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EFFECT OF OIL CONCENTRATION ON THE MICROENCAPSULATION OF FLAXSEED OIL BY SPRAY DRYING

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Abstract: This work aimed to evaluate the influence of emulsion composition, as well as its associated properties (stability, viscosity and droplet size), on the microencapsulation of flaxseed oil by spray drying, using gum Arabic as wall material. Emulsions were prepared with a fixed total solid content (30% w/w), with different oil concentrations (10, 20, 30 and 40%). The increase in the oil concentration led to the formation of emulsions with larger droplets and lower viscosity, and, consequently, in the production of particles with higher surface oil and higher lipid oxidation.

Keywords: flaxseed oil, emulsion properties, encapsulation efficiency, lipid oxidation.

INTRODUCTION

Flaxseed oil is recognized as one of the greatest Omega-3 sources in nature. Oils rich in polyunsaturated chains have a positive effect on human health, acting in the prevention of cardiovascular diseases. However, during processing, distribution and handling, they can easily oxidize, due to their high instauration degree. Oxidation leads to the formation of unpleasant tastes and odors and, consequently, to the reduction of product's shelf life.

Microencapsulation of oils in a polymeric matrix is an alternative for protecting them against lipid oxidation and increasing their shelf life. In the case of foods, the most common procedure for microencapsulation is spray drying. It involves the atomization of emulsions into a drying medium with high temperature, resulting in a quick crust formation and in a quasi-instantaneous entrapment of the core material (Gharsallaoui et al., 2007).

Gum Arabic is one of the most traditional wall materials used in the microencapsulation of oils and flavors. It is a natural gum with good emulsifying properties, since it has a little protein content in its composition. Moreover, it exhibits high solubility and low viscosity in aqueous solution, which facilitates the spray drying process.

In the last years, special attention has been given to the studies aiming at improving the encapsulation efficiency during spray drying of food flavors and oils, by minimizing the amount of unencapsulated oil present at the surface of powder particles and thus preventing lipid oxidation (Desai and Park, 2005).

According to Jafari et al. (2008), the main factors that affect encapsulation efficiency of microencapsulated oils and flavors are: type of wall material, properties of the core materials (concentration, volatiliy), the characteristics of the infeed emulsion (total solids, viscosity, droplets size) and the conditions of the spray drying process (atomization type, inlet air temperature, air flow, humidity). Thus, it is important to evaluate some process variables, in order to obtain the minimal surface oil in the powder particles.

The objective of this work was to study the influence of the oil concentration on the microencapsulation of flaxseed oil by spray drying, using gum Arabic as wall material. Emulsions were produced with different oil concentrations and evaluated for stability, droplets size and viscosity. The powdered particles were analyzed with respect to surface oil and, from this result, encapsulation efficiency was determined. Powders lipid oxidation was also measured.

MATERIAL AND 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.

Gum Arabic Instantgum BA®, kindly donated by Colloïdes Naturels Brazil (São Paulo, Brazil), was used as wall material.

Emulsion preparation

Gum Arabic was dissolved in distilled water under magnetic agitation, one day before emulsification. Coarse emulsions were prepared by blending flaxseed oil and the wall solution, using a rotor-stator blender (Ultra-turrax IKA T18 Basic, Wilmington, USA), at 15500 rpm for 5 min. Total solid concentration was fixed at 30% and four different oil concentrations were used: 10, 20, 30 and 40% (w/w).

Emulsion preparation

Immediately after the emulsion preparation, 25 mL aliquots of each sample were transferred to graduated cylinders of 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:

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

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

Emulsion viscosity

Emulsion viscosity was measured through the determination of steady-shear flow curves, using a controlled stress Physica MCR301 rheometer (Anton Paar, Graz, Austria) with stainless steel plate-plate geometry with a diameter of 75 mm and a gap of 0.2 mm. Three flow ramps (up, down and up-cycles) were obtained in a range of shear stress corresponding to shear rates from 0 to $300s^{-1}$, in order to eliminate any possible thixotropy effect. Trials were performed in triplicate, using a new sample for each repetition. Rheograms were analyzed according to empirical models and viscosity was calculated as the relationship 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 under 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.

Encapsulation efficiency

Encapsulation efficiency (EE) was determined according to the method described by Bae and Lee (2008). 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.

Lipid oxidation

Lipid oxidation was evaluated by determination of the peroxide value. The oil was extracted according to the method described by Partanen et al. (2008). Peroxide value was determined spectrophotometrically, according to the IDF standard method (Shanta and Decker, 1994) with some modifications. A portion of the extraction medium (600 μl) was added to 2.8 ml of a chloroform/methanol (7:3) mixture. For color formation, 30 μL of an ammonium thiocyanate/iron (II) chloride solution (1:1) were added. The sample was vortexed, reacted in the dark for 20 minutes, and absorbance was measured at 500 nm. Peroxide values were determined using a Fe^{+3} standard curve with iron concentration varying from 1 to 25 μg.

RESULTS AND DISCUSSION

Emulsion properties

The % of separation, droplets mean diameter (D_{32}) and viscosity of the emulsions prepared with different concentrations of flaxseed oil are presented in Table 1.

Table 1: Properties of the emulsions produced with different oil concentrations.

% Oil	Separation (%)	D_{32} (µm)	Viscosity (mPa.s)
10%	0	$1{,}854 \pm 0{,}008^{d}$	$111{,}1 \pm 4{,}3^{a}$
20%	0	$2{,}191 \pm 0{,}002^{c}$	$92{,}0 \pm 0{,}8^{b}$
30%	0	$2{,}479 \pm 0{,}003^{b}$	$75{,}5 \pm 0{,}1^{c}$
40%	2,8	$3{,}464 \pm 0{,}064^{a}$	$60{,}5 \pm 0{,}1^{d}$

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

Emulsion stability

Only the emulsion prepared with 40% oil was kinetically unstable, with a little region of phase separation. All the other emulsions were stable, showing no phase separation. This confirms gum Arabic as a good encapsulating agent, which has low viscosity in aqueous solution, making easier the system homogenization with the rotor-stator homogenizer, and being able to cover the oil droplets in the emulsion, avoiding the cremeation phenomena.

Emulsion viscosity

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.

This same behavior was observed by Bae and Lee (2008) in emulsions containing avocado oil + maltodextrin/whey protein isolate. Buffo and Reineccius (2002) also observed a Newtonian behavior in beverage emulsions composed by orange oil and seven types of gum Arabic.

Emulsion viscosity decreased with the increase in oil concentration. This can be attributed to the lower amount of gum Arabic present in the emulsions formed with higher oil concentration, for the same total solid content. Gum Arabic is generally used as a thickening agent in foodstuffs, making them more viscous. Yu et al. (2007) also verified a decrease of viscosity with the increase in the mass ratio of phospholipid to wall material, in emulsions subjected to microencapsulation by spray drying, using maltodextrin added of sodium caseinate, gelatin or soy protein as encapsulating agents.

Emulsion droplet size

The increase in the oil concentration led to higher droplet mean diameters. Higher amounts of oil implies in lower gum Arabic content, for the same total solid content. Since gum Arabic has emulsifying properties, the lower concentration of this wall material may have resulted in a less efficient emulsification. An emulsifier is a surface-active substance that is able to adsorb to an oil-water interface and protecting emulsion droplets from flocculation and/or coalescence (McClements, 2005). Thus, the decrease of gum Arabic content may have promoted higher droplets coalescence in the emulsions studied, resulting in the formation of larger droplets.

The higher mean diameters of the emulsions produced with higher oil content can also be related to the emulsions viscosity. Emulsions produced with higher oil concentration were less viscous than those produced with lower oil concentration, for the same total solid content. The more viscous is an emulsion, the more difficult is the droplets movements inside it. This may have reduced the droplets coalescence, resulting in larger droplet sizes.

Encapsulation efficiency

Encapsulation efficiency of the samples obtained from emulsions produced with different oil concentrations are shown in Figure 1.

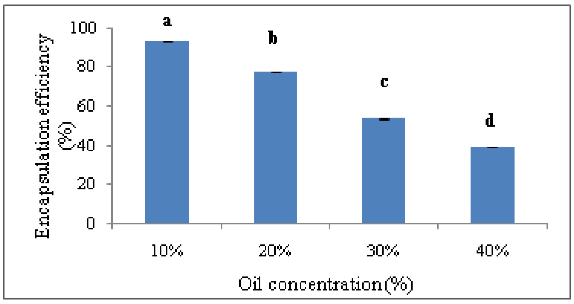

Fig. 1. Encapsulation efficiency as a function of oil concentration.

Oil concentration strongly affected encapsulation efficiency, showing a negative effect on this response. The increase of oil concentration led to an increase in the amount of unencapsulated surface oil. A similar behavior was observed by Huynh et al. (2008) in the microencapsulation of lemon myrtle oil, using modified starch + maltodextrin and whey protein concentrate + maltodextrin as wall materials. Tan et al. (2005) also verified that high oil loadings resulted in lower process yield and lower encapsulation efficiency for microencapsulated fish oil by spray drying.

According to Jafari et al. (2008), the lower encapsulation efficiency related to higher oil loads can be attributed to the greater amount of core material close to the drying surface, which makes short the diffusion path length to the air/particle interface, thus increasing the surface oil content. Minemoto et al. (2002), working with microencapsulation of linoleic acid, also observed that encapsulation efficiency decreased when the weight ratio of core to wall material increased. According to the authors, at higher ratios, the amount of gum Arabic may be insufficient for fully covering the oil droplets and this insufficiency might result in a decrease in the encapsulation efficiency.

The negative influence of oil concentration on the encapsulation efficiency may be related to the emulsion droplet size, which was lower for higher oil content. Many studies have shown that the encapsulation efficiency of oils and flavors is improved with decreasing emulsion droplet size (Soottitantawat et al., 2005; Liu et al., 2001). According to Jafari et al. (2008), the higher surface oil in the particles produced from emulsions with larger droplets can be attributed to the droplets breakdown during atomization. Moreover, lower oil content resulted in higher emulsion viscosity, which reduces the oil droplets diffusion inside the atomized droplets, making difficult the oil migration to the particle surface and thus increasing the encapsulation efficiency.

Lipid oxidation

Figure 2 shows the lipid oxidation of the samples obtained from emulsions produced with different oil concentrations.

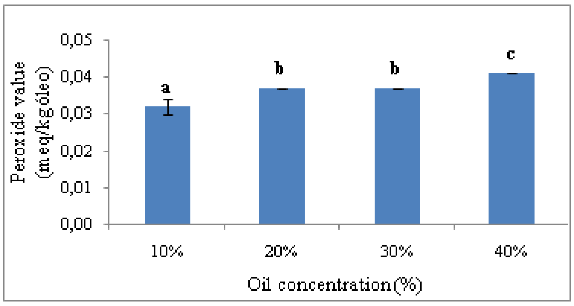

Fig. 2. Lipid oxidation as a function of oil concentration.

Lipid oxidation was also affected by the oil concentration. In general, higher oil concentration led to higher peroxide values. This can be related to the lower encapsulation efficiency obtained at these conditions, which leads to poorer oil protection against oxidation. From Figures 1 and 2, it is clear that the peroxide values increased as the encapsulation efficiency decreased. The lower the encapsulation efficiency, the higher is the amount of oil present in the particles surface. This unencapsulated oil, when in contact with the oxygen, is more susceptible to lipid oxidation than the encapsulated one.

CONCLUSIONS

Both encapsulation efficiency and lipid oxidation were affected by the oil concentration, and these results could be related to the emulsions properties. The increase in the oil concentration resulted in emulsions with larger droplets and higher viscosity, which negatively affected the surface oil content and, consequently, the lipid oxidation.

NOMENCLATURE

H_0	emulsion initial height	cm
H_1	upper phase height	cm
EE	encapsulation efficiency	%
TO	total oil content	g
SO	surface oil content	g
D_{32}	droplet mean diameter	μm

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