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Influence of spray-drying operating conditions on sunflower oil powder qualities

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ABSTRACT

The main aim of the study was to develop a vegetable oil (VO) microencapsulation process using a spray drying technique with the aid of the response surface methodology (RSM). The specific objectives were to evaluate the operating condition effect on VO powder qualities and analyze the microstructure and rancidity on VO microcapsules obtained under set conditions that lead to high solid yields. Maltodextrin and hydroxypropylmethylcellulose were used as wall materials. The influence of spray drying process variables over solid yield (SY), moisture content (MC), surface oil (SO) and encapsulation efficiency (EE) was studied. All experiments led to high values of EE, while SY and MC were significantly affected by modifying spray drier conditions, allowing the enhancement of SY when the optimization process was applied. The optimized VO microcapsules presented an external surface with a continuous wall and no apparent pores; with low moisture content, high VO content retained into microcapsules and low peroxide value.

1. Introduction

Unsaturated fatty acids (UFA) (omega-3, omega-6 and omega-9) are essential fatty acids commonly found in marine and vegetable oils (VO). They are nutritionally important for good health and are especially beneficial for individuals suffering from coronary heart disease, diabetes, and immune response disorders [1,2]. For this reason, the use of these fatty acids in health food formulations was increased in the last years. Nevertheless, the susceptibility of UFA to oxidative degradation during food processing and storage is always a concern. Fatty acids are chemically unstable in the presence of oxygen, light, moisture and heat. Microencapsulation of oils in polymeric matrices has been used in order to protect UFA against oxidative degradation [3,4]. The efficiency of protection or controlled release of the core material mainly depends on the composition and structure of the wall material [5,6]. However, the operating conditions (temperature, pH, pressure, humidity) employed during the encapsulation process may strongly affect the encapsulation efficiency, the stability of microcapsules and the shelf-life of the core material. Biopolymer blends have been successfully used as wall materials [7,8]. Among them, carbohydrates have showed to improve the yield and efficiency of microencapsulation [9,10]. Lactose, sucrose, cellulose, maltose, maltodextrins, cyclodextrins and gums are the most commonly used [11]. Kolanowski et al. [12] and Davidov-Pardo et al. [13] have reported that the use of modified celluloses, such as methylcellulose and hydroxypropyl methylcellulose improves the stability, encapsulation efficiency and morphology of fish oil powders obtained by spray drying. Several researches also indicate that maltodextrins have protective effects on microencapsulated oils [14,3]. The use of maltodextrin has several advantages such as low viscosity, good solubility and low cost [15,16].

Spray drying involves the atomization of emulsions into a drying medium with high temperature which leads to very fast water evaporation, resulting in quick crust formation and quasi-instantaneous entrapment of the core material [3,16,17]. The advantages of this method are its ability to handle heat-sensitive materials, its availability of machinery and its variety, its good keeping qualities of microcapsules, a variety of particle sizes that can be produced and an excellent dispensability of particles in aqueous media. The disadvantage of this technology is the high temperature conditions necessary for drying and access of air [18]. Spray drying technology requires well-adjusted operating conditions as well as adequate composition of the solution that contains the active compounds. The former include factors such as inlet air temperature, atomization airflow, liquid flow rate, aspirator suction velocity and solid concentration, among others [19]. The response surface methodology (RSM) is a statistical analysis tool that predicts appropriate levels of independent variables for optimizing response variables. Factorial designs are frequently used in experiments involving several factors where it is necessary to study the joint effect of the factors on a response. In this study, RSM was applied to obtain a high solid yield of sunflower oil microcapsules by using a spray drying process. The specific objectives of the work were to evaluate the influence of operating

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conditions on VO powder qualities and analyze the microstructure and rancidity in the optimized VO microcapsules.

2. Materials and methods

2.1. Materials

Sunflower (*Helianthus annuus*) oil (Natura, AGD, Córdoba, Argentina), certified as organic product, was used as a model of VO for microencapsulation experiments. This VO was rich in polyunsaturated essential fatty acids (64%) and monounsaturated fatty acids (26%). Hydroxypropyl methylcellulose (HPMC, Methocel K99, Ciclo Química, Argentina) and maltodextrin (MD, DE15, Distribuidora Nicco, Argentina) were used as wall materials. Soya lecithin (food grade, Distribuidora Nicco, Argentina) was used as emulsifying agent.

2.2. Spray drying emulsion preparation

A suspension of MD/HPMC was prepared as follow: MD (6%) was dissolved in distilled water at room temperature; HPMC (3%) was slowly added, the whole mixture was blended for 5 min using a typical kitchen mixer at 200 r.p.m. (Philips, China), and it was stored for 24 h at 4 °C. For the emulsion preparation, a blend of soybean lecithin (0.15%) and sunflower oil was incorporated to the MD/HPMC/water suspension at a ratio of 2:1 (MD/HPMC:sunflower oil), by using an Ultraturrax T18 homogenizer at 18000 r.p.m. for 10 min at 10 °C. The obtained emulsion (200 mL) was stored at 4 °C until use.

2.3. Emulsion viscosity

Emulsion viscosity was measured at 10 °C by means of steady-shear flow curves (shear stress × shear rate), using a controlled stress Physica MCR301 rheometer (Anton Paar, Graz, Austria) with stainless steel plate–plate geometry (25 mm in diameter and 2 mm in gap). Three flow ramps (up, down and up-cycles) were obtained in a range of shear stress corresponding to shear rates from 0 to 100 s^{-1}, in order to eliminate any possible thixotropic effect. Trials were performed in triplicate by 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.

2.4. Spray drying and experimental design

The spray drying process was performed by using a laboratory-scale Mini Spray Dryer (Büchi B-290, Büchi Labortechnik AG). A two-fluid nozzle with a cap orifice diameter of 0.5 mm was used. Air atomizing pressure was kept constant at 6 bars for all the experiments.

The following parameters were selected as independent variables: (A) drying air inlet temperature, (B) atomization air volumetric flow rate, (C) feed volumetric flow rate, and (D) drying air volumetric flow rate. Table 1 summarizes the levels of the operating variables which were selected on the basis of what was recommended for the experimental unit [20], and from several trial experiments [19].

Experiments were planned applying a ratable central composite design (response surface methodology, RSM, Statgraphics plus 5.0) with four factors and five levels (2^{-4} design) to assess the effect of the operating variables on the responses: solid yield (SY, percentage of solid material recovered in the dryer), surface oil (SO, percentage of VO on the microcapsules surface), encapsulation efficiency (EE, percentage of VO present into the microcapsules) and moisture content (MC, moisture content on SY). All experimental runs were performed in randomized order to eliminate any possible sources of bias. Four replicates were made at the center point of design to allow the estimation of the pure error at the sum of the square. The experiment design is shown in Table 2.

Results were analyzed by a multiple regression method. The quality of the models' fitness was evaluated by ANOVA (Statgraphics plus 5.0, USA). The experimental results were applied to obtain the regression models. The fit of each model to the experimental data was given by the determination coefficient (R^2) which explains the extent of the variance in a modeled variable that can be understood with the model. Multiple regression equations included only significant coefficients ($p < 0.05$). Only models with high determination coefficients were included in this study. Three-dimensional response surface plots were generated for each response variable.

Calculation of the optimal processing conditions for the solid yield production was performed using a multiple response method called desirability [21]. This optimization method incorporates desires and priorities for each of the variables.

2.5. Powder analysis

2.5.1. Solid yield (SY)

It was calculated as the ratio of the powder weight collected after every spray drying experiment to the initial amount of solids in the sprayed dispersion volume (g of solid in 200 mL of emulsion).

Table 1
Process variables levels.

Parameter independent formulation	Low (−)	High (+)	Units
A (air inlet temperature)	130	200	°C
B (atomization air flow rate)	400	800	L/h
C (pump setting)	5	15	%
D (aspirator setting)	80	100	%

Table 2
Experimental matrix according to a 2^{-4} central composite design and studied responses.

Run	A	B	C	D	MC*	SY*	SO*	EE*
1[a]	165	600	10	90	3.95	23.47	20.22	79.78
2	200	400	15	80	3.24	33.69	25.66	74.34
3	200	800	15	80	4.31	15.04	22.62	77.38
4	200	400	5	100	2.96	39.34	22.13	77.87
5	130	800	15	80	4.86	9.00	19.09	80.91
6[a]	165	600	10	90	3.73	23.41	16.31	83.69
7	165	600	2	90	3.56	20.17	19.42	80.58
8	200	800	15	100	3.47	26.13	23.65	76.35
9	130	400	5	100	2.35	38.95	18.18	81.82
10	130	400	5	80	2.55	29.41	24.46	75.54
11	130	800	5	80	3.97	10.30	13.93	86.07
12	130	400	15	100	3.88	27.35	26.87	73.13
13	200	800	5	80	3.88	5.44	15.68	84.32
14	200	800	5	100	3.21	17.87	20.99	79.01
15	165	279	10	90	2.93	38.68	22.90	77.10
16	165	600	18	90	4.67	19.91	22.62	77.38
17	109	600	10	90	4.12	22.03	18.73	81.27
18	130	800	5	100	4.78	23.07	21.02	78.98
19	200	400	15	100	3.18	39.88	17.75	82.25
20	130	800	15	100	4.41	18.51	17.45	82.55
21	130	400	15	80	4.63	21.76	18.69	81.31
22[a]	165	600	10	90	3.09	25.56	16.37	83.63
23	221	600	10	90	2.63	28.63	15.44	84.56
24	165	600	10	74	3.14	15.42	13.70	86.30
25	165	600	10	106	3.27	33.21	16.16	83.84
26[a]	165	600	10	90	3.41	25.44	13.00	87.00
27	200	400	5	80	2.71	33.05	25.69	74.31
28	165	921	10	90	3.94	10.90	17.60	82.40

A: Drying air inlet temperature. B: Atomization air volumetric flow rate. C: Feed volumetric flow rate. D: Drying air volumetric flow rate.

[a] Central point.

* Experimental responses: MC (moisture content, %); SY (solid yield, %); SO (surface oil, %); EE (encapsulation efficiency, %).