which was not expected. This can be related to the lower stability shown by the emulsions produced with higher protein concentration, which may have resulted in poorer encapsulation efficiency.

Particles characterization

The results obtained for moisture content, bulk density and mean diameter of particles produced with different wall materials in different proportions, are presented in Table 3.

Table 3. **C**haracterization of particles prepared with different wall materials, in different concentrations.

Formulation	Moisture content (%)	Bulk density (g/cm^3)	D_{43} (µm)
25%MD / 75% HC	1.171 ± 0.007 A	0.354 ± 0.027 A	19.79 ± 0.05 A
50%MD / 50% HC	1.508 ± 0.006 A	0.360 ± 0.008 A	16.01 ± 0.67 B
75%MD / 25% HC	1.364 ± 0.003 A	0.376 ± 0.010 A	14.53 ± 0.31 C
25%MD / 75% WPC	1.560 ± 0.001 A	0.283 ± 0.010 B	17.98 ± 0.88 D
50%MD / 50% WPC	1.295 ± 0.002 A	0.311 ± 0.006 B	20.80 ± 0.28 A
75%MD / 25% WPC	1.665 ± 0.008 A	0.355 ± 0.004 A	33.85 ± 0.56 E

Different letters indicate significant difference at $p \leq 0.05$.

Moisture content did not show a significant defference when different wall materials and different concentrations of them were used. Then, the results obtained may be related to drying conditions. Similar behavior was obtained by Soottitantawat et al. [9], when studying the influence of emulsion droplet size on volatiles retention.

The microcapsules produced from mixing of Maltodextrin/Hi-Cap did not show any variation in bulk density. However, there was a slight increase in bulk density with increasing maltodextrin concentration in the mixing of Maltodextrin/Whey protein concentrate. Results were similar to the obtained by Bae & Lee [7] in the microencapsulation of avocado oil, where the powders bulk density increased with increasing maltodextrin proportion. The authors attributed this result to the high power of clustering of maltodextrin that can generate a decrease in particles volume.

The particle mean diameters ranged between 14.5 and 33.85 µm. The microcapsules produced from mixtures of maltodextrin and whey protein showed greater size, probably due their higher emulsion viscosity. Bae & Lee [7], for avocado oil encapsulated with mixtures of wall materials (whey protein and maltodextrin), obtained values of diameter ranging from 1 to 10 µm. Both the proportion of wall materials studied and the different types of wall materials influenced on particles size distribution. Figure 2 shows the particle size distribution of powders produced with different combinations of wall materials.

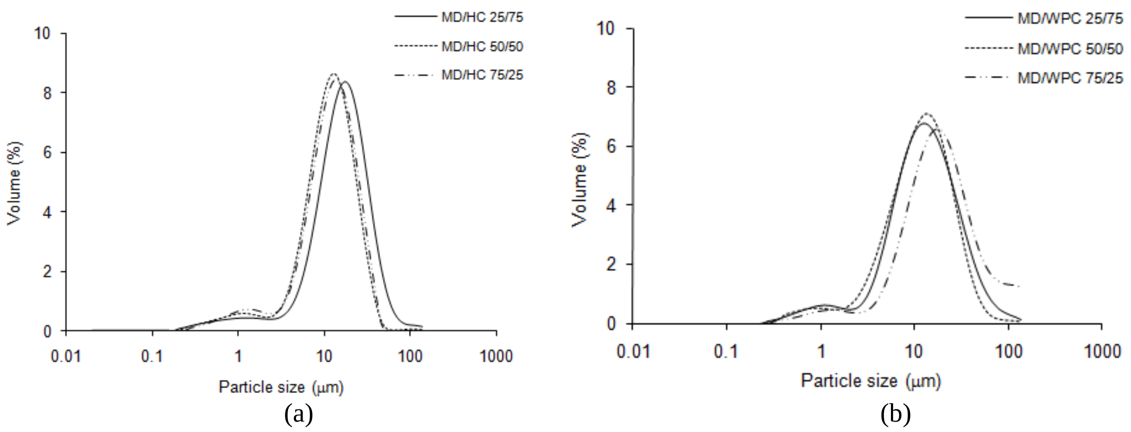

Figure 2. Size distribution of emulsions prepared with maltodextrin and HiCap (a) and emulsions prepared with maltodextrin and whey protein concentrate (b).

The particles exhibited a very large size range, with diameters varying from 0.2 to 160.0 µm, approximately, and showed a bimodal distribution with two distinct peaks, each one representing a predominant size. This is particularly interesting in the case of powders, once the "population" of smaller particles can penetrate into the spaces between the larger ones, thus occupying less space.

CONCLUSION

This work showed that the type and proportion of wall materials have significant influence on the emulsion properties and on the encapsulation efficiency of flaxseed oil. Among the mixtures of wall materials evaluated, the combination of maltodextrin with Hi-Cap obtained better results of encapsulation efficiency, showing that the addition of about 50% of maltodextrin does not damage the retention of the active material and also may decrease the wall material cost. The use of different combinations of wall materials resulted in emulsions with different viscosities and different droplet sizes, and resulted in particles with different bulk density and particles mean diameter. The combination of maltodextrin with WPC and Hi-Cap can be an economically viable alternative, since these last two wall materials are more expensive and the replacement of part of them by maltodextrin is beneficial to product cost.

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