Igual Ramo, M.; Ramires, S.; Mosquera, LH.; Martínez Navarrete, N. (2014). Optimization of spray-drying conditions for lulo (Solanum quitoense L.) pulp. Powder Technology. 256:233-238. doi:10.1016/j.powtec.2014.02.003.

Optimization of spray drying conditions for lulo (*Solanum quitoense L.*) pulp

Igual, M [a]., Ramires, S [b]., Mosquera. L.H[c]., Martínez-Navarrete, N [a,*]

[a] *Universitat Politècnica de València, Food Technology Department, Food Investigation and Innovation Group, Camino de Vera s/n, 46022 Valencia, Spain*

[b] *Universidade Católica Portuguesa, Escola Superior de Biotecnologia, Rua Dr. António Bernardino de Almeida 4200-072 Porto, Portugal*

[c] *Grupo de Investigación en Valoración y Aprovechamiento de la Biodiversidad, Universidad Tecnológica del Chocó, Carrera 22, No. 18 B – 10 Quibdo, Colombia.*

Abstract

The spray drying of lulo was optimized by using the central composite design of the response surface methodology, to study the effect of inlet air temperature (120–180 °C), arabic gum concentration (0-10% w/w), and maltodextrin DE16.5-19.5 concentration (0-10% w/w) on some product and process aspects. Arabic gum and maltodextrin, more than inlet air temperature, improved the product yield, reduced the hygroscopicity and the water content of the obtained powder, and contributed to the retention of its nutritive and functional properties through an increase in ascorbic acid, vitamin C, total phenol and total flavonoid content and antioxidant capacity. Significant ($p<0.05$) response surface models were obtained in every case, with the linear terms of solute concentration being the factors that affected the response variables most significantly. The overall optimum spray drying conditions for obtaining lulo powder were 125 °C inlet air temperature, 3% (w/w) arabic gum, and 13.4% (w/w) maltodextrin DE16.5-19.5.

---

[*] Corresponding author: Tel.: +34 96 3879362; fax: +34 96 387 73 69. E-mail address: nmartin@tal.upv.es (Martínez- Navarrete, N).

Keywords: lulo powder, functional value, hygroscopicity, vitamin C, antioxidant activity, phenolic compounds

1. Introduction

Lulo (*Solanum quitoense L.*) is cultivated on a very small scale and, in local areas, it is put to traditional uses due to its highly nutritive value and/or medicinal properties [1]. Lulo, also known as "naranjilla" belongs to the huge Solanaceae family [2]. This 1-2.5 m high shrubby perennial is native to the Andes. The geographical distribution of *Solanum quitoense* stretches from Venezuela to Peru. It is generally cultivated at a height of between 1,000 and 1,900 m above sea level [3, 4]. The naranjilla plant produces a spherical fruit with a diameter that ranges from 3 to 8 cm [5]. The skin (exocarp) is orange and it is usually covered with short, prickly, stiff hairs (or "spines") that easily rub off. The skin is peeled and discarded in food preparation. The fruit's internal structure is similar to that of the tomato: the yellow-green flesh (mesocarp and endocarp) forms four compartments separated by membranous partitions and filled with translucent green or yellowish pulp, very juicy and acid [6]. The naranjilla fruit is rarely eaten fresh mainly due to its acidity, but is most commonly used to make flavored drinks, preserves and desserts. The fresh juice is also processed into frozen concentrates and can be fermented to make wine [5, 6]. The fruit appears to have considerable nutritional potential due to its high content in vitamins, proteins and minerals [6, 7].

Spray drying is a well-established and widely used method for transforming a wide range of liquid food products into powder form. The process involves spraying finely atomized solutions into a chamber where hot dry air rapidly evaporates the solution leaving the spray-dried particles. Spray-dried powders can be stored at room temperature for prolonged periods without compromising the powder's stability [8]. Powders are cheaper to transport and easier to handle in manufacturing plants. Spray-dried powders are economical to produce compared to other processes, such as freeze-drying [9]. Spray drying has many applications, particularly in the food, pharmaceutical and agrochemical industries [10-13]. The conversion of high value food materials, such as fruit and vegetable extracts, into particulate form is not easy due to the presence of a high proportion of low molecular weight sugars in their composition [13], which lead to the problem of stickiness [14, 15]. The particles stick to one another, to the dryer and to cyclone walls and remain there, forming thick wall deposits, while very little product comes out at the dryer's exit. This might lead to low product yield and operating problems [10, 16]. In general, the stickiness causes considerable economic loss and limits the application of spray drying on foods as well as on pharmaceutical materials [11, 17]. In order to reduce stickiness, different solutes have been used as carriers and coating agents for the spray drying [18-23]. Some examples of these are arabic gum, maltodextrins, starches, gelatin, methyl cellulose, gum tragacanth, alginates, pectin, silicon dioxide, tricalcium phosphate, glycerol monostearate and mixtures of some of them. Of these additives, maltodextrin offers a good compromise between cost and effectiveness. It has been found that it contributes to the retention of some food properties, such as nutrients, colour and flavour, during spray drying and storage [14]. There are numerous reports on the spray drying of different fruits with maltodextrin: aril fruit [24], cactus pear [14, 25, 26], watermelon [27], black carrot [28], pineapple [29] and mango [30]. Nevertheless, arabic gum has been proved to be more effective than maltodextrin at improving the handling of the borojó powder [31]. On the other hand, the feed flow rate, the inlet and outlet air temperatures, atomizer speed, feed concentration, feed temperature and inlet air flow rate are important factors that have to be controlled in a spray drying process [32]. Among them, the inlet and/or outlet air temperatures are the most effective factors in spray drying to be optimized [33]. The aim of this work was to optimize the spray drying conditions of lulo (*Solanum quitoense L.*) pulp in order to favour the process yield and to produce a stable powder with high nutritional and functional values. The inlet air temperature and maltodextrin and arabic gum concentration were considered as the process variables.

2. Materials and methods

2.1. Raw material

This study was carried out with *f*rozen lulo *(Solanum quitoense L.)* pulp supplied by Jota Jota Alimentos Global S.L. (Valencia, Spain). Maltodextrin DE16.5-19.5 (MD) and arabic gum (AG) added to the pulp were purchased from Sigma-Aldrich (USA).

2.2. Preparation of feed mixture and spray drying conditions

The frozen lulo pulp was thawed and mixed with a water solution containing MD and/or AG. Solutes were added according to the generated experimental designobtained from the response surface methodology (RSM) (Table 1) and commented on below. To incorporate the solutes, a solution in water was previously prepared. The amount of each one of these solutions was 200 g, which were added to 200 g of lulo pulp. ºBrix of lulo pulp and the mixture with the solute solution were measured. The mixture was stirred for 30 min until homogeneity was reached. After that, ºBrix were measured with a refractometer at 20 ºC (Zeiss, ATAGO model NAR-3T, Japan), it was fed into a Büchi B-290 (Switzerland) mini spray dryer with the following operating conditions: aspirator rate 90% (35 m^3/h); atomisation air rotameter 40 mm (473 L/h) with a co-current flow; pump rate 30% (9 mL/min). Drying air inlet temperature was varied according to experimental design (Table 1). After the completion of the experiment and when the air inlet temperature fell below 50 ºC, the samples were collected from the product collection vessel.

2.3. Experimental Design

For this study, RSM was used to evaluate the effect of three process independent variables on eleven response variables mainly related to the profitability of the process and the quality of the powder. As independent variables, the inlet air temperature (x_1, 120– 180 °C) and the concentration of arabic gum (x_2, 0-10 g AG/100g lulo pulp) and maltodextrin (DE16.5-19.5) (x_3, 0-10 g MD/100g lulo pulp) were selected. The response variables taken into consideration were those of outlet temperature (Y_1), product yield (Y_2), drying ratio (Y_3), productivity (Y_4), water content (Y_5), hygroscopicity (Y_6), ascorbic acid content (Y_7), vitamin C content (Y_8), phenolic content (Y_9), flavonoid content (Y_{10}) and antioxidant capacity (Y_{11}) of spray-dried lulo powder. Twenty-three experimental runs were generated based on the corresponding rotatable and orthogonal central composite design (Table 1). The experiments were randomized.

2.4. Analysis of response variables

Y_2 was defined as the ratio of the mass of solutes present in the lulo powder obtained at the end of each spray drying period, to the mass of solutes present in the mixture prior to spray drying [34]. Y_3 and Y_4 were calculated for spray drying by using Cai and Corke [35] but with a slight modification. The drying ratio and the productivity (g/h) were calculated by equations (1) (powder solid content / feed solid content) and (2), respectively.

$$Y_3 = \frac{\left(X_w^i + 1\right)}{\left(X_w^f + 1\right)} \quad (1)$$

where X_w^i is the mixture feed moisture (dry basis), and X_w^f is the powder moisture (dry basis).

$$Y_4 = \frac{Fr}{Y_3} \quad (2)$$

where Fr is the feed rate (g/h), calculated from the mass of mixture feed (g) and the process time (h).

The mass fraction of water (g/100g) was obtained by vacuum drying the samples in a vacuum oven (Vaciotem, J.P. Selecta, Spain) at 60 ºC ± 1 ºC under a pressure of <100 mm Hg until constant weight. For hygroscopicity [35], samples (about 2 g in a Petri dish) of each powder were placed at 25 °C in an airtight plastic container containing a Na_2SO_4 saturated solution (81% RH) at the bottom. After one week, each sample was weighed and hygroscopicity was expressed as g of water gained per 100 g dry solids.

Ascorbic acid (AA) and total vitamin C (ascorbic + dehydroascorbic acids) were determined by HPLC (Jasco, Italy). To determine the ascorbic acid, a 1 g sample was extracted with 9 mL 0.1% oxalic acid for 3 min [36] and immediately filtered through a 0.45 μm membrane filter before injection. The procedure employed to determine total vitamin C was the reduction of dehydroascorbic acid to ascorbic acid, using DL-dithiothreitol as the reductant reagent [37]. A 0.5 mL aliquot sample was taken to react with 2 mL of a 20 g/L dithiothreitol solution for 2 h at room temperature and in darkness. Afterwards, the same procedure as that used for the ascorbic acid method was performed. The HPLC conditions were: Ultrabase-C18, 5 μm (4.6x250 mm) column (Análisis Vínicos, Spain); mobile phase 0.1 % oxalic acid, volume injection 20 μL, flow rate 1mL/min, detection at 243 nm and at 25 ºC. AA standard solution (Panreac, Spain) was prepared.

The total quantity of phenols (TP) was analysed by using the method reported by Benzie & Strain [38] based on the Folin-Ciocalteu method, which involves the reduction of the reagent by phenolic compounds with the concomitant formation of a blue complex. Total flavonoids (TF) were measured spectrophotometrically, following the method described by Djeridane et al., [39] based on the formation of a flavonoids-aluminium complex. For the extraction of TP and TF, 35 g of the sample were homogenized (T25D Ultra-turrax, IKA, Germany) for 5 min with 40 mL of methanol, 10 mL of HCl (6 N) and NaF (2 mM) to prevent phenolic degradation caused by polyphenol oxidase action; the homogenate was centrifuged at 10,000 rpm, 10 min, 4 ºC. For TP quantification, 15 mL of distilled water and 1.25 mL of Folin Ciocalteu reagent (Sigma-Aldrich, Germany) were added to 250 μL of the supernatant. The samples were mixed and allowed to stand for 8 min in darkness before 3.75 mL of 7.5 % sodium carbonate aqueous solution was added. Water was added to adjust the final volume to 25 mL. Samples were allowed to stand for 2 h at room temperature before absorbance was measured at 765 nm in a UV-visible spectrophotometer (Thermo Electron Corporation, USA). The total phenolic content was expressed as mg of gallic acid equivalents (GAE) per gram of sample, using a standard curve range of 0-800 mg of gallic acid (Sigma-Aldrich, Germany)/mL. For TF quantification, 1 mL of the extract was mixed with 1 mL of 20g/L $AlCl_3$ methanolic solution. After incubation at room temperature for 30 min in darkness, the absorbance of the reaction mixture was measured at 430 nm using the aforementioned spectrophotometer. The total content in flavonoids was expressed as mg of rutin equivalents (RE) per gram of sample, using a standard curve range of 0-50 mg of rutin (Sigma-Aldrich, Germany)/L.

Antioxidant capacity (AOC) was assessed using the free radical scavenging activity of the samples evaluated with the stable radical DPPH [37]. Briefly, the samples were homogenized and centrifuged (Selecta Medifriger-BL, Spain) at 10,000 rpm for 10 min at 4 °C. 0.1 mL of supernatant diluted in methanol was added to 3.9 mL of DPPH diluted in methanol (0.030 g/L, Sigma-Aldrich, Germany). At 25 ºC, the same spectrophotometer mentioned before was used to measure the absorbance at 515 nm at 0.25 min intervals until the reaction reached the steady state. Appropriately diluted samples were used on the day of preparation. The percentage of DPPH was calculated following equation (3):

$$\%\,\mathrm{DPPH} = \frac{(A_{control} - A_{sample})}{A_{control}} \times 100 \tag{3}$$

where $A_{control}$ is the absorbance of the control (initial time) and A_{sample} the absorbance of the sample at the steady state.

The final results were expressed as milimole trolox equivalents (TE) per 100 grams (mmol TE/100 g) using a trolox calibration curve in the range 6.25-150 mM (Sigma-Aldrich, Germany).

Every analysis of the Y_5 to Y_{11} variables was carried out in triplicate. To characterize the lulo pulp, the results of Y_7 to Y_{11} were referred to 100 g of lulo pulp, while for the statistical study, all of them were expressed as mg of each compound/100 g total lulo solids (TLS).

2.5. Statistical analysis

An analysis of variance and a regression surface analysis were conducted to fit a regression relationship relating the experimental data to the independent variables. The generalized polynomial model proposed for the prediction of the response variables as a function of the independent variables was that given by equation (4):

$$Y_i = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x^2_1 + \beta_{22} x^2_2 + \beta_{33} x^2_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \qquad (4)$$

where Y_i was the response value predicted by the model; β_0 was a constant; β_1, β_2, and β_3 were the regression coefficients for the linear effects; β_{11}, β_{22}, and β_{33} were those for the quadratic effects; and β_{12}, β_{13}, and β_{23} were those which included interaction effects. In this model, x_1, x_2, and x_3 were the independent variables. Only the model terms found to be statistically significant ($p<0.05$) were included in the final reduced model. The terms which were statistically non-significant ($p>0.05$) were dropped from the initial models, and the experimental data were refitted only to the significant ($p<0.05$) independent variable effects to obtain the final reduced model [40]. The fact that none of the selected final models provided a significant lack of fit ($p>0.05$) confirmed the suitability of the fitted model and the non-significance of the Durbin-Watson statistic proved that there was no significant autocorrelation in the residuals. The goodness of the fit of the final reduced models to the experimental data was evaluated from the adjusted coefficient of determination (R^2_{adj}) and the standard error (SE) between the predicted and experimental values.

In the present study, for multiple response optimization, a response optimizer was used to determine the combination of input variable settings that jointly optimized the response of a set of variables. Through this optimization procedure, a combined level of the considered spray drying independent variables was obtained to produce a spray-dried powder with the most desirable powder properties (i.e., the highest content of ascorbic acid, vitamin C, phenols flavonoids, the greatest antioxidant capacity, product yield and productivity and the lowest outlet temperature, drying ratio, water content and hygroscopicity value).

The correlation between the antioxidant activity and every studied bioactive component with a 95% significance level was also analysed.

All statistical analyses were performed using Statgraphics Plus 5.1 [41].

3. Results and Discussion

3.1. *Lulo (Solanum quitoense L.) pulp characterization*

The mean values (with standard deviation in brackets) of pH, ºBrix and water content of lulo pulp were 3.29 (0.02), 8.5 (0.2) and 90.3 (1.2) g/100g, respectively. The soluble solids consist of about 7.5 g sugars/100g lulo pulp and 1g organic acids /100 g lulo pulp (data not shown). The non-dissolved material (1.2 %) is expected to be non-soluble carbohydrates and some lipids. The total phenol content was 81.1 (1.6) mg GAE/ 100 g lulo pulp. Within the group of phenolic compounds, the total flavonoid content in lulo pulp was 16.16 (0.06) mg RE / 100g. The vitamin C content was 120 (4) mg /100g and the value for ascorbic acid was 61.4 (1.5) mg/100g). These results coincide with the values obtained for naranjilla by other authors [1, 6, 42].

3.2. *Response Surface Model*

The experimental results obtained for each response variable is shown in Table 1. The final reduced models relating each response variable with the independent variables are shown in Table 2. The results indicated the significance ($p<0.05$) of the response surface model, with high coefficients of determination values ranging from 0.74 to 0.97. Thus, more than 74% of the response variation may be accurately explained as a function of the three independent spray drying variables selected. The adequacy of the response surface equation was checked by the comparison of experimental and predicted values (data not shown).

The outlet temperature ranged between 67 to 119 ºC and it was mainly affected ($p<0.05$) by the inlet temperature with a positive linear and a negative quadratic effect. Moreover, a negative quadratic effect of maltodextrin concentration on Y_1 was observed (Table 2). If the results of Y_1 are observed (Table 1), it may be seen that the lower the inlet temperature, the lower the outlet temperature. Lulo pulp contains sugars, which make the spray drying process difficult, mainly due to the basic physical characteristics of the low molecular weight sugars present in fruits, essentially sucrose, glucose and fructose. Moreover, the presence of organic acids, such as tartaric, malic, and citric acid, also contributes to the problem of stickiness in the powder [10]. In this work, it was extremely difficult to obtain powder at the exit of the dryer in samples without added high molecular weight solutes and large deposits were formed on the main chamber and cyclone walls. For example, Table 1 shows the lower/lowest values of product yield for these samples (runs 3 and 14). The addition of high molecular weight solutes, such as arabic gum and maltodextrin, prior to spray drying was necessary in order to obtain powders. Y_2 was positively affected by the concentration of arabic gum and maltodextrin. Despite there being a negative quadratic effect of these solutes and of their interaction, an increase in the solute concentration in lulo pulp improved the product yield in spray drying.

The drying ratio decreased linearly when the concentration of any of the added solutes rose, while the productivity mainly increased when there was a rise in the concentration of arabic gum.

Generally, food powders with lower hygroscopicity and water content are considered a good powdered product. Goula and Adamopoulos [43] suggested that adding maltodextrin decreased powder hygroscopicity. In the present study, the hygroscopicity values of spray-dried lulo powders showed values between 80 and 114%, which decreased to 35–60% when the considered solutes were added (Table 1). The hygroscopicity of lulo powder was decreased ($p<0.05$) due to an increase in the concentration of both arabic gum and maltodextrin (Table 2). The lower degree of hygroscopicity of lulo powders when the solutes were added could be related to the less hygroscopic nature of maltodextrin and arabic gum. Similar observations were reported by other researchers [14, 22, 31, 35, 44]. In general, the hygroscopicity values of samples with solutes were similar to those obtained by Rodriguez-Hernandez et al. [14] and Cai and Corke [35] in their studies on spray-dried cactus pear juice powder (36–49%) and spray-dried Amaranthus powder (45–50%), respectively. The inlet air temperature also influenced the hygroscopicity of the powder. Increasing the inlet temperature led to a lower degree of powder hygroscopicity. A similar observation was reported by Goula and Adamopoulos [43, 44]; Moreira et al. [45], and Jaya and Das [22]. However, in the final model, this factor had a less significant effect than the incorporation of solutes (Table 2). Moreira et al. [45] reported that the higher degree of hygroscopicity of the powders produced at lower temperatures seems to be related to their higher water content. Water content has a prominent effect on powder stability.

The water content of spray-dried lulo powders varied from 0.9% to 7%. As shown in Table 1, the higher the inlet air temperature and arabic gum/maltodextrin concentration, the lower the water content of the spray-dried lulo powder. At higher inlet air temperatures, there was a greater temperature gradient between the atomized feed and the drying air, resulting in a higher rate of heat transfer for water evaporation, thus producing low-water powders [23, 44]. The highest water content presented in Table 1 corresponds to lulo without added solutes and spray-dried at lower inlet temperatures. The inlet temperature effect was observed by León-Martínez et al. [46]; Kha et al. [24]; Quek et al. [27]; Ersus and Yurdagel [28]; Rodríguez-Hernández et al. [14]; Chegini and Ghobadian [32]; Goula et al. [47]; Cai and Corke [35] working with spray-dried nopal mucilage powder, gac fruit powder, watermelon powder, black carrot powder, cactus pear juice powder, orange juice powder, tomato powder, and amaranthus pigment powder, respectively. The water content of lulo powder exhibited an inverse relationship with increasing arabic gum/maltodextrin concentration, which was also reported by other authors [14, 22, 31, 35, 44]. Both the water content and the hygroscopicity showed a positive interaction between both of the solutes considered in the final model (table 2).

Maltodextrins and gums are also added during the production of food powders in order to act as encapsulating or wall materials, contributing to keep the desired functional properties in the finished product, such as stability against oxidation, ease of handling, improved solubility, controlled release, and extended shelf-life [47, 48]. According to Qi and Xu [49], high-DE maltodextrins show high reducing capacity, providing protection against oxidation. The ability of maltodextrins to protect encapsulated products against oxidation is attributed to their film-forming capacity and plastic properties, being also largely used as wall materials due to a good compromise between cost and effectiveness, being bland in flavor, and having a low viscosity at a high solid ratio. On the other hand, arabic gum is the gum which is most commonly used as a flavour encapsulating material, mainly due to its solubility, low viscosity, emulsification characteristics, and its good retention of volatile compounds [50-52]. Reinnecius [53] found that combinations of maltodextrin with arabic gum provided good protection against the oxidation of encapsulated aromas. In Table 1, it can be observed that samples with no solutes added to feed prior to spray drying (runs 3 and 14) showed the lowest values of bioactive compounds (Y_7, Y_8, Y_9, Y_{10} and Y_{11}). High weight molecular solutes reduced the powder's stickiness and helped the retention of nutritive and functional properties. Moreover, in samples 3 and 14 there was a significant ($p<0.05$) decrease in the functional value when the inlet temperature during spray drying was increased (Table 1). Quek et al. [27] reported that the spray drying of watermelon juice at over 165 °C led to inferior products due to nutrient loss, and spray drying at temperatures of over 180 °C were not suitable for Amaranthus betacyanins pigments [35].

When high molecular weight solutes were added to lulo pulp prior to spray drying, a temperature effect was also observed. For example, when spray drying of samples with 5% arabic gum and 5% maltodextrin was carried out at 200 ºC, a greater loss of bioactive compounds was observed as compared to those spray-dried at 150 or 100 ºC (Table 1). In general, every studied bioactive compound and antioxidant capacity (Y_7, Y_8, Y_9, Y_{10}, Y_{11}) was positively affected by an increase in solute concentration and a decrease in inlet temperature (Table 2).

Pearson's statistical correlation analysis was used to establish correlations between the antioxidant capacity and the studied bioactive compounds. The obtained results showed that the most significant contribution to antioxidant capacity was provided by total flavonoid content (0.7762, $p<0.05$), followed by total phenol content (0.7204, $p<0.05$), ascorbic acid (0.6980, $p<0.05$) and vitamin C (0.6359, $p<0.05$). In apricot [54] and grapefruit [55] the most significant contribution to antioxidant capacity was provided by total phenol.

3.3 Optimization procedure for predicting an optimum spray-dried lulo pulp

Spray-dried lulo powder could be considered an optimum product if the criteria applied to achieve the optimization resulted in (1) the highest ascorbic acid, vitamin C, total phenol and total flavonoid content and antioxidant capacity, as well as the greatest product yield and productivity and (2) the lowest outlet temperature, drying ratio, water content and hygroscopicity. Multiple response optimization suggested that the optimal conditions for producing the best spray-dried lulo powder were reached by combining an inlet air temperature of 125 °C, 3% arabic gum, and 13.4% maltodextrin DE16.5-19.5. Under these optimum conditions, the predicted responses for the obtained powder are: ascorbic acid = 225 mg/100g lulo pulp solids, vitamin C = 444 mg/100g lulo pulp solids, total phenols = 957 mg/100g lulo pulp solids, total flavonoids = 128 mg/100g lulo pulp solids, antioxidant capacity = 115 mg/100g lulo pulp solids, product yield = 35 %, productivity = 51 g/h, outlet temperature = 78.6 ºC, drying ratio = 7, water content = 2.2 g water/100g and hygroscopicity = 54 g/100g.

4. Conclusion

The optimization of the spray drying conditions for the lulo pulp was successfully executed using the central composite design of the RSM. The optimum powder was the one with a high nutritive and functional value, low hygroscopicity and low water content. Significant empirical equations ($R^2>0.74$) have been developed for describing and predicting the variation of each response variable studied. The concentrations of arabic gum and maltodextrin were the factors that most significantly ($p<0.05$) contributed to the increase in the product yield and the decrease in both the hygroscopicity and the water content in powder. These solutes also contributed to retain the nutritive and functional properties of the fruit. The inlet temperature had the mildest effect on these variables. The multiple response optimization predicted that the use of an inlet air temperature of 125 °C, 3% arabic gum, and 13.4% maltodextrin DE16.5-19.5 provided the overall optimum parameters for the spray drying of the lulo pulp.

Acknowledgment

The authors thank the Universidad Politécnica de Valencia for the financial support given throughout the Project ADSIDEO-COOPERACIÓN 2010 “Adaptación de procesos de secado para favorecer la comercialización de super frutas de origen colombiano”.

References

1. Gancel, A.L.; Alter, P.; Dhuique-Mayer, C;, Ruales, J.; Vaillant, F. Identifying carotenoids and phenolic compounds in naranjilla (Solanum quitoense Lam. var. puyo hybrid), an andean fruit. J. Agric Food Chem 56 (2008) 11890–11899
2. Heiser, C. B.; Anderson, G. “New” Solanums. Perspectives on new crops and new use Janick, J., Ed.; ASHS Press: Alexandria, VA, 1999, 379-384.
3. Heiser, C. B. The relationships of the naranjilla, Solanum quitoense. Biotropica, 1972, 4 77–84.
4. Dennis, F. G. J.; Herner, R. C. Naranjilla: a potential cash crop for the small farmer in Latin America. Acta Hortic 158 (1985) 475–481
5. National Research Council. Naranjilla (Lulo). Lost crops of the Incas: Little known plants of the Andes with promise of the world cultivation. National Academy Press: Washington, 1989, 267-275.
6. Morton, J. (1987). Naranjilla. Fruits of warm climates. Morton, J. F., Ed.; Miami, F.L, 1987, 425-428.
7. Griffiths, F. P. Processing procedure to retain Vitamin C in Naranjilla, Solanum quitoense, products. J. Rio Grande Valley Hortic Soc 19 (1965) 33-36.
8. Jayasundera, M.; Adhikari, B.; Adhikari, R.; Aldred, P. The effects of proteins and low molecular weight surfactants on spray drying of model sugar-rich foods: Powder production and characterisation. J Food Eng 104 (2011) 259–271

9. Knorr, D. Technology aspects related to microorganisms in functional foods. Trends Food Sci Technol 9 (1998) 295–306

10. Bhandari, B.R.; Datta, N.; Howes, T. Problems associated with spray drying of sugar-rich foods. Drying Technol 15 (1997) 671–684

11. Maa, Y. F.; Nguyen, P. A. T.; Hsu, S. W. Spray drying of air–liquid interface sensitive recombinant human growth hormone. J Pharm Pharm Sci 87(2) (1998) 152–159

12. Vega, C.; Goff, H. D.; Roos, Y. H. Spray drying of high-sucrose dairy emulsions: Feasibility and physicochemical properties. J Food Sci 70 (2005) 244–251

13. Adhikari, B.; Howes, T.; Shrestha, A. K.; Bhandari, B. R. Development of stickiness of whey protein isolate and lactose droplets during convective drying. Chem Eng Process 46 (2007) 420–428

14. Rodríguez-Hernández, G. R.; Gonzalez-Garcia, R.; Grajales-Lagunes, A.; Ruiz-Cabrera, M. A.; Abud-Archila, M. Spray-drying of cactus pear juice (Opuntia streptacantha): effect on the physicochemical properties of powder and reconstituted product. Drying Technol 23(4) (2005) 955–973.

15. Roustapour, O.R.; Hosseinalipour, M.; Ghobadian, B. An experimental investigation of lime juice drying in a pilot plant spray dryer. Drying Technol 24 (2006) 181–188

16. De Oliveira, M.A.; Maia, G.A.; De Figueiredo, R.W.; De Souza, A.C.R.; De Brito, E.S.; De Azeredo, H.M.C. Addition of cashew tree gum to maltodextrin-based carriers for spray drying of cashew apple juice. International J Food Sci Tech 44 (2009) 641–645

17. Boonyai, P.; Bhandari, B; Howes, T. Stickiness measurement techniques for food powders: A review. Powder Technol 145 (2004) 34–46

18. Glicksman M. Gum Technology in the Food Industry. Food Science & Technology A Series of Monographs. Academic Press, Inc., New York, NY, 1969.

19. Revie, G. N.; Thomas, B. R. J. Powdered flavors offer many advantages. Food Manufacture 37(1) (1972) 40-41

20. Main, J.H.; Clydesdale, F.M.; Francis, F.J. Spray drying anthocyanin concentrates for use as food colorants. J Food Sci 43 (1978) 1693-1694, 1697

21. Francis FJ. Handbook of Food Colorant Patents. Food & Nutrition Press, Inc., Trumbull, Conn, 1986, 14-19.

22. Jaya, S.; Das, H. Accelerated storage, shelf life and colour of mango powder. J Food Process Preserv 29 (2005) 45–62.

23. Tonon, R.V.; Brabet, C.; Pallet, D.; Brat, P.; Hubinger, M.D. Physicochemical and morphological characterisation of ac¸ai powder produced with different carrier agents. International J Food Sci Tech 44 (2009)1950–1958

24. Kha, T. C.; Nguyen, M. H.;Roach, P. D. Effects of spray drying conditions on the physicochemical and antioxidant properties of the Gac (Momordica cochinchinensis) fruit aril powder. J Food Eng, 98 (2010) 385–392

25. Moßhammer, M. R.; Stintzing, F. C.; Carle, R. Evaluation of different methods for the production of juice concentrates and fruit powders from cactus pear. Innov Food Sci Emerg Technol 7(4) (2006) 275–287

26. Obon, J. M.; Castellar, M. R.; Alacid, M.; Fernández-López, J. A. Production of a red–purple food colorant from Opuntia stricta fruits by spray drying and its application in food model systems. J Food Eng 90 (2009) 471–479

27. Quek, S. Y.; Chok, N. K.; Swedlund, P. The physicochemical properties of spray-dried watermelon powders. Chem Eng Process 46 (2007) 386–392.

28. Ersus, S.; Yurdagel, U. Microencapsulation of anthocyanin pigments of black carrot (Daucuscarota L.) by spray drier. J Food Eng 80 (2007) 805–812

29. Abadio, F. D. B.; Domingues, A. M.; Borges, S. V.; Oliveira, V. M. Physical properties of powdered pineapple (Ananas comosus) juice-effect of maltdextrin concentration and atomization speed. J Food Eng 64 (2004) 285–287

30. Cano-Chauca, M.; Stringheta, P. C.; Ramos, A. M.;Cal-Vidal, J. Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. Innov Food Sci Emerg Technol 6 (2005) 420–428

31. Mosquera, L.H.; Moraga, G; Martínez-Navarrete, N. Critical water activity and critical water content of freeze-dried strawberry powder as affected by maltodextrin and arabic gum. Food Res Int 47 (2012) 201-206

32. Chegini, G. R.; Ghobadian, B. Effect of spray-drying conditions on physical properties of orange juice powder. Drying Technol 23(3) (2005) 657–668

33. Liu, Z.; Zhou, J.; Zeng, Y.; Ouyang, X. The enhancement and encapsulation of Agaricus bisporus flavor. J Food Eng 65 (2004) 391–396

34. Vandir, H.; Yasar, M. Optimisation of pomegranate (Punica Granatum L.) juice spray-drying as affected by temperature and maltodextrin content. Int J Food Sci Technol 47 (2012) 167–176

35. Cai, Y.Z.; Corke, H. Production and properties of spraydried amarantus betacyanin pigments. J Food Sci 65 (2000) 1248–1252

36. Xu, G.; Liu, D.; Chen, J.; Ye, X.; Ma, Y.; Shi, J. Juice components and antioxidant capacity of citrus varieties cultivated in China. Food Chem 106 (2008) 545–551

37. Sánchez-Moreno, C.; Plaza, L;, De Ancos, B.; Cano, M. P. Quantitative bioactive compounds assessment and their relative contribution to the antioxidant capacity of commercial orange juices. J Sci Food Agric 83 (2003) 430–439

38. Benzie, I. F. F.; Strain, J. J. Ferric reducing/antioxidant power assay: Direct measure of total antioxidant activity of biological fluids and modified version for simultaneous measurement of total antioxidant power and ascorbic acid concentration. Methods Enzymol 299 (1999) 15–27

39. Djeridane, A.; Yousfi, M.; Nadjemi, B.; Boutassouna, D.; Stocker, P.; Vidal, N. Antioxidant activity of some Algerian medicinal plants extracts containing phenolic compounds. Food Chem 97(4) (2006) 654-660

40. Mirhosseini, H.; Tan, C. P.; Hamid, N. S. A.; Yusof, S.; Chern, B. H. Characterization of the influence of main emulsion components on the physicochemical properties of orange beverage emulsion using response surface methodology. Food Hydrocoll 23 (2009) 271–280

41. Statgraphics Plus 5.1. for Windows (2000). Statistical Graphics Corporation. Virginia, USA: StatPoint, Inc.

42. Vasco C.; Ruales J.; Kamal-Eldin A. Total phenolic compounds and antioxidant capacities of major fruits from Ecuador. Food Chem 111(4) (2008) 816-823

43. Goula, A. M.; Adamopoulos, K. G. Effect of maltodextrin addition during spray drying of tomato pulp in dehumidified air: II. Powder properties. Drying Technol 26(6) (2008) 726–737

44. Goula, A. M.; Adamopoulos, K. G. A new technique for spray drying orange juice concentrate. Innov Food Sci Emerg Technol 11 (2010) 342–351

45. Moreira, G. E. G.; Costa, M. G. M.; de Souza, A. C. R.; de Brito, E. S.; de Medeiros, M. F. D.; de Azeredo, H. M. C. Physical properties of spray dried acerola pomace extract as affected by temperature and drying aids. Lebenson Wiss Technol 42 (2009) 641–645

46. León-Martínez, F. M.; Méndez-Lagunas, L. L.; Rodríguez-Ramírez, J. Spray drying of nopal mucilage (Opuntia ficus-indica): Effects on powder properties and characterization. Carbohydr Polym 81 (2010) 864–870

47. Claude, J.; Ubbink, J. Thermal degradation of carbohydrate polymers in amorphous states: A physical study including colorimetry. Food Chem 96 (2006) 402 -410

47. Goula, A. M.; Adamopoulos, K. G.; Kazakis, N. A. Influence of spray drying conditions on tomato powder properties. Drying Technol 22(5) (2004) 1129–1151.

48. Pérez-Alonso, C.; Beristain, C.I.; Lobato-Calleros, C.; Rodríguez-Huezo, M.E.; Vernon-Carter, E.J.. Thermodynamic analysis of the sorption isotherms of pure and blended carbohydrate polymers. J Food Eng 77 (2006) 753-760

49. Qi, Z. H.; Xu, A. Starch-based ingredients for flavor encapsulation. Cereal Foods World 44 (1999) 460–465

50. Madene, A.; Jacquot, M.; Scher, J.; Desobry, S. Flavour encapsulation and controlled release – a review. Int J Food Sci Technol 41 (2006) 1-21

51. Righetto, A.M.; Netto, F.M. Vitamin C stability in encapsulated green West Indian cherry juice and in encapsulated synthetic ascorbic acid. J Sci Food Agric 86 (2006) 1202-1208

52. Desobry, S.A.; Netto, F.M.; Labuza, T.P. Comparison of spray-drying, drum-drying and freeze-drying for β-carotene encapsulation and preservation. J Food Sci 62 (1997) 1158-1162

53. Reinnecius, G.A.. Carbohydrates for flavor encapsulation. Food Technol 46 (1991) 144-152

54. Igual, M.; García-Martínez, E.; Martín-Esparza, M. E.; Martínez-Navarrete, N. Effect of processing on the drying kinetics and functional value of dried apricot. Food Res Int 47 (2012) 284–290

55. Igual, M.; García-Martínez, E.; Camacho, M. M.; Martínez-Navarrete, N. Effect of thermal treatment and storage on the stability of organic acids and the functional value of grapefruit juice. Food Chem 118 (2010) 291–299

Highlights

- Spray-drying lulo optimization according to inlet air temperature and solutes
- High nutritive and functional value, and lowest hygroscopicity and water content
- 125°C, 3% arabic gum and 13.4% maltodextrin were found to be the optimum conditions
- Inlet temperature had the lesser effect on the studied variables
- Solutes promoted fruit bioactive compounds retention during spray-drying

Table 1

Table 1. Matrix of the central composite design (x_i: independent variables) and experimental data obtained for the response variables studied (Y_j)

Run	x_1	x_2	x_3	Y_1	Y_2	Y_3	Y_4	Y_5	Y_6	Y_7	Y_8	Y_9	Y_{10}	Y_{11}
1	150	5	5	100	32	10	37	3	48	227	325	881	125	99
2	150	5	5	97	42	10	38	4	40	223	319	881	131	100
3	180	0	0	106	0.4	21	18	4	114	31	37	366	61	47
4	150	5	5	101	34	10	37	3	35	234	366	924	124	100
5	150	5	5	97	42	10	38	4	40	226	342	948	125	100
6	180	10	10	108	35	7	61	0.9	47	215	358	813	120	85
7	150	5	5	95	38	10	38	2	38	216	371	825	131	97
8	150	5	5	98	42	10	38	4	39	223	378	860	125	101
9	120	10	0	77	34	10	38	2	56	181	413	661	88	100
10	150	0	5	91	20	14	27	4	55	186	362	848	100	71
11	150	13.4	5	95	32	7	53	1.3	44	243	405	855	119	94
12	200	5	5	115	34	10	36	2	60	193	354	744	91	76
13	180	0	10	119	16	10	37	2	52	197	381	861	118	94
14	120	0	0	77	9	21	19	7	82	41	48	644	87	71
15	120	10	10	78	29	7	59	2	51	229	413	933	136	147
16	100	5	5	67	27	10	37	4	56	205	362	819	113	97
17	150	5	5	101	34	10	37	3	35	231	374	949	124	99
18	180	10	0	106	42	10	37	2	50	175	370	814	101	95
19	150	5	5	100	32	10	37	3	48	220	316	930	126	99
20	150	5	0	95	19	14	28	5	52	152	355	700	90	72
21	120	0	10	82	36	10	37	3	50	211	415	994	120	101
22	150	5	13.4	93	30	7	55	1	44	242	412	890	128	92
23	150	5	5	98	42	10	38	4	39	222	381	925	131	101

Y_1 to Y_{11}: response variables of outlet temperature (ºC), product yield (g solutes in the powder/ 100 g solutes in the mixture), drying ratio (powder solid content/ feed solid content), productivity (g/h), water content (g/100g), hygroscopicity (g $_{\text{water gain}}$ /100 $_{\text{dry solids}}$), ascorbic acid content (g/100g_{TLS}), vitamin C content (g/100g_{TLS}), total phenolic content (g GAE/100g_{TLS}), total flavonoid content (g RE/100g_{TLS}) and antioxidant capacity (mmol TE/100g_{TLS}), respectively.
x_1, x_2 and x_3 independent variable of inlet temperature (ºC), arabic gum (g/ 100 g pulp) and maltodextrin (g/ 100 g pulp), respectively.

Table 2

Table 2. Regression coefficients and adjusted R^2 for the final reduced models

Regression	Outlet temperature	Product yield	Drying ratio	Productivity	Water content	Hygroscopicity	Ascorbic acid	Vitamin C	Total phenols	Total flavonoids	Antioxidant capacity
coefficient	(Y_1)	(Y_2)	(Y_3)	(Y_4)	(Y_5)	(Y_6)	(Y_7)	(Y_8)	(Y_9)	(Y_{10})	(Y_{11})
Constant											
b_0	-38.0119	2.8869	18.2515	35.2978	9.1564	197.272	-259.489	114.781	-16.8269	-67.0776	118.474
Linear											
b_1	1.3052	-	0.0169	-0.1134	-0.0238	-1.3052	4.1687	-	11.3169	2.1885	-0.3275
b_2	-	5.3933	-1.3506	1.63101	-0.3105	-7.324	21.9153	30.9427	-41.1455	4.5094	2.3135
b_3	-	5.1108	-1.0779	-0.9649	-0.2861	-7.217	29.0628	31.7247	75.6376	7.2362	2.2676
Square											
b^2_1	-0.0028	-	-	-	-	0.0042	-0.0138	-	-0.048	-0.0079	-
b^2_2	-	-0.1942	0.0286	-	-	0.3352	-0.7902	-	-	-0.2674	-
b^2_3	-0.0771	-0.1996	0.0389	-	-	0.2567	-1.2078	-	-3.3875	-0.3537	-
Interactions											
b_{12}	-	-	-	-	-	-		-	0.37	-	-
b_{13}	-	-	-0.0034	0.0227	-	-		-	-	-	-
b_{23}	-	-0.3815	0.0905	-	0.0188	0.4516	-1.2438	-3.6147	-2.8702	-	-
R^2 adj	0.96	0.776	0.97	0.866	0.801	0.837	0.971	0.756	0.792	0.852	0.744

b_i: the estimated regression coefficient for the main linear effects. b^2_i: the estimated regression coefficient for the quadratic effects. b_{ij}: the estimated regression coefficient for the interaction effects. i=1: Inlet temperature; i=2: Arabic gum; i=3: Maltodextrin.