Effect of oil in emulsion and homogenization pressure on the microencapsulation of basil oil

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ABSTRACT

Microencapsulation of flavors is the technology of converting liquid flavor materials into easy-to-handle solids. It also provides protection against degradation reactions and prevents the loss of volatiles compounds. The capsule wall material along with the emulsion properties (i.e. viscosity and droplets size) and the drying process conditions are some of the responsible factors for the flavor retention during the encapsulation process. The objective of this work was to evaluate the influence of oil concentration and homogenization pressure on the emulsion properties and oil retention during the microencapsulation of basil essential oil by spray drying, using gum Arabic as wall material. Experiments were planned according a 2^2 central composite design. The independent variables were total oil concentration with respect to total solids (10 – 25%) and homogenization pressure (0 – 100MPa) and the analysed responses were droplet size, emulsion viscosity and oil retention in the microcapsules. The increase in the homogenization pressure up to 50 MPa decreased the emulsion droplet size. However, the use of pressures above 85MPa resulted in the formation of droplets with larger size. The homogenization pressure, as well as the oil concentration, had no effect on viscosity. Since the use o gum Arabic as wall material results in the formation of emulsions with low viscosity (in the order of 0.08 Pa.s), no significant reduction in emulsion viscosity is obtained with the use of different homogenization pressures. Oil retention was positively affected by the homogenization pressure and negatively affected by the oil content. Higher flavor loads resulted in poorer flavor retention. Microencapsulation of basil essential oil using gum Arabic as wall material showed to be a suitable process to obtain powdered flavors. It is possible to obtain higher oil retention with the use of lower oil concentration in the emulsion and higher pressure.

Keywords: microencapsulation; basil; spray-dryer; essential oil

INTRODUCTION

Essential oils are natural liquid products obtained from plants by hydro or steam distillation. The essential oils components are volatile substances, sensitive to oxygen, light, moisture and heat [1]. Stability of essential oils can be increased by using microencapsulation, which consists of the entrapment or coating of those substances within another material or system. [2]

Microencapsulation is of great importance in the flavoring and food industries, since in this technique, flavors in the liquid form are entrapped in a carrier matrix in order to obtain a dry flavor powder, which is easy to handle. The advantages of this technology are not only in providing protection against degradative reactions and prevention of flavor loss, but also promoting the flavor controlled release during food processing and storage [3].

Successful spray-drying microencapsulation relies in achieving high core material retention during processing [2]. The stability of emulsions is an important factor to be considered for the encapsulation of flavors, since these substances are generally insoluble in water [4]. The goal of emulsification is to produce droplets as small as possible and various techniques can be used for this purpose. The high pressure homogenization is widely used to emulsify, disperse, homogenize and to reduce the average droplets size in order to obtain more stable emulsions to coalescence [5].

Among the encapsulation methods, spray drying is the most popular method of producing flavor powders [3, 6].

Gums are generally used as wall material in the microencapsulation process because they present film forming capacity and are able to stabilize emulsions. Among all gums, gum Arabic stands out due to its excellent emulsification properties and low viscosity, even at high concentrations. The emulsification properties of gum Arabic are attributed to the presence of a little protein fraction in its composition [7, 8].

The objective of this work was to evaluate the influence of emulsion composition (oil concentration) and homogenization pressure on the microencapsulation of basil essential oil by spray drying, using gum Arabic as wall material.

MATERIALS & METHODS

Materials
Basil essential oil was obtained from Linax Essential Oil Extraction (Votuporanga, Brazil). The wall material used was gum Arabic Instantgum BA® supplied by Colloides Naturels Brasil (São Paulo, Brazil).

Methods
Preparation of emulsions
The carrier solution was prepared by dispersing gum Arabic in water, until complete dissolution. After that, basil essential oil was emulsified in the gum Arabic solution, using a rotor-stator homogenizer (Ultra Turrax Ika, T18 Model, Staufen, Germany) operating at 14,000 rpm for 5 minutes. For some of the studied conditions, a second emulsification step was made on a high pressure homogenizer (PANDA2K, Niro Soave S.P.A., Parma, Italy). Total solid content in the emulsions was fixed in 30% (w/w).

Emulsion droplet size
The determination of the average emulsion droplet size was made by optical microscopy , in a Jenaval optical microscope (Carl Zeiss, Oberkochen, Germany). The diameter of 500 droplets were measured with the image processor system ImageJ 1.38x and were used to calculate the Sauter mean diameter (D_{32}):

$$D_{32} = \frac{\sum_i z_i D_i^3}{\sum_i z_i D_i^2}$$

(1)

Where z_i is the number of droplets with diameter D_i.

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 0.5 mm. Three flow ramps (up, down and up-cycles) were obtained in a range of shear stress corresponding to shear rates from 0 to $300 s^{-1}$, 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.

Microencapsulation by spray drying
Emulsions were spray-dried in a laboratorial dryer (mini spray dryer, Labmaq, MSD 1.0 model, Ribeirão Preto, Brazil) equipped with a dual fluid nozzle of 1.2 mm diameter. The emulsion was fed into the main chamber through a peristaltic pump, feed flow rate was 0.7L/h, drying temperature was 180ºC and compressor air pressure was 0.4 MPa.
Eleven tests were made, according to a 2^2 central composite design. Total oil concentration with respect to total solids (10 – 25%) and homogenization pressure (0 – 100MPa) were the independent variables. The analysed responses were emulsion droplet size, emulsion viscosity and oil retention in the microcapsules. Five levels of each variable were chosen for the trials, including 4 axial points and 3 repetitions of central point, giving a total of 11 studied conditions. The following polynomial equation was fitted to data:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + + \beta_{12} x_1 x_2$$

(2)

Where β_n are constant regression coefficients; y is the response, and x_1, and x_2 are the coded independent variables (oil retention and homogenization pressure, respectively).
The analysis of variance (ANOVA), test for the lack of fit, determination of R^2 and the generation of three-dimensional graphs were carried out using the Statistica 7.0 software (StatSoft, Tulsa, USA).

Analysis of powders

Oil retention

The oil retained in the microencapsulated powders was determined by hydrodistillation in a Clevenger apparatus, in triplicate, according to method described by Bhandari et al. [9], with some modifications. About 5 g of powder were dissolved in 150 mL of distilled water in a round bottom flask of 500 mL. About 0.5 ml of antifoam was added to the solution. The distillation was performed for 40 minutes and the volume of distilled essential oil was directly read in Clevenger. The mass of oil in the microcapsules can be obtained by multiplying the volume of oil read by the density of basil essential oil (0.8986 g /cm^3). The total oil retained in the microcapsules was calculated according to Equation (3):

$$Oil\, retenti\ \ on\ (\%)\ = \left(\frac{Oil_{Clevenger}\ \times Density}{Oil_{initial}} \right) \times 100 \tag{3}$$

The analysis of variance (ANOVA), test for the lack of fit, determination of R^2 and the generation of three-dimensional graphs were carried out using the Statistica 7.0 software (StatSoft, Tulsa, USA).

RESULTS & DISCUSSION

Response surface analysis

The trials performed for the central composite design and the obtained responses are shown in Table 1.

Table 1. Trials and responses of experimental design.

Trials	Oil concentration (%)	Homogenization pressure (MPa)	D_{32} (μm)	Viscosity ($\times 10^3$ Pa.s)	Oil Retention (%)
1	12.2 (-1)	85 (1)	1.27	8.75	88.28
2	12.2 (-1)	15 (1)	1.83	8.34	75.69
3	22.8 (1)	85 (-1)	1.56	9.73	61.83
4	22.8 (1)	15 (-1)	1.97	10.03	64.47
5	10 (-1.41)	50 (0)	1.28	9.63	90.61
6	25 (+1.41)	50 (0)	1.34	7.39	57.50
7	17.5 (0)	0 (-1.41)	2.87	11.75	56.43
8	17.5 (0)	100 (+1.41)	1.17	6.94	78.79
9	17.5 (0)	50 (0)	1.22	7.93	72.73
10	17.5 (0)	50 (0)	1.21	6.61	80.78
11	17.5 (0)	50 (0)	1.42	8.38	75.80

Table 2 shows the regression coefficients for the coded second-order polynomial equation, the F values and the determination coefficients (R^2). Some non-significant terms were eliminated and the resulting equations were tested for adequacy and fitness by the analysis of variance (ANOVA). The fitted models were suitable, showing significant regression, low residual values, no lack of fit and satisfactory determination coefficients.

Table 2. Coded second-order regression coefficients for droplet mean size, viscosity and oil retention

Coefficient	D_{32} (μm)	Viscosity ($\times 10^3$ Pa.s)	Oil Retention (%)
β_0	1.28	0.076	76.74
β_1	N.S.	N.S.	- 10.57
β_2	-0,42	N.S.	+ 5.20
β_{11}	N.S.	N.S.	N.S.
β_{22}	+0,38	N.S.	N.S.
β_{12}	N.S.	N.S.	N.S.
R^2	0.86775	---	0.81607
F	26.25	---	17.75

N.S. Non-significant.

The surface responses for droplets mean diameter and oil retention are shown in Figure 1. Emulsion droplet mean diameter varied from 1.17 to 2.87 µm and was significantly affected by homogenization pressure (MPa). Total oil concentration had no influence on this response. When the homogenization pressure varied from 0 to 50 MPa the oil droplet size decreased with the increase in the homogenization pressure, probably due to the higher amount of energy provided to break the emulsion droplets. However, the use of pressures above 85 MPa resulted in the formation of droplets with larger size. An similar behavior of droplets diameter in relation to changes in homogenization pressure was observed by Huynh et al [10], that studied the microencapsulation of lemon myrtle oil. This phenomenon in which the particle size increases with increasing energy emulsification is termed as over-processing and can be attributed to the an increase in Brownian motion of the droplets and underperformance of emulsifiers [5, 11]. The emulsion droplet size is reduced due to droplet disruption resulting from high energy input by the high pressure homogenization. However, newly disrupted droplets are thermodynamically unstable due to the Brownian motions and high-intensity turbulence on the equipment. This increases the probability of collision and coalescence of freshly formed droplets to form bigger droplets [12]. Another reason can be the increase in interfacial area during homogenization. If the interfacial area becomes too large, the wall materials (emulsifiers) can no longer sufficiently cover the droplets, thereby exposing the hydrophobic part (LMO). This is called ''threshold'' of depletion [10]. Such thresholds cause a recoalescence process [13] As a result, final emulsions would have a bigger droplet size. Also, the increase in the homogenization pressure increases the viscous stress caused by the high fluid flow in the homogenizer. This mechanical energy is partially dissipated as heat in the fluid, thus increasing the emulsion temperature. Therefore, certain types of emulsifiers, like proteins, can lose their ability to stabilize emulsion droplets against coalescence, when they are heated above a critical temperature, resulting in the formation of emulsions with larger droplets [14].
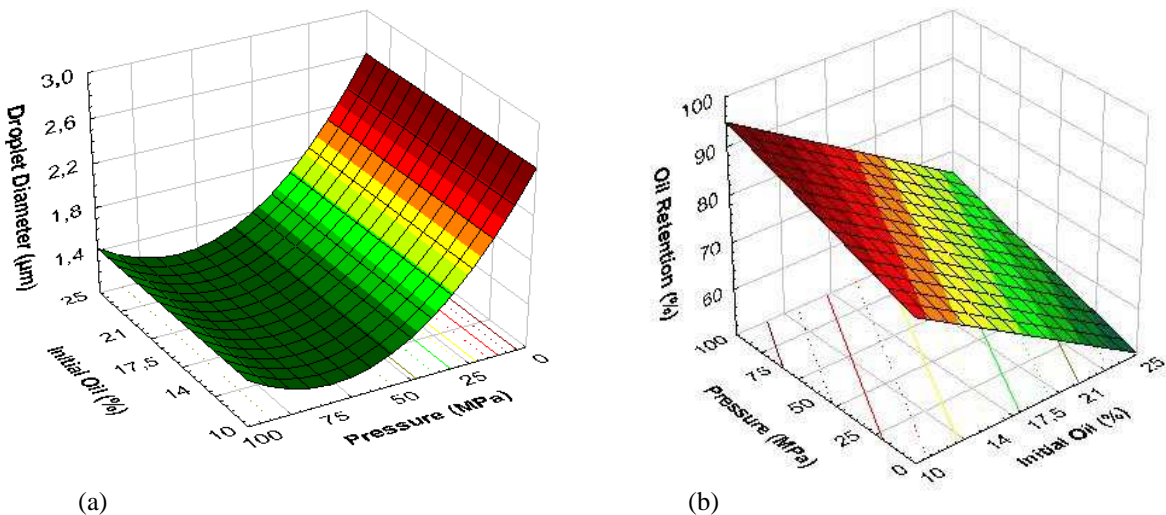

(a)   (b)

Figure 1. Response surfaces for (a) Droplet mean size; (b) Oil retention.

Both the homogenization pressure and the oil concentration had no effect on emulsions viscosity. Since the use of gum Arabic as wall material results in the formation of emulsions with low viscosity, even at high concentrations, no significant reduction in the emulsion viscosity was observed with the use of different homogenization pressures and oil concentrations. Thus, as none of the independent variables were statistically significant on viscosity, no model was obtained for this response.

Oil retention varied from 56.43 to 90.61% and was positively affected by the homogenization pressure and negatively affected by oil content. Higher flavor loads resulted in poorer flavor retention since higher oil loads lead to greater proportions of volatiles close to the drying surface, thereby shortening the diffusion path length to air and favoring the flavor loss. Moreover, the increase in the oil concentration implies in a decrease in the amount of wall material (for a fixed total solid content), which may be not enough to cover the oil droplets, making easier the loss of volatiles compounds. On the other hand, the increase in oil retention

resulting from the increase of homogenization pressure can be related to the droplets size, since higher homogenization pressures resulted in smaller droplet diameters. Similar results were obtained by Soottitantawat et al [6], which observed higher oil retention for small emulsions droplets when compared with large emulsions droplets, in the microencapsulation of d-limonene by spray drying. Evaporation of flavor during atomization seems to be easier with large emulsion size [3]. The large atomized droplets have reduced surface area to volume ratio which would result in better d-limonene retention. However it also takes a longer time for film formation around the large atomized droplets in the drying process and the longer is the time necessary for the film formation, the greater is the loss of volatile flavors [15].

CONCLUSION

Microencapsulation of basil essential oil using gum Arabic as wall material is a suitable process to obtain powdered flavors. It is possible to obtain higher oil retention with the use of lower oil concentration in the emulsion and higher homogenization pressure. The study of the emulsions properties is of great importance in the production of particles with high flavor retention. The best conditions for basil oil encapsulation, aiming at achieving high oil retention, in the range of the independent variables studied, were: oil concentration between 10 and 12% and homogenization pressures above 50 MPa.

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