



# Goldenberry powder processing: analysis by a response surface methodology

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**ABSTRACT.** Goldenberry (*Physalis peruviana*) is a fruit of great interest for its nutritional properties and bioactive compounds, such as carotenoids. This study aimed to determine the ideal conditions for the development of a goldenberry powder. A Central Composite Design (CCD) was adopted to obtain response surfaces. For processing, different temperatures (50 - 70°C) and times (27.18 - 32.82 hours) were used for dehydration of the fruits, evaluating the content of total carotenoids as a response and the moisture content around 15%. Data obtained were tested by analysis of variance (ANOVA) and fitted to a second-order polynomial equation using multiple regression analysis. An optimization study was carried out and the desirability function methodology was applied to find the ideal process condition. The optimization was determined at 52°C and in a time of 27.18 hours, in which the experimental value obtained for total carotenoids was  $12656.5 \pm 527.22 \mu\text{g } 100 \text{ g}^{-1}$  and moisture content of  $15.00 \pm 0.26\%$ . In this condition, the global desirability value was 1.000 and the experimental values agreed with the predicted ones. Second-order polynomials were able to predict the carotenoids content in goldenberry powder, as well as the moisture content of the powder. The CCD and response surface tools were effective in optimizing the process. The production of goldenberry powder under these experimental conditions represents a viable alternative for adding value to the fruits, enabling the production of a potential food ingredient with carotenoid retention.

**Keywords:** cape gooseberry; fruit powder; carotenoids; optimization; desirability; central composite design.

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## Introduction

*Physalis peruviana* is a yellow-orange fruit with a juicy berry belonging to the family Solanacea and to the genus *Physalis*. The largest producer in the world is Colombia, followed by South Africa, and its cultivation has been expanding in tropical and subtropical countries, places where the plant has good adaptation (Bazana et al., 2019). Despite being recent in Brazil, the crop has been standing out in the southern region of the country, representing a production alternative with potential nutritional and economic value (Muniz et al., 2014).

Known as goldenberry or cape gooseberry, the nutritional value of *Physalis peruviana* and their byproducts makes them potential candidates for use by the cosmetic industry, in the production of functional foods and as components in phytomedicine (Nocetti, Núñez, Puente, Espinosa, & Romero, 2020).

Highly healthy and tasty, the fruit of *Physalis peruviana* has nutraceutical characteristics such as, anti-inflammatory (Dong et al., 2019); antimicrobial (El- Beltagi, Mohamed, Safwat, Gamal, & Megahed, 2019), and anticancer properties (Ballesteros-Vivas et al., 2019). Its main bioactive compounds are vitamins, physalins, carotenoids and flavonoids (Carrillo-Perdomo, Aller, Cruz-Quintana, Giampieri, & Alvarez-Suarez, 2015). Carotenoids are fat-soluble pigments responsible for the yellow, orange and red colors in many fruits and vegetables. They play an important role in the diet due to vitamin A activity and antioxidant function (Saini, Nile, & Park, 2015).

*Physalis peruviana* is a climacteric fruit with difficulties in retaining an acceptable level of quality attributes during storage. Temperature has a great effect on the shelf life of the fruit and the removal of the calyx stimulates an increase in moisture on the fruit surface, promoting the growth of fungi (Olivares-Tenorio, Dekker, van Boekel, & Verkerk, 2017).

Dehydration of fruits and vegetables is a widely used process, as it reduces the moisture content and water activity of these products, considerably increasing their shelf life. Drying also adds value to fruits and

vegetables and reduces storage and transportation costs. If fruits and vegetables are dried in the place where they are produced, food losses are reduced and this helps small farmers to earn a higher income (Roratto, Monteiro, Carciofi, & Laurindo, 2021).

In this aspect, the processing of fruits or byproducts into flours or powders enables the reduction of chemical and microbiological reactions through the removal of free water. Therefore, they become important ingredients for their food safety, while increasing the number of bioactive compounds, dietary fiber and minerals in foods (Soquetta et al., 2016). Some examples of fruit flours are reported in the literature, such as mutamba flour (*Guazuma ulmifolia*) (Assis et al., 2019); buriti fruit (*Mauritia flexuosa* L. f.) (Resende, Franca, & Oliveira, 2019); and breadfruit (*Artocarpus altilis*) (Huang, Roman, Martinez, & Bohrer, 2021).

Food processing influences the degradation of bioactive compounds, like carotenoids. Oxidation and isomerization of carotenoids can occur in various thermal and non-thermal processing methods. Convective and infrared drying at 80°C (Puente et al., 2021), as well convective drying at 60°C (Vega-Gálvez et al., 2015) and refractance window drying operated at 70°C (Puente et al., 2020) proved to be viable options for drying goldenberry pulp. However, the proper selection of technique and processing conditions must be evaluated along with technological investment, processing cost and product quality. Nevertheless, this kind of general consideration of the process (and optimization) is rarely reported in the literature (Ngamwonglumlert, Devahastin, Chiewchan, & Raghavan, 2020).

Since carotenoids have different biological characteristics, properties and chemical stability, further studies need to investigate the influence of processing and storage to optimize the conditions for goldenberry products to retain carotenoids and health promoting properties (Etzbach, Pfeiffer, Weber, & Schieber, 2018). In this context, the objective of this study was to determine the best condition of time and temperature during physalis dehydration, by evaluating the content of total carotenoids and moisture, using a Central Composite Design (CCD) and the response surface analysis tool.

## Material and methods

### Fruits

For this study, intact and ripe goldenberry (*Physalis peruviana* L.) fruit were used, cultivated in the municipality of São Francisco de Paula, state of Rio Grande do Sul, Brazil (Latitude: 29° 26' 52" S and Longitude: 50° 35' 02" W), from the 2020 growing season, harvested in March, April and May, and sold frozen by the company Itaberry Frutas Finas, Itá, state of Santa Catarina, Brazil. Fruit samples with a longitudinal diameter of  $15.7 \pm 0.21$  mm and a mass of  $2.822 \pm 1.097$  g (mean  $\pm$  standard deviation;  $n = 10$ ), were stored at temperatures between -10 and -15°C for 15 days, in polyethylene packages containing 1,000 g goldenberry in each, transported to the laboratory in polystyrene boxes, containing reusable ice. In the laboratory, they were kept frozen, until analysis.

### Production of goldenberry powder

For the development of the goldenberry powder, fruit were thawed under refrigeration for 24 hours. Portions of approximately 500 g were weighed and dehydrated in an oven with air circulation (SSD – CR 110L, Eurobrás, state of São Paulo, Brazil), according to the time and temperature established by the experimental planning described below. The objective was to optimize the best combination of time and temperature, ensuring the greatest preservation of carotenoids and a moisture content around of 15% (value established by technological interest). After drying, whole fruit (including the seeds) were ground in a Wiley knife mill (Star FT 50, Fortinox, state of São Paulo, Brazil) with a 10 mesh sieve. The fruit powders produced, according to each treatment, were packed in polyethylene containers hermetically sealed, protected from light, and kept refrigerated.

### Experimental design for the production of goldenberry powder

For the production of goldenberry powder, a CCD with two independent variables was used. Time and temperature were evaluated using a  $2^2$  factorial design, with a triplicate at central point and four axial points, for the calculation of effects and response surface analysis. Dependent variables (responses) were the content of total carotenoids and the moisture content of goldenberry powders. The levels of each variable were set from preliminary fruit dehydration tests and are listed in Table 1.

**Table 1.** Values used in the CCD for the production of goldenberry powder.

Test	Coded levels		Uncoded levels	
	$x_1$	$x_2$	Temperature (°C)	Time (hour)
1	-1	-1	53	28
2	1	-1	67	28
3	-1	1	53	32
4	1	1	67	32
5	-1.41	0	50	30
6	1.41	0	70	30
7	0	-1.41	60	27.18
8	0	1.41	60	32.82
9 to 11	0	0	60	30

Preliminary tests for drying the fruit were carried out using three different temperatures (50, 60 and 70°C), starting from a minimum established time of one day (24 hours) and then increasing to 28 hours. The maximum temperature of 70°C was determined due to the stability of carotenoids (Ngamwonglumlert & Devahastin, 2019). After dehydration, moisture was determined in triplicate.

### Determination of moisture content and total carotenoids of goldenberry powders

The moisture content of each fruit powder produced was evaluated immediately after preparation, using method 44-15.02 of the American Association of Cereal Chemistry International (AACC, 2010) and expressed in % moisture ( $\text{m m}^{-1}$ ). Total carotenoid content was determined as described by Rodriguez-Amaya (1999). Extraction was carried out with acetone and separation in petroleum ether. Absorbance was read at 450 nm in a spectrophotometer (UV – 9200, Rayleigh, Beijing, China). The calculation was performed using the absorption coefficient of  $\beta$ -carotene in petroleum ether (2,592) and the results expressed as all-trans- $\beta$ -carotene (the predominant carotenoid in *Physalis peruviana*). The result was multiplied by 100 and expressed in  $\mu\text{g } 100 \text{ g}^{-1}$ . Analyses were performed in triplicate.

### Statistical analysis

The performance of processes for the production of fruit powders was evaluated by analyzing the responses (Y), which depend on the input factors  $x_1$ ,  $x_2$ ,  $x_k$  and the relationship between the response and the process parameters, being described by Equation 1.

$$Y = f(x_1, x_2 \dots x_k) + e \quad (1)$$

where:

'f' is the actual response function, whose format is unknown and 'e' is the error describing the differentiation. The response surface behavior was investigated for the response function ( $Y_i$ ) using a second order polynomial equation, whose generalized response surface model is described in Equation 2.

$$Y = \beta_0 + \sum_{j=1}^k \beta_j x_j + \sum_{j=1}^k \beta_{jj} x_j^2 + \sum_i \sum_{j=2}^k \beta_{ij} x_i x_j + e_i \quad (2)$$

where:

Y is the answer;  $x_i$  and  $x_j$  are variables (i and j range from 1 to k);  $\beta_0$  is the model's intercept coefficient;  $\beta_i$ ,  $\beta_{jj}$  and  $\beta_{ij}$  are linear, quadratic and second order interaction coefficients, respectively; k is the number of independent parameters (k = 2 in this study); and  $e_i$  is the error (Sridhar, Sivakumar, Prince, & Maran, 2012).

Calculations required to determine the model, comparisons of experimental results with predicted values, and generation of response surfaces, were performed in Statistica 12 software (Statsoft Inc., USA). Analyses of variance were performed with an  $\alpha$  level of 0.05 to determine statistical significance.

### Verification of optimized conditions and predictive model for the development of goldenberry powder

The ideal conditions of time and temperature for a better preservation of carotenoids in the production of goldenberry powder were obtained through response surface methodology. The total carotenoid content was determined after production of the powder under optimized conditions. The experimental and predicted values were also compared to determine the validity of the model.

### Physical and chemical analysis

Some physical and chemical parameters were evaluated in the goldenberry powder after production under optimized conditions, including pH, water activity and titratable acidity. The pH values were measured using the potentiometric method (Association of Official Analytical Chemists [AOAC], 2006) using a calibrated digital pH meter (MA 522, Marconi, Piracicaba/state of São Paulo, Brazil). To determine the water activity (Aw), the LabTouch - LabMasteraw equipment (Novasina, Lachen, Switzerland) was used with direct reading of the samples. Titratable acidity was measured using the reference described by AOAC (2006), and the results were expressed in percentage of citric acid. Analyses were performed in triplicate

## Results and discussion

### Preliminary results of moisture content of dehydrated fruits

Table 2 lists the results of moisture content (%) of the fruit after dehydration. From these values, the temperatures 53, 60 and 67°C to be used in the CCD were determined, and 28 hours was used as minimum time, as it presented moisture content results around 15%.

**Table 2.** Results of moisture content (mean  $\pm$  standard deviation) of dehydrated fruits.

Test	Temperature (°C)	Time (hour)	Moisture (%)
1	50	24	19.09 $\pm$ 0.47
2	60	24	15.32 $\pm$ 0.27
3	70	24	14.25 $\pm$ 0.18
4	50	28	17.75 $\pm$ 0.69
5	60	28	13.44 $\pm$ 0.29
6	70	28	11.68 $\pm$ 0.30

### Normality tests

Considering that the main statistical tests used for analysis of clinical and experimental data are based on theoretical models that assume normal distribution of data, the results obtained were tested for normality. Thus, the Shapiro-Wilk test was applied and all data were presented in a normal distribution curve.

For the results of total carotenoids and moisture of the goldenberry powder samples, the p-values were 0.0604 and 0.1391, respectively. Thus, it was possible to confirm the normal distribution of data, since  $p > 0.05$ .

### Construction of second-order models and statistical analysis

The relationship between experimental results obtained based on the CCD and the input variables were expressed by a second-order polynomial equation with interaction terms. The final equations obtained in terms of coded factors are presented by Equation 3 e 4.

$$\text{Totalcarotenoids} \left( \frac{g}{100g} \text{ goldenberrypowder} \right) = 11276.26 - 499.92x_1 + 106.77x_1^2 - 179.12x_2 + 28.73x_2^2 + 207.21x_1x_2 \quad (3)$$

$$\text{Moisture}(\% \text{ goldenberrypowder}) = 11.96 - 2.08x_1 - 0.07x_1^2 - 0.47x_2 + 0.36x_2^2 + 0.07x_1x_2 \quad (4)$$

The adequacy and fit of the model were tested by analysis of variance (ANOVA) and regression analysis. A complete analysis of variance, which takes into account the lack of fit of the model and the pure error, requires that the number of different tests be greater than the number of model parameters and that such tests be performed in replicate, as conducted in this study. The ANOVA results (Table 3) indicated that the equation adequately represented the real value of the relationship between the independent variables and the responses.

Values of p were lower than 0.05, indicating that the terms are statistically significant, with the exception of time ( $x_2$ ) in the quadratic model in relation to the content of total carotenoids and temperature ( $x_1$ ) in the quadratic model, as well as the interaction between the factors ( $x_1x_2$ ) in relation to the moisture content of the fruit powders. These factors had no significant influence on the response variables, but they are part of the model. High values of F in the models indicate that a large part of the variation in the responses can be explained by the regression equation. From the p-values of the model, it can be seen that these were highly significant from a statistical point of view ( $p < 0.05$ ).

**Table 3.** ANOVA and statistical parameters of the model for the production of goldenberry powder.

Factor	Coefficient	SS	DF	MS	F	p
Carotenoid in goldenberry powder						
(x <sub>1</sub> ) Temperature (L)	-499,92	6059237.1	1	6059237.10	554.94	0.000000
Temperature (Q)	106,77	199269.05	1	199269.05	18.25	0.000264
(x <sub>2</sub> ) Time (L)	-179.12	767695.49	1	767695.49	70.31	0.000000
Time (Q)	28.73	13819.60	1	13819.60	1.27	0.271705
x <sub>1</sub> x <sub>2</sub>	207.21	515211.09	1	515211.09	47.19	0.000000
Lack of fit		412364.39	3	137454.80	12.59	0.000038
Pure Error		262049.38	24	10918.72		
Total SS		8216079.28	32			
Model	11276.26					
Moisture of goldenberry powder						
(x <sub>1</sub> ) Temperature (L)	-2.084	105.33	1	105.33	1000.59	0.000000
Temperature (Q)	-0.075	0.10	1	0.10	0.93	0.343793
(x <sub>2</sub> ) Time (L)	-0.466	5.19	1	5.19	49.33	0.000000
Time (Q)	0.357	2.14	1	2.14	20.31	0.000146
x <sub>1</sub> x <sub>2</sub>	0.065	0.05	1	0.05	0.48	0.494348
Lack of Fit		5.97	3	1.99	18.91	0.000002
Pure Error		2.53	24	0.11		
Total SS		121.82	32			
Model	11.96					

The verification of the model fit through the coefficient of determination ( $R^2$ ) showed a high degree of correlation between the response and the independent variables (experimental and predicted values). The  $R^2$  values for total carotenoid content and moisture content of goldenberry powders were 0.9179 and 0.9302, respectively, indicating that the model is a good fit for the data. Despite high coefficients of determination and significant effects (high values of F for the assessed confidence level), the analysis of variance showed, also by the F-test, that the lack of fit was significant in both ( $p < 0.05$ ). This was observed even considering a model with all terms evaluated (linear and quadratic). Other complex models were not considered, as they have a higher number of parameters than the number of tests performed at different levels, which would make analysis of variance impossible.

### Experimental design for the production of goldenberry powders

Experiments were carried out according to the design in order to find the best conditions and study the effect of process variables on the development of goldenberry powder. The predicted values were obtained by a Statistica software model fit technique and were shown to be correlated with the observed values.

Table 4 lists the results observed experimentally and the predicted values for the content of total carotenoids and moisture content of goldenberry powders. It is important to confirm the fit of the model to make sure it provides a sufficient approximation to the real values. The results suggest that the models used here were able to identify operational conditions for the development of a goldenberry powder, with optimization of the content of total carotenoids and moisture content.

Goldenberry powders showed total carotenoid values ranging from  $10,670.57 \pm 10.21$  to  $12,372.98 \pm 163.73$   $\mu\text{g } 100 \text{ g}^{-1}$ , indicating the potential to become an ingredient with bioactivity. Regarding the moisture content, all experiments showed values around 15%.

Fruit used for the production of powders exhibited a total carotenoid value of  $2,966.28 \pm 116.13$   $\mu\text{g } 100 \text{ g}^{-1}$  fresh fruit. The results obtained in the literature for total carotenoids in *P. peruviana* show a wide range of variation, with values from 1,240 to 25,500  $\mu\text{g } 100 \text{ g}^{-1}$  (Luchese, Gurak, & Marczak, 2015; Pereda, Nazareno, & Viturro, 2019). The high variation in the content of carotenoids in fruit is influenced by their variety, maturity, growing conditions and season of the year in which they are produced. In addition, there is great variability in the results according to the analysis method used (Saini et al., 2015).

According to Rodriguez-Amaya, Kimura, and Amaya-Farfan (2008), foods that contain more than 20  $\mu\text{g } \text{g}^{-1}$  carotenoids are rich sources of this pigment. To obtain products with high concentrations of carotenoids, the ripening stage of the fruits must be considered. Due to degradation, the nutritional value in terms of carotenoid content of goldenberry fruits is affected during ripening. In a study, ripe and very ripe *P. peruviana* fruit presented values of 200 and 140  $\mu\text{g } \text{g}^{-1}$  (dry basis), respectively, for total carotenoids (Etzbach et al., 2018).



**Table 4.** Matrix of the experimental design with the respective experimental values mean  $\pm$  standard deviation) and the predicted values of total carotenoids and moisture in goldenberry powders.

Test	Temperature	Time	Total carotenoid ( $\mu\text{g } 100 \text{ g}^{-1}$ )			Moisture (%)		
	( $^{\circ}\text{C}$ )	(hour)	Experimental*	Predicted	Error (%)	Experimental*	Predicted	Error (%)
1	53	28	12,088.93 $\pm$ 21.39	12,297.99	-1.7	14.9 $\pm$ 0.32	14.86	0.28
2	67	28	10,870.46 $\pm$ 64.86	10,883.75	-0.12	10.98 $\pm$ 0.16	10.56	4.01
3	53	32	11,482.25 $\pm$ 74.98	11,525.35	-0.37	13.00 $\pm$ 0.63	13.8	-5.8
4	67	32	11,092.60 $\pm$ 36.67	10,939.93	1.39	9.34 $\pm$ 0.25	9.76	-4.28
5	50	30	12,372.98 $\pm$ 163.73	12,208.31	1.35	15.24 $\pm$ 0.37	14.79	3.05
6	70	30	10,670.57 $\pm$ 10.21	10,779.98	-1.01	8.75 $\pm$ 0.10	8.83	-0.94
7	60	27.18	11,732.00 $\pm$ 48.57	11,585.93	1.26	12.92 $\pm$ 0.18	13.33	-3.06
8	60	32.82	10,991.48 $\pm$ 99.96	11,080.82	-0.81	12.80 $\pm$ 0.21	12.01	6.54
9	60	30	11,226.65 $\pm$ 167.74	11,276.26	-0.44	11.60 $\pm$ 0.32	11.96	-3.01
10	60	30	11,243.43 $\pm$ 186.51	11,276.26	-0.29	12.05 $\pm$ 0.07	11.96	0.77
11	60	30	11,359.48 $\pm$ 38.93	11,276.26	0.74	12.23 $\pm$ 0.15	11.96	2.28

\*Standard deviation refers to the replicate of analyses. Analyses were performed in triplicate.

In comparison with goldenberry juice (4134  $\mu\text{g } 100 \text{ g}^{-1}$  of juice), a study by Etzbach et al. (2020) showed that there was a significant reduction (38.9 - 69.7%) in the total content of carotenoids after the fruits were sprayed through a spray dryer, depending on the carrier agent used. In the case of the production of goldenberry powder, there was a concentration of total carotenoids, resulting a higher content of these compounds.

### Effects of process variables on the content of total carotenoids and moisture in goldenberry powders

The regression coefficients and analysis of variance of the variables of the mathematical models are described in Table 5. The p values show that there was an influence of the variables (temperature and time) on the content of total carotenoids ( $p < 0.05$ ), both in isolation and between the interaction of both, with the exception of the 'time' factor in the quadratic model. This can also be seen in the response surface graph (Figure 1), which did not show curvature, evidencing the non-dependence of this model.

Regarding the moisture content of goldenberry powders, only time showed statistical significance ( $p < 0.05$ ), in both models (linear and quadratic). The temperature did not exert a significant influence on the powder moisture nor the interactive effect between the two factors.

The 3D plots of response surfaces (Figure 1) were used to represent the effect of process variables on the content of total carotenoids (a) and moisture content of the powders (b). The response surface method aims to find the ideal response and understand how the response changes in a given direction by adjusting the design variables (Núñez-Gómez, Lapolli, Nagel-Hassemer, & Lobo-Recio, 2017).

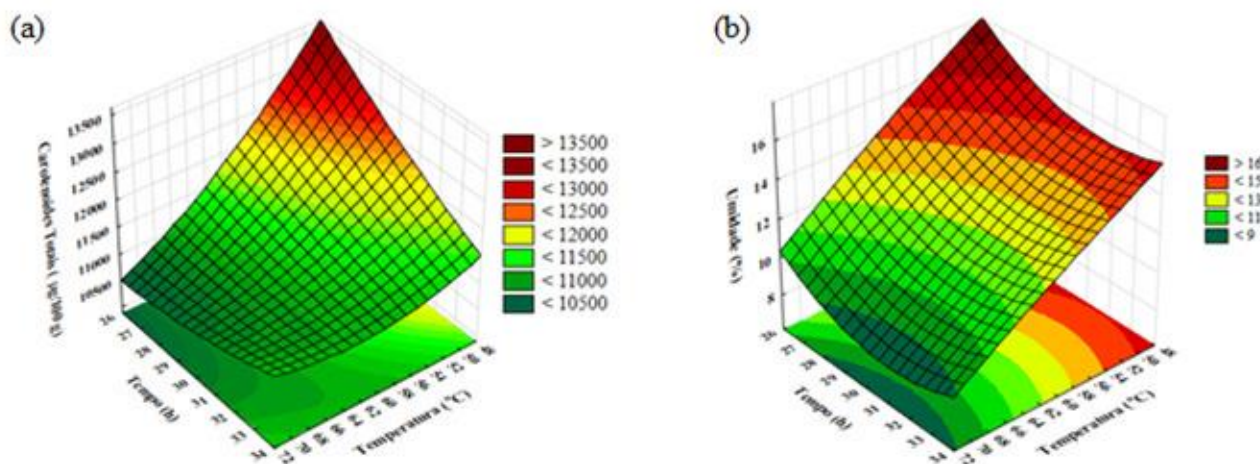
The stability of carotenoids is affected by temperature and exposure time, degrading with the increase in these two factors, as can be seen in the graph of response surface of the carotenoid content of goldenberry powders. At temperatures above 64 $^{\circ}\text{C}$ , there was a gradual decrease in carotenoid content, even with narrow ranges of variation in the drying process time.

This degradation is mainly caused by oxidation and isomerization reactions. Most carotenoids in plants are trans isomers and trans to cis isomerization occurs during food processing. Heat and light, as well as acids, are the main factors that can promote this isomerization, which leads to slightly reduced biological activities and color saturation. Oxidation, on the other hand, leads to complete loss of activities and color of carotenoids (Ngamwonglumlert, Devahastin, & Chiewchan, 2017).

The optimal condition was a time from 27 to 28 hours, with temperatures between 50 and 52 $^{\circ}\text{C}$ , where the moisture content was also around 15%.

**Table 5.** Regression coefficients (RC) and p-values of the regression model for the content of total carotenoids and moisture of goldenberry powders.

Factor	Carotenoids in goldenberry powders		Moisture of goldenberry powders	
	RC	p-value	RC	p-value
Mean/Interc. ( $\beta_0$ )	59197.41	0.000000	120.07	0.000032
( $x_1$ ) Temperature (L)	-776.9	0.000000	-0.2535	0.368436
Temperature (Q)	2.18	0.000264	-0.0015	0.343793
( $x_2$ ) Time (L)	-1408.56	0.001923	-5.8722	0.000095
Time (Q)	7.18	0.271705	0.0893	0.0001468
$x_1.x_2$	14.80	0.000000	0.0046	0.494348



**Figure 1.** (a) Graph of response surface for the content of total carotenoids ( $\mu\text{g } 100 \text{ g}^{-1}$ ) of the goldenberry powders according to the different conditions of temperature ( $^{\circ}\text{C}$ ) and time (hour). Total carotenoids =  $59,197.41 - 776.90 x_1 + 2.18 x_1^2 - 1,408.56 x_2 + 7.18 x_2^2 + 14.80 x_1 x_2$ ;  $R^2 = 0.9179$ ;  $x_1$  = uncoded variable for temperature and  $x_2$  = time. (b) Graph of response surface for the moisture content (%) of goldenberry powders according to the different conditions of temperature ( $^{\circ}\text{C}$ ) and time (hour). Moisture =  $120.0756 - 0.2535 x_1 - 0.0015 x_1^2 - 5.8722 x_2 + 0.0893 x_2^2 + 0.0046 x_1 x_2$ ;  $R^2 = 0.9302$ ;  $x_1$  = uncoded variable for temperature and  $x_2$  = time.

During a heating process, Olivares-Tenorio et al. (2017) evaluated the behavior of  $\beta$ -carotene in *P. peruviana*. At a temperature of  $40^{\circ}\text{C}$ , there was no change in its content. Between  $60$  and  $80^{\circ}\text{C}$ , there was a slight apparent increase with also a suggestion of isomerization from trans to cis form. At temperatures between  $100$  and  $120^{\circ}\text{C}$ , however, there were clear signs of isomerization, leading to an equilibrium between the two forms. The apparent increase in  $\beta$ -carotene is probably related to the release from the food matrix after heat treatment. At  $100^{\circ}\text{C}$ , ascorbic acid,  $\beta$ -carotene, catechin and epicatechin were still present after heat treatment, as well as antioxidant properties. Thus, it becomes evident that goldenberry has bioactive compounds that are not fully degraded after heating, and may be a potential source of such compounds.

### Determination of optimal conditions

Ideal conditions of time and temperature were set to reach the highest proportion of carotenoids and a moisture content around 15% for the production of goldenberry powder. For this, the desirability function was used, where first, each response ( $Y_i$ ) is converted into an individual desirability function ( $d_i$ ), which varies in a range of  $0 \leq d_i \leq 1$ . Then, the individual needs are combined to obtain the global desirability function ( $D$ ) and, finally, one should maximize the  $D$  and identify the optimal configurations of the factors. The function varies between 0 (completely undesirable response) and 1 (completely desirable response) (Amami et al., 2017).

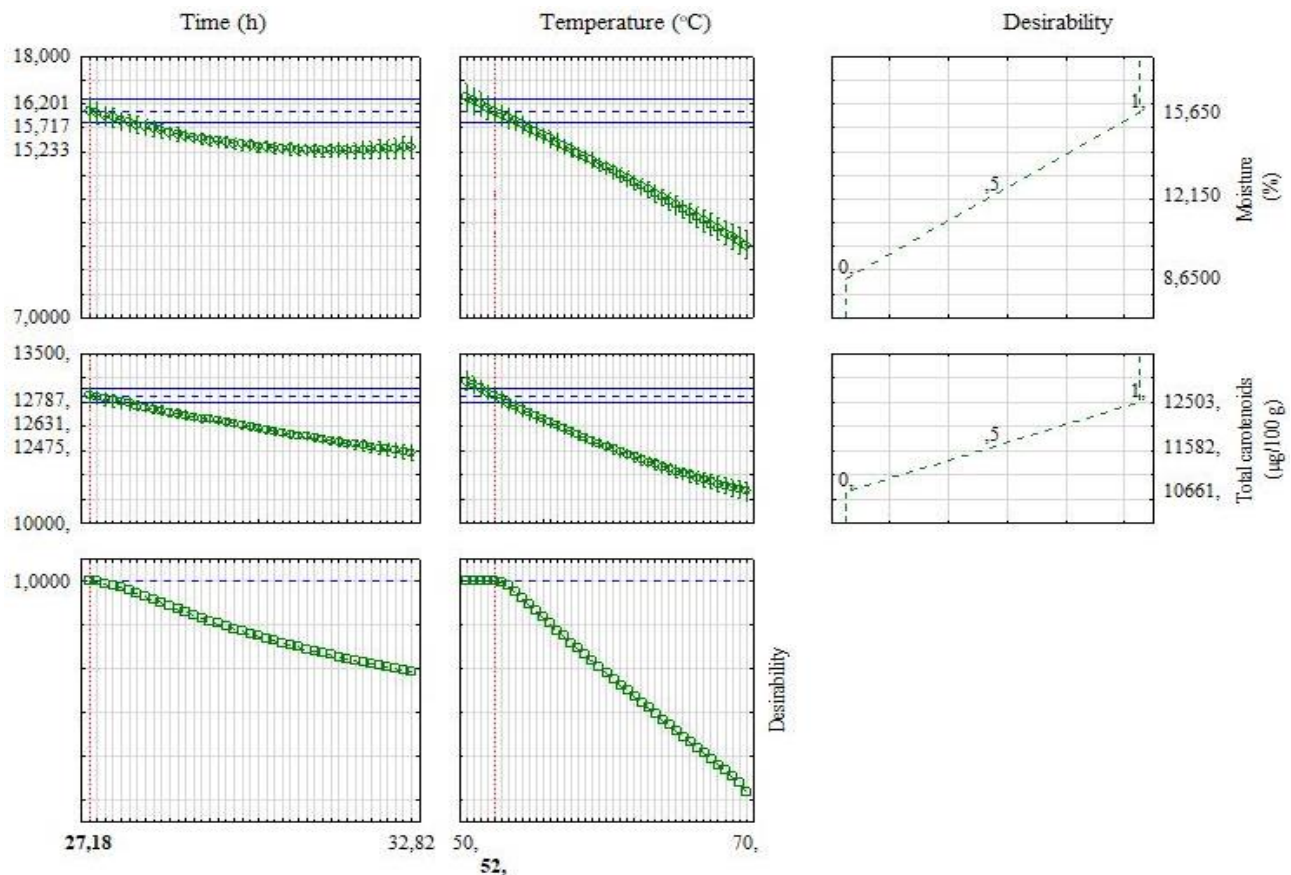
To optimize the development of goldenberry powder, the following parameters were used: (1) temperature ( $50 - 70^{\circ}\text{C}$ ) and (2) time ( $27.18 - 32.82$  hours), respectively, for greater efficiency in carotenoid content with moisture content around 15%. Applying the methodology of the desirability function, the optimal level was set at a temperature of  $52^{\circ}\text{C}$  and for a time of 27.18 hours, theoretically obtaining from these conditions, a total carotenoid content of  $12,630.61 \mu\text{g } 100 \text{ g}^{-1}$  and 15.72% moisture. For this optimization, the global desirability value was 1.0000. Figure 2 illustrates curves developed from optimal points via numerical optimization.

### Verification of optimized conditions and validation of the predictive model

The adequacy of the model equations to predict the best response values in the development of goldenberry powder was tested under the following conditions: temperature of  $52^{\circ}\text{C}$  and time of 27.18 hours. This condition was determined as optimal by response surface analysis and used to confirm the validity of the optimized process.

The experiment was carried out to compare the results with the predicted values of the responses using the model equations (Table 6). The experimental value obtained for the content of total carotenoids is in accordance with the 95% confidence interval range of the predicted values (Table 6).

The experimental result of the goldenberry powder moisture content was slightly lower than the range stipulated by the confidence interval, however, it is adequate for the powder fruit. Despite this small variation, it is possible to verify the adequacy of the system developed by the model.



**Figure 2.** Desirability function for optimization of the CCD for the development of goldenberry powder.

**Table 6.** Predicted and experimental values of responses under ideal conditions.

Optimum levels of process parameters	Optimized value <sup>1</sup> (predicted value)	Experimental value <sup>2</sup>
goldenberry powder	Total carotenoids ( $\mu\text{g } 100 \text{ g}^{-1}$ )	Total carotenoids ( $\mu\text{g } 100 \text{ g}^{-1}$ )
Temperature ( $^{\circ}\text{C}$ ) = 52	12,630.61 (12,474.69 – 12,786.53) <sup>3</sup>	12,656.5 $\pm$ 527.52
Time (hour) = 27.18		
	Moisture (%)	Moisture (%)
Temperature ( $^{\circ}\text{C}$ ) = 52	15.72 (15.23 – 16.20) <sup>3</sup>	15.00 $\pm$ 0.26
Time (hour) = 27.18		

<sup>1</sup>Value predicted by the response surface model; <sup>2</sup>Mean  $\pm$  standard deviation of triplicate determinations of experiments; <sup>3</sup>Confidence interval.

### Physical and chemical analysis

Table 7 lists the results of physical and chemical parameters evaluated in goldenberry powder. The pH value was below 4.5, which favors its preservation, since below this limit, the growth of microorganisms, mainly bacteria, is restricted, as it is characterized as acidic. Likewise, the  $A_w$  result was below 0.600, which is the lowest  $A_w$  at which the growth of microorganisms in food occurs (Sagrin & Chong, 2013). The titratable acidity is related to the concentration of organic acids present in the fruit. The main organic acids in goldenberry fruit are citric, malic and oxalic (Galvis, Fischer, & Gordillo, 2005).

**Table 7.** Results (mean  $\pm$  standard deviation) of the physical and chemical parameters in goldenberry powder.

Physical and chemical parameters	Goldenberry powder
pH	3.65 $\pm$ 0.14
$A_w$	0.320 $\pm$ 0.002
Titratable acidity (% citric acid)	3.65 $\pm$ 0.14

### Conclusion

The CCD and the response surface analysis proved to be effective in determining the ideal condition for the production of goldenberry powder. The results showed that the conditions of the drying process had a significant influence on the content of total carotenoids and moisture in goldenberry powders.



The values ensure a satisfactory fit of the regression model to the experimental data. Despite a significant lack of fit for both models (F test), it was possible to observe through response surfaces a combination of parameters to obtain the ingredient with high content of total carotenoids and desired moisture content.

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