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# Spray-drying of *Escontria chiotilla* fruit juice: effect of the process variables on yield and physicochemical properties

# Secado por aspersión de jugo de *Escontria chiotilla*: efecto de las variables del proceso sobre el rendimiento y las propiedades fisicoquímicas

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## Abstract

*Escontria chiotilla* (*jiotilla*) fruit pulp has a high content of sugars, vitamins, antioxidants and betalains. However, the *jiotilla* fruits have a high susceptibility to microbial attack and physicochemical degradation during storage. Spray-drying of *jiotilla* fruit juice rich in betalains was carried out using maltodextrin as carrier material. The effects of the inlet (140 and 160 ºC) and outlet (80 and 90 ºC) temperatures and maltodextrin levels (1 and 2 g·g juice solids$^{-1}$) on the process yield and physicochemical properties of *jiotilla* powders were studied. The maltodextrin concentration significantly affected ($p < 0.05$) all responses evaluated, except $a_w$ and ºHue values. While drying temperatures did not affect any response. An increase in process yields and luminosity, and a decrease in chroma values and betalains content, were observed when the maltodextrin concentration was increased. Physicochemically stable powders ($a_w$ <0.25), with a color tendency to red (ºHue: 11-13º), an intermediate cohesiveness, a flowability fair-to-good, a high solubility (92.16%), a low hygroscopicity (16.28 g $H_2O$·100 g db$^{-1}$) and a high content of betacyanins (82.4-127.1 mg BC·100 g db$^{-1}$) and betaxanthins (174.7-275.3 mg BX·100 g db$^{-1}$) were obtained. *Jiotilla* powder could be applied to the development of food products, even outside the *jiotilla* fruits production season.

*Keywords*: *Escontria chiotilla*, fruit juice, spray-drying, betalains, maltodextrin.

## Resumen

La fruta de *Escontria chiotilla* (*jiotilla*) tiene alto contenido de azúcares, vitaminas, antioxidantes y betalaínas. Sin embargo, presentan alta susceptibilidad al ataque microbiano y degradación fisicoquímica durante el almacenamiento. Se realizó el secado por aspersión de jugo de *jiotilla* utilizando maltodextrina como material acarreador. Se estudiaron los efectos de las temperaturas de entrada (140 y 160 ºC) y salida (80 y 90 ºC), y los niveles de maltodextrina (1 y 2 g·g sólidos de jugo$^{-1}$) sobre el rendimiento del proceso y las propiedades fisicoquímicas de los polvos obtenidos. La concentración de maltodextrina afectó significativamente ($p$ <0.05) todas las respuestas evaluadas, excepto los valores de $a_w$ y ºHue. Mientras que las temperaturas de secado no afectaron ninguna respuesta. Al incrementar la concentración de maltodextrina se aumentaron los rendimientos del proceso y la luminosidad, y se disminuyeron los valores de croma y el contenido de betalaínas. Se obtuvieron polvos fisicoquímicamente estables ($a_w$ < 0.25), de color con tendencia al rojo (ºHue: 11-13º), cohesividad intermedia, fluidez regular a buena, alta solubilidad (92.16%), baja higroscopicidad (16.28 g $H_2O$·100 g bs$^{-1}$) y alto contenido en betacianinas (82.4-127.1 mg BC·100 g bs$^{-1}$) y betaxantinas (174.7-275.3 mg BX·100 g bs$^{-1}$). El polvo de *jiotilla* se puede aplicar al desarrollo de productos alimenticios, incluso fuera de la temporada de producción de frutos de *jiotilla*.

*Palabras clave*: *Escontria chiotilla*, jugo de fruta, secado por aspersión, betalaínas, maltodextrina.

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# 1 Introduction

Cactus fruits are a source of hydro-soluble pigments and bioactive compounds for food industry (Obón *et al*., 2009; Otálora *et al*., 2015). However, these fruits have little industrial utilization due to their $a_w > 0.8$ and, therefore have high susceptibility to microbial and physicochemical degradations (Castro-Muñoz *et al*., 2015; León-Martínez *et al*., 2010). The cactus fruit juices could be recovered in form of dry particles by spray-drying, which offers short contact times with drying gas and relatively low outlet temperatures, therefore tends to preserve the physicochemical and biological properties of foods, and the nutrients, bioactive compounds and antioxidant activity (Darniadi *et al*., 2019; Goyal *et al*., 2020; Kinalski and Noreña, 2019; Shishir and Chen, 2017; Wang *et al*., 2020). Spray-drying parameters such as drying temperatures, feed flow, carrier material (both type and content), have shown effects on the physicochemical properties of fruit juice powders and reconstituted products as moisture content, particle size, bulk density, flowability, color, hygroscopicity, and solubility (Koç and Dirim, 2018; Shishir and Chen, 2017; Tontul and Topuz, 2017; Wang *et al*., 2020). The principal problem during spray-drying of fruit juices is the adhesion of product on the both dryer chamber and cyclone due to the stickiness associated with a high content of low molecular-weight sugars and organic acids, so low yields and operating problems could be shown (Bazaria and Kumar, 2017; Rodríguez-Hernández *et al*., 2005; Sun-Waterhouse and Waterhouse, 2015). Various carrier materials like gums, modified starches, maltodextrins, fibers, and other materials of high molecular-weight are used in the spray-drying of fruit juices to avoid those problems (Mishra *et al*., 2014; Sarabandi *et al*., 2017; Suhag *et al*., 2016; Wang *et al*., 2020).

The maltodextrins are used as carrier and wall materials in the food industry. These materials are obtained by acid hydrolysis of starches such as corn, potato, and others. In general, maltodextrins have high water solubility, and their water solutions are colorless and have low viscosity and bland flavor (Bazaria and Kumar, 2017; Islam *et al*., 2021; Mishra *et al*., 2014; Shishir and Chen, 2017). The maltodextrins are usually classified according to their degree of hydrolysis, expressed as dextrose equivalent (DE) and typically possess a DE < 20 for food applications (Marchal *et al*., 1999). Examples of spray-dried cactus fruit juices with maltodextrins include cactus pear from *Opuntia streptacantha* (10 and 20 DE) (Rodríguez-Hernández *et al*., 2005), cactus pear from *Opuntia ficus-indica* (10 and 20 DE) (Saénz *et al*., 2009), red pitaya (*Hylocereus polyrhizus*) (Tze *et al*., 2012) and purple cactus pear from *Opuntia stricta* (10-DE) (Castro-Muñoz *et al*., 2015). In these studies, maltodextrin improved the stability of fruit juice powders, reducing stickiness and agglomeration problems during processing and storage.

*Escontria chiotilla* (F. A. C. Weber) Rose (*jiotilla*) is a culturally and economically important cactus from arid and semiarid areas in Central Mexico (Oaxaca-Villa *et al*., 2006). *Jiotilla* fruits are an excellent source of natural colorants and functional compounds (Soriano-Santos *et al*., 2007; Soto-Castro *et al*., 2019). *Jiotilla* fruits are red, characteristically covered by scales which give them a greenish or grayish appearance. The edible sweet pulp is more commonly red, although white, pink, and purple colors can sometimes be observed or referred by local people. The harvest of *jiotilla* fruit is an important economic activity, mainly occurring between April and June (Arellano and Casas, 2003). *Jiotilla* is consumed crisp, and their flavor is preferred over fruit of other cacti species due to its high content of sugars, vitamins, and antioxidants. Jams, ice cream, the wine called "*nochoctli*", and the essence called "*vinillo*" can also be prepared (Arellano and Casas, 2003; Oaxaca-Villa *et al*., 2006; Soriano-Santos *et al*., 2007).

To the best of our knowledge, there are no studies of the maltodextrin effect as carrier material and spray-drying conditions on the physicochemical properties of *jiotilla* juice powder and the reconstituted product. Some authors have studied the pigment content in *jiotilla* fruit and its microencapsulation by spray-drying. Soriano-Santos *et al*. (2007) identified the red pigment of this fruit as betalains, which are used as natural colorants for red shades in the food industry (Bazaria and Kumar, 2017; Franco-Vásquez *et al*., 2023; Ravichandran *et al*., 2014; Stintzing *et al*., 2005). These authors identified that betalains from *jiotilla* pulp were composed of 119 ± 3.1 mg betaxanthins/kg fruit and 89 ± 6.5 mg betacyanins/kg fruit, these values represent the 19.7 ± 2.3 % of betacyanin and 41.8 ± 0.6 % of betaxanthins compared to beet, which is the only betalain-based commodity commercially used (Bazaria and Kumar, 2017; Ravichandran *et al*., 2014; Stintzing *et al*., 2005). Soto-Castro *et al*. (2019) achieved the retention of betalains at more than 90 % during the spray-drying of betalain rich-extracts from *Escontria chiotilla* using cactus mucilage as carrier material, the process only was carried out at inlet drying air temperature of 140 °C and outlet air temperature of 70 °C.

Therefore, the aim objective of this study was to evaluate the effects of the process temperatures and the DE-10 maltodextrin concentrations used for the spray-drying of *jiotilla* juice on the process yield and the physicochemical properties of powder. An understanding of the phenomena around the process will allow its subsequent escalation or propose new lines of research. The spray-drying of *jiotilla* juices rich in betalains has a high potential to design new food products from *jiotilla* powder, and moreover will promote both sustainable production and economic benefits to social groups that recollect these fruits.

## 2 Materials and methods

### 2.1 Materials

*Jiotilla* fruits were harvested in September 2018 from Santa María Camotlán, Oaxaca, México (17° 49' - 17° 55' north latitude; 97° 34' - 97° 44' west longitude). Only commercial mature fruits were collected according to local farmers recommendations. The fruits were stored at 4 °C and processed in less than 48 h. Maltodextrin 10-DE was purchased from Fabpsa® (CDMX, México). All chemicals (analytical grade) were purchased from Sigma Chemical Co. (St. Louis, MO, USA) and all solvents (analytical grade) from JT Baker (CDMX, Mexico). Distilled water was used in this work.

### 2.2 Extraction of juice from jiotilla fruits

*Jiotilla* fruits first were washed, and then the fruit pulp was manually separated from pericarp. After that, the pulp was pressed manually. In this stage is important avoid the rupture of the seeds because a mucilage could be extracted, and the viscosity of the juice could be increase. Finally, the seeds were removed by filtration through a number 100 metallic sieve. The *jiotilla* juice (11.7 ± 0.1 ºBrix) was stored at 4 ºC until spray-drying. The juice was processed in less than 48 h. The solid fraction in *jiotilla* juice was calculated by mass difference: aliquots (10 g) of *jiotilla* juice were placed in a vacuum oven at 65 ºC and 1.4 kPa until constant mass was reached (Vázquez-León *et al.*, 2021).

### 2.3 Preparation of feed mixture and spray-drying process

Different amounts of maltodextrin 10-DE per gram of fed juice solids (g M/g JS) are listed in Table 1. Maltodextrin was added directly to the *jiotilla* juice (150 g in all cases). The resulting mixture was homogenized at 50 ºC and 200 rpm, for 15 min (Thermo Scientific, MaxQ 4450; Ohio, USA). All feed mixtures were magnetically stirred throughout the drying process (Soto-Castro *et al.*, 2019; Vázquez-León *et al.*, 2021). A laboratory-scale spray dryer (Büchi, B-190; Flawil, Switzerland) equipped with a 0.7 mm standard-diameter nozzle and a 1.5 mm diameter nozzle screw cap was used in the present work. The atomization air flow was 538 $L \cdot h^{-1}$ (35 mm on the gas rotameter indicator) and the atomization pressure was $6 \times 10^5$ Pa. The air aspiration rate was maintained at 35 $m^3 \cdot h^{-1}$. Table 1 shown the process temperatures that were evaluated. The outlet air temperature was controlled with the feed rate through a peristaltic pump (Vázquez-León *et al.*, 2021). The powders obtained were kept in polyurethane bags that were sealed under vacuum and then stored at 25 ºC until analyses.

### 2.4 Process yield

Process yield (%) of each experiment was calculated with the Eq. (1), where $W_1$ is the total solids in the feed mixture, and $W_2$ is the total mass of the recovered dry product after drying process (Soto-Castro *et al.*, 2019; Vázquez-León *et al.*, 2021; Wang *et al.*, 2020):

$$\text{Process yield (\%)} = \frac{W_2 \times 100}{W_1} \tag{1}$$

Table 1. Experimental conditions to evaluate the spray drying of *Escontria chiotilla* fruit juice, final composition of the fed mixtures, process yield, $a_w$ values and betalains content.

| Inlet/outlet temperature (°C) | g M/g JS | Final composition (%) | | Process yield (%) | $a_w$ | mg BC / 100 g db | mg BX / 100 g db |
|---|---|---|---|---|---|---|---|
| | | Water | Solids | | | | |
| **160/90** | 2 | 79.5 | 20.5 | $43.5 \pm 4.2^{ab}$ | $0.234 \pm 0.01^{a}$ | $89.5 \pm 0.5^{b}$ | $191.0 \pm 5.5^{b}$ |
| **160/80** | 2 | 79.5 | 20.5 | $49.8 \pm 12.5^{ab}$ | $0.198 \pm 0.01^{a}$ | $89.4 \pm 9.9^{b}$ | $187.7 \pm 14.0^{b}$ |
| **140/90** | 2 | 79.5 | 20.5 | $46.0 \pm 11.3^{ab}$ | $0.198 \pm 0.00^{a}$ | $100.3 \pm 13.6^{ab}$ | $209.8 \pm 28.2^{ab}$ |
| **140/80** | 2 | 79.5 | 20.5 | $62.4 \pm 12.8^{a}$ | $0.194 \pm 0.03^{a}$ | $89.4 \pm 8.6^{b}$ | $185.1 \pm 14.8^{b}$ |
| **160/90** | 1 | 85.4 | 14.6 | $32.9 \pm 1.8^{b}$ | $0.223 \pm 0.01^{a}$ | $107.9 \pm 1.3^{ab}$ | $223.2 \pm 6.43^{ab}$ |
| **160/80** | 1 | 85.4 | 14.6 | $33.5 \pm 11.6^{b}$ | $0.208 \pm 0.04^{a}$ | $115.6 \pm 4.7^{ab}$ | $242.8 \pm 19.3^{ab}$ |
| **140/90** | 1 | 85.4 | 14.6 | $34.4 \pm 14.6^{b}$ | $0.212 \pm 0.05^{a}$ | $124.9 \pm 3.1^{a}$ | $263.3 \pm 17.0^{a}$ |
| **140/80** | 1 | 85.4 | 14.6 | $30.4 \pm 6.0^{b}$ | $0.241 \pm 0.07^{a}$ | $113.6 \pm 9.1^{ab}$ | $236.7 \pm 19.1^{ab}$ |

g M/g JS: grams of maltodextrin per gram of fed juice solids. BC: betacyanins. BX: betaxanthins.
The values are the mean ± standard deviation ($n = 2$).
Values in columns that do not share a letter are significantly different ($p < 0.05$).

## 2.5 Physicochemical analyses

### 2.5.1 Water activity and color measurement

Water activity ($a_w$) at 25 ºC was evaluated with an AquaLab Series 3 equipment (Decagon Devices Inc., Pullman, WA, USA) for each spray-dried powder (Vázquez-León *et al*., 2021). The color of the samples was measured with a color reader (Hunter Associates Laboratory Inc.; MiniScan XE Plus; Reston, VA, USA). Powder color was reported as the luminosity (L*), hue angle (ºHue) and chroma (C*) values (Bazaria and Kumar, 2017).

### 2.5.2 Quantification of betacyanins and betaxanthins

Betacyanins (BC) and betaxanthins (BX) content in both *jiotilla* juice and resuspended powder samples was evaluated with the method reported by Soriano-Santos *et al*. (2007), Castro-Muñoz *et al*. (2015) and Khatabi *et al*. (2016). Briefly, 1 ± 0.1 g of dry powder (or 1 mL of *jiotilla* juice) was diluted in 25 mL of buffer solution (citrate-phosphate) adjusted to pH 5.8. Then 1 mL of the mixture was diluted with 9 mL of the same buffer. The absorbance of the solutions was measured in a spectrophotometer at 538 nm (for BC) and 476 nm (for BX) (Thermo Scientific, Mod. Genesys 10S; Waltham, MA, USA). The measurements were made using a quartz cell of 1 cm. The mg of BC or BX per g of dry basis (mg/(g db)) were calculated with the Eq. (2).

$$(mg/(gdb)) = \frac{A \cdot DF \cdot V}{\varepsilon \cdot W} \tag{2}$$

where $A$ is the absorbance for each pigment (BC or BX), DF is the dilution factor, $V$ is the aliquot volume, $W$ is the dry weight (g db) of the sample and $\varepsilon$ is the extinction coefficient (BC, 1120; BX, 750) [L mol$^{-1}$ cm$^{-1}$].

### 2.5.3 Bulk density ($\rho_b$) and tapped density ($\rho_t$)

Spray-dried powders were loaded into a tared graduated cylinder, then the bulk density ($\rho_b$) was calculated as the relationship mass/volume (g·cm$^{-3}$) of each sample (Fernandes *et al*., 2014; Wang *et al*., 2020). Then the cylinder with the loaded sample was repeatedly tapped until a negligible difference in volume was observed. Finally, the tapped density ($\rho_t$) was calculated as the relationship mass/tapped volume (g·cm$^{-1}$) (Fernandes *et al*., 2014; Koç and Dirim, 2018).

### 2.5.4 Hausner ratio and Carr index

Hausner ratio ($HR$, Eq. (3)) and Carr index (CI, Eq. (4)) were calculated with both $\rho_t$ and $\rho_b$ values, in according to Tze *et al*. (2012) and Koç and Dirim (2018).

$$HR = \rho_t/\rho_b \tag{3}$$

$$CI = (\rho_t - \rho_b)/\rho_t \tag{4}$$

### 2.5.5 Solubility and hygroscopicity

For the determination of solubility, the method reported by Castro-Muñoz *et al*. (2015) was followed with light modifications. 1 g of dry sample was dispersed in 10 mL of distilled water at 25 °C. The suspension was mixed in a vortex for 5 min and then the mixture was centrifuged (4000 ×g; 5 min). The supernatant was emptied in a constant-weight aluminum tray, weighed, and then placed in a drying oven (65 ºC, 1.4 kPa) until constant mass. The solubility (g·100 mL$^{-1}$) was calculated with the mass of the dried solids and the volume of the supernatant.

The hygroscopicity of the powders was calculated by the method reported by Tonon *et al*. (2008). 1 g of powdered juice was weighed in a constant-weight aluminum tray. The sample was placed in a desiccator with a NaCl saturated solution (76% relative humidity at 25 ºC). After 7 days, the samples were weighed every 24 h until reaching a constant weight. The hygroscopicity was expressed as g of adsorbed water per 100 g of dry solids (g $H_2O$·100 g db$^{-1}$).

## 2.6 Experimental design and statistical analysis

A $2^3$ factorial design was used to evaluate the effect of spray-drying conditions on the process yield and the physicochemical properties of *jiotilla* juice powder (Table 1). Inlet and outlet air temperatures (Tin, $x_1$; Tout, $x_2$; respectively), and maltodextrin concentration into feed (g M/g JS, $x_3$) were the process variables under study. All treatments were carried out in triplicate. The response variables ($y_i$) were the process yield, $a_w$, L*, C*, ºHue and both betacyanins and betaxanthins content. The $y_i$ were fitted to the model shown in Eq. (5):

$$y_i = \beta_{i0} + \beta_{i1}x_{i1} + \beta_{i2}x_{i2} + \beta_{i3}x_{i3} + \beta_{i12}x_{i1}x_{i2} + \beta_{i13}x_{i1}x_{i3} + \beta_{i23}x_{i2}x_{i3} \tag{5}$$

where $\beta_{ij}$ are the regression coefficients calculated by multivariate linear regression for each response $i$; $x_1$, $x_2$ and $x_3$ are the coded independent variables. Significant terms ($p < 0.05$) in the model for each $y_i$ were determined by multivariate analysis of variance (Pui *et al*., 2020; Vázquez-León *et al*., 2021) in Minitab 18 statistics package (Minitab Inc., State College, PA, USA). Some results were finally analyzed by a Tukey's pairwise test to identify significant difference ($p < 0.05$) between the means of the experimental data.

# 3 Results and discussion

## *3.1 Spray-drying of jiotilla juice*

In this work, spray-drying (SD) of *jiotilla* juice with the DE-10 maltodextrin addition as carrier material was studied. Table 2 shows the regression coefficients ($\beta_{ij}$) for the proposed model (Eq. (5)) calculated for each response variable ($y_i$) and the studied SD conditions that significantly affected ($p < 0.05$) them. Before of an escalation process it is important to understand the phenomena that around the spray-drying. Thus, the effects of the process variables on $y_i$ were visualized and discussed from response surfaces generated for each fitted model (Figs. 1-3), and in according to the results of the statistical analysis (Table 2).

### *3.1.1 Effects on process yield*

The results analysis showed in the present research that only maltodextrin concentration significantly affected ($p < 0.05$) the process yields under the conditions evaluated (Table 2). The drying yield of *jiotilla* juice was in the range from 30.4% to 62.4% (Fig. 1 and Table 1). Process yield ≥ 50 % obtained in this work can be considered to be efficient (Koç and Dirim, 2018; Tontul and Topuz, 2017). Obón *et al.* (2009) reported an optimal yield of 58 % for *Opuntia stricta* fruit juice at 160 °C (Tin) and using glucose syrup (29-31 DE; 10 % w/v) as encapsulant agent, however the juice concentration (1.2 °Brix) was lowest than in the present work (11.7 ± 0.1 °Brix). Saénz *et al.* (2009) evaluated the spray-drying of an *Opuntia ficus-indica* fruit juice of highest concentration (14 °Brix), but they did not report the process yield. Drying yields obtained in this research were higher than Castro-Muñoz *et al.* (2015) and Otálora *et al.* (2015). Castro-Muñoz *et al.* (2015) reported values from 7.76 to 14.86 % for purple cactus pear (*Opuntia stricta*) clarified juice powders, which were dried at Tin from 110 to 140 °C and with gelatin-maltodextrin composite as the carrier material. Otálora *et al.* (2015) reported values of 16.4 and 25.4 % when maltodextrin 20-DE and maltodextrin/mucilage were used as carrier materials (Tin, 170 ± 2 °C; Tout, 98 ± 4 °C), during the spray-drying of *Opuntia ficus-indica* fruit juice. These authors used feed mixtures with total solids of 10 % (Castro-Muñoz *et al.*, 2015) and 30 % (Otálora *et al.*, 2015), approximately. The difference in the yield values can be attributed to the spray-drying conditions (inlet/outlet temperatures), juice composition, carrier material type, carrier/juice ratio, atomization speed or pressure, and/or solids content (Bazaria and Kumar, 2017; Islam *et al.*, 2021; Koç and Dirim, 2018; Vázquez-León *et al.*, 2021; Wang *et al.*, 2020).

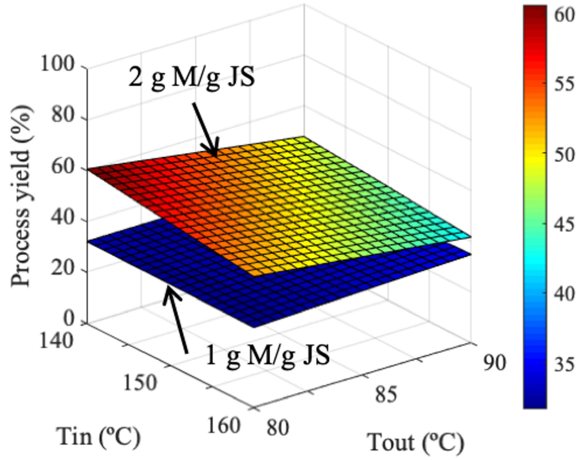


Figure 1. Spray drying process of jiotilla juice: effects on process yield.

Soto-Castro *et al.* (2019) reported yield values from 42.24 to 66.74 % when spray-drying the betalain rich extracts from *Escontria chiotilla* using mucilage as carrier material (Tin, 140 °C; Tout, 70 °C), similar results than the present research, however the total solids into feed mixtures were in the range of 1.2-1.3 %, approximately (according to the solids content of both mucilage and fruit pulp). In the present work was feasible the spray-drying of feed mixtures with total solids of 14.6-20.5 % (according to the amount of maltodextrin added and the *jiotilla* juice solids, view Table 1), under the conditions evaluated. This represents an advantage because higher solids content in feed mixture decreases the consumption of energy during spray-drying, due to the decrease in water content for evaporation (Shishir and Chen, 2017; Tontul and Topuz, 2017; Vázquez-León *et al.*, 2021). So, the maltodextrin 10-DE could be a carrier material feasible and competitive to spray-drying of *jiotilla* juice with high solids content or to other cactus fruits with similar composition.

The selection of a carrier material and its concentration doses is crucial to minimize stickiness problems and increase the process yield (Bazaria and Kumar, 2017; Vázquez-León *et al.*, 2021). Generally, the process yield increases when the carrier material content increases, such as was reported for sumac extract (Caliskan and Dirim, 2013) and *Sideritis stricta* extract (Nadeem *et al.*, 2011). Nadeem *et al.* (2011) reported that an increases in the carrier material amount from 0 to 3 g/100 g and 3 to 5 g/100 g tend to an increment of 217 % and 36 % of the process yield, respectively. Spray-drying of *jiotilla* juice without the addition of maltodextrin was carried out at 140 °C (Tin) and 80 °C (Tout) as preliminary test, however the process was not feasible because the product was totally adhered to the walls of the cyclone and the collection vessel. The product obtained from *jiotilla* juice under these conditions was rubbery and highly hygroscopic, which can be associated with low glass transition temperatures ($Tg$) of the fruit components, such as low molecular-weight sugars and organic acids, which led to stickiness problems (Islam *et al*., 2021; Tontul and Topuz, 2017; Vázquez-León *et al*., 2021; Wang *et al*., 2020). The highest yields (43.5-62.4 %) in this research were obtained when the maltodextrin proportion was increased from 1 to 2 g M/g JS (Fig. 1) into the feed mixture. The carrier materials as carbohydrate-based are added to the extracts and fruit juices to increase $Tg$ of the fed mixtures to spray dryers due to its high molecular weight (Bazaria and Kumar, 2017; Islam *et al*., 2021; Tontul and Topuz, 2017; Vázquez-León *et al*., 2021; Wang *et al*., 2020), so with the maltodextrin a higher $Tg$ could be obtained and therefore a higher process yield. Some feed mixtures with high sugar content tend to have low yields at high process temperatures, due to effects on the thermoplastic properties of the materials (León-Martínez *et al*., 2010; Otálora *et al*., 2015). León-Martínez *et al*. (2010) reported that Tin ≥ 170 ºC could reduce the process yield of mucilaginous products due that powder tendency to melt and adhere to the chamber wall. However, both Tin and Tout values evaluated in this work did not significantly affect ($p > 0.05$) the process yields (Table 2), which suggests that the doses of maltodextrin added are enough to ensure the thermoplastic stability of *jiotilla* juice during spray-drying under the temperatures studied. Moreover, a higher solid content in the feed mixture could increase the efficiency of the heat and mass transfer and assure a higher yield. Because a sample with a high solids content has a lower water mass to evaporate (Table 1), and therefore a lower impact of wet particles on the spray-dryer walls could occur (Arellano and Casas, 2003; Goyal *et al*., 2020; Shishir and Chen, 2017; Tontul and Topuz, 2017).

More studies are necessary to increase the process yield of spray-drying of *jiotilla* juice. For example, the process yields could increase with the addition of a flow regulator (e.g., silicon dioxides) (Vázquez-León *et al*., 2021) or carry out a mixture of the maltodextrin with other carrier materials (e.g., modified starches).

### *3.1.2 Effects on water activity*

Water activity ($a_w$) values from 0.20 to 0.40 ensure the stability of the dried products against autooxidation, lipid oxidation, browning and hydrolytic reactions, and enzymatic activity (Sun-Waterhouse and Waterhouse, 2015). The process variables studied did not show a significant effect ($p > 0.05$) on the $a_w$ values of *jiotilla* juice powders (Table 2); this finding can be attributed to that the operating conditions evaluated allowed the fast and equal removal of the moisture (Vázquez-León *et al*., 2021). Therefore, the response surface was not made. The $a_w$ of *jiotilla* juice powders was in the range from 0.194 ± 0.032 to 0.241 ± 0.072 (Table 1), thus can be considered as physical, microbiological and oxidatively stable (Ozkan *et al*., 2019) and also into the interval of industrially spray-dried products (~0.2) (Wang *et al*., 2020). The $a_w$ values of *jiotilla* juice powders observed in this work are similar than others spray-dried fruit juices. Quek *et al*. (2007) reported $a_w$ of 0.20-0.29 for watermelon powders, while Fazaeli *et al*. (2012) and Wang *et al*. (2020) found $a_w$ from 0.15 to 0.32 for black mulberry juice powders, and Cardona *et al*. (2023) reported $a_w$ values of 0.15 to 0.25 for pineapple powder. Castro-Muñoz *et al*. (2015) reported $a_w$ values of 0.19-0.29 for *Opuntia stricta* juice powders and Sun-Waterhouse and Waterhouse (2015) obtained kiwifruit powders with $a_w$ of 0.26-0.28. Soto-Castro *et al*. (2019) were not reported the $a_w$ values of the spray-dried betalain rich extracts from *Escontria chiotilla*.

### *3.1.3 Effects on powder color*

In juice powders and natural colorants, the color represents its quality and sensory attractiveness (Quek *et al*., 2007; Tze *et al*., 2012). ºHue value refers to the circular scale colors seen in the spectrum, C* indicates the color intensity, and L* is associated with the lightness (Jiménez-Aguilar *et al*., 2011). The color parameters L* and C* of the spray-dried *jiotilla* juice powders only were significantly affected ($p < 0.05$) by the maltodextrin concentration (Table 2). L* and C* values of the *jiotilla* juice powders were in the range from 44.06 ± 5.19 to 56.17 ± 1.89 and 35.36 ± 0.47 to 43.50 ± 1.70, respectively (Fig. 2). While ºHue values of the *jiotilla* juice powders were not significantly affected ($p > 0.05$) by any independent variable evaluated (Table 2). Therefore, the response surface for ºHue values was not made. ºHue parameter was in the range from 11.23º ± 1.85º to 13.14º ± 0.16º.

According to the ºHue values (11-13º), the color of the *jiotilla* juice powders obtained in the present work tend to red tone, which is the characteristic color of betalains from *jiotilla* fruits (Soriano-Santos *et al*., 2007; Soto-Castro *et al*., 2019) and purple cactus pear from *Opuntia stricta* (Castro-Muñoz *et al*., 2015) and *Opuntia ficus-indica* (Otálora *et al*., 2015). While Soto-Castro *et al*. (2019) reported a higher tendency to reddish yellow tonalities (ºHue values: 37-41º) for spray-dried betalain rich extracts from *Escontria chiotilla* when cactus mucilage was used as carrier material; the color measurements were carried out after 3 months of storage. The differences observed between both works could been associated with the fruit composition, storage time and the carrier material type. The maltodextrins at different DE values tend to increase the pigments retention in the spray-dried products, because change both thermoplastic and hygroscopic properties of the powders (Bazaria and Kumar, 2017; Lao and Giusti, 2017; Tontul and Topuz, 2017; Wang *et al*., 2020).

Table 2. Regression coefficients for each response variable ($\beta_{ij}$) of spray dried *Escontria chiotilla* fruit juice and $p$-values for the variables evaluated.

| RC[a] | Process yield | | $a_w$ | | L* | | C* | | °Hue | | Betacyanins content | | Betaxanthins content | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | RC-value | p-value | RC-value | p-value | RC-value | p-value | RC-value | p-value | RC-value | p-value | RC-value | p-value | RC-value | p-value |
| $\beta_{i0}$ | 41.61 | **<0.01** | 0.21 | **<0.01** | 49.82 | **<0.01** | 39.15 | **<0.01** | 12.11 | **<0.01** | 103.81 | **<0.01** | 217.44 | **<0.01** |
| $\beta_{i1}$ | -1.69 | 0.58 | 0 | 0.8 | 0 | 1 | 0.03 | 0.97 | -0.02 | 0.96 | -3.23 | 0.11 | -6.27 | 0.16 |
| $\beta_{i2}$ | -2.43 | 0.43 | 0 | 0.72 | 2.29 | 0.16 | -0.5 | 0.52 | 0.39 | 0.33 | 1.82 | 0.35 | 4.37 | 0.32 |
| $\beta_{i3}$ | 8.81 | **0.02** | -0.01 | 0.42 | 5.22 | **0.01** | -2.64 | **0.01** | -0.07 | 0.86 | -11.67 | **<0.01** | -24.04 | **<0.01** |
| $\beta_{i12}$ | 0.69 | 0.82 | 0.01 | 0.31 | 1.19 | 0.45 | -0.29 | 0.7 | -0.42 | 0.29 | -3.74 | 0.07 | -8.42 | 0.07 |
| $\beta_{i13}$ | -2.11 | 0.49 | 0.01 | 0.39 | 0.36 | 0.82 | 0.36 | 0.64 | -0.01 | 0.98 | 0.52 | 0.79 | 2.22 | 0.61 |
| $\beta_{i23}$ | -3.27 | 0.29 | 0.01 | 0.45 | -1.24 | 0.43 | 0.35 | 0.65 | 0.24 | 0.53 | 0.92 | 0.63 | 2.61 | 0.54 |

[a]Regression coefficients ($\beta_{ij}$) of the proposed model (Eq. (5)). Subscripts denote: 1, Tin; 2, Tout; 3, g M/g JS; i, response variable.
The terms were considered statistically significant at $p < 0.05$.

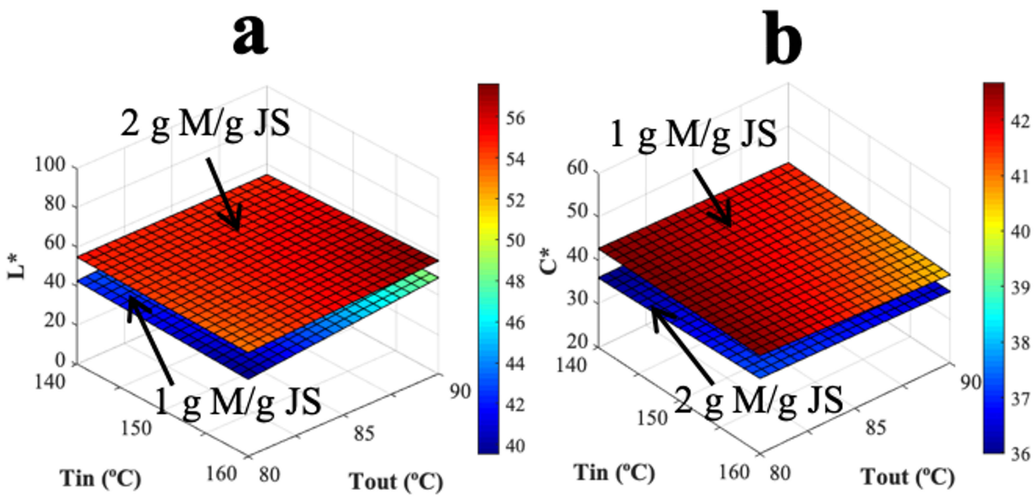


Figure 2. Effect of spray drying process on color parameters of jiotilla juice powders: luminosity (L*) and chroma (C*).

In this work, an increase in the maltodextrin amount in the feed mixture tended to increase the L* values, but the C* values were decreased (Table 2 and Fig. 2). The color of the *jiotilla* juice powders became lighter and less saturated at higher maltodextrin concentration. In the spray drying of the mountain tea extract similar results were obtained (Nadeem *et al*., 2011). Tontul and Topuz (2017) suggested that these results could be associated with a dilution effect of the fed solution solids with the carrier material (L* and C* values of maltodextrin 10-DE used in this work: 90.27 and 4.02, respectively).

Generally, the highest drying temperatures tend to have a higher loss of color due to the non-enzymatic browning reactions (Shishir and Chen, 2017; Tontul and Topuz, 2017). In the present research, both Tin and Tout values used did not significantly affect ($p >$ 0.05) the color parameters (Table 2), which suggests that the amount of maltodextrin added tend to preserve the characteristic color of *jiotilla* fruit juice during spray-drying, under conditions evaluated. Bazaria and Kumar (2017) used maltodextrins with 10 and 20-DE and arabic gum as carrier materials for the beetroot juice. The authors reported that the reason of color retention with these materials might be the formation of capsules that improve the stability of the pigments during the spray drying and storage.

### *3.1.4 Effects on betaxanthins and betacyanins content*

Characterization of *jiotilla* juice before the spray-drying process was carried out in terms of betaxanthins and betacyanins content. The total betalains content was 364.7 mg/100 g of *jiotilla* juice, where 118.7 ± 0.2 mg were betacyanins (BC) and 245.7 ± 0.2 mg were betaxanthins (BX). These values were higher than those reported by Saénz *et al*. (2009), who reported values of 41.05 mg BC/100 g and 18.65 mg BX/100 g, for cactus pear (*Opuntia ficus-indica*); and by Soriano-Santos *et al*. (2007), who collected *jiotilla* fruits in the Mixteca Baja (Oaxaca, México) region and obtained 89 ± 6.5 mg BC/kg pulp and 119 ± 3.1 mg BX/kg pulp. Castro-Muñoz *et al*. (2015) also reported a lower betalains content of 37.73 ± 0.34 mg/L for purple cactus pear juice from *Opuntia stricta*. The differences observed in the betalains content can be associated with different agroclimatic factors (variety, maturity, climatic conditions, production soil) (Celli and Brooks, 2017; Saénz *et al*., 2009). However, the differences also may be due to the sample preparation and the techniques used for the identification and quantification of these compounds. The authors cited used the spectrophotometric method as a feasible assay due to that both compounds have different absorption maxima. When the amino acid of the BX is

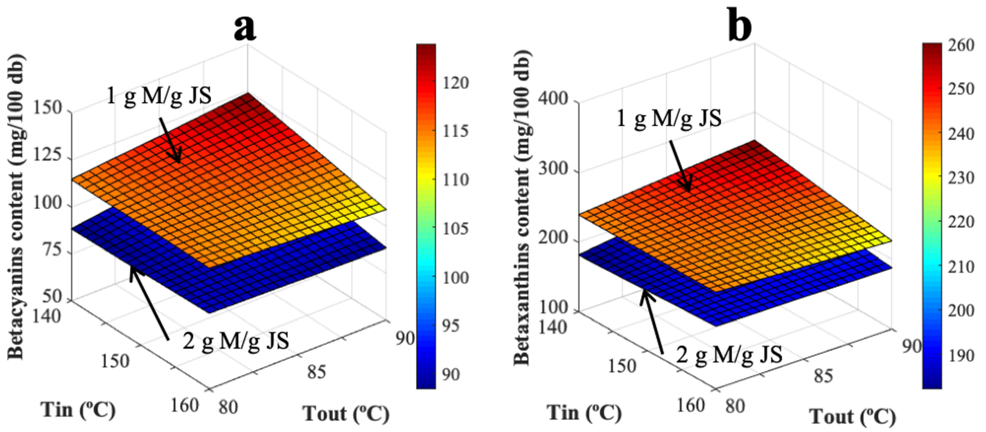


Figure 3. Spray drying process of jiotilla juice: effects on betacyanins (a) and betaxanthins (b) content.

replaced by an auxochromic indole group, at the end of the 1,7-diazaheptamethine conjugated system the BC are obtained. This change in the functional group alters the visible absorption maximum (Marañón-Ruiz *et al*., 2011). Despite thus, the HPLC analysis is the tool more recommended for confirmed the results obtained because it separates the compounds and avoid the overestimation. An example of this analysis could be the methodology proposed by Soriano-Santos *et al*. (2007).

Once the *jiotilla* juice was characterized, it was spray dried according to Table 1. The BC and BX content in the *jiotilla* juice powders were from 89.4 ± 8.6 to 124.9 ± 3.1 mg BC/100 g dry basis (db) and from 185.1 ± 14.8 to 263.7.3 ± 19.1 mg BX/100 g db, respectively (Table 1). Both BC and BX content only were significantly affected ($p < 0.05$) by the maltodextrin concentration during the spray-drying of *jiotilla* juice (Table 2). An increase in the concentration of maltodextrin caused a decrease in betalains (BC and BX) (Fig. 3 and Table 1). It can be associated with the dilution of *jiotilla* juice solids in maltodextrin solids: the *jiotilla* juice solids represent approximately 50 % (1 g M/g JS) and 33.3 % (2 g M/g JS) of the total dry weight in the spray-dried products obtained. Similar results were reported by Tze *et al*. (2012) and Mishra *et al*. (2014) for total betalains content and total phenolics content of *Hylocereus polyrhizus* and *Emblica officinalis* juice powders, respectively.

In the Figure 3 can be observed a negative tendency in the betalains content when the Tin increase, mainly at lower maltodextrin content (1 g M/g JS). However, the statistical analysis shown that both Tin and Tout values evaluated did not have a significant effect ($p > 0.05$) on the BC and BX content (Table 2). Despite that, the total betalains content obtained in this work for *jiotilla* powders (274.6-388.2 mg/100 g db) was higher than reported by Castro-Muñoz *et al*. (2015), who found 11-35 mg betalains/100 g db for spray-dried juice of *Opuntia stricta* fruits; and by Otálora *et al*. (2015), reported values of 49.5-49.7 mg betalains/100 g db for spray-dried purple cactus pear from *Opuntia ficus-indica*. Bazaria and Kumar (2017) carried out the spray-drying of beetroot juice using maltodextrins (10 and 20-DE), gum arabic and mixtures of them; its results showed a lower concentration of betalains (160-208.06 mg/100 g db) compared to the results obtained in the present work for *jiotilla* powders (258.0-402.3 mg/100 g db), which suggests a greater efficiency in the retention of betalains in addition to being a more effective method because these values were obtained using only maltodextrin. The differences observed can be associated with betalains source, initial betalains content, spray-drying conditions, carrier material type and carrier/betalains ratios (Caliskan and Dirim, 2013; Castro-Muñoz *et al*., 2015; Fazaeli *et al*., 2012; Ravichandran *et al*., 2014; Vázquez-León *et al*., 2021). Both BC and BX values of *jiotilla* juice powders obtained in this research shown values range of spray-dried betalain rich extracts from *Escontria chiotilla* reported by Soto-Castro *et al*. (2019). These authors used cactus mucilage (*Opuntia ficus indica*) as carrier material and reported total betalains content from 308.36 to 392.70 mg/100 g db, where 135.80-170.80 mg were BC and 172.56-221.86 mg were BX. A considerable loss of betalains also was observed by all these authors (Bazaria and Kumar, 2017; Castro-Muñoz *et al*., 2015; Otálora *et al*., 2015; Soto-Castro *et al*., 2019) when the spray-drying of betalain rich fruits was carried out, which could be attributed to the degradation of its molecular structure by the thermal process (Celli and Brooks, 2017). Maltodextrin is used as a wall material in different food products to increases its shelf life, and also has been used for betalain-rich powders from cactus fruits (Celli and Brooks, 2017; Otálora *et al*., 2015) and beetroot (Bazaria and Kumar, 2017; Ravichandran *et al*., 2014). Different authors inform that maltodextrin during spray-drying tended to form a wall around bioactive compounds, that protect to the pigments from oxygen and moisture, while that the juice solids without maltodextrin are exposed to the external factors that affect its physicochemical stability. Ravichandran *et al.* (2014) evaluated different wall materials (e.g., pectin, guar gum, xanthan gum, Arabic gum) in combination with maltodextrin during the encapsulation of betalains from beetroots by spray drying. The results of these authors indicated that the mixtures of the wall materials evaluated tend to increase the stability of betalains. Spray-drying process of *jiotilla* fruits juice with maltodextrin gives a feasible process yield and a significant betalains content in the spray-dried product, so it is a good method of large-scale production in a continuous way. However, more studies are necessary to characterize the capsule formation and technical feasibly of possible mixtures of maltodextrin with other polymers to spray-drying of *jiotilla* fruits juice.

## 3.2 *Characterization of jiotilla juice powders*

In order to evaluate the particle characteristics, the *jiotilla* juice powder obtained with the spray-drying conditions that allowed a process yield > 50 % was selected (Figure 1 and Table 1): 2.0 g M/g JS, 140 ºC (Tin) and 80 ºC (Tout). The process yield obtained with this operation conditions show a significant statistical difference ($p < 0.05$) than others mean (Table 1). However, is necessary to evaluate other carrier materials to improve the performance of the spray drying of *jiotilla* juice. The particle characterization results are shown in Table 3. Analyses were done in triplicate.

Table 3. Physicochemical properties of jiotilla (*Escontria chiotilla*) juice powders.

| **Physicochemical properties** | **Values** |
|---|---|
| **Color Lab** | |
| **Bulk density (g cm$^{-3}$)** | 0.39 ± 0.01 |
| **Tapped density (g cm$^{-3}$)** | 0.48 ± 0.01 |
| **Hausner ratio** | 1.25 ± 0.01 |
| **Carr index** | 20.00 ± 0.08 |
| **Solubility (%)** | 92.16 ± 0.19 |
| **Hygroscopicity (g $H_2O$ 100 g db$^{-1}$)** | 16.28 ± 0.85 |

The values are the mean ± standard deviation ($n = 3$). Spray drying conditions: Tin, 140 °C; Tout, 80 °C; 2 g M/g JS.

### 3.2.1 *Bulk and tapped densities*

A factor associated to the packaging, transport and storage of powders is the density (Fernandes *et al.*, 2014; Tontul and Topuz, 2017; Tze *et al.*, 2012). Both average bulk density ($\rho_b$) and tapped density ($\rho_t$) for *jiotilla* juice powder are showed in Table 3. These values were similar to the reported by Fazaeli *et al.* (2012) and Wang *et al.* (2020) who reported $\rho_b$ = 0.35-0.55 and 0.26-0.48 g cm$^{-3}$ for black mulberry powders, respectively; and slightly higher than those reported for soymilk powders ($\rho_b$ = 0.21-0.32 g cm$^{-3}$ and $\rho_t$ =0.35-0.47 g cm$^{-3}$) (Jinapong *et al.*, 2008), rosemary essential oil microencapsulated ($\rho_b$ =0.28-0.34 g cm$^{-3}$ and $\rho_t$ =0.38-0.48 g cm$^{-3}$) (Fernandes *et al.*, 2014) and for pitaya juice powders ($\rho_b$ =0.29-0.34 g cm$^{-3}$) (Tze *et al.*, 2012). The differences could be attributed mainly to the fruit juice physicochemical properties, operating parameters, and carrier material type, that could affect to the particle size and $\rho_b$ of powders (Tontul and Topuz, 2017; Wang *et al.*, 2020). High $\rho_b$ is desirable to reduce shipping and packaging costs. Furthermore, at higher $\rho_b$ should has a more free-flowing powder as the interparticle forces between particles become weaker (Abdullah and Geldart, 1999; Tontul and Topuz, 2017).

### 3.2.2 *Flow properties*

The Carr index (*CI*) and Hausner ratio (*HR*) of the *jiotilla* juice powders obtained are presented in Table 3. The CI and HR values of dried *jiotilla* particles indicates they have a cohesiveness intermediate and a flowability from fair to good according to the classification of powders flowability and cohesiveness reported by Jinapong *et al.* (2008). While Tze *et al.* (2012) reported pitaya juice powders with poor flowability (*CI*, 39.04-48.16; HR, 1.64-1.93). The flow properties could be associated to the particle size and the $\rho_b$ of the powders. Tze *et al.* (2012) point out that the lower the $\rho_b$ is, the more cohesive a powder will be and the greater the compressibility and the difficulty of fluidity. Jinapong *et al.* (2008) reported a low cohesiveness and a good flowability for soymilk powders as resulted of largest particle size.

### 3.2.3 *Solubility and hygroscopicity*

To be practical, the juice powders should be exhibit an appropriate solubility in water (Jayasundera *et al.*, 2011). The *jiotilla* powders showed a solubility of 92.16%, it is higher than the values reported by Fazaeli *et al.* (2012), Cano-Chauca *et al.* (2005) and Rivas Reyes (2016) for black mulberry (*Morus nigra*), mango (*Mangifera indica* L.) and custard apple (*Annona cherimola* Mill) juice powders, who reported solubilities of 75-87%, 77-90% and 75-79% respectively. These differences can be attributed to the dextrose equivalent and the maltodextrin amount used as wall material. Cardona *et al.* (2023) observed that the solubility of the pineapple powder depended on the maltodextrin amount into the product. The powders hygroscopicity tend to reduce at highest values of maltodextrin (Suzihaque *et al*., 2015). The lowest hygroscopicity confirm the positive effect of the carrier material because its solubility increase.

Hygroscopicity should be a factor to consider for the storage of powders (Nadeau and Puiggali, 1995). The *jiotilla* juice powder obtained a hygroscopicity value of 16.28 g of water/100 g db, this value is lower than that found by Rodríguez-Hernández *et al*. (2005) and Castro-Muñoz *et al*. (2015) during the reconstitution of cactus pear juice powder of *Opuntia streptacantha* (36.30 to 48.93 g water/100 g db) and *Opuntia stricta* (19.34-28.13 g water/100 g db), respectively. But was slightly larger than reported by Tonon *et al*. (2008) and Rivas Reyes (2016) for acai (*Euterpe oleracea*) (13-15 g water/100 g db) and custard apple (4-8 g water/100 g db) juices, respectively. These differences could be due to the amount of sugars present in the fruit (Sebhatu *et al*., 1994), concentration and type of carrier material (Rodríguez-Hernández *et al*., 2005) and particle size of the powder (Nadeau and Puiggali, 1995).

## Conclusions

Betalains rich juice powders, physico-chemically stable, were obtained by spray-drying of *Escontria chiotilla* fruit juice with maltodextrin 10-DE. Both process yields and the properties of the *jiotilla* juice powders only were found to depend on the amount of maltodextrin added to *jiotilla* juice; under spray-drying conditions evaluated. Spray-drying of betalains rich juice with maltodextrin as carrier material may be a feasible method to obtain products with color tendency red, an intermediate cohesiveness, a flowability from fair to good, a high solubility, a low hygroscopicity and a final high content of betacyanins and betaxanthins. This technology could be scalable and highly suitable for producing betalains rich powders. However other carrier materials should be evaluated to improve the process yield. *Escontria chiotilla* fruit juice is a feasible source of betalains (betacyanins and betaxanthins) that could be used as a natural colorant. More studies are necessary to evaluate the physicochemical stability of *jiotilla* powders during storage and to definite both technical and economic feasibility of this conservation strategy.

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