



Optimization and physicochemical characterization of a spray-dried microencapsulated *Origanum vulgare* L. extract

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ABSTRACT: This study employed response surface methodology to optimize the spray-drying conditions for a microencapsulated oregano extract (*Origanum vulgare* L.) using a maltodextrin-gum Arabic wall-material system. Feed flow rates of 0.5, 0.6, and 0.7 L h⁻¹ and inlet air temperatures of 130, 140, and 150 °C were evaluated. The oregano leaves were dried, ground, and extracted by maceration with 90% (v/v) ethanol at a 1:10 mass-to-solvent ratio at 60 °C for 24 h under agitation. Process yield, encapsulation efficiency, and moisture content were evaluated. The optimal conditions within the experimental domain were a feed flow rate of 0.5 L h⁻¹ and an inlet air temperature of 150 °C. Independent validation under these conditions resulted in a process yield of 94.97%, encapsulation efficiency of 94.06%, and moisture content of 3.15%, with relative prediction errors of 0.13, 0.60, and 0.73%, respectively. The resulting spray-dried microencapsulated oregano extract showed 92.0% solubility, a bulk density of 0.24 g mL⁻¹, a tapped density of 0.48 g mL⁻¹, a Carr index of 50%, a Hausner ratio of 2.00, an angle of repose of 31.58°, and hygroscopicity of 21.49 g 100 g⁻¹. Its ferric reducing antioxidant power was 402 µmol Fe²⁺/g sample, and its total polyphenol content was 0.15 mg g⁻¹. Overall, the optimized process produced a spray-dried microencapsulated oregano extract with high process yield and encapsulation efficiency and measurable antioxidant-related activity, supporting its further evaluation as a functional food ingredient.

Keywords: microencapsulation; oregano extract; spray drying; antioxidant capacity.

Otimização e caracterização físico-química de extrato de *Origanum vulgare* L. microencapsulado por secagem por atomização

RESUMO: Este estudo empregou a metodologia de superfície de resposta para otimizar as condições de secagem por aspersão de um extrato de orégano (*Origanum vulgare* L.) microencapsulado, utilizando um sistema de materiais de parede à base de maltodextrina e goma arábica. Foram avaliadas vazões de alimentação de 0,5, 0,6 e 0,7 L h⁻¹ e temperaturas do ar de entrada de 130, 140 e 150 °C. As folhas de orégano foram secas, trituradas e submetidas à extração por maceração com etanol a 90% (v/v), na proporção massa/solvente de 1:10, a 60 °C durante 24 h, sob agitação. Foram avaliados o rendimento do processo, a eficiência de encapsulação e o teor de umidade. As condições ótimas no domínio experimental foram uma vazão de alimentação de 0,5 L h⁻¹ e uma temperatura do ar de entrada de 150 °C. A validação independente nessas condições resultou em rendimento do processo de 94,97%, eficiência de encapsulação de 94,06% e teor de umidade de 3,15%, com erros relativos de predição de 0,13; 0,60; e 0,73%, respectivamente. O extrato de orégano microencapsulado, obtido por secagem por aspersão, apresentou solubilidade de 92,0%, densidade aparente de 0,24 g mL⁻¹, densidade compactada de 0,48 g mL⁻¹, índice de Carr de 50%, razão de Hausner de 2,00, ângulo de repouso de 31,58° e higroscopicidade de 21,49 g 100 g⁻¹. Sua capacidade antioxidante de redução férrica foi de 402 µmol Fe²⁺/g de amostra, e o teor de polifenóis totais foi de 0,15 mg g⁻¹. De modo geral, o processo otimizado produziu um extrato de orégano microencapsulado por secagem por aspersão, com elevado rendimento e eficiência de encapsulação, além de atividade antioxidante mensurável, o que justifica sua posterior avaliação como ingrediente alimentício funcional.

Palavras-chave: microencapsulação; extrato de orégano; secagem por atomização; capacidade antioxidante.

1. INTRODUCTION

Aromatic plants of the *Origanum* genus are noted for their high content of phenolic and terpenoid compounds, with recognized antioxidant, antimicrobial, and anti-inflammatory properties. Among them, *Origanum vulgare* L. is

one of the most widely used species in the pharmaceutical and food industries, where it has traditionally been used as a natural preservative due to its richness in carvacrol, thymol, and rosmarinic acid, among other bioactive compounds (LEYVA-LÓPEZ et al., 2017). However, the direct use of

plant extracts in food formulations presents technological limitations associated with their volatility, low stability against environmental factors, and potential unwanted interactions with the food matrix (PUDŽIUVELYTĖ et al., 2025).

Microencapsulation is a widely used protection strategy to increase the stability and functionality of naturally occurring bioactive compounds (Rojas-Molina et al., 2024). Among the available techniques, spray drying is the most widely used method in the food industry due to its cost-effectiveness, reproducibility, and potential for industrial scaling (GHARSALLAOUI et al., 2007). Furthermore, this technique transforms liquid extracts into stable powders with improved technological properties such as fluidity, solubility, and ease of storage (ROJAS-MOLINA et al., 2022).

Several studies have reported the effectiveness of spray drying in encapsulating extracts rich in antioxidant compounds, using matrices such as maltodextrin and gum Arabic, which favor protection against oxidation and thermal degradation (BARANAUSKAITE et al., 2016). In the specific case of *O. vulgare* extracts, combining encapsulating materials and optimizing process variables, such as inlet temperature and feed flow, are determining factors in improving encapsulation efficiency and process yield (SÁNCHEZ-OSORNO et al., 2023).

However, despite the growing interest in the use of microencapsulated natural extracts as functional ingredients or food preservatives, there is still limited literature addressing the simultaneous optimization of the microencapsulation process of oregano extracts using response surface modeling (RSM), especially in formulations based on maltodextrin and gum Arabic. Furthermore, it is important to evaluate not only encapsulation performance and efficiency variables, but also the physicochemical properties of the resulting powder, which determine its industrial applicability.

In this context, the objective of this research was to apply the response surface methodology to characterize the process performance and physicochemical properties of a microencapsulated oregano (*O. vulgare*) extract by spray drying.

2. MATERIALS AND METHODS

2.1. Materials

Maltodextrin (Sigma-Aldrich, USA; DE 16.5–19.5) and gum Arabic (Sigma-Aldrich, USA; molecular weight approximately 250,000) were used as wall materials. Analytical-grade ethanol, acetic acid, hydrochloric acid, sodium carbonate, sodium acetate, sodium chloride, ferric chloride hexahydrate, 2,4,6-tripyridyl-s-triazine (TPTZ), Folin–Ciocalteu reagent, and other reagents used for the phytochemical screening and antioxidant and total polyphenol determinations were obtained from Merck KGaA, Darmstadt, Germany. Gallic acid was used as the reference standard for total polyphenol determination.

2.2. Collection and identification of plant material

A total of 500 g of leaves with the same size and color characteristics and no visible defects were collected. The selected leaves were oven-dried (Memmert) for 3 days at 65 °C to prevent thermal degradation of sensitive active

compounds (ZUBIA et al., 2023). The dried leaves were subsequently pulverized in a manual mill (Corona). The dried yield was 237.34 g.

2.3. Preparation of the hydroalcoholic extract of oregano

The dried leaves, in a 1:10 mass/solvent ratio, were macerated in 90% v/v ethanol at 60 °C for 24 hours with shaking in a sieve (GAVILANEZ; ROJAS, 2024). The solution was vacuum-filtered (Rocker-300), and the filtrate was concentrated in a rotary evaporator (SHB-III) at 50 °C, 50 min⁻¹ and 0.065 MPa until a final volume of 120 mL was obtained. The resulting concentrated hydroalcoholic extract was stored in an amber bottle at 4 °C until spray drying. The extract before drying had a total polyphenol content of 0.111 mg L⁻¹ and an antioxidant capacity of 10,340.55 mg L⁻¹, determined according to Gavilanez; Rojas (2024).

2.4. Phytochemical profile

Preliminary phytochemical analysis of the plant extracts was performed according to the procedure described by Terán et al. (2021) to identify the presence of secondary metabolites, including alkaloids, flavonoids, steroids, glycosides, cardiac glycosides, quinones, tannins, and saponins.

2.5. Response surface methodology

A D-optimal response surface design was generated using Design-Expert 8.0.6 (Stat-Ease Inc., Minneapolis, USA). Statistical significance was evaluated at $\alpha = 0.05$. The oregano powder with the highest yield, the most efficient microencapsulation process, and the lowest moisture content was selected. A D-optimal design was selected instead of a full factorial design because it provides an efficient estimation of the effects of the selected factors and their interactions using fewer experimental runs, thereby reducing experimental effort and material consumption.

The experimental domain (Table 1) consisted of 9 runs, including two replicates (runs 2 and 7), and considered two independent factors evaluated under controlled spray-drying conditions. The first factor, feed flow (A), was treated as a discrete numeric variable, with levels ranging from 500 to 700 mL h⁻¹. The second factor, inlet air temperature (B), was also defined as a discrete numeric variable, ranging from 130 to 150 °C. These experimental conditions were established to analyze their effects on the microencapsulation process of oregano extract, specifically on process yield, encapsulation efficiency, and moisture content of the resulting powder.

Table 1. Experimental design matrix for spray drying

Tabela 1. Matriz de planejamento experimental para secagem por aspersão

Run	Feed flow (mL h ⁻¹)	Inlet air temperature (°C)
1	600.0	150.0
2*	600.0	140.0
3	700.0	150.0
4	700.0	130.0
5	500.0	130.0
6	500.0	150.0
7*	600.0	140.0
8	700.0	140.0
9	500.0	140.0

*Experimental replicate runs at the center point (600 mL h⁻¹ and 140 °C).

*Réplicas de ensaios experimentais no ponto central (600 mL h⁻¹ e 140 °C).

2.6. Spray drying

The feed solution was prepared by mixing 70 g of gum Arabic with 290 g of distilled water and maintained at 65 °C until completely dissolved. Subsequently, 70 g of maltodextrin and 70 g of concentrated oregano extract were added, obtaining a final feed mass of 500 g.

Thus, the maltodextrin:gum Arabic ratio was 1:1, the wall-material:extract ratio was 2:1, and the total solids content was 42%. The mixture was homogenized using an Ultra-Turrax for 3 min at 10,000 min⁻¹. A total of 500 g of feed with 42% total solids was prepared according to the method described by Nguyen et al. (2024).

Spray drying was performed using a Mobile MinorTM spray dryer (Niro Atomizer Ltd., Copenhagen, Denmark). The feed temperature was maintained at 65 °C. The atomizer speed was set at 25,000 min⁻¹, and the outlet air temperature was maintained at 75 ± 2 °C. Each run processed 500 g of feed. The resulting powder was recovered from the dryer outlet, placed in airtight bags, and stored in a desiccator at 20 °C.

The process yield (MY) was calculated as the ratio between the mass of total solids recovered at the dryer outlet as powder and the mass of total solids fed into the spray dryer (equation 1), expressed as a percentage (KRISHNAIAH et al., 2012):

$$MY (\%) = \frac{M_M}{M_{TSF}} \quad (01)$$

where: MY, microencapsulation yield; M_M , microparticle mass; M_{TSF} , total mass of total solids in the feed.

The polyphenolic compounds were extracted following the method of Tiozon Jr et al. (2025) with some modifications. A mass of 100 mg of microencapsulated powder was dissolved in 2 mL of an ethanol/acetic acid/water (50:8:42) mixture to break up the spray-dried particles and facilitate the release of phenolic compounds. This mixture was stirred using a Vortex-Genie 2 for 1 min and filtered through a nylon microfilter mesh (0.45 µm). Total polyphenol content was determined using the Folin-Ciocalteu reagent, following the methodology described by Gavilanez; Rojas (2024). The reaction mixture was prepared by adding 50 µL of the sample to 2.5 mL of diluted Folin-Ciocalteu reagent, allowed to stand for 5 min, and then 7.5% (m/v) Na₂CO₃ (2 mL) was added. The mixture was incubated for 2 h before reading the absorbance at 765 nm in a spectrophotometer (Rayleigh UV-1601, Beijing). The results were expressed as milligrams of gallic acid equivalents per gram of sample.

Encapsulation efficiency (EE) was calculated as the percentage of extract retained within the microcapsules relative to the total extract present in the spray-drying feed, according to equation 2 (RIOS-AGUIRRE; GIL-GARZÓN, 2021):

$$EM (\%) = \frac{M_{MOE} - M_{SOE}}{M_{MOE}} \quad (02)$$

where: EM, efficiency of the microencapsulation process (%); M_{MOE} , mass of microencapsulated oregano extract; and M_{SOE} , mass of surface oregano extract recovered from the microcapsules.

2.7. Powder characterization

2.7.1. Humidity (% m/m)

The moisture content of the spray-dried microencapsulates was determined by estimating the mass loss of the powder after drying in a thermobalance (Sartorius Mod. MA-40, Germany) at 105 °C to constant mass.

2.7.2. Powder fluidity

The flowability of a powder is an important variable during bulk compression and depends on many factors, such as particle size and shape, density, surface properties, and also on processing conditions such as moisture content, temperature, electrical load, and applied shear/strain. A higher Hausner ratio (HR) indicates greater interparticle cohesion and poorer flowability (SUHAG et al., 2024). The HR was calculated as the ratio of tapped density to bulk density (equation 03) (MOONDRRA et al., 2018), with higher values indicating greater interparticle cohesion and poorer flowability:

$$\text{Hausner ratio} = \frac{\text{Tapped density}}{\text{Bulk density}} \quad (03)$$

The apparent density or poured density of powder (loosely packed at random) is defined as the ratio of the sample's mass to its total volume, which includes the interstices between the particles. The tapped density is the apparent density value after tapping the container several times. The Carr index, or compressibility index, is calculated as indicated in equation 04 (MOONDRRA et al., 2018):

$$\text{Carr index (\%)} = \frac{\text{Tapped density} - \text{Bulk density}}{\text{Tapped density}} * 100 \quad (04)$$

To determine the initial bulk density, the sample was poured into a glass cylinder, and the volume and mass were determined. The sample was then subjected to a series of tappings (N = 1500 times) using a Copley JV200 volumetric density apparatus.

2.7.3. Angle of repose

The angle of repose (°) is the simplest method for expressing the fluidity of powders. The angle of the heaped cone of a free-standing powder is the angle of repose. When a powder is dropped onto a flat surface from a funnel placed at a certain height, the funnel gradually moves upward to maintain a fixed height between the tip of the powder and the bottom of the funnel. The angle produced by the powder on the surface is the angle of repose (MÜLLER et al., 2021).

The angle of repose provides information about the flow characteristics and sliding behavior of the material; the Carr index is an indication of the compressibility of a powder; and the Hausner ratio is a number that indicates the flowability of granular/powdered material and is used in a wide variety of industries as an indication of powder flowability (MOONDRRA et al., 2018).

2.7.4. Hygroscopicity

Hygroscopicity was determined by placing 1 g of powder in a container at 25 °C with a saturated NaCl solution, obtaining a relative humidity of approximately 75%. The experimental period was 168 h, and after this time, the container was weighed. The hygroscopicity value was calculated based on the moisture absorption capacity,

expressed in grams of moisture absorbed per 100 g of dry matter (ARYA; KUMAR, 2023).

2.7.5. Solubility

The solubility of the microencapsulates was determined according to the methodology described by Cano-Chauca et al. (2005). One gram of the sample was mixed with 100 mL of distilled water, and the mixture was stirred with a magnetic stirrer (MS01) for 30 min. The solution was then centrifuged (ELMI CM, Riga, Latvia) at 3500 min⁻¹ for 5 min. A 25 mL aliquot of the supernatant was transferred to a pre-weighed Petri dish and immediately dried in a Universal Oven UF55 (Memmert GmbH+Co.KG, Germany) at 105 °C for 5 h. The solubility was calculated by mass difference and expressed as a percentage of the total mass.

2.7.6. Iron-reducing antioxidant capacity

Total polyphenol content (mg g⁻¹) and ferric reducing antioxidant power (FRAP) assay were performed as described by Gavilanez; Rojas (2024). Briefly, 100 mg of the sample was weighed and mixed with 10 mL of distilled water. To prepare the reagent, acetate buffer (0.3 M, pH 3.6), TPTZ (10 mM in 40 mM HCl), and FeCl₃.6H₂O solution (20 mM)

were mixed in a 10:1:1 (v/v) ratio. To determine the ferric reducing antioxidant power (FRAP), 2.85 mL of FRAP reagent was added to 0.15 mL of the diluted sample. The reaction mixture was incubated in the dark for 30 minutes, and the absorbance was measured at 593 nm using a UV-Vis spectrophotometer with distilled water as a blank. The reducing power was expressed as micromoles of iron per gram (μM Fe²⁺/g sample).

3. RESULTS

3.1. Phytochemical profile of the crude drug oregano

Phytochemicals are a heterogeneous class of plant secondary metabolites whose content depends on various intrinsic and extrinsic factors, which could be related to the differences between the results of the present study and those reported in the literature. Fatty compounds and triterpenes /steroids were present in the ether extract (Table 2). Terpenoids comprise the second largest group of secondary metabolites and are widespread in terrestrial plants; they also possess a wide range of biological activities, including antimicrobial, anti-inflammatory, antioxidant, and anticancer properties (RODRIGUEZ-GARCIA et al., 2017).

Table 2. Phytochemical profile of the ethereal, ethanolic, and aqueous extracts of *O. vulgare*.

Tabela 2. Perfil fitoquímico dos extratos etéreo, etanólico e aquoso de *O. vulgare*.

Metabolite	Test	Ethereal extract	Ethanolic extract	Aqueous extract
Fatty compounds	Sudan	+++	NA	NA
Alkaloids	Dragendorff	-	++	++
Lactonic grouping	Baljet	-	+++	NA
Triterpenes/steroids	Lieberman. B	+++	+++	+++
Catechins	Catechins	NA	+++	NA
Resins	Resins	NA	-	NA
Reducing sugars	Fehling	NA	+++	+++
Saponins	Foam	NA	-	+-
Phenolic compounds	Ferric (III) chloride	NA	+++	+++
Free amino acids/amines	Nihrin	NA	+++	NA
Quinones/benzoquinones	Bortranger	NA	+++	NA
Flavonoids	Shinoda	NA	+++	+++
Cardiotonic glycosides	Kedde	NA	-	NA
Anthocyanins	Anthocyanidins	NA	+-	NA
Mucilages	Mucilages	NA	NA	+-
Bitter principles	Bitter principles	NA	NA	+++

+: weak presence; ++: moderate presence; +++: strong presence; ±: weak or doubtful presence; -: absence; NA: not applicable.

+: presença fraca; ++: presença moderada; +++: presença forte; ±: presença fraca ou duvidosa; -: ausência; NA: não aplicável.

The ethanolic extract was positive for alkaloids, lactonic grouping, triterpenes, catechins, reducing sugars, phenolic compounds, free amino acids/amines, quinones/benzoquinones, and flavonoids. In addition, the regular presence of anthocyanidins was detected. The presence of terpenes was confirmed in a study by Bañuelos-Valenzuela et al. (2018), who identified carvacrol, thymol, terpinene, linalool, and limonene in the ethanolic extracts of *O. vulgare*. Although these compounds have been reported in *O. vulgare* extracts in previous studies, they were not specifically quantified in the present study.

Flavonoids and phenolic acids are the main types of phenolic compounds present in oregano. They are classified based on the number and arrangement of their carbon atoms into flavonoids (anthocyanidins) and non-flavonoids (phenolic acids). They are commonly found conjugated with sugars and organic acids. Flavones have been identified as the

most abundant flavonoid in *O. vulgare* (SOLTANI et al., 2021).

Triterpenes, steroids, reducing sugars, phenolic compounds, flavonoids, and bitter principles were detected in the aqueous extract. Antoniadou et al. (2024) also found alkaloids, flavonoids, and terpenoids in extracts of *O. vulgare*. Alkaloids are the most important group of nitrogen-containing secondary metabolites (RODRIGUEZ-GARCIA et al., 2017).

3.2. Regression models and statistical analysis

The relationships between feed flow and inlet air temperature with respect to the dependent variables of microencapsulation process yield, microencapsulation process efficiency, and humidity were analyzed. Table 3 shows the experimental design and results for each variable. As can be seen, run 6 presented the highest values for yield

(95.15%) and microencapsulation process efficiency (94.0%), while also exhibiting the lowest humidity value (3.15%).

The regression models were expressed in terms of coded independent variables, where A represents the coded feed flow rate, and B represents the coded inlet air temperature. The variables were coded according to the following transformation: $x_i = (X_i - X_0) / \Delta X_i$, where X_i is the actual value of the independent variable, X_0 is the value at the center of the experimental range, and ΔX_i is the step change. The results for the dependent variables were related to the independent variables through linear mathematical equations. The best fit to the model for each of the variables was:

$$Y = 89.6 - 1.92A + 3.33B \quad (05)$$

$$EE = 88.48 - 2.99A + 3.16B \quad (06)$$

$$H = 3.89 + 0.35A - 0.37B \quad (07)$$

where: Y, process yield (%); EE, encapsulation efficiency (%); H, moisture content (%); A, coded feed flow rate; and B, coded inlet air temperature.

Table 4 presents the analysis of variance and the significance of the estimated coefficients for the linear model, by response variable. The degree of fit of the experimental data to the suggested models was evaluated using the

parameters F, p, R^2 , and lack of fit. Since the F-values of the models were significant for the three dependent variables, it can be stated that the fitted models showed significant regression terms for the evaluated responses ($p \leq 0.05$). The adjusted R^2 values indicated that the models explained a substantial proportion of the variability in the responses, accounting for approximately 97%, 81%, and 97% of the variability in process yield, encapsulation efficiency, and moisture content, respectively. However, model adequacy was further assessed using residual diagnostics and should be interpreted considering the limited number of experimental runs.

It is evident that both the feed flow rate and the inlet air temperature significantly affected the humidity, yield, and efficiency of the microencapsulation process. The lack of fit, as indicated by the F and p values, was not significant, suggesting that the model adequately describes the functional relationship between the experimental factors and the response variables.

In the case of the microencapsulation process, the R^2 values for performance and humidity were greater than 95%, while the efficiency of the microencapsulation process was 85.63%. The R^2 value increases each time significant factors are incorporated into the model; however, this is not the case for the adjusted R^2 .

Table 3. Effect of independent variables on microencapsulation process yield, microencapsulation process efficiency, and humidity.
Tabela 3. Efeito das variáveis independentes no rendimento, na eficiência e na umidade do processo de microencapsulação.

Run	Feed flow (mL h ⁻¹)	Inlet air temperature (°C)	Microencapsulation process yield (%)	Microencapsulation process efficiency (%)	Humidity (%)
1	600	150	93.23 ± 0.40	91.01 ± 0.47	3.56 ± 0.05
2	600	140	89.01 ± 0.46	90.86 ± 0.54	3.99 ± 0.06
3	700	150	91.13 ± 0.40	88.25 ± 0.53	3.85 ± 0.06
4	700	130	85.13 ± 0.60	81.00 ± 0.80	4.56 ± 0.07
5	500	130	88.12 ± 0.53	88.00 ± 0.60	3.98 ± 0.07
6	500	150	95.15 ± 0.53	94.00 ± 0.60	3.15 ± 0.06
7	600	140	89.12 ± 0.53	91.11 ± 0.62	3.92 ± 0.06
8	700	140	87.16 ± 0.54	85.00 ± 0.70	4.22 ± 0.07
9	500	140	91.67 ± 0.56	90.22 ± 0.63	3.43 ± 0.07

Mean ± standard deviation (n = 3, replicates of analytical determinations).

Média ± desvio-padrão (n = 3, réplicas de determinações analíticas).

Table 4. Analysis of variance of the coded linear models for process yield, encapsulation efficiency, and moisture content.

Tabela 4. Análise de variância dos modelos lineares codificados para o rendimento do processo, a eficiência de encapsulamento e o teor de umidade.

Source	Microencapsulation process yield		Efficiency of the microencapsulation process		Humidity	
	F value	p-value	F value	p-value	F value	p-value
Model	136.51	< 0.0001	17.88	0.0030	127.64	< 0.0001
A	79.04	0.0001	18.77	0.0049	130.22	< 0.0001
B	193.99	< 0.0001	16.99	0.0062	125.06	< 0.0001
Lack of fit	55.31	0.1017	109.90	0.0723	2.49	0.4462
R^2 adjusted	0.9713	-	0.8084	-	0.9694	-
R^2 predicted	0.9411	-	0.7460	-	0.9488	-
CV (%)	0.5880	-	1.9062	-	1.9229	-
Adequate	34.3938	-	12.5852	-	33.6586	-
Standard	0.5290	-	1.6932	-	0.0741	-
Mean	89.9689	-	88.8278	-	3.8511	-
PRESS	4.5985	-	30.4146	-	0.0734	-

A, coded feed flow rate; B, coded inlet air temperature; CV, coefficient of variation; PRESS, predicted residual error sum of squares. Statistical significance was evaluated at $p \leq 0.05$.

A, vazão de alimentação codificada; B, temperatura do ar de entrada codificada; CV, coeficiente de variação; PRESS, soma dos quadrados dos erros residuais de previsão. A significância estatística foi avaliada com $p \leq 0,05$.

In this sense, according to this parameter, the adjusted models of microencapsulation process performance and

humidity explained more than 95.0% of the data variability, as well as 80% of the variability for microencapsulation

process efficiency. The residuals are calculated primarily to detect outliers, which are far from the straight line. The residuals followed a normal distribution, suggesting the adequacy of the models; in each case, they fit a straight line (Figure 1).

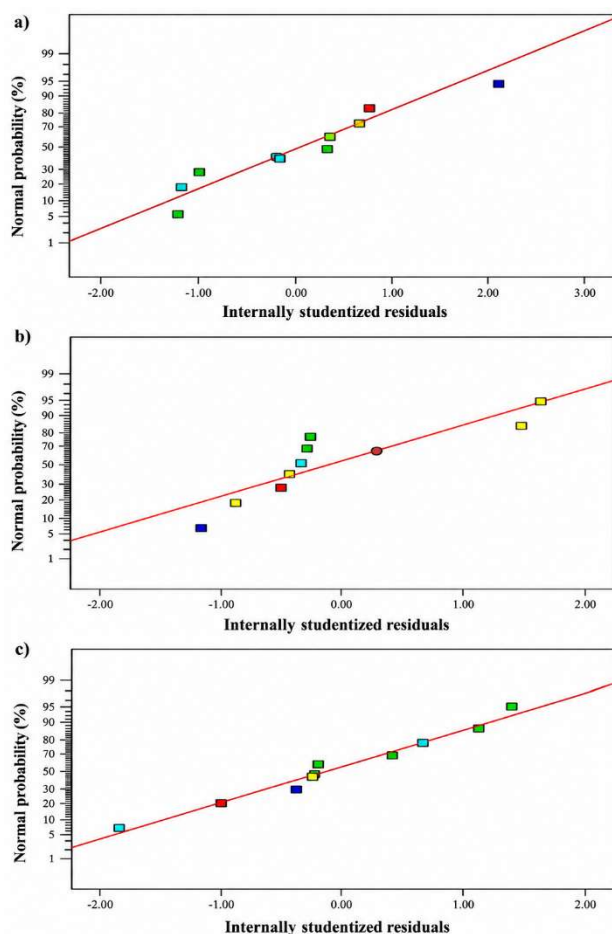


Figure 1. Normal probability plot of the Studentized residuals for the response variables: a) microencapsulation process yield; b) microencapsulation process efficiency; c) powder moisture. Figura 1. Probabilidade normal dos resíduos studentizados para as variáveis de resposta: a) rendimento do processo de microencapsulação; b) eficiência do processo de microencapsulação; c) umidade do pó.

3.3. Influence of feed flow and inlet air temperature on the performance of the microencapsulation process

From the statistical analysis of the experimental data, it was observed that both the feed flow and the inlet air temperature significantly influenced the performance of the microencapsulation process (Table 4). Equation 5 shows that the effect of feed flow was linear and negative, such that as this factor increased, the performance of the microencapsulation process decreased.

On the other hand, the inlet air temperature had a positive influence, which caused an increase in the response variable. The highest performance value for the microencapsulation process (95.15%) was obtained for run 6, with a feed flow of 500 (mL h⁻¹) and an inlet air temperature of 150 °C (Figure 2a).

According to Bernal-Millán et al. (2022), the performance values ranged from 34.73 to 71.85%, with the highest values reported for 140 °C inlet air temperature. Contrary to the results of this study, the quadratic model was the one that

proved significant; however, the coefficient of determination was 79.36%.

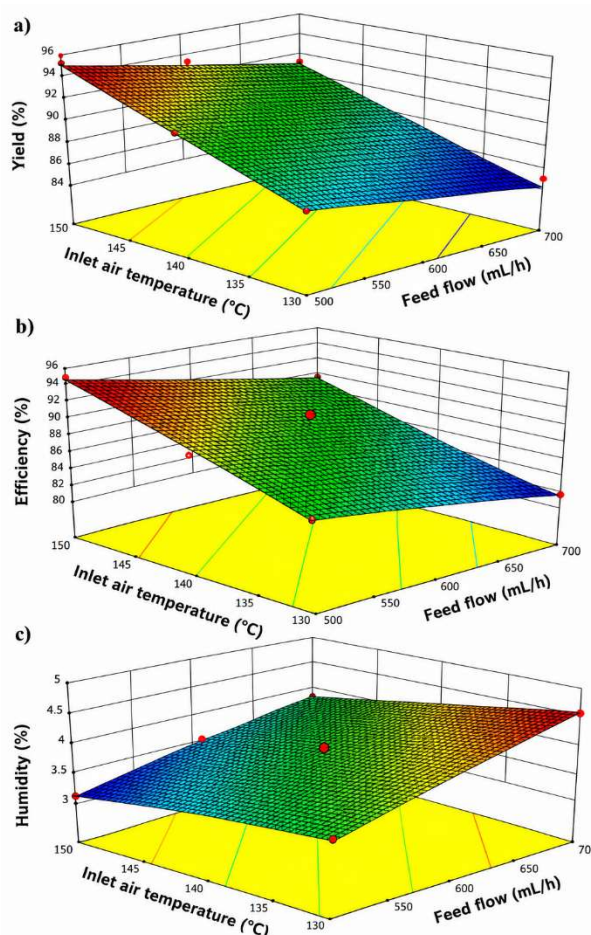


Figure 2. Variation of a) microencapsulation yield; b) efficiency; and c) humidity as a function of feed flow and inlet air temperature.

Figura 2. Variação de a) rendimento de microencapsulação; b) eficiência; e c) umidade em função da vazão de alimentação e da temperatura do ar de entrada.

3.4. Influence of feed flow and inlet air temperature on the efficiency of the microencapsulation process

As observed in Table 4, both factors significantly influenced the response variables. According to Equation 06, the feed flow had a linear and negative impact, whereas the inlet air temperature had a linear but positive impact.

Thus, while an increase in the feed flow decreases the efficiency of the microencapsulation process, an increase in the Inlet air temperature increases the efficiency of the microencapsulation process.

The feed flow and inlet air temperature factors had an equal impact on the performance and efficiency of the microencapsulation process. The highest efficiency of the microencapsulation process corresponded to run 6, with 94% efficiency, under conditions of 500 mL h⁻¹ as feed flow and 150 °C inlet air temperature (Figure 2b).

The efficiency of the microencapsulation process is one of the most important parameters in microencapsulation. If the extract is completely encapsulated by the wall material, the number of active compounds in a given mass of microcapsules can be calculated from the composition of the dispersion used for spray drying. However, in practice, materials subjected to microencapsulation are not completely encapsulated.

According to the results of Baranauskaite et al. (2016), this factor was mainly affected by the concentration of the wall material (maltodextrin and gum Arabic mixture), while the influence of the feed flow rate and inlet air temperature was unclear. Uclaray et al. (2022) reported a 93% encapsulation efficiency of a phenolic extract of wild oregano.

3.5. Influence of feed flow and inlet air temperature on powder moisture

The linear terms for feed flow and inlet air temperature were found to have significant effects on humidity (Table 4). The coefficients in Eq. 7 show that feed flow had a positive impact, while inlet air temperature had a negative impact. However, in the case of humidity, the goal is to minimize this value. Thus, the lowest humidity value would correspond to a decrease in feed flow and an increase in inlet air temperature (Figure 2c). According to Table 3, the lowest humidity value was recorded for run 6 (500 mL h⁻¹ and 150 °C).

Similar to this work, Baranauskaite et al. (2016) proposed that the highest temperature (190 °C) resulted in the lowest moisture content, which is related to the fact that at higher temperatures, evaporation of the solution is faster and more efficient.

Contrary to this work, they observed a similar trend in the feed flow rate. However, it has been suggested that with increasing feed flow rate, due to the large amount of product to be dried, the contact time of the dispersion with the drying air is insufficient to cause the evaporation of all the water

(Barańska-Dolomisiewicz et al., 2025), increasing the moisture content. Botrel et al. (2012) reported similar moisture content values (1.3-3.65%) for a microencapsulated oregano essential oil.

3.6. Optimal conditions for the microencapsulation

The ideal conditions for drying the oregano extract were defined to maximize the yield and efficiency of the microencapsulation process while minimizing powder moisture within the evaluated range of independent variables. Desirability was also used to optimize the responses simultaneously. The optimization model generated three feasible solutions that simultaneously maximized process performance and minimized moisture content in the microencapsulated oregano extract.

All proposed solutions maintained a constant feed flow of 500 mL h⁻¹, while the inlet air temperature varied slightly between 148 and 150 °C (Table 5). Solution 1 showed the best overall performance, achieving a microencapsulation yield of 94.85%, efficiency of 94.63%, and humidity of 3.17%, with the highest desirability index (0.98). Solutions 2 and 3 also demonstrated satisfactory responses, though with marginally lower desirability values (0.95 and 0.94, respectively), confirming the robustness of the optimized operating conditions.

For comparison, Baranauskaite et al. (2016) reported different optimal operating conditions for the microencapsulation of oregano extract, including an inlet air temperature of 170 °C and a feed flow rate of 40 mL min⁻¹.

Table 5. Optimization solutions for the microencapsulation process
Tabela 5. Soluções de otimização para o processo de microencapsulamento

Solution	Feed flow (mL/h)	Inlet temperature (°C)	Predicted yield (%)	Predicted EE (%)	Predicted moisture (%)	Desirability
1*	500	150	94.8509	94.6286	3.17318	0.984494
2	500	148	94.3511	94.1551	3.22937	0.954079
3	500	148	94.1594	93.9736	3.25091	0.941643

*Solution 1 was selected by the optimization software. EE: encapsulation efficiency.

*A Solução 1 foi selecionada pelo software de otimização. EE: eficiência de encapsulamento.

An independent validation run was performed under the conditions specified in Solution 1. The experimental values obtained for yield, encapsulation efficiency (EE), and moisture were 94.97, 94.06, and 3.15%, respectively, compared with predicted values of 94.85, 94.63, and 3.17%. The relative prediction errors were 0.13, 0.60, and 0.73%, respectively, indicating good agreement between the experimental and predicted values.

3.7. Powder characterization

Under optimal drying conditions, the microencapsulated oregano extract exhibited suitable physicochemical properties for use as a functional powder ingredient (Table 6).

The product showed low moisture content ($3.15 \pm 0.2\%$) and high solubility ($92.0 \pm 0.8\%$), indicating good stability and reconstitution capacity. The bulk and tapped densities were 0.24 ± 0.01 g mL⁻¹ and 0.48 ± 0.02 g mL⁻¹, respectively, resulting in a Carr index of 50% and a Hausner ratio of 2.0 ± 0.08 , both of which classify the powder as cohesive with very poor flowability/high cohesiveness according to conventional Hausner ratio and Carr index classifications.

Table 6. Physical and chemical properties of the powder obtained under the optimal drying conditions

Tabela 6. Propriedades físicas e químicas do pó obtido nas condições ótimas de secagem

Parameter	Mean $\pm$ SD
Moisture (% w w ⁻¹)	3.15 $\pm$ 0.20
Solubility (%)	92.0 $\pm$ 0.80
Bulk density (g mL ⁻¹)	0.24 $\pm$ 0.01
Tapped density (g mL ⁻¹)	0.48 $\pm$ 0.02
Carr's index (%)	50.0 $\pm$ 1.50
Hausner ratio	2.00 $\pm$ 0.08
Angle of repose (°)	31.58 $\pm$ 0.80
Hygroscopicity (g 100g ⁻¹)	21.49 $\pm$ 0.50
Yield (%)	94.97 $\pm$ 0.60
Ferric reducing antioxidant power (μ mol Fe ²⁺ /g sample)	402 $\pm$ 10
Total polyphenols (mg g ⁻¹)	0.15 $\pm$ 0.01

*Replicates of analytical determinations.

*Rélicas de determinações analíticas.

The angle of repose ($31.58 \pm 0.8^\circ$) suggested acceptable flow behavior, while the hygroscopicity value (21.49 ± 0.5 g 100g⁻¹) indicated a moderate tendency to absorb moisture. Additionally, the independent validation run resulted in a

process yield of $94.97 \pm 0.6\%$, confirming the model prediction. It retained its bioactive properties, with an iron-reducing antioxidant capacity of $402 \pm 10 \mu\text{mol Fe}^{2+}/\text{g}$ and a total polyphenol content of $0.15 \pm 0.01 \text{ mg g}^{-1}$, confirming the partial preservation of antioxidant compounds after spray drying.

Humidity is an important parameter in evaluating powder quality and shelf life. High humidity can induce stickiness in the particles, leading to the joining and subsequent agglutination of microcapsules, as well as the collapse and oxidation of the encapsulated material (BARANAUSKAITE et al., 2016). The moisture content in this study was within the range (3.11-4.95%) reported by Baranauskaite et al. (2016).

Solubility is also considered an important factor in determining powder quality. The powder solubility value was within the range (71.4-97.2%) reported by Baranauskaite et al. (2016) and Cardona et al. (2021), who reported a solubility greater than 90%.

The bulk density of the microparticles ranged from 0.182 to 0.310 g mL^{-1} , as reported for the microencapsulation of a Turkish oregano extract (BARANAUSKAITE et al., 2016). The apparent density can be affected by the inlet air temperature, since higher evaporation rates can produce more porous powders with less droplet shrinkage during drying (JANISZEWSKA; WŁODARCZYK, 2013).

According to the Carr index and the Hausner ratio, the powder was cohesive and poorly flowing. However, the angle of repose was 31.58° , whereas the Carr index (50%) and Hausner ratio (2.0) indicated high cohesiveness and poor flowability. Because these indicators assess different aspects of powder flow, the discrepancy should be interpreted cautiously. A low Carr index value indicates good powder fluidity, while a high Hausner ratio corresponds to a more cohesive powder with reduced ability to flow freely (MOONDRA et al., 2018).

According to Baranauskaite et al. (2016), the Carr index and the Hausner ratio are affected by the concentration of wall material and, probably, by the size of the microcapsules. In this sense, smaller particles could result in a large surface area per unit mass. The presence of a high contact surface between particles enables greater cohesion and the formation of an attrition force, resulting in increased flow resistance. The surface composition of the powder particles also plays an important role in this property, as flowability involves overcoming surface interactions between particles (FERNANDES et al., 2013). Dry products that capture more than 5% by mass, between 40 and 90% RH at 25°C , are described as hygroscopic. Materials that capture less than 1%, under the same conditions, are considered non-hygroscopic.

The determination of hygroscopicity is a critical factor in the development of dry products, as it affects the flow, compaction, dissolution, stability, storage, and processing of the final product during formulation and packaging (MOONDRA et al., 2018). The value obtained in this work ($21.49 \text{ g } 100\text{g}^{-1}$) was similar to, although higher than, the 19.53% reported by Mahdavi et al. (2016) for drying with maltodextrin and gum Arabic. This parameter was inversely affected by the moisture content.

4. DISCUSSION

The optimization of spray drying conditions for microencapsulating *O. vulgare* hydroalcoholic extract

demonstrated that feed flow rate and inlet air temperature are critical factors governing process performance and powder quality. The linear regression models obtained (equations 5 to 7) revealed opposite effects of the two variables: increasing feed flow reduced both process yield and encapsulation efficiency, whereas raising the inlet temperature improved them, while moisture content followed an inverse trend. These findings are mechanistically consistent with the principles of spray drying technology. Higher feed flows shorten the droplet residence time inside the drying chamber, limiting complete particle drying and promoting wall deposition, which lowers yield (GHARSALLAOUI et al., 2007). Conversely, elevated inlet temperatures enhance the heat transfer rate, accelerating solvent evaporation and favoring the formation of dry, non-sticky particles that are efficiently recovered (BARANAUSKAITE et al., 2016).

Similar trends have been observed for other plant extracts; for instance, Bernal-Millán et al. (2022) reported that increasing inlet temperature from 130 to 150°C raised the yield of microencapsulated oregano polyphenols, although their absolute yield values (34.7-71.9%) were lower than those obtained here, likely due to differences in wall material composition and total solids content. The high total solids (42%) and the synergistic combination of maltodextrin and gum Arabic used in the present study probably contributed to the superior yields (exceeding 95% under optimal conditions) and encapsulation efficiencies (close to 95%) by providing adequate viscosity and film-forming properties that prevent droplet rupture before complete drying (KRISHNAIAH et al., 2012).

Regarding powder moisture, the lowest value was achieved at the highest inlet temperature and lowest feed flow, a direct consequence of more intense and prolonged drying. This is in full agreement with the work of Botrel et al. (2012) on oregano essential oil microcapsules, where moisture contents ranged from 1.3% to 3.65%, and with Baranauskaite et al. (2016), who found that increasing inlet temperature from 130°C to 190°C reduced moisture from 4.95% to 3.11%. Low residual moisture (here around 3%) is technologically desirable because it minimizes the risk of microbial proliferation, particle agglomeration, and oxidative degradation of labile phenolics during storage, thereby extending shelf life (BARAŃSKA-DOŁOMISIEWICZ et al., 2025).

The physicochemical properties of the powder obtained under optimal conditions deserve attention in relation to its potential food applications. The powder showed a solubility of 92% under the experimental conditions described by Cano-Chauca et al. (2005). This value represents the fraction of solids recovered from the aqueous dispersion under the assay conditions and should not be interpreted as evidence of beverage stability or sedimentation resistance, although a solubility greater than 90% is considered excellent for powdered ingredients intended for reconstitution in aqueous food systems such as beverages, sauces, or marinades (CARDONA et al., 2021).

However, the flowability indicators (Carr index of 50%, Hausner ratio of 2.0) suggested a cohesive powder with limited free-flowing behavior. This apparent contradiction with the angle of repose (31.6° , indicating acceptable flow) can be resolved by considering that the Carr index and Hausner ratio are more sensitive to interparticle cohesive forces, which are magnified when particle size is small and surface area is large – a common outcome of spray drying at

high inlet temperatures (JANISZEWSKA; WŁODARCZYK, 2013). Fernandes et al. (2013) reported similar findings for rosemary essential oil microcapsules, where higher drying temperatures produced finer particles with increased cohesion. For industrial handling, the use of flow conditioners (e.g., small amounts of silicon dioxide) or agglomeration steps could be implemented to improve powder flowability without compromising bioactivity. The hygroscopicity value (21.5 g 100g⁻¹) classifies the powder as hygroscopic, which is typical for maltodextrin-based encapsulates containing low molecular weight sugars from the extract (MAHDAVI et al., 2016). This implies that packaging under low relative humidity (e.g., using moisture barrier films with desiccants) is necessary to prevent caking and maintain dispersibility.

The microencapsulated powder exhibited a measurable reducing capacity by the FRAP assay (402 µmol Fe²⁺/g) and a total polyphenol content of 0.15 mg g⁻¹. These findings indicate that antioxidant-related compounds were detectable after spray drying, although the pre-drying and post-drying results were expressed on different concentration bases, which limits their comparability. Moreover, their preservation, antimicrobial activity, release behavior, and effectiveness in food matrices were not directly evaluated in the present study.

These findings indicate that the spray-dried microencapsulated oregano extract has potential as a functional food ingredient with antioxidant-related properties. However, antimicrobial activity, bioactive-compound retention during storage, controlled release, and efficacy in real food matrices were not directly evaluated in the present study. Therefore, further studies are required to determine its suitability for specific food applications.

The physicochemical properties observed suggest that the powder may be suitable for further evaluation as a functional food ingredient. Nevertheless, application-specific studies are required to assess its stability, sensory impact, bioactive-compound retention, release behavior, and technological performance in different food matrices.

5. CONCLUSIONS

Under the experimental conditions evaluated, lower feed flow rates and higher inlet air temperatures favored process yield and reduced the moisture content of the spray-dried microencapsulated *O. vulgare* extract. The best-performing condition within the evaluated range was 500 mL h⁻¹ and 150 °C, and independent validation showed good agreement between predicted and experimental responses.

The resulting microencapsulated powder exhibited high solubility and measurable reducing capacity, but also high cohesiveness and hygroscopicity, which may limit its handling and storage. Further studies are required to determine bioactive-compound retention, storage stability, release behavior, and efficacy in food matrices.

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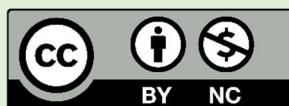
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