

OPTIMIZATION OF THE SPRAY DRYING PROCESS FOR OBTAINING CAPE GOOSEBERRY POWDER: AN INNOVATIVE AND PROMISING FUNCTIONAL FOOD

OPTIMIZACIÓN DEL PROCESO DE SECADO POR ASPERSIÓN PARA LA OBTENCIÓN UCHUVA EN POLVO: UN ALIMENTO FUNCIONAL INNOVADOR Y PROMISORIO

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ABSTRACT

Background: currently, functional foods are the type of foods of most interest to the modern consumer, due to the health benefits they provide. **Objectives:** Optimize the spray drying process to obtain cape gooseberry powder added with active compounds. **Methods:** A process of spray drying was carried out to obtain a powder from cape gooseberry suspensions added with vitamin C, iron, folic acid, isolated soy protein and dietary fiber. The drying process was optimized according to the characteristics of food formulations and operating conditions, obtaining a product with low hygroscopicity, high solubility and high levels of physiologically active compounds. Response surface methodology was used, considering a central composite design with four factors: maltodextrin (0-40%p/p), inlet air temperature (170-210°C), atomizer disc speed (16000-24000 rpm) and outlet air temperature (75-95°C). **Results:** The results showed a higher retention of vitamin C ($69.7 \pm 0.7\%$), folic acid ($90.9 \pm 1.8\%$) and iron ($90.8 \pm 1.0\%$) with the food formulation containing a 24.4% of maltodextrin and the drying process defined by an atomizer disc speed of 19848 rpm and inlet and outlet air temperature of 194.2°C and 87.7°C, respectively. **Conclusions:** The spray drying process is an effective technology that provides added value to the fruit of cape gooseberry, allowing the incorporation and conservation of active compounds such as iron, folic acid and ascorbic acid.

Keywords: *Physalis peruviana* L., aguaymanto, fruits, functional foods, dehydration.

RESUMEN

Antecedentes: Actualmente, los alimentos funcionales son la gama de alimentos de mayor interés para el consumidor moderno, debido a los beneficios saludables que estos proporcionan. **Objetivo:** Optimizar el proceso de secado por aspersión para la obtención de uchuva en polvo adicionado con componentes activos. **Métodos:** Suspensiones de uchuva adicionadas con vitamina C, hierro, ácido fólico, proteína de soya texturizada y fibra fueron sometidas a secado por aspersión para la obtención de un producto en polvo fortificado. El proceso de secado fue optimizado de acuerdo con las características de formulación de la suspensión y condiciones de operación del secador con el objetivo de obtener un producto en polvo

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con baja higroscopicidad, alta solubilidad y altos niveles de compuestos fisiológicamente activos. Se utilizó la metodología de superficie de respuesta, considerando un diseño central compuesto con 4 factores: maltodextrina (0-40%p/p), temperatura del aire de entrada (170-210°C), velocidad del disco atomizador (16000-24000 rpm) y temperatura del aire de salida (75-95°C). **Resultados:** Los resultados mostraron una mayor retención de vitamina C ($69,7\pm 0,7\%$), ácido fólico ($90,9\pm 1,8\%$) e hierro ($90,8\pm 1,0\%$) con la formulación de la suspensión que contiene un 24,4% de maltodextrina y condiciones de operación del proceso de secado definidas por una velocidad de disco atomizador de 19848 rpm y temperatura del aire a la entrada y salida de 194,2°C y 87,7°C, respectivamente. **Conclusiones:** El proceso de secado por aspersión es una tecnología efectiva que proporciona valor agregado al fruto de uchuva, permitiendo la incorporación y conservación de componentes activos como hierro, ácido fólico y ácido ascórbico.

Palabra clave: *Physalis peruviana* L., aguaymanto, frutas, alimentos funcionales, deshidratación.

INTRODUCTION

Fruit *Physalis peruviana* L., commonly called in Colombia as “Uchuva” is one of the most known species of this genre. Both the fruit and leaves extracts of the plant have been used in traditional medicine, showing important anticancer, antimycobacterial, antipyretic, diuretic and immunomodulatory antibiotics, antioxidants and anti-inflammatory activities, corroborated by the phytochemical studies of this genre, with the presence of Witanolides, steroids, alkaloids, glycosides, and flavonoids. The fruit has a pleasant flavor and characteristic aroma, which makes it interesting for transformation, conservation and use as a functional food by the addition of compounds with physiological activity: ascorbic acid (potent antioxidant), folic acid (formation of structural proteins and hemoglobin), minerals like iron (synthesis of red blood cells), fiber (promotes intestinal evacuation and maintenance of the intestinal microbial flora), and soy protein (high amount of essential amino acids and isoflavones that help control women's estrogens), which complements its nutritional value and provide it with healthy attributes (1- 5).

Different methods of food production are highlighted by significantly increasing shelf life and allowing the development of new products. The spray drying process is an interesting alternative for the preservation of food; besides it represents an important perspective for the industrial sector in view of the current demand of natural and easy to prepare products, as well as the possibility of using them as natural flavoring in various food formulations (cakes, shakers, creams, among others). Compared to other dehydration processes, the spray drying is notable for its applicability to heat-sensitive products, due to the fast evaporation

of water which keeps the droplet temperature low and short drying time, decreasing thermal damages, and loss of nutrients, resulting in good quality, and stability products with low storage and transport costs. (6, 7).

Dehydration of sugary foods such as juices or fruit purees is a technically complex process due to the hygroscopicity, and thermoplasticity of this products in the conditions of high humidity and temperature of the drying air. These conditions promote an amorphous (rubbery) and non-stable state in liquid feed, such state is highly adherent to the walls of spray dryer, decreasing the formation of powder and therefore the yield of the product (7, 8). The rubbery behavior correlates with drying temperatures above of glass transition temperature (T_g), this is the temperature in which a material goes from glassy state to rubbery state (8, 9). Therefore, through a special conditions such as low temperatures, partial crystallization of sugars, addition of additives, cooling the wall in the powder collection section, recirculation of fine powders into the chamber, addition of scraper surfaces, and modification of the dimension and shape of spray dryer, the possibility of having powdered fruits with higher yields and better quality, has been achieved (8, 9).

Using additives such as Arabic gum, pectins, maltodextrin, and other products with high molecular weight, the T_g is increased, therefore high temperatures can be used in order to obtain low moisture and more stable products. In the other hand, the concentration of the products in the feed should not exceed the technical operating limits of the equipment, such situation can increase the viscosity and decrease the performance of the spray or alter the taste. Nutritional value, physic properties related with the ease of reconstitution (moisture,

density, and porosity, surface area), instantizing properties (penetration, wettability, dispersibility, and solubility), droplet size or droplet size distribution of the powder, among others, are influenced by the nature of the feed (solid content, viscosity, temperature), type of spray, operating speed or pressure, and inlet and outlet temperatures of drying air (7, 10-14).

The aim of this research was to optimize the spray drying process to obtain cape gooseberry powder added with vitamin C, iron, folic acid, isolate soy protein and dietary fiber.

MATERIALS AND METHODS

Raw Material

Fresh cape gooseberries (FC), Colombia eco-type (La Unión-Antioquia) were used. The fruits free of external damages and with a commercial maturity in the scale color of 3-4 according to the Colombian technical norm NTC 4580 (15), were previously selected. The pulp was extracted by an Ultra-Turrax T25 (IKA) homogenizer, at 8000 rpm for 2 minutes, which grinds the fruit with its seed and peel, then the pulp was filtered with a 500 μm mesh. After the pulp was obtained, it was mixed under homogenization at 2000 rpm for 5 minutes, with maltodextrin of equivalent dextrose (DE) 19-20 (Shandong Boalingbao Biotechnology Co Ltd) and physiologically active compounds such as vitamin C, folic acid, iron, dietary fiber and isolate soy protein.

Spray drying process and optimization.

Spray drying process was carried out in a spray dryer (Vibrasec S.A., model Pasalab 1.5) with an evaporation ratio capacity of 1.5 Lh^{-1} , atomizer disc speed, air inlet temperature, air inlet flow, and feed temperature regulation systems. The system was operated under a vacuum pressure of 0.5 inches of water. Response surface methodology was used, considering a central composite design with four factors or independent variables: maltodextrin (0-40%p/p), inlet air temperature (170-210°C), atomizer disc speed (16000-24000 rpm) and outlet air temperature (75-95°C), and dependent variables: recovery percentage of vitamin C, folic acid, and iron, besides solubility and hygroscopicity. Twenty two experiments with six central points were generated from the statistical design, and the data were analyzed by Design Expert 6.0. software.

Optimization results were used to re-formulate the feed to spray dryer, with the objective of obtaining in 100 g of powder, 30 mg of ascorbic acid, 200 μg of folic acid and, 9 mg of iron which corresponds to 50% levels of the daily reference value (VDR) according to the Colombian technical standards. Response variables were adjusted to the following model:

$$Y = b_0 + \sum_{i=1}^k b_i X_i + \sum_{i=1}^k b_{ii} X_i^2 + \sum_{i=1}^{k-1} \sum_{\substack{j=2 \\ j>i}}^k b_{ij} X_i X_j \quad (\text{Equation 1})$$

Where: b_i are constants from the statistical adjustment of the model, X_i statistic factors of the model (A = % maltodextrin; B = inlet air temperature; C = atomizer disc speed; D = outlet air temperature), and k number of model factors.

Vitamin C, folic acid, and iron quantification

The quantification of vitamin C and folic acid (vitamin B₉) was carried out in high-performance liquid chromatograph (Shimadzu Prominence 20A) in reverse phase, equipped with a Premier C18 column, 5 μm droplet size, 250 mm x 4.6 mm, 1 mL/min flow, UV/VIS detector with diode array. The extraction of vitamin C was performed by diluting the sample in distilled water and filtering on Whatman paper 45 μm , a wavelength of 244 nm was detected in aqueous phase with oven temperatures of 35°C, and mobile phase (MP) of buffer KH_2PO_4 (0.02 molar, adjusted to pH = 3.0 with orthophosphoric acid) (16). Folic acid extraction was performed with sample hydrolysis with 2M NaOH, a wavelength of 283 nm was detected with an oven temperature of 35°C, and a PM buffer solution of KH_2PO_4 (0.05 molar, adjusted to pH = 3.0 with orthophosphoric acid/acetonitrile (90/10)) (17-19).

Iron was determined by the flame atomic absorption spectrometric method according to Colombian technical standards, NTC 4807 (20) for determination of minerals in fruits and vegetables.

Solubility

An amount corresponding to 1 g of powder was solubilized in 10 mL of distilled water this solution was homogenized in a vortex for 30 s and centrifuged at 3000 rpm for 15 minutes. Subsequently, the insoluble residue was determined by the difference in weight of the sample and the insoluble residue in the 10 mL of added water (21).

Hygroscopicity

For each experiment, samples of 1 g were placed in a sealed vessel with a saturated solution of CuCl_2 (67.5% HR) at 30°C. Samples were weighed after reaching equilibrium, and their hygroscopicity was expressed in grams of the total moisture per 100 g of sample (g/100g) (22).

RESULTS

Table 1 shows the results obtained from response variables for each experiment. The percentage of recovery of vitamin C presented significant differences ($p < 0.05$) respecting to factors A, B, and D. Quadratic interactions were significant for all factors, so were AB, AD, BC, and CD, except AC and CD. The percentage of recovery of folic acid showed significant differences ($p < 0.05$) due to the combined effect of four factors. Quadratic interactions were significant ($p < 0.05$), so were AD, BC, and CD, showing that this combination of variables may improve the percentage of recovery of folic acid (22, 23, 24). The percentage of iron recovery presented significant differences ($p < 0.05$) due to the effect of A, C, and D factors. Quadratic

interactions were significant ($p < 0.05$) for all four factors, so were AB, AD, BC, BD, and CD interactions, except AC interaction, consequent with the smaller possibility to generate attraction forces between these two compounds. Solubility did not show significant differences ($p > 0.05$) due to the effect of the study factors, reaching an average value of $97.42 \pm 0.43\%$. This result is mainly due to the high solubility of maltodextrin (ED 19-20) at the concentrations used (25, 26).

Hygroscopicity showed significant differences ($p < 0.05$) respecting to A, and C factors, and AA, AC, BC, BD, and CD interactions, highlighting a decrease in the hygroscopicity of the powder added with compounds with physiological activity (CPA) with maltodextrin increasing and the negative interaction between maltodextrin and atomizer disc speed at high content of maltodextrin in the feed suspension.

Figure 1, showed the response surfaces for vitamin C, folic acid, and iron recovery, as a function of study factors. Figure 2, showed response surfaces of solubility and hygroscopicity percentage of the powder as a function of study factors.

Table 1. Experiment results in spray drying process.

Experiment	Factor A Maltodextrin (%)	Factor B Inlet air temperature (°C)	Factor C Disc speed (rpm)	Factor D Outlet air temperature (°C)	Vitamin C (%)	Folic acid (%)	Iron (%)	Solubility (%)	Hygroscopicity (%)
1	20	190	20000	85	68.12	89.96	85.39	97.75	14.65
2	20	190	20000	85	66.35	91.27	87.16	96.76	14.80
3	20	190	20000	85	66.26	92.34	85.75	98.05	14.62
4	20	190	20000	85	67.30	93.37	85.41	96.94	14.51
5	20	190	20000	85	66.48	91.03	84.42	97.76	14.58
6	20	190	20000	85	67.40	95.54	84.08	96.75	14.32
7	20	190	20000	95	46.88	69.80	80.59	97.51	14.58
8	20	190	20000	75	29.97	53.84	55.64	97.68	15.26
9	20	190	24000	85	41.69	66.82	79.11	97.81	13.50
10	20	210	20000	85	89.38	41.57	79.99	97.50	14.72
11	20	170	20000	85	74.42	27.00	74.20	97.66	15.46
12	20	190	16000	85	34.55	56.40	63.21	97.46	14.79
13	10	200	18000	90	44.02	36.06	74.64	97.15	16.19
14	10	200	22000	90	27.69	16.87	54.50	96.96	18.16
15	10	180	18000	80	15.04	13.34	37.14	96.70	17.70
16	10	180	22000	80	28.91	59.50	90.59	96.82	17.29
17	30	200	22000	80	42.14	39.45	64.42	97.80	13.43
18	30	180	22000	90	57.95	64.95	68.87	97.17	13.65
19	30	200	18000	80	32.51	17.70	49.01	97.82	14.67
20	30	180	18000	90	54.63	45.61	65.10	97.62	14.42
21	40	190	20000	85	33.48	15.55	46.22	98.06	12.84
22	0	190	20000	85	10.60	5.31	26.63	97.61	25.08

■ Maxima □ Minima

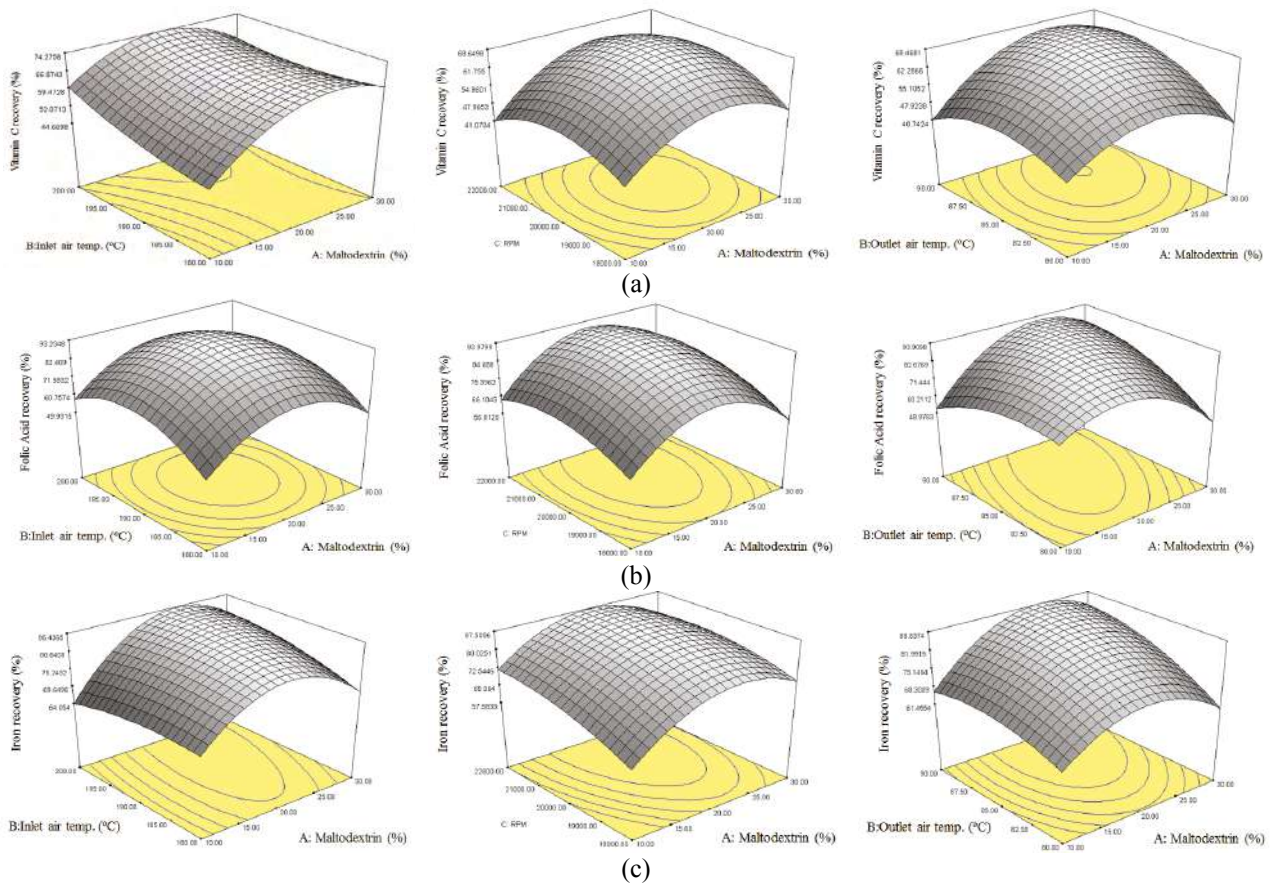


Figure 1. Recovery of vitamin C (a), folic acid (b), and iron (c) as a function of study factor.

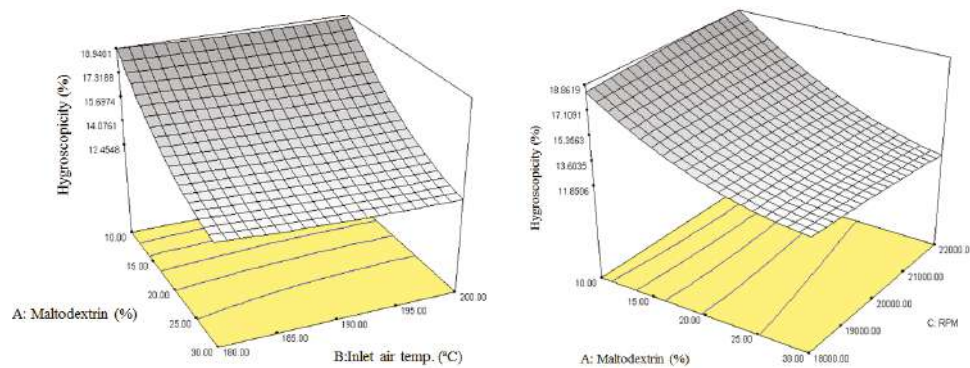


Figure 2. Surface response plots of hygroscopicity of the powder, as a function of study factors.

DISCUSSION

It is considered that vitamin C has a high molecular affinity with maltodextrin, due to the OH molecules they have in common, decreasing losses by the effect of the process (10, 13). Inlet and outlet air temperatures have an effect on the surface droplet temperature during spray drying, where increasing them decreases the percentage of recovery (7,

8). The mean value for the percentage of recovery for vitamin C at central points (A: 20%, B: 190°C, C: 20000 rpm y D: 85°C) was $66.75 \pm 0.49\%$.

On the other hand, folic acid presents in its chemical structure, carbonyl and amino groups, which interact with soy protein and maltodextrin, being strongly fixed with these compounds, protecting them against thermal effect (inlet and outlet air temperatures), and rotation speed (27, 28). The

mean value of the percentage of folic acid recovery at the central points was $92.25 \pm 1.82\%$.

The behavior of the percentage of iron recovery can be attributed to the fact that the iron is a non-thermosensitive mineral, otherwise, it is affected by humidity and droplet size, hence the influence of the outlet temperature. The iron (added as amino-chel in the liquid mixture) recovery showed a great number of statistic interactions, because its chemical structure allows interacting with mixture compounds by molecular attraction forces, protecting them and decreasing the negative effects of the process (29). Isolate soy protein interacts with bisglycinate iron which has amino and carbonyl groups, allowing the interaction by the hydrogen bonds. The pH of cap gooseberry pulp (3.4 ± 0.1) favors the charge of amino groups, allowing these two compounds to interact strongly (30).

The behavior of the hygroscopicity respecting to the study factors can be attributed to the decrease in the moisture of the powder, due to the higher content of maltodextrin and therefore higher percentage of solids in the feed suspension (27). The atomizing disc speed defines the droplet size and helps to increase or decrease the functionality of maltodextrin as protective agent (25). In this case, a lower hygroscopicity was obtained due to the high-speed operation for atomizer disc that generated smaller droplet size, and the high percentage of maltodextrin (22, 31-33).

Vitamin C is a thermolabile compound; however, results showed a greater recovery with the increase of inlet air temperature, probably due to the shorter residence time of the droplets under conditions of faster drying kinetics. Patil *Et al.* (2014) (39) reported vitamin C recovery levels for guava between 66.7% y 80.5%, being favorable the effect of the increase of inlet air temperature. Mendoza *Et al.* (2016) (40) evaluated the effect of different process variables on vitamin C content of a mango and whey beverage, with recovery levels of 60.97%. Naddaf *Et al.* (2012) (41) reported vitamin C recovery levels of 51.66% in the drying of orange pulp. Similar values were reported by Carrillo-Navas *Et al.* (2011) (42). The effect of A, C, and D factors on vitamin c recovery showed a similar behavior (figure 1a), where inflexion points located near the central points were observed, 20–25% de maltodextrin, 20000 rpm, and 85°C, with the minimum recovery percentages of 18000 and 22000 rpm, 10% maltodextrin, and outlet air temperatures of 80 and

90°C, because at high percentages, the encapsulant effect of maltodextrin is weakened and at low percentages is not enough to encapsulate the vitamin C in the suspension.

The high retention the encapsulation process of CPA is essential for its industrial application which is why some authors have suggested the use of soy protein in a spray drying process of a mixture formulated with maltodextrin and soy protein from legumes, where the vitamin C retention was 69 y 65%, with a significant difference due to the effect of proteins type because of the specific amino acids profile for each protein which has different charges increasing the molecular interactions with vitamin C (34). In this context, the presence of soy protein (2.5%) in the formulation, favors the recovery percentage for vitamin C, reaching a maximum of 89.4% (experiment 10, table 1). Dib *et al.*, 2003 (10) found interactions for vitamin C between a formulation with arabic gum and maltodextrin and the process variables, during the spray drying of camu-camu (*Myrciaria dubia*) pulp.

The effect of the four factors on the acid folic recovery was similar (figure 1b), showing inflexion points where the maxima tend to be located close to the central points and the minima close to the extremes (10 and 30% of maltodextrin, 180 and 200°C inlet air temperature, 22000 and 18000 rpm, and 80 and 90°C outlet air temperature). The sixth experiment was the most effective (95.54%), being higher than the obtained by Lopera *et al.*, 2009 (27), using arabic gum and maltodextrin in a ratio of 50:50, the highest encapsulation efficient was 97.3%, while with only arabic gum it was 92.4%, similar to the results found in this study, when 20 % maltodextrin was used. These results can be explained by the differences in the functionality of the wall materials used (28). The effect of the factors on the iron recovery percentage is shown in figure 1c, which is similar to that observed in others CPA. The interaction between the maltodextrin and inlet air temperature shows the maximum recovery ($\approx 86.4\%$) near the central coordinates: maltodextrin (22–24%) and 200°C; however, a decrease of the temperature in the zone of maximum recovery causes a reduction up to approximately 80%. The lowest effectiveness in the recovery percentage is presented in the maximum and minimum percentage of maltodextrin in the formulation. AC and AD interaction are similar to AB interactions, where the decrease in atomizing disc speed causes a reduction

in the recovery percentage, being more effective at speeds of 22000 rpm and outlet air temperatures of 85°C. The maximum and minimum iron recovery percentage corresponded to experiment 16 (90.59%) and 22 (26.63%), being higher than those found by García *et al.*, 2004 (30).

Some research determined an influence of study factors: Tonon *et al.*, 2008 (35) found that the anthocyanins recovery in the acai fruit (*Euterpe oleracea* Mart.) showed significant differences due to the inlet air temperature where at temperature of 138°C the recovery percentage was 88%; recovery values for folic acid and iron in the present study was similar at inlet and outlet air temperatures of 190 y 85°C respectively, with 20% maltodextrin, while at lower temperatures there is a tendency to form agglomerates due to the higher moisture content (31,32,33), especially for sugary products. Such powder agglomerations reduce the exposure to oxygen, preventing the oxidation of the pigments.

Other research have reported a decrease in CPA with the increase of inlet air temperature, like lycopene and β -carotene in watermelon juice (23), lycopene in tomato (31,32,33), anthocyanins in carrots (decrease also associated with the degree of ED in maltodextrin)(36), and also greater loss of the pigments in *Amaranthus betacianina* (22). Temperatures above 180°C are not good for spray drying of betacyanins (31-33). For β -carotene, the behavior is similar, because carotenoids are heat-sensible and easily oxidized due to their unsaturations in chemical structure (37). On the other hand, the use of proteins, allows a stronger structure during microencapsulation, such phenomena was reported by Oliveira *et al.*, 2007 (29) when elaborating microcapsules by coacervation complexes using casein and pectin as wall material, followed by spray drying. The observed structure (without cracks and interruptions) revealed the strength of the solid film, which is a property of isolated soy protein, explaining the high levels of iron recovery, since it is a high molecular compound as bisglycinate iron.

The analysis of AB, AC, and AD interactions on solubility, identifies a slight increase in this variable from 96.7 to 98.06% with the increase of maltodextrin percentage; while the decrease in atomizing disc speed and the increase in outlet air temperature do not have a significant effect on the solubility, ranging between 97.1 a 97.8%.

The influence of the molecular weight distribution of maltodextrins with different ED has

been evaluated in order to encapsulate flavorings (38); the higher ED the greater protection against oxidative processes and the greater solubility of the product (25, 26). Such situation could be favoring the solubility of the cape gooseberry powder by increasing of maltodextrin content, which has an ED between 19 and 20.

The analysis of AB, AC, and AD interactions, on the hygroscopicity, showed that the water absorption by the powder added with CPA decreased between 25.0 and 12.8% with increasing of maltodextrin content; while the other factors did not present a significant influence. Research reported in nopal juice and betacyanin pigments (22, 25) presented a similar behavior, where the reduction of hygroscopicity was presented to higher contents of maltodextrin (values close to 20%). It is considered that inlet and outlet air temperatures did not influence on the hygroscopicity, due to the effective evaporation that allowed to obtain products with a moisture ranging between 2 and 4% and a_w between 0.235 and 0.652 (31, 32, 33).

Spray drying process optimization

Since the solubility did not present significant differences ($p>0.05$), it was not considered to establish objective function in the optimization process. Thus, the optimization process was defined by maximizing CPA levels and minimizing the hygroscopicity. Results showed that a product with the following conditions: hygroscopicity of $13.0\pm 0.1\%$, solubility of $97.4\pm 0.4\%$, and recovery percentages for iron of $90.8\pm 1.0\%$; for folic acid of $90.9\pm 1.8\%$, and for ascorbic acid of $69.7\pm 0.7\%$, can be obtained by operating the spray dryer with an inlet and outlet air temperature of **194.2°C and 87.7°C** respectively, and an atomizer disc speed of 19848 rpm.

CONCLUSIONS

The spray drying process is an effective technological alternative, that provides added value to cape gooseberry fruit, making it a good product for food industry, gastronomy, and homemade food; at the same time, allows its fortification with CPA (iron, folic acid, and ascorbic acid), keeping high amount of them in the final powder, mainly due to the short time of the process. The experimental optimization of spray drying process by statistical analysis is a useful tool for agro-industry, it allows to improve

the quality attributes of powder products, taking into account the independent variables. In general, the quality of cape gooseberry powder added with CPA is affected by the process conditions and by the content of maltodextrin in the feed suspension.

CONFLICT OF INTERESTS

The authors declare not to have conflict of interest with this publication

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AUTHORS' CONTRIBUTIONS

Conception and design of study: M. Cortés R.; acquisition of data: G. Hernández S.; analysis and/or interpretation of data: M. Cortés R., G. Hernández S. and E. M. Estrada M.

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