

## Spray drying of liquid extracts of curcumin: process performance and product quality properties

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## Secado por aspersión de extractos líquidos de cúrcuma: Evaluación del proceso y propiedades de calidad del producto

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### Abstract

The aim of this work was to evaluate the operation of spray drying to obtain powder curcumin from turmeric extract blended with maltodextrin. An experimental design (central composite design) with two statistical factors was used. These factors were the inlet air temperature (140-160°C) and outlet air temperature (75-95°C), with the rotation speed kept constant (28000 RPM). Statistical optimization was established by considering the response surfaces analysis where the hygroscopicity was minimized and the curcumin concentration was maximized. The optimal conditions for the spray drying process were inlet and outlet air temperatures of 149°C and 75°C, respectively, at an atomization speed of 28000 RPM. These settings provided the following results: hygroscopicity ( $11.71\% \pm 0.03$ ) and final concentration of curcumin ( $9.03 \pm 0.44$  mg/g), which were statistically significant.

**Keywords:** *Chromatography, Curcuma longa, spray drying, powder.*

### Resumen

El objetivo del trabajo fue evaluar las condiciones del proceso de secado por aspersión para obtener curcumina en polvo a partir de extracto de cúrcuma mezclado con maltodextrina. Se empleó un diseño central compuesto que consideró la influencia de dos factores: temperatura del aire de entrada (140 - 160°C) y temperatura del aire de salida del secador por aspersión (75-95°C), y la velocidad de atomización fue constante en 28000 RPM. El proceso de secado se optimizó mediante superficies de respuesta minimizando la higroscopicidad y maximizando la concentración de curcumina en el polvo, ya que las demás propiedades de calidad no fueron estadísticamente significativas. Los resultados mostraron que las condiciones óptimas para el proceso de secado por aspersión de curcumina fueron: temperatura del aire a la entrada de 149°C y 75°C a la salida, para las cuales se obtiene en el producto final: higroscopicidad ( $11,71\% \pm 0,03$ ) y concentración ( $9,03 \pm 0,44$  mg/g), que fueron significativas.

**Palabras clave:** *Cromatografía, Curcuma longa, secado por aspersión, polvo.*

## 1. Introduction

Curcumin, or the active ingredient of turmeric, is a polyphenol derived from the rhizome of the *Curcuma longa* plant. It is widely used as a natural food coloring agent for achieving an orange-yellow color. Additionally, it possesses antioxidant, anti-inflammatory, antimicrobial, and anticancer properties and rapidly degrades in neutral or alkaline pH conditions with light and temperature exposure. Turmeric is used in between 3 and 5% of coloring material in the form of curcumin crystals (curcuminoids) (Gomez et al., 2012). Turmeric is very important for the food industry because it retains freshness through food processing and provides a distinctive flavor. Curcumin is used as a natural food coloring for ice cream, sauces, soups, confectionery, desserts, cheeses, precooked foods, beverages, condiments and others (Ríos et al., 2009).

Spray drying is a commonly used technique for acquiring powder from plant extracts. In this process, the product to be dried (emulsion, suspension or solution) is converted into a dry solid product (Shaikh et al., 2006; Gharsallaoui et al., 2007). This drying process protects the compound oxidation and volatilization processes, and it is highly recommended for products that have high thermolability (Shaikh et al., 2006).

Importantly, very few literature reports have evaluated the performance variables of the spray drying process and product quality. Considering the importance of industry-wide development and application of natural dyes in the food industry, the objective of this research was to evaluate the effect of the spray drying conditions on the final product quality (powdered dye) and process performance variables for turmeric extracts using liquid systems formulated with high molecular weights (maltodextrin).

## 2. Materials and methods

The formulation of the suspension, drying process and characterization of the powder dye were performed at the facilities of the Tecnas SA and

the Universidad Nacional de Colombia-Sede Medellín. The raw materials used for suspension were turmeric extract (in organic solvent), glycerin, maltodextrin and water. The suspension that underwent spray drying consisted of a mixture of maltodextrin (DE 10) and a concentrated water extract of turmeric. The concentrate was obtained from a mixture of glycerine and organic solvent extract, and this mixture was subjected to the rotary evaporation process (Heidolph2 rotary evaporator) at a temperature of 50°C in a glycerin bath and speed of 90 RPM. This concentrate was stored at 4°C and protected from light. After obtaining the concentrated curcumin, the sample was subjected to the drying process. The drying suspension consisted of a mixture of concentrated curcumin (13.86%), maltodextrin (28.14%) and water (58%), which were mixed using a homogenizer (Ultra-Turrax IKA-T25) at 7000 RPM for 1 minute. The following variables were determined for the dryer feed suspension:

**Viscosity:** Viscosity was measured using a Brookfield Rheometer DV-III Ultra at 25°C while considering a maximum cutting speed of 200 RPM. It was determined by analyzing the rheograms or product flow curves (Frascareli et al., 2012; Tonon et al., 2011).

**pH Measurement:** The pH was determined according to the NTC 4092 standard given by ICONTEC (2009) using a potentiometer and immersing the electrode in a sample (Hanna automatic titrator pH 211) that was previously calibrated with buffer solutions (pH 4 and 7) at 25°C.

**Color:** The methodology system for uniform color space CIE L\*a\*b\* and h° (hue angle) was used. The measurement was performed using a sphere spectrophotometer X-Rite D65 illuminant at a 10° observation angle. Before reading, calibration was achieved with black and white (CIE, 2004; HunterLab, 2008).

**Quantification of curcumin:** The curcumin concentration in the concentrated product and the powder (dye) was evaluated using the liquid

chromatography technique (HPLC: High performance liquid chromatography). The mobile phase consisted of water/phosphoric acid (1%) at a flow of 0.8 ml/min and injection volume of 10 microliters of acid. Standard curcumin (33 mg) was diluted in 0.5 mL of distilled water and then 10 mL of methanol. It was stirred using vortexing and ultrasonication; finally, it was centrifuged at 2800 rpm for 5 min. Quantification was achieved using a standard calibration solution of 100 ppm curcumin in 10 mL of methanol chromatograph AGILENT (Paramera et al., 2011).

Drying was performed in a spray dryer (VIBRASEC S.A) with a water evaporation capacity of 1.5 L h<sup>-1</sup>. The control variables were the drying air temperature at the inlet and the drying air and temperature at the outlet; the atomization velocity was kept constant. These operating conditions were independently controlled according to automated programming equipment. The drier operating parameters correspond to a drying air temperature at the inlet of 140-160°C, outlet between 75-95°C and speed of the atomizer disk of 28000 rpm. A central composite design with 11 treatments and three repetitions at the central point (inlet air temperature: 85°C and outlet air temperature: 150 °C) was defined. The following variables were measured to evaluate both the overall performance of the drying process and quality conditions of the resulting dried product (powder dye):

Process yield. The amount of product obtained with drying divided by all solids in the suspension according to weight (Tonon et al., 2008):

$$\text{Process yield} = \frac{\text{Total final solids}}{\text{Total initial solids}} * 100 \quad (1)$$

Percentage of material adhering to the dryer surface. This value was calculated based on the difference between the input and output materials according to the amount of dust that adhered in the

dryer. In this case, the loss of fines (negligible) was considered by the cyclone (Tonon et al., 2008).

Water activity of the dry product (*A<sub>w</sub>*). A 3-g sample was collected using a hygrometer with a dew point at 25°C (AquaLab -3TE, Decagon, Devices, Pullman, WA, USA), and the water activity was measured (Cortes et al., 2007).

Moisture content. This was measured according to the AOAC 925.45 / 97 methodology (Ríos et al., 2009).

Solubility. The solubility was calculated according to a modified version of the Eastman & Moore (2005) method. It was based on 1 g of dry sample powder homogenized in 50 ml distilled water and vortexed for 30 seconds. The solution was placed in a tube, and the suspension was centrifuged at 3000 RPM for 5 minutes at room temperature (25°C). A 25-ml aliquot of the supernatant was collected, transferred to previously weighed Petri dishes and dried in an oven at 105°C for 5 hours. The solubility percent was calculated as the difference between the initial and final weights divided by the initial weight (Cano-Chauca., 2005; Li et al., 2013).

Hygroscopicity. This value was determined using the gravimetric method. The samples (5 g) were placed inside an airtight container that had an atmosphere of constant relative humidity, which was controlled by the presence of a supersaturated solution of KI (65% at 20°C). To check the status of the sample-atmosphere equilibrium, the sample weights were determined until they reached a constant weight ( $\Delta\text{weight} = \pm 0.001 \text{ g/g sample}$ ). The hygroscopicity was expressed in terms of the % moisture (wet basis) (Arazola et al., 2014).

Color measurement. This measurement was performed according to the methodology described above (CIE L\*a\* b\* and h°). The powdered product and 0.1% and 0.01% product solutions were evaluated.

Quantification of curcumin in the powder. HPLC was performed according to the methodology previously cited for concentrated curcumin.

Morphological characterization. The microstructure of the dried product was evaluated using scanning electron microscopy (SEM). The powder was placed on the SEM slide using double-sided adhesive tape (Nisshin EM, Tokyo, Japan) at an acceleration voltage of 20 kV after the sample was coated with Pt-Pd atomized by a sputter coater magnetron MSP-1S (vacuum device, Tokyo, Japan) (Soottitantawat et al., 2005).

The central composite design was optimized according to the response surface to operating conditions of the spray dryer with two factors. These factors were the air temperature at the inlet (140-160°C) and outlet (75-95°C) while keeping the atomizer disk speed at 28000 rpm. The process was optimized under the following conditions: hygroscopicity minimization and maximization of the curcumin concentration. There were no statistically significant findings ( $p > 0.05$ ) for any of the other variables, such as the moisture content, water activity of the final product, percentage of material adhering to the dryer surface, curcumin color, solubility and drying performance. The following generalized second-order polynomial model was used to analyze the response surface (Bezerra et al., 2008):

$$Y = \beta_0 + \beta_A A + \beta_B B + \beta_{A^2} A^2 + \beta_{B^2} B^2 + \beta_{AB} AB \quad (2)$$

where  $\beta_0$  is a constant;  $\beta_A$ ,  $\beta_B$ ,  $\beta_C$  and  $\beta_D$  are the coefficients of each factor;  $\beta_{A^2}$ ,  $\beta_{B^2}$ ,  $\beta_{C^2}$  and  $\beta_{D^2}$  are the coefficients of double interactions for each factor; and  $\beta_{AB}$ ,  $\beta_{AC}$ ,  $\beta_{AD}$ ,  $\beta_{BC}$ ,  $\beta_{BD}$ , and  $\beta_{CD}$  are the coefficient products of the factor interactions.

The experimental design results were analyzed using analysis of variance ( $p = 0.05$ ), the lack of fit test, determination of regression coefficients and response surfaces with Design Expert 8.0 software.

### 3. Results and discussion

The physicochemical parameters were evaluated for the concentrated extract of curcumin formulated

with maltodextrin, a matrix that was considered the drying carrier (Table 1). Rheograms demonstrate a viscosity value of  $109.4 \pm 1.68$  mPa-s with typical Newtonian behavior and  $R^2 > 0.98$ . For viscosity, Cretu et al. (2011) reported lower values (13.65 mPa-s) when they analyzed a linseed oil microemulsion, including Tween 80, alcohol and curcumin. The formulation's low viscosity is due to structuring of the microemulsion continuous phase with interactions between molecules. The pH for drying the suspension was 7.6, corresponding to an alkaline pH and an orange color with a tendency to yellow. According to Vargas (2011), a brown color has an acid pH, yellow a neutral pH, and orange to red an alkaline pH; in addition, it is stable at neutral and basic pH values.

With respect to color, the color parameters for the concentrated extract of curcumin formulated with maltodextrin were L:  $26.83 \pm 0.37$ , indicating the luminosity, a trend towards black or dark colors. Additionally,  $h^\circ$ :  $51.81 \pm 0.91$ ,  $a^*$ :  $26.34 \pm 0.54$  and  $b^*$   $33.51 \pm 1.80$ , and the  $h^\circ$  angle and coordinates  $a^*$  and  $b^*$  show that the color is yellow-orange, which is characteristic of curcumin and matches the color parameters for the extract pH observed by Vargas (2011). For color parameters, Cretu et al. (2011) reported L values of the mixture and microemulsion with curcumin as nearly white (90.54) and  $h^\circ$  angle as 91.58; these are higher values than those in the present study because linseed oil increases the luminosity values. Additionally, their reported curcumin was more likely pale-yellow, while ours was more orange-yellow.

The total solids in the drying suspension were up to 40.7%, representing the contribution of solids from maltodextrin. This value is within the range recommended by Vargas (2011), who stated that the range should be between 30 and 50% to avoid low drying yields. The curcumin concentration in the concentrated extract was  $17.53 \text{ mg/g} \pm 1.43$ , which is less than that obtained by Vargas (2011), whose turmeric oleoresin value was 165.16 mg/g. Therefore, our study produced a low curcumin yield from the extract.

**Table 1.** Characterization of the concentrated extract.

Parameter	Concentrated curcumin
Viscosity 25°C	109.4 ± 1.68 mPa-s
pH	7.6 ± 0.1
Color	L: 26.83 ± 0.37. h°: 51.81 ± 0.91. a*: 26.34± 0.54 y b* 33.51± 1.80
Total solids	40.72 ± 2.30 %

According to statistical analysis of the process yield, the material that adhered to the dryer as well as its

solubility, final moisture content, water activity, color parameters (for powdered product, diluted solution at 0.1% and diluted solution at 0.01%), there were no statistically significant differences ( $p > 0.05$ ). Thus, the drying temperatures do not influence these response variables. However, statistical analysis (Table 2) demonstrated significant differences ( $p < 0.05$ ) for the curcumin concentration and hygroscopicity, resulting in a high regression coefficient ( $R^2 > 0.97$ ) and no significant lack of fit ( $p > 0.05$ ). The results for each of the response variables are defined (Table 3).

**Table 2.** Main effects of the polynomial regression model on the hygroscopicity and concentration.

Regression coefficients	Hygroscopicity (%)	Concentration (mg/g)
$\beta_0$	6.05	1.71
$\beta_A$	0.022	0.013
$\beta_B$	5.14	0.87
$\beta_{AB}$	0.84	0.021
$\beta_A^2$	0.019	0.77
$\beta_B^2$	0.012	0.17
<b>R<sup>2</sup></b>	<b>0.8552</b>	<b>0.9393</b>
Model (p-valor)	0.0368*	0.048*
Lack of fit (p-valor)	0.1471	0.0610

\*Significant at 5%

**Table 3.** Variable process and quality properties of dry curcumin extracts obtained using spray drying.

Test	Tin (°C)	Tout (°C)	Yield (%)	Material adhered (%)	Moisture (%)	Aw	Solubility (%)	Hygroscopicity (%)	Concentration (mg/g)
1	140	95	89	0.74	3.38 ± 0.00	0.24 ± 0.02	98.10 ± 0.00	12.74 ± 0.00	4.92 ± 0.27
2	140	85	95.88	0	3.21 ± 0.00	0.27 ± 0.02	98.11 ± 0.00	10.99 ± 0.00	4.76 ± 0.49
3	150	95	86.16	0	2.79 ± 0.00	0.19 ± 0.03	97.86 ± 0.00	11.22 ± 0.00	5.32 ± 0.40
4	150	85	96.89	0	3.12 ± 0.00	0.18 ± 0.00	97.98 ± 0.00	11.03 ± 0.00	5.40 ± 0.22
5	160	85	78.63	0	2.16 ± 0.00	0.23 ± 0.00	98.24 ± 0.00	10.63 ± 0.00	5.17 ± 0.50
6	150	75	75.8	2.75	2.41 ± 0.00	0.23 ± 0.00	98.16 ± 0.00	10.36 ± 0.00	6.23 ± 0.14
7	150	85	81.94	7.14	2.17 ± 0.00	0.24 ± 0.01	98.13 ± 0.00	10.72 ± 0.00	5.49 ± 0.31
8	140	75	61.96	9.86	3.16 ± 0.00	0.27 ± 0.00	98.15 ± 0.00	9.47 ± 0.00	5.44 ± 0.26
9	160	75	83.49	0	2.94 ± 0.00	0.18 ± 0.00	98.15 ± 0.00	10.39 ± 0.00	5.54 ± 0.05
10	150	85	85.03	0	2.09 ± 0.00	0.20 ± 0.00	98.09 ± 0.00	11.17 ± 0.00	5.43 ± 0.34
11	160	95	88.97	0	1.87 ± 0.00	0.17 ± 0.00	98.06 ± 0.00	11.82 ± 0.00	4.70 ± 0.36

The yield for the drying process ranged from 61.96 to 96.89% (Table 3). Arrazola et al. (2014) reported yield values up to 90.74% for spray drying of anthocyanins, which is lower than those found in the present investigation, which can be explained by water solubility of the maltodextrin and concentrated curcumin extract as well as their good affinity. As a result, there was less solid adhesion to the dryer surface. Bhusari et al. (2014) reported yields of 46.5 to 76.23% for drying tamarind powder and maltodextrin, while Obón et al. (2009) reported yields of 58% for a powdered dye obtained from *Opuntia stricta* fruits due to difficulties with collecting small compounds because they do not deposit as efficiently in the cyclone.

The moisture content of the product varies between 1.38 and 3.38% (w.b) based on values reported in several studies (Arrazola et al., 2014; Obón et al., 2009; Jittanit et al., 2011). Bagchi (2012), reported

a value of moisture content of 5% for a dry turmeric powder, which is higher than those presented here. According to Frascareli et al. (2012) with the increase in the inlet temperature, there is a higher temperature gradient between the air and product for drying, allowing for greater heat and mass transfer, favoring an increased water evaporation rate and decreasing the moisture content of the final product. The water activity values ranged between 0.24 and 0.27, showing a similar moisture content behavior. Furthermore, the values are similar to those reported by Jittanit et al. (2011) and Fang & Bhandari (2011).

The solubility showed no significant influence of the temperature factor; it ranged from 97.86 to 98.24, showing good solubility. Maltodextrin has a high solubility in aqueous solutions. For this research, the curcumin powder evaluation was conducted directly in water. Similar solubility values have been found by Rivas (2010).

**Table 4.** Color parameters for the curcumin dye powder that was diluted using the spray drying process.

Test	Tin (°C)	Tout (°C)	Color (CIELAB Method)											
			Powder				Solution (0.1 %)				Solution (0.01%)			
			L	h <sup>0</sup>	a*	b*	L	h <sup>0</sup>	a*	b*	L	h <sup>0</sup>	a*	b*
1	140	95	39.51± 1.35	58.38± 0.31	24.83± 0.76	40.35± 1.73	43.72± 0.51	93.28± 1.17	-2.41± 0.68	42.71± 2.91	50.19± 0.65	116.67± 0.14	-3.81± 0.05	7.58± 0.12
2	140	85	53.82± 0.76	67.44± 0.07	21.07± 0.36	50.71± 0.75	46.28± 0.04	95.27± 0.26	-3.82± 0.26	41.31± 0.70	50.86± 0.58	114.40± 0.99	-4.43± 0.26	9.80± 1.06
3	150	95	52.52± 0.36	65.58± 0.27	21.81± 0.53	48.04± 1.79	45.22± 1.07	95.00± 0.17	-3.58± 0.10	40.93± 0.35	47.80± 4.06	113.86± 1.15	-3.88± 0.24	8.77± 0.06
4	150	85	53.13± 0.18	67.17± 0.45	22.36± 0.18	53.15± 0.76	46.60± 0.15	96.61± 0.13	-4.51± 0.15	38.93± 0.85	49.47± 0.12	116.14± 0.13	-3.81± 0.12	7.76± 0.29
5	160	85	52.45± 0.17	65.20± 0.39	21.82± 0.75	47.22± 1.28	45.45± 0.09	93.82± 0.04	-2.86± 0.04	42.89± 0.38	50.19± 0.36	113.56± 0.87	-4.11± 0.18	9.45± 0.76
6	150	75	55.04± 0.27	66.53± 0.87	19.39± 1.16	44.75± 4.28	42.95± 0.17	94.89± 0.19	-3.51± 0.03	41.03± 1.50	49.88± 1.15	114.42± 1.65	-4.12± 0.28	±9.13± 1.28
7	150	85	56.73± 2.39	67.46± 0.33	19.02± 0.65	45.80± 0.86	45.53± 0.18	94.25± 0.18	-3.05± 0.11	41.12± 1.65	50.40± 0.94	113.87± 0.61	-3.95± 0.05	8.92± 0.37
8	140	75	57.77± 0.14	68.54± 0.40	19.06± 0.40	48.50± 1.39	45.18± 0.21	94.78± 0.21	-3.41± 0.21	40.76± 0.44	48.89± 1.64	113.27± 0.35	-3.80± 0.10	8.83± 0.30
9	160	75	56.79± 0.30	68.05± 0.23	18.48± 0.36	45.87± 0.47	43.67± 0.15	94.02± 0.15	-2.88± 0.16	41.04± 2.94	49.26± 1.03	112.16± 0.30	-4.02± 0.11	9.88± 0.43
10	150	85	55.39± 2.40	67.52± 0.46	18.97± 1.16	45.91± 3.81	43.62± 0.29	94.24± 0.29	-3.05± 0.27	41.21± 1.25	49.62± 0.74	112.61± 1.46	-3.63± 0.46	8.81± 1.66
11	160	95	56.46± 0.15	67.82± 0.35	19.28± 0.37	47.28± 0.19	45.75± 0.14	94.31± 0.15	-3.02± 0.06	40.14± 0.69	49.29± 2.58	110.85± 0.51	-4.25± 0.18	11.16± 0.77

The color parameters for the dye in dry powder form and diluted in aqueous medium are reported (Table 4). The L parameter corresponds to the luminosity (white or black); the lower luminosity value for the powder was 39.51,  $a^*$  color tending toward black, and the highest value was 57.77, corresponding to a white color. The angle  $h^\circ$  (hue) for powder (yellow/orange) ranged between 58.38 and 68.54, and for the parameters  $a^*$  and  $b^*$ , the ranges were 18.48-24.83 and 40.35-53.15, respectively. For luminosity (L), the values are lower than those reported by Gómez et al. (2012) (62.1 for commercial curcumin), and they are likely similar to those found by Arrazola et al. (2014) when they evaluated anthocyanins (41 and 56.2). Additionally, the  $h^\circ$  angle for the powder is similar to the  $h^\circ=62.6$  reported by Gómez et al. (2012). For the 0.1% diluted solution, the L value was 42.95-46.60 and the  $h^\circ$  angle varied between 93.28 and 96.61; the  $a^*$  and  $b^*$  parameters were between -2.41-4.51 and 40.14-42.89, respectively. For the diluted solution at this concentration, Gómez et al. (2012) reported values for commercial curcumin of 87.6. The difference in the value of L with respect to this research is that these authors performed their dilution in milk; therefore, the luminosity tends to be whiter in color. Additionally, the same authors reported an  $h^\circ$  angle of 108.5, describing it as a yellow hue, while that value in this research has a yellow to orange hue. For the 0.01% diluted solution, the L values ranged from 47.80 to 50.86, which were higher than the 0.1% solution, while the  $h^\circ$  angle increased compared to the 0.1% concentration (10.85-116.67). The parameters  $a^*$  and  $b^*$  were -3.63-4.43 and 7.58-11.16, respectively. This increase in the L parameter indicates that the luminosity is higher for dilute solutions, as they allow for easier passage of light through the sample and also because the lower concentration of curcumin tends towards light colors. On the other hand, with respect to  $a^*$ ,  $b^*$  and  $h^\circ$  angle, these parameters are indicative of the tone. The lower the presence of curcumin in the solution, the less intense a pale-yellow color the solution will be.

The response surface for the variables that were significant in this study, that is, hygroscopicity and concentration of curcumin, are given in Figure 1. Higher curcumin concentrations in the powder are

present for the intermediate inlet air temperatures (150°C) and low outlet air temperatures (75°C), indicating higher retention of curcuminoids in low drying temperatures (Figure 1a). There is a significant effect of drying temperatures on the curcumin concentration in the dry powder (Table 2), ranging from 4.92 to 6.23 mg/g (Table 3). Therefore, there is degradation of the curcumin present due to the thermal effects of processing. In this study, analysis of curcumin was quantified by curcuminoids present in the sample, and a higher percentage retention of low temperature drying at entry was observed. The degradation and impact of the thermal conditions of the drying process are evident, showing a decrease between 35.53% and 28.06% relative to the initial curcumin content. According to Ko et al. (2015), curcumin retention in the wall material is determined by the relationship between the molecules of the material with curcumin. Moreover, the driving force that occurs between the material and curcumin involves Van der Waals forces and hydrophobic interactions as well as contributions of the hydrogen bonds derived from the curcumin OH groups, contributing to the lower degradation of the curcumin compounds. Moreover, when the intermolecular forces of the auxiliary agent (maltodextrin) are greater than those of curcumin, the balance of the suspension is destroyed and the binding ability of the curcumin compounds decreases, increasing the curcumin degradation. The ratio between curcumin and maltodextrin becomes smaller and does not act as a protective agent. Chen et al. (2014) studied the thermal degradation of curcumin and suggested that degradation occurs in two stages; the first is given by the decomposition of the substituent groups of curcumin, and the second is due to the decomposition of two curcumin benzene rings. When evaluating the concentration of powdered curcumin, Molina et al. (2013) studied the curcuminoid content in microparticles obtained by spray drying turmeric extract. The content ranged from 17.15 to 19.57 mg/g, which is higher than the levels found in this investigation. This difference may be from the lower curcumin level (17.53 mg/g) at the start of the research.

With respect to the hygroscopicity property, lower values are shown for inlet air temperatures

between 140-150°C and outlet temperatures below 75°C (Figure 1b). If high drying temperatures are used, the driest products are generated in storage conditions that favor the absorption of more water. In this case, low hygroscopicity values are due to the use of lower drying temperatures. Similarly, at lower entrance drying temperatures, the hygroscopicity increases considerably with a higher outlet temperature. The hygroscopicity of the product ranged from 9.47 to 12.74% (Table 3). High drying temperatures decrease the product's moisture content, making it more hygroscopic

because it absorbs more water, thus favoring the water concentration gradient between the product and air (Arrazola et al., 2014; Tonon et al., 2008). The obtained values are lower than those reported by other authors, which is possibly because maltodextrin was used as an auxiliary agent and has low hygroscopicity. Additionally, there was a decrease in the powder yield (Valenzuela & Aguilera, 2015; Caparino et al., 2012). Bhusari et al. (2014) obtained values ranging from 16.61 to 28.96%; thus, the maltodextrin has a positive effect by decreasing the hygroscopicity.

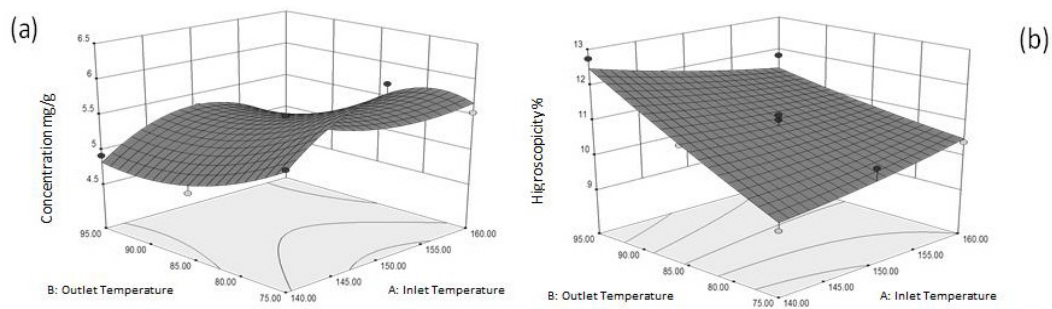


Figure 1. Response surface for curcuminoid concentration in the powder (a) and hygroscopicity (b).

For application at the industrial level and to identify the best operating conditions for the drying process, a process to optimize the drying process (inlet and outlet air temperatures) as a function of variables that had statistically significant effects, such as the hygroscopicity and concentration was performed. The concentration was maximized, based on high levels of curcumin in the powder, and hygroscopicity was minimized by keeping the powder in optimal storage conditions. The obtained optimum drying conditions were an inlet temperature of 149°C and outlet temperature of 75°C with a spray rate of 28000 RPM. The values experimentally validated the process and were compared with the model shown in Table 5. For the performance variables, the solubility, hygroscopicity, final curcumin concentration, and L parameters (h0, a\* and b\*) for the 0.1% color solution and the same parameters for the 0.01% color solution in the theoretical model exhibit lower experimental values with higher values for the material adherence, final moisture content,

water activity, and color parameters for powder (L, a\*, b\* and h0) (Table 5). The powder obtained and the respective dilutions at 0.1 and 0.01% are shown (Figure 2).

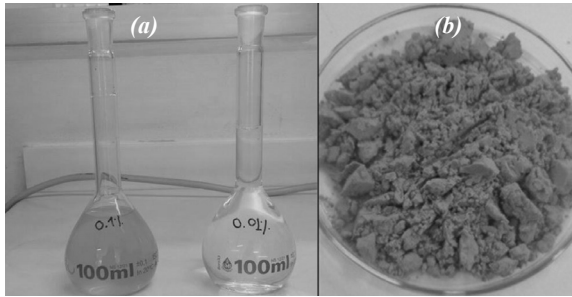
Table 5. Experimental validation of drying process.

Variable	Experimental	Model value
Yield (%)	80.45	73.30
Material adhered (%)	2.48	4.96
Moisture (%) (w.b)	2.63±0.02	2.89
Aw	0.175±0.00	0.218
Solubility (%)	98.15±0.02	98.07
Hygroscopicity (%)	11.71±0.03	9.97
Concentration mg/g	9.03 ±0.44	6.10
Powder color	L	53.93±0.31
	a*	18.63±0.10
	b*	46.49±0.22
	h <sup>0</sup>	62.87±0.26
Solution color: 0.1%	L	45.96±1.67
	h <sup>0</sup>	98.23±0.04



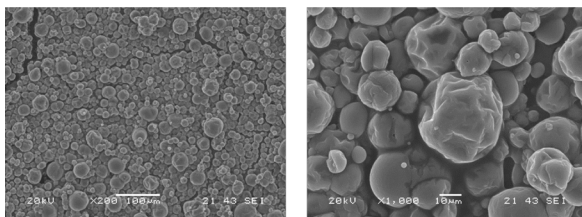
\*Continued table 5

	a*	-3.54±0.34	a*	-3.57
	b*	40.35±0.28	b*	40.31
Solution color: 0.01%	L	50.98±0.46	L	48.92
	h <sup>0</sup>	117.88±0.36	h <sup>0</sup>	113.56
	a*	*-3.84±0.57	a*	-3.98
	b*	8.78±0.41	b*	8.78



**Figure 2.** (a) Curcumin powder in 0.1% and 0.01% diluted solutions. (b). Curcumin powder optimum.

In Figure 3, the analysis by scanning electron microscopy (SEM) at different magnifications is shown. According to the results, curcumin powder has a compact microstructure structure with a shape that approximates a sphere and a particle size estimated as 17.38  $\mu\text{m}$  (some particles are larger because they form aggregates) in some cases. They are corrugated and lack evidence of fragmented areas, indicating that the final product has an adequate structure. In addition, they are different sizes, which is characteristic of powders obtained by spray drying. In the microstructure, some pore surfaces, which are undesirable because they allow for permeability of gases such as oxygen, are not observed. Gómez et al. (2012) reported that encapsulated corn zein shares structural similarity with curcumin.



**Figure 3.** Scanning electronic microscopy of curcumin powder.

## 4. Conclusions

The use of spray drying as technological process to obtain a powder from curcumin liquids extracts formulated with maltodextrin affects curcuminoids concentration in the final product. There is a degradation of the curcumin in the powder due to the thermal effects where a higher percentage retention is obtained when a low inlet air temperature is used.

This research work shows the necessity to study the incorporation of another carrier agents different to maltodextrin in the spray drying process of turmeric extracts with the objective to evaluate the effect of wall material upon the quality characteristic of the powder and dryer performance variables.

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