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original scientific paper

Optimization of Spray Drying Parameters for Producing Red Pitahaya Powder Using Response Surface Methodology

Running head: Spray Drying of *Hylocereus polyrhizus* Powder

Yuli Lorena Callata Aparicio^{1*}, María Mercedes Valeriano Taco¹, Jose Antonio Villalobos Taucaya¹,
William Edgar Heredia Peña¹ and Jose Manuel Prieto²

¹National University of Saint Augustine of Arequipa, Santa Catalina Nro. 117, 04001 Arequipa, Perú

²National University of Juliaca, Av. Nueva Zelandia Nro. 631, Urb. La Capilla, 21101 Juliaca Puno, Perú

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SUMMARY

Research background. Red pitahaya (*Hylocereus polyrhizus*) is an exotic tropical fruit rich in antioxidants and betalains, but it is characterised by a relatively short shelf life. An effective and safe means of processing this fruit is the production of dehydrated products that can be readily reconstituted. Therefore, this study aimed to optimise the spray drying parameters for producing red pitahaya powder with suitable physicochemical and reconstitution properties.

Experimental approach. Response surface methodology based on a central composite design was applied to evaluate the effects of inlet air temperature, feed flow rate and maltodextrin mass per

*Corresponding author:
Phone: +51924629654
E-mail: ycallataa@unsa.edu.pe

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volume ratio. The experimental data were modelled using a second-order polynomial equation to describe and predict the assessed responses: yield, moisture, water activity, hygroscopicity, flowability, cohesion and solubility. In the optimised sample, betalains, total phenols and antioxidant capacity were quantified as complementary quality indicators. Particle size and morphology were analysed using scanning electron microscopy.

Results and conclusions. The quadratic models adequately described the main physicochemical responses, including yield, moisture content, water activity, flowability, cohesion and solubility. The selected processing conditions resulted in a powder with suitable physicochemical characteristics and reconstitution properties, including appropriate powder recovery, moisture content, water activity, flowability and solubility. The powder also showed measurable levels of betalains, phenolic compounds and antioxidant capacity. Microscopic observations revealed irregular particles with smooth surfaces and no visible cracks or fractures.

Novelty and scientific contribution. The results demonstrate that inlet air temperature, feed flow rate and maltodextrin mass per volume ratio influenced the physicochemical and reconstitution properties of red pitahaya powder. The scientific contribution of this work lies in defining suitable processing conditions for a juice matrix rich in low-molecular-mass sugars and natural pigments, where powder recovery, moisture control, water activity, flowability and solubility must be balanced. These findings provide specific information for the production of red pitahaya powder with suitable technological properties and support its potential use as an ingredient in food formulations.

Keywords: red pitahaya; spray drying; response surface methodology; maltodextrin; scanning electron microscopy

INTRODUCTION

Purple dragon fruit (*Hylocereus polyrhizus*) [1], also referred to as pitahaya, is a tropical exotic fruit belonging to the Cactaceae family [2]. It is rich in vitamin C and antioxidants, particularly betalains and polyphenols [3]. In recent years, consumer interest has increased due to its distinctive flavour, vivid appearance, and notable nutritional and biofunctional properties [4].

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However, due to its perishable nature, the fruit has a short shelf life, typically no longer than three to four days at room temperature or about one week at 20 °C [5]. Besides being consumed fresh, pitahaya is also used in juices, purées, and innovative culinary applications designed to meet consumer preferences and demands [6].

Juices from fruits and vegetables undergo dehydration to obtain a practical and stable form that can be easily reconstituted, preserving the original nutritional and functional qualities [7]. Spray drying is a fast dehydration technique that transforms liquid feeds into powders and is recognised as the most economical and widely adopted method, allowing precise control of temperature and airflow [8]. The process occurs in three main steps: atomisation of the feed, drying with hot air, and powder recovery [9]. The characteristics of the final powder are largely determined by both the feed formulation and the drying parameters [10]. Spray drying is considered optimal when strict quality standards are required, especially regarding particle size distribution, residual moisture, and bulk density [7].

During the spray drying of fruit juices, a common challenge is adhesion and stickiness on the chamber walls, caused by the abundance of organic acids and simple sugars of low molecular weight. This issue complicates the drying process, making the use of a carrier agent essential [11]. Carrier agents are fundamental in spray drying; examples include maltodextrin, gum arabic, pectin and whey proteins, all recognised for their effective carrier performance [12]. Maltodextrin is particularly popular for encapsulating fruit juices and juice-based products because of its excellent solubility, reduced hygroscopicity, and affordability, which make it highly suitable for this purpose [13]. Spray drying has been successfully applied to obtain powdered fruit pulp from various sources, such as apple [14], cupuaçu [15], strawberry [16], and *Opuntia ficus-indica* peels [17]. The resulting powder is easier to handle, package, and transport than fresh fruit, while also offering a high-quality product with extended shelf life [7].

This study sought to optimise spray-drying parameters through response surface methodology (RSM) for producing red pitahaya (*Hylocereus polyrhizus*) powder. The independent variables were inlet air temperature, feed flow rate, and maltodextrin level. The response variables comprised yield, moisture content, water activity, hygroscopicity, flowability and cohesion, solubility, colour, and morphology. Bioactive properties (betalains, total phenols, and antioxidant capacity) were also assessed, and particle size was characterised using scanning electron microscopy (SEM). RSM was chosen for its structured approach to process optimisation, supporting the identification of drying

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conditions that favour suitable physicochemical and reconstitution properties in spray-dried pitahaya powder.

MATERIALS AND METHODS

Sample preparation

Fresh red pitahaya (*Hylocereus polyrhizus*) fruits were obtained from Nueva Esperanza Market, part of the Andrés Avelino Cáceres commercial platform in Arequipa, Peru. The juice was obtained mechanically using a juicer (HR-1858/90, Philips, Amsterdam, Netherlands) and filtered through a cloth. The remaining pulp was subjected to an additional filtration step to maximise juice recovery. The final juice was stored in pre-sterilised glass containers at 4 °C until further use.

Concentration of red pitahaya juice using vacuum microwave assistance

Approximately 400 g of red pitahaya (*Hylocereus polyrhizus*) juice was placed in a 2 L glass flask and processed in a microwave oven (MS32J5133AM/PE, Samsung Electronics, Suwon, South Korea; manufactured in Malaysia) connected to a vacuum pump (W2V10, Woosung Automa Co., Ltd., Hwaseong-si, South Korea), as shown in Fig. S1. The juice was concentrated to 20 °Brix at 300 W. Soluble solids (°Brix) and temperature were measured every 10 min, with the temperature reaching up to 55 °C.

Spray drying

The concentrated red pitahaya (*Hylocereus polyrhizus*) juice was spray-dried using a laboratory atomiser (SD-2000, ZOIBKD Supply, Changzhou, Jiangsu, China). The nozzle had an internal diameter of 0.5 mm, and the feed flow rate was adjusted via pump speed. The inlet air temperature was set at 90–110 °C, and the feed rate at 600–800 mL/h. The drying air fan was operated at 100 %, corresponding to an approximate air flow rate of 80 m³/h, and the outlet air temperature ranged from approximately 54–64 °C during the drying process. Commercial food-grade maltodextrin (Maltodextrin DE 10-12; Qinhuangdao Lihua Starch Co., Ltd., China) was added at mass per volume ratios of 10–30 % to facilitate drying. The resulting powder was sealed in polyethylene bags and stored in a

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desiccator containing silica gel at 25 °C until analysis. The process was carried out at 3 824 m a.s.l. (Juliaca; UTM 19L 378554.045 8286979.815).

Powder yield

Following the spray-drying process, the powder yield (in %) was determined as the ratio of the recovered solids to the total solids present in the feed suspension [18]:

$$Y = \left(\frac{m(\text{recovered})}{m(\text{feed})} \right) \cdot 100 \quad /1/$$

where $m(\text{recovered})$ is the recovered solid mass (in g), and $m(\text{feed})$ is the initial solid mass in the feed suspension (in g).

Moisture

The moisture content (in %) of the powder was determined by oven drying. Approximately 3 g of the sample was accurately weighed into a Petri dish using an analytical balance (Pioneer, Ohaus Corporation, Parsippany, NJ, USA) and placed in a hot-air oven (ODHG-9030B, KertLab, USA) at 105 °C for 24 h, until a stable mass was achieved [19]. The results were expressed as percentage moisture content, calculated as:

$$w(\text{moisture}) = \left(\frac{m_0 - m}{m_0} \right) \cdot 100 \quad /2/$$

where m_0 represents the initial mass of the sample (in g), and m corresponds to the mass of the dried product (in g).

Water activity

The water activity (a_w) of the samples was measured using a water activity meter (WA-160A, Guangzhou Amittari Instruments Co., Ltd., Guangzhou, China). This represents a key parameter in evaluating product shelf-life and for controlling the growth of yeasts, moulds, and fungi, which is crucial for maintaining food quality and safety.

Hygroscopicity

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Hygroscopicity was assessed according to the method described by Cai and Corke [20], with minor modifications. Approximately 2 g of powder was weighed into a small plastic dish, which was then placed in a container with saturated KCl solution (86.3 % RH) at 20 °C. The mass gain was recorded after 1, 2, 3, 5, 7, 20, 22, 23, and 24 h, and hygroscopicity (in %) was calculated as:

$$w(\text{hygroscopicity}) = \left(\frac{m_t - m_i}{m_i} \right) \cdot 100 \quad /3/$$

where the m_t is the sample mass at time t (in g), and m_i is the initial mass of the dry sample (in g).

Flowability and cohesion

To characterise the spray-dried powder, bulk density and tapped density were determined. For bulk density, approximately 3 g of powder was transferred into a 10 mL graduated cylinder, and the corresponding volume was recorded. For tapped density, the cylinder was tapped 25 times on a smooth, cushioned surface, and the final volume was noted. Both densities were calculated by dividing the powder mass by the respective volume, before tapping (ρ_{bulk}) and after tapping (ρ_{tapped}) [8]. From these values, Carr's index (CI) and Hausner ratio (HR) were calculated to assess powder flowability and cohesion [21].

$$\text{CI} = \left(\frac{\rho_{\text{tapped}} - \rho_{\text{bulk}}}{\rho_{\text{tapped}}} \right) \cdot 100 \quad /4/$$

$$\text{HR} = \frac{\rho_{\text{tapped}}}{\rho_{\text{bulk}}} \quad /5/$$

where ρ_{bulk} is the bulk density (in g/mL), ρ_{tapped} is the tapped density (in g/mL), CI is Carr's index (in %), and HR is the Hausner ratio (dimensionless).

Water solubility

Water solubility was determined following the method described by Cano *et al.* [22], with slight modifications as outlined below. A mass of 0.1 g powder sample was accurately weighed and dissolved in 10 mL of distilled water within a test tube, then mixed using a variable speed vortex mixer (Four E's Scientific, Guangzhou, Guangdong, China) at 1500 min⁻¹ for 5 min. The resulting suspension was subsequently centrifuged at 1000×*g* for 5 min using a 12-tube digital centrifuge (K-CF0202003, Four E's Scientific, Guangzhou, Guangdong, China). Finally, a 2.5 mL portion of the supernatant was

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transferred into pre-weighed Petri dishes and dried in an oven at 105 °C for 5 h. The solubility percentage was calculated from the mass difference according to the following equation:

$$S = \left(\frac{m_{\text{solids}}}{m_{\text{sample}}} \right) \cdot 100 \quad /6/$$

where m_{solids} represents the mass of solids in the supernatant (in g), and m_{sample} corresponds to the mass of the sample (in g).

Colour

Colour was determined by digital image analysis based on the conversion of the RGB colour space to the CIELAB colour space, following the procedure described by Castro *et al.* [23], with modifications related to the image processing software and sample type. Approximately 2 g of powder from each treatment were placed in small plastic sample containers and photographed under controlled conditions inside an image acquisition chamber with internal walls measuring 50 cm×40 cm, covered with matte black material to minimise light reflection. Illumination was provided by three 20 W daylight lamps, arranged equidistantly and positioned 10 cm from the chamber edges. Images were captured without flash using a Logitech C930e webcam (Logitech, Newark, CA, USA) at a resolution of 1920 × 1080 pixels and stored in JPG format. A fixed vertical distance of 40 cm was maintained between the camera lens and the sample surface for all image acquisitions.

Images were processed using Python 3.10 [24], with OpenCV [25], NumPy [26] and scikit-image [27] libraries used for image reading, numerical processing and colour space conversion. Regions of interest corresponding to the sample surface were selected from each image, and the mean red, green and blue values were extracted, normalised and converted to the XYZ colour space using the D65 standard illuminant and the 2° standard observer. The XYZ values were then transformed into the CIELAB colour space to obtain L^* , a^* and b^* , where L^* represents lightness, a^* the green-to-red axis and b^* the blue-to-yellow axis.

Particle morphology by optical microscopy

Particle morphology was examined using a binocular optical microscope (B120, AmScope, Irvine, CA, USA) equipped with a 40× objective and a digital camera (MObyat 48 MP HDMI USB

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H1605, China). Powder samples from all treatments were placed on clean glass slides, gently dispersed with a spatula to form a thin layer and minimise particle overlapping, and then covered with coverslips. All samples were observed under the same magnification conditions for qualitative morphological comparison [28].

Experimental design

A total of 12 experimental runs were conducted under different inlet air temperatures (90–110 °C), feed flow rates (600–800 mL/h), and maltodextrin concentrations (10–30 % *m/V*), as summarised in Table 1. The full set of treatments and the experimental responses obtained for each run are provided in Table S1. The experimental design employed response surface methodology (RSM) with a central composite design (CCD), including two centre points and an alpha value of 1. Model fitting and optimisation of the physical parameters were carried out using Design-Expert® 13 software [29]. In this setup, inlet air temperature, feed flow rate, and maltodextrin mass per volume ratio were considered as independent variables, while the responses were evaluated according to their target direction (maximisation or minimisation). Regression analysis applied a second-order polynomial model to describe the relationship between the dependent response (*Y*) and the independent variables (*x*). The significance of the models was assessed using analysis of variance (ANOVA) and lack-of-fit tests at the 95 % confidence level. The software then suggested the optimal values for the control factors.

$$\gamma = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_1 X_2 + \beta_5 X_1 X_3 + \beta_6 X_2 X_3 + \beta_7 X_1^2 + \beta_8 X_2^2 + \beta_9 X_3^2 \quad /7/$$

Physicochemical evaluation of the optimised pitahaya powder

Betalains

Total betalain content was determined in the optimised spray-dried red pitahaya powder as the sum of betacyanins and betaxanthins using the multicomponent spectrophotometric method of Nilsson, as applied by García-Segovia *et al.* [30], with modifications in sample type, extraction conditions and centrifugation parameters. Briefly, 0.5 g of spray-dried powder was extracted with 6 mL of phosphate buffer at pH=6.5. The mixture was vortexed for 1 min at 120 min⁻¹ using a vortex mixer (Four E's Scientific, Guangzhou, Guangdong, China) and stored in the dark at 3 °C for 24 h.

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The extract was then centrifuged at $2200\times g$ for 30 min at 3 °C using a digital centrifuge (K-CF0202003, Four E's Scientific, Guangzhou, Guangdong, China), and the supernatant was analysed using a UV-Vis spectrophotometer (Lambda 365, PerkinElmer, Waltham, MA, USA). Absorbance was recorded at 476, 538 and 600 nm, using distilled water as blank. Betacyanin and betaxanthin contents were calculated using molar extinction coefficients of 60 000 and 48 000 L/(mol·cm), respectively, with a 1 cm path length. Total betalains were calculated as the sum of betacyanins and betaxanthins and expressed as mg of total betalains per g of dry sample.

Total phenol content

Total phenolic content was determined in the optimised powder sample using the Swain and Hillis method, as reported by Repo de Carrasco and Encina Zelada [31], with modifications in the sample amount, extraction procedure and reaction conditions. Briefly, 0.5 g of spray-dried powder was transferred to a Falcon tube and extracted with 5 mL of 95 % (by volume) methanol. The mixture was homogenised and stored at 4 °C for 24 h. After extraction, the sample was centrifuged at $1000\times g$ for 15 min, and 0.5 mL of the clear supernatant was collected. This aliquot was mixed with 8 mL of ultrapure water and 0.5 mL of 0.25 N Folin-Ciocalteu reagent. After 3 min, 1 mL of 0.5 M sodium carbonate was added, and the reaction was allowed to proceed for 10 min. The absorbance was then recorded at 725 nm using a UV-VIS spectrophotometer (Lambda 365, PerkinElmer, Waltham, MA, USA). Total phenolic content was quantified using a gallic acid calibration curve and expressed as mg gallic acid equivalents per 100 g of dry sample.

Antioxidant capacity

Antioxidant capacity was determined in the optimised spray-dried red pitahaya powder using the 2,2-diphenyl-1-picrylhydrazyl radical assay, following the method of Brand-Williams *et al.* [32], with modifications in sample volume and reaction conditions. Different volumes of the extract prepared from the powder sample, ranging from 0.02–0.200 mL, were mixed with 1.0 mL of 2,2-diphenyl-1-picrylhydrazyl solution, and methanol was added to reach a final volume of 2.0 mL. The mixture was kept in the dark for 30 min, and absorbance was measured at 515 nm using a UV-VIS spectrophotometer (Lambda 365, PerkinElmer, Waltham, MA, USA). The analyses were performed in triplicate. Radical inhibition was calculated in relation to the control, and antioxidant capacity was

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expressed as IC_{50} , corresponding to the concentration of powder extract required to decrease the initial absorbance of the 2,2-diphenyl-1-picrylhydrazyl radical by 50 %.

Scanning electron microscopy particle size distribution

Particle size was observed by scanning electron microscopy (SEM) using a Scios 2 DualBeam System (Thermo Fisher Scientific, Waltham, MA, USA). The powder samples were affixed onto double-sided carbon adhesive tape and subsequently coated with a thin layer of gold using a metalliser. The equipment was operated at 10 kV with magnifications of 250 $\times$, 1000 $\times$ and 5000 $\times$. Representative micrographs were selected, and the particle size was determined from the SEM images using ImageJ software [33].

RESULTS AND DISCUSSION

Powder yield of spray-dried pitahaya powder

Fig. 1a shows that the lowest powder yield was obtained in treatment T7 (9.67 %) at an inlet air temperature of 100 °C. Under these conditions, drying was incomplete, resulting in partially dried droplets adhering to the chamber walls and consequently reducing powder recovery [34]. Similarly, relatively low yields were observed in T8 (11.69 %) and T3 (13.66 %). This behaviour may be attributed to the natural composition of pitahaya juice, which contains low-molecular-mass sugars and organic acids that can reduce the glass transition temperature and increase particle stickiness during spray drying. As a result, powder deposition on the drying chamber walls is promoted, thereby decreasing the amount of product recovered.

Increasing the inlet air temperature improved powder recovery, probably due to more efficient heat and mass transfer, in agreement with previous studies [35]. The highest yields were obtained in T11 (35.19 %), T12 (31.84 %) and T6 (26.01 %), indicating that both drying conditions and carrier concentration influenced process efficiency. In particular, treatments containing 30 % (*m/V*) maltodextrin (T5, T6 and T11) consistently produced higher yields than those containing 10 % maltodextrin (*m/V*) (T1, T8 and T10), suggesting that the carrier played an important role in reducing stickiness and improving powder recovery. Maltodextrin enhances yield by increasing the total soluble solids content of the feed and reducing particle adhesion through encapsulation [36]. Therefore,

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further improvements in yield could potentially be achieved by increasing the total solids content of the feed, optimising carrier concentration, minimising wall deposition through appropriate drying conditions, and employing carrier systems with higher glass transition temperatures, while preserving the quality attributes of the final product. The second-order polynomial equation for powder yield (in %), derived from the coded values of the independent variables, is presented below:

$$Y = 23.34 + 5.19A - 4.31B + 6.61C - 2.72AB - 1.76AC - 5.24BC + 3.14A^2 - 9.14B^2 - 4.24C^2 \quad /8/$$

Moisture content and water activity of spray-dried pitahaya powder

Powdered products exhibiting higher moisture levels and increased water activity (a_w) are more prone to microbial degradation, leading to a shorter storage life. To ensure stability, the food industry generally recommends that the moisture content of powders should not exceed 4 % [37]. In this study, the moisture content of spray-dried samples ranged from 3.27–5.81 % (Fig. 1b). Across the tested temperature range, higher inlet air temperatures facilitated the outward diffusion of surface water from the microcapsules, leading to lower residual moisture levels [38]. However, the inlet air temperature represents the drying air condition rather than the actual particle temperature, since evaporation during spray drying reduces the thermal exposure of the droplets. The recorded outlet air temperature ranged from approximately 54–64 °C.

Water activity (a_w) is a key parameter in spray-dried samples, as it strongly influences the shelf life of the resulting powder. High a_w indicates that more water is available for chemical and microbial reactions, thereby reducing shelf life. Values of a_w range from 0-1, and foods with values below 0.60 are generally considered microbiologically stable [39]. In this study, a_w values between 0.22 and 0.33 were obtained (Fig. 1c), suggesting relative microbiological stability under the tested conditions. Table S2 showed a significant quadratic model with a non-significant lack of fit. The second-order polynomial equations generated from the coded values of the independent variables for powder moisture (in %) and water activity are presented below, providing insight into how inlet air temperature, feed flow rate, and maltodextrin mass per volume ratio affect these properties.

$$w(\text{moisture}) = 3.66 - 0.48A - 0.11B - 0.91C - 0.18AB + 6.12AC + 1.76BC + 0.30A^2 + 0.65B^2 + 0.90C^2 \quad /9/$$

$$a_w = 0.255 - 0.019A - 0.010B - 0.045C - 0.033AB - 0.027AC + 0.024BC + 0.004A^2 + 0.005B^2 + 0.030C^2 \quad /10/$$

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Hygroscopicity of spray-dried pitahaya powder

Hygroscopicity refers to the ability of powdered foods to absorb moisture from the surrounding environment. Low hygroscopicity is desirable, as high values promote water absorption and stickiness [40]. Hygroscopicity, measured as the hygroscopicity rate (Fig. 2), increased over time. After 24 h, values ranged from 10.92–14.58 %. The best results were obtained in T5 (11.65 %) and T11 (10.92 %) with 30 % (m/V) maltodextrin, while the highest value (14.58 %) was observed at 90 °C and 20 % (m/V) maltodextrin. Maltodextrin mass per volume ratio significantly affected hygroscopicity, decreasing it as its concentration increased, as suggested previously [41]. Hygroscopicity also varied with moisture content and inlet air temperature: powders with lower moisture content showed higher hygroscopicity due to a greater water gradient with the environment. Conversely, decreasing inlet temperature reduced hygroscopicity [42]. Tonon *et al.* [40] reported values of 12.48–15.79 %, which are comparable to those obtained in this study.

Flowability and cohesion of spray-dried pitahaya powder

Hausner ratio (HR) and Carr's index (CI) are widely used to characterise powder properties during processing and storage, particularly flowability [43]. Flowability and cohesion are key quality attributes of spray-dried powders and depend on multiple factors such as temperature, particle size, moisture content, hardness, elasticity, and inlet air conditions [44]. CI values between 0–10 % indicate excellent flow, 10–15 % good flow, 16–20 % fair flow, 21–25 % possible flow, and above 26 % poor flow. Similarly, HR values <1.2 reflect low cohesion, while values of 1.2–1.4 and >1.4 correspond to intermediate and high cohesion, respectively [43]. These measurements indicate that controlling particle characteristics and processing conditions is crucial for achieving optimal flow and handling of spray-dried powders.

The HR values ranged from 1.191–1.409, while CI values ranged from 16.06–29.03 % (Fig. 3a and Fig. 3b). The best results were obtained in treatments T10 (CI=16.06 %, HR=1.191) and T11 (CI=17.45 %, HR=1.211). According to AlYammahi *et al.* [43], these values indicate acceptable flowability and low cohesion, suggesting that under these conditions the powder would be easier to handle and less prone to aggregation during storage. Table S2 showed a significant quadratic model with a non-significant lack of fit. The second-order polynomial equations generated from the coded values of the independent variables for HR and CI are presented below:

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$$HR = 1.32 - 0.007A - 0.057B - 0.037C + 0.011AB - 0.031AC + 0.009BC - 0.077A^2 + 0.032B^2 - 0.017C^2 \quad /11/$$

$$CI = 23.94 - 0.37A - 3.22B - 2.08C + 1.1AB - 1.64AC + 0.53BC - 4.72A^2 + 1.75B^2 - 0.94C^2 \quad /12/$$

Water solubility of spray-dried pitahaya powder

Solubility is an important quality parameter of powders, as it indicates their capacity to disperse or dissolve in water. Maltodextrin is widely used in spray drying due to its high solubility, which results from low-molecular-mass compounds formed during starch hydrolysis [45]. In this study, solubility ranged from 82.79–92.15 % (Fig. 3c), values comparable to those reported for guava powder (85.87–95.43 %) by Patil *et al.* [46]. According to Table S1, powders produced with 10 % (m/V) maltodextrin generally showed the lowest solubility values, whereas higher values were obtained with 20–30 % (m/V) maltodextrin, with the highest solubility recorded in the optimised treatment containing 30 % (m/V) maltodextrin. This trend was supported by the analysis of variance, which showed a significant effect of maltodextrin concentration on solubility. The additional response surface plot in Fig. S2 further supports this behaviour, showing that the interaction between feed flow rate and maltodextrin mass per volume ratio also contributed to the solubility response. The positive effect of maltodextrin can be attributed to its high-water solubility and chemical structure, which contains polar hydroxyl groups capable of interacting with water molecules. In addition, maltodextrins with higher dextrose equivalent values usually contain shorter chains and more hydrophilic sites, favouring powder hydration and dissolution [47].

Although the powders showed high solubility, complete dissolution was not expected because red pitahaya juice may still contain fine suspended pulp residues and naturally occurring insoluble or slowly dispersible solids. In addition, the physical structure of the spray-dried particles may affect dispersion and hydration during reconstitution. Therefore, solubility values below 100 % may be related to both the composition of the juice-based feed and the structural characteristics of the powder.

The second-order polynomial equation obtained from the coded values of the independent variables for powder solubility (in %) is presented below. Overall, the high solubility observed indicates that the powders produced under the tested conditions would rehydrate easily, facilitating their potential use in food applications.

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$$S = 89.69 + 1.47A + 0.69B + 2.68C - 2.33AB - 1.36AC - 2.31BC - 0.002A^2 - 0.07B^2 - 4.11C^2 / 13/$$

Colour parameters of spray-dried pitahaya powder

Colour is recognised as a key optical property with a strong influence on consumer perception and acceptance of packaged food products. In this study, the powders obtained showed limited functionality as bulking agents; however, they also provided the additional advantage of contributing partial colouring when incorporated into appropriate food formulations, which is highly relevant since visual attributes frequently determine preference and marketability. As emphasised by Atalar *et al.* [48], further investigations should focus on the persistence of this colouring effect during storage, as well as the way in which chromatic parameters evolve across different processing conditions and food matrices, in order to gain a more comprehensive understanding of their stability and technological potential. The colourimetric measurements obtained for the powders, specifically a value ranging from 22.52–68.66 and *b* values between –36.31 to –15.54, place the samples within the fourth quadrant of the CIELAB system (Fig. S3), with a clear tendency towards red rather than blue, suggesting that these materials may contribute to the enhancement of visual appeal in food products where reddish tones are desirable. These findings are broadly aligned with those reported by Vieira *et al.* (2) (*a*=16.64, *b*=7.41), supporting the reliability of the colour characteristics observed in this work.

Zuanon *et al.* [49] noted that betalains are mainly used to impart desirable pink-to-red hues in dairy, confectionery, and meat products, and also reported brightness (*L**) values between 58.99 and 76.34 in red beet extract dried by spray using carriers based on polysaccharides, consistent with the range found in this study, where *L** varied from 46.84 to 87.93 and increased with higher maltodextrin concentration and drying flow rate. No significant influence of the process variables was detected on *L**, *a**, or *b** values. This suggests that, within the evaluated drying conditions, the colour parameters were not markedly affected by inlet air temperature, feed flow rate or maltodextrin concentration.

Optical microscopy of spray-dried pitahaya powder

Optical microscopy (40×) was used to investigate the morphology of spray-dried pitahaya juice powder with maltodextrin mass per volume ratios of 10–30 % (Fig. 4). Samples in Fig. 4a and Fig. 4h exhibited irregular, porous structures with larger particle sizes. In contrast, Fig. 4j showed pronounced agglomeration, likely resulting from the combination of low feed temperature (90 °C), high flow rate

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(800 mL/h), and low maltodextrin content (10 % *m/V*). Samples produced with higher maltodextrin mass per volume ratio (20–30 %) revealed micrometric, homogeneous, irregularly shaped, multi-walled particles, confirming effective microencapsulation. As reported by Mousavi and Ghandiha [50], maltodextrin as a coating agent can also introduce surface irregularities. These findings indicate that both maltodextrin mass per volume ratio and drying parameters strongly influence particle morphology, and that careful optimisation is required to improve powder quality for functional applications.

Optimisation and validation of spray-drying conditions

Based on the methodology, one of the Design-Expert software solutions was selected, yielding a desirability of 0.705. The optimal drying conditions were determined as an inlet air temperature of 110 °C, a feed flow rate of 600 mL/h, and a maltodextrin mass per volume ratio of 30 %. Under these parameters, **Table 2** presents a comparison between predicted and experimental response values, along with the relative percentage error. The close agreement between predicted and actual data demonstrates the effectiveness of this approach in optimising the spray drying of red pitahaya juice using maltodextrin as a carrier.

Betalain content of the optimised powder

Betalains are valuable not only as natural food colourants but also for their antioxidant properties. Red pitahaya betalains therefore represent a promising resource for industrial applications. Under the optimal spray-drying conditions (110 °C, 600 mL/h, 30 % *m/V* maltodextrin), the total betalain content measured in T11 was (1.98±0.01) mg/g (**Table 3**), which is comparable to the 1.66 mg/g reported by Vieira *et al.* [2]. According to Bazaria and Kumar [51], increasing inlet air temperature tends to reduce betalain content in powders, most likely due to pigment degradation via hydrolytic cleavage into cyclo-Dopa-glucoside (CDG) and betalamic acid (BA).

Phenolic content and antioxidant capacity of the optimised powder

The total phenolic content in the powder was determined using the Folin-Ciocalteu method, yielding (112.14±0.80) mg GAE/100 g under the optimised conditions, which involved vacuum

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microwave concentration followed by spray drying. This value is slightly higher but still comparable to that reported by Fathordoobady *et al.* [52], who evaluated the influence of solvent type and proportion on betacyanins and antioxidant activity in *Hylocereus polyrhizus* extracts. Although their study employed supercritical fluid and solvent extraction techniques, the similarity of the values suggests that the combined approach of vacuum microwave concentration and spray drying allowed the production of a powder with measurable phenolic content under the optimised conditions.

Dietary intake of antioxidant-rich foods can counteract free radical formation, whereas excessive radical generation within cells disrupts the antioxidant defence system and has been associated with the onset of several diseases [53]. The DPPH assay is a widely applied *in vitro* method for assessing antioxidant capacity. The 50 % inhibitory concentration (IC_{50}) reflects the effectiveness of a compound in limiting radical activity; lower IC_{50} values correspond to stronger scavenging potential. As shown in Table 3, red pitahaya powder demonstrated an IC_{50} of (5.81 ± 0.03) mg/mL, which is lower than the value reported by Choo and Yong [54] for red pitahaya pulp extracts (9.39 mg/mL), suggesting relevant radical-scavenging capacity in the optimised powder. However, differences in matrix composition and extraction conditions should be considered when comparing these values with those reported in previous studies.

Morphology and particle size distribution of the optimised powder by SEM

Fig. 5 shows the SEM micrographs and particle size distribution of the spray-dried pitahaya powder obtained under the optimised conditions of 110 °C inlet air temperature, 600 mL/h feed flow rate and 30 % (*m/V*) maltodextrin. The micrographs, recorded at 250, 1000 and 5000× magnification, showed particles with mainly irregular morphology, smooth surfaces and no visible cracks or fractures. Some spherical particles were also observed, particularly at higher magnification (Fig. 5c), suggesting adequate particle formation during spray drying. The presence of particle bridges may be associated with residual moisture or moisture absorption from the surrounding environment [2]. The particle size was mainly distributed between 6 and 10 µm. In general, microcapsule size can be influenced by spray-drying conditions, including inlet air temperature, residual moisture, and the type and concentration of carrier material, highlighting the importance of process optimisation to obtain more consistent particle morphology and size distribution.

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CONCLUSIONS

This study identified suitable spray-drying conditions for red pitahaya juice with maltodextrin as a carrier and evaluated the effects of the experimental variables on the resulting powder properties. The optimised treatment, obtained at 110 °C inlet air temperature, 600 mL/h feed flow rate and 30 % (m/V) maltodextrin, provided the most favourable balance among powder recovery, moisture content, water activity, flow-related properties and solubility. These results indicate that the selected process conditions improved the technological quality and reconstitution behaviour of the powder. In the optimised sample, measurable levels of betalains, phenolic compounds and antioxidant capacity were obtained, while SEM analysis showed mainly irregular particles with smooth surfaces and a particle size concentrated between 6 and 10 µm. Therefore, spray drying under the selected conditions represents a feasible approach for producing red pitahaya powder with potential application as a food ingredient.

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The authors have used ChatGPT (GPT-5.5 Thinking, OpenAI, San Francisco, CA, USA) solely to improve the clarity, grammar and style of the manuscript. The tool was not used to generate research data, perform analyses or draw scientific conclusions. All AI-assisted suggestions were reviewed, edited and verified by the authors, who take full responsibility for the accuracy and integrity of the final manuscript.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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SUPPLEMENTARY MATERIALS

Supplementary materials are available at www.ftb.com.hr.

AUTHORS' CONTRIBUTIONS

Y.L. Callata Aparicio, and M.M. Valeriano Taco, J.A. Villalobos Taucaya conceived and designed the study, conducted the experiments, collected and analysed the data, interpreted the results, drafted the manuscript, and approved the final version for submission.

W.E. Heredia Peña supervised the research process, provided guidance on experimental design and data interpretation, contributed to the discussion of results, critically revised the manuscript, and approved the final version for submission.

J.M. Prieto provided research facilities, equipment, and software, offered methodological guidance, contributed to manuscript revision, and approved the final version for submission.

ORCID ID

Y.L. Callata Aparicio <https://orcid.org/0000-0002-7258-8101>

M.M. Valeriano Taco <https://orcid.org/0009-0009-2419-7665>

J.A. Villalobos Taucaya <https://orcid.org/0009-0008-3643-3349>

W.E. Heredia Peña <https://orcid.org/0000-0003-3337-0342>

J.M. Prieto <https://orcid.org/0000-0001-6280-7844>

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Table 1. Ranges of independent and dependent variables for red pitahaya (*Hylocereus polyrhizus*) powder

Variables	Response	Variable level		Target	Desirability
		Lower limit	Upper limit		
Independent variables	A: $T(\text{inlet})/^{\circ}\text{C}$	90	110	in range	3
	B: $V(\text{feed})/(\text{mL/h})$	600	800	in range	3
	C: $w(\text{maltodextrin})/\%$	10	30	in range	3
Dependent variables	Y/%	3.36	35.199	maximize	3
	$w(\text{moisture})/\%$	3.27	5.81	minimize	3
	a_w	0.22	0.33	minimize	3
	HR	1.191	1.409	minimize	3
	CI/%	16.06	29.03	minimize	3
	S/%	82.79	92.15	maximize	3
	L^*	46.84	87.93	minimize	3
	a^*	22.52	68.66	maximize	3
	b^*	-36.31	-15.54	maximize	3

Table 2. Predicted and observed response values obtained during the spray-drying optimisation of red pitahaya (*Hylocereus polyrhizus*)

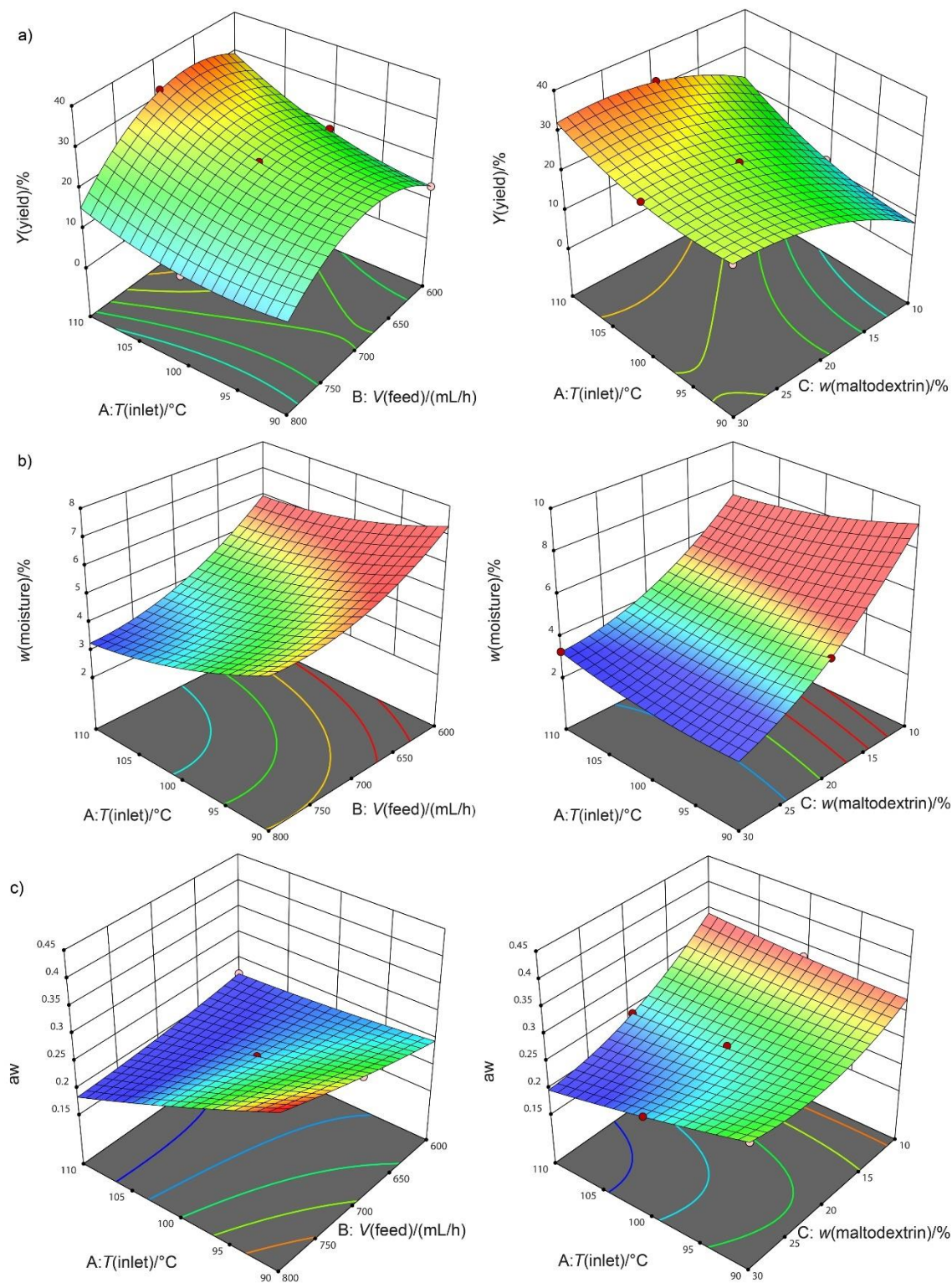
Response	Predicted value	Experimental value	Relative error/%
Y/%	35.20	34.98	0.62
$w(\text{moisture})/\%$	3.27	3.31	1.13
a_w	0.22	0.21	2.33
HR	1.21	1.22	1.16
CI/%	17.53	17.65	0.68
S/%	91.09	92.10	1.11

Table 3. Quality indicators of the optimised spray-dried red pitahaya (*Hylocereus polyrhizus*) powder

Parameter	Experimental value
$w(\text{betalains})/(\text{mg/g})$	1.98 ± 0.01
$w(\text{total phenolics as GAE})/(\text{mg/100 g})$	112.14 ± 0.80
$\text{IC}_{50}/(\text{mg/mL})$	5.81 ± 0.03

Results are expressed as mean value $\pm$ standard deviation ($N=3$)

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Fig. 1. Three-dimensional response surface plots showing the effects of inlet temperature, feed flow rate and maltodextrin mass per volume ratio on: a) yield, b) moisture content, and c) water activity of spray-dried pitahaya powder

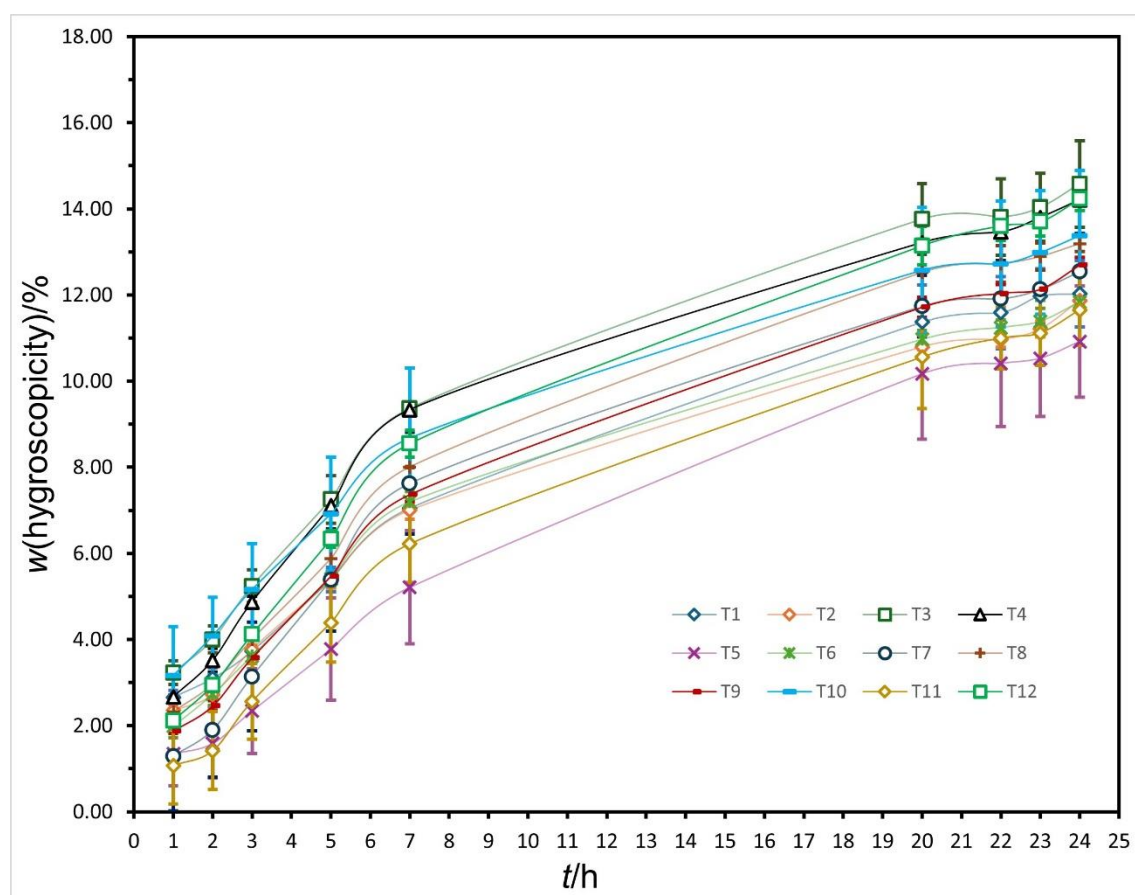


Fig. 2. Hygroscopicity of spray-dried pitahaya powder as a function of exposure time for the 12 treatments

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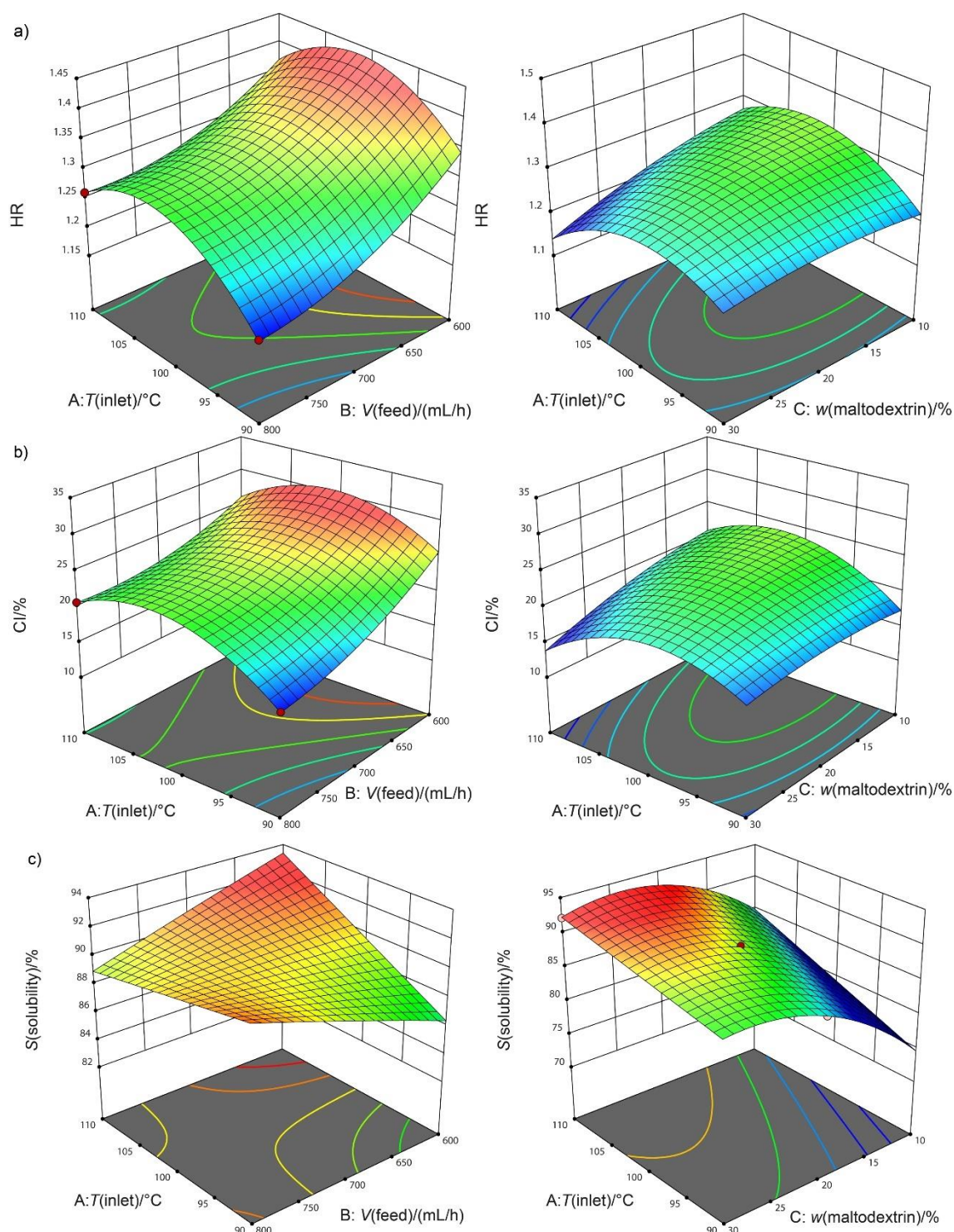


Fig. 3. Three-dimensional response surface plots showing the effects of inlet temperature, feed flow rate and maltodextrin mass per volume ratio on: a) Hauser ratio (HR), b) Carr's index, and c) solubility of spray-dried pitahaya powder

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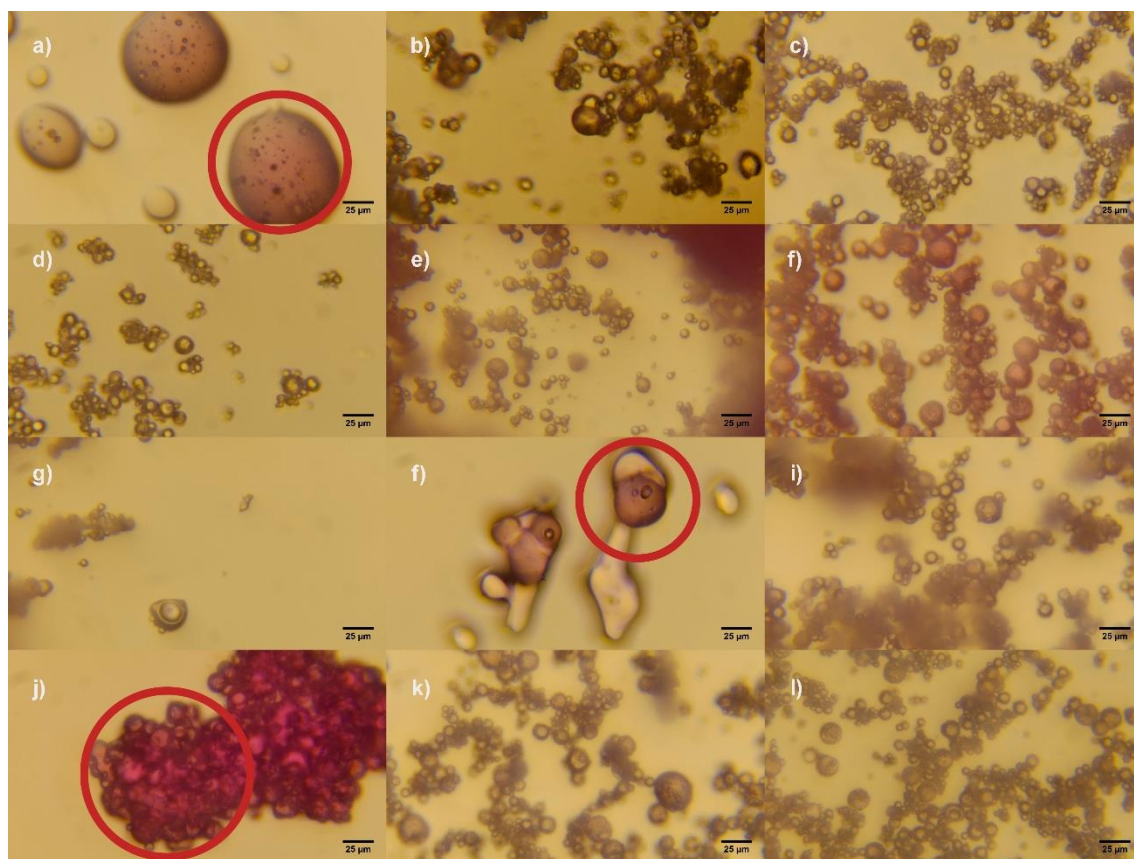
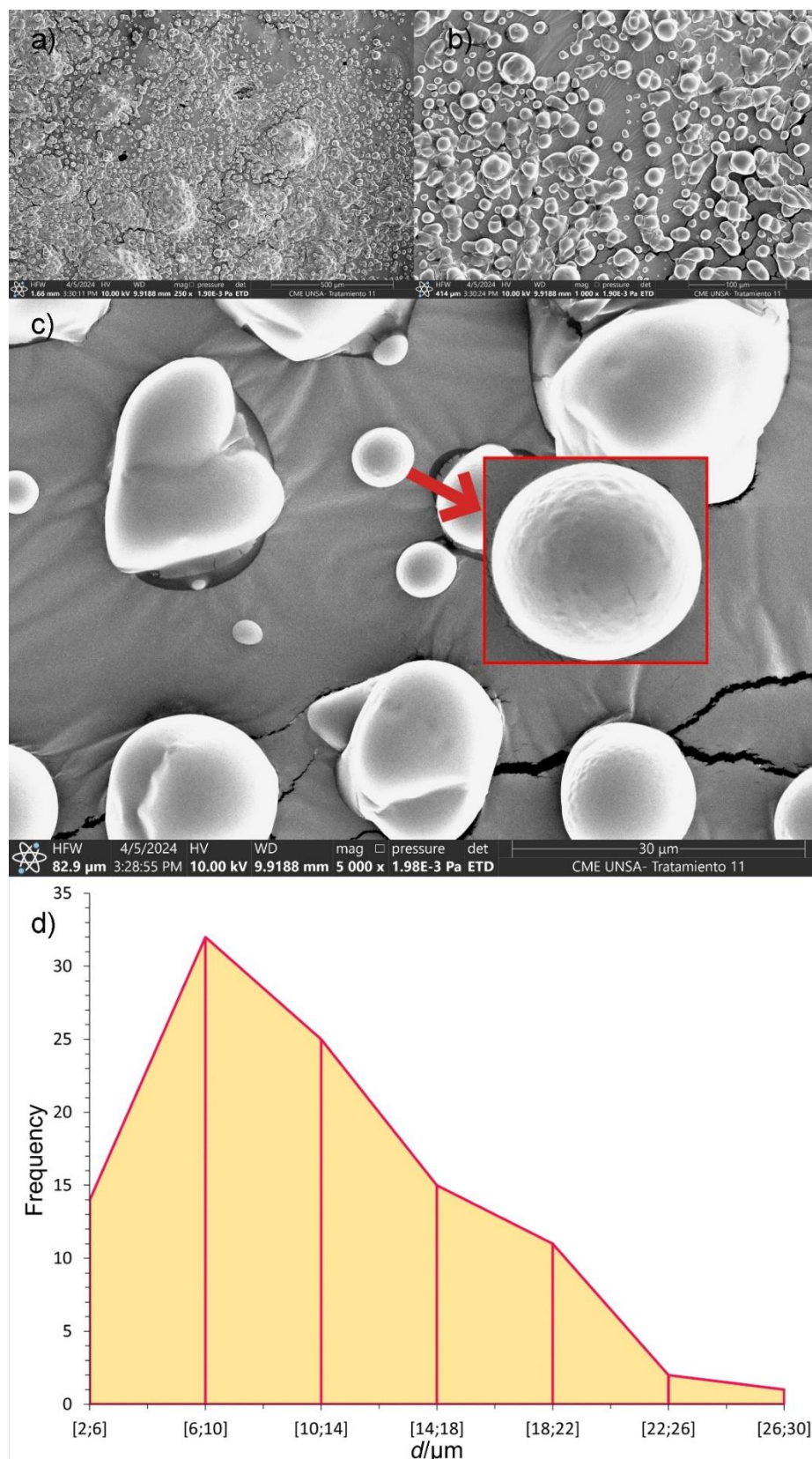


Fig. 4. Optical microscopy images of pitahaya powder at 40× magnification for treatments T1 (a) to T12 (l)

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Fig. 5. SEM images and particle size distribution of spray-dried pitahaya powder obtained under the optimised conditions: 110 °C inlet air temperature, 600 mL/h feed flow rate, and 30 % maltodextrin. Images show: a) 250×, b) 1000×, and c) 5000× magnification, and d) particle size distribution ($N=100$)

SUPPLEMENTARY MATERIALS

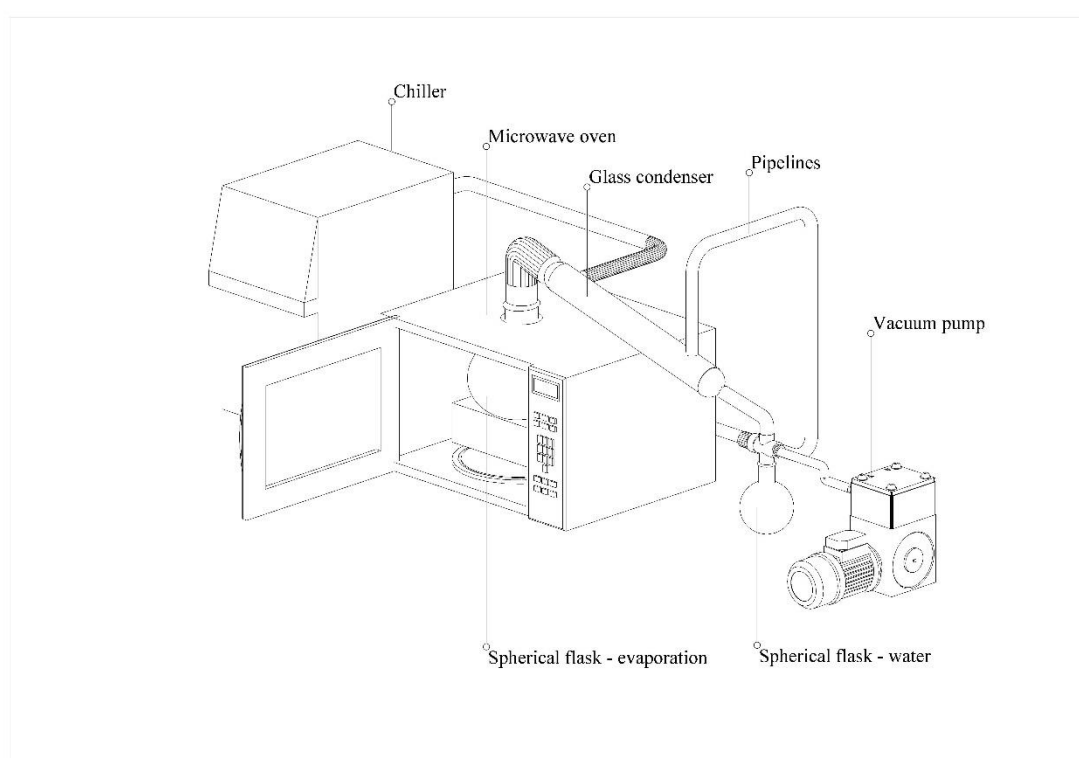


Fig. S1. Schematic representation of the microwave-assisted vacuum system

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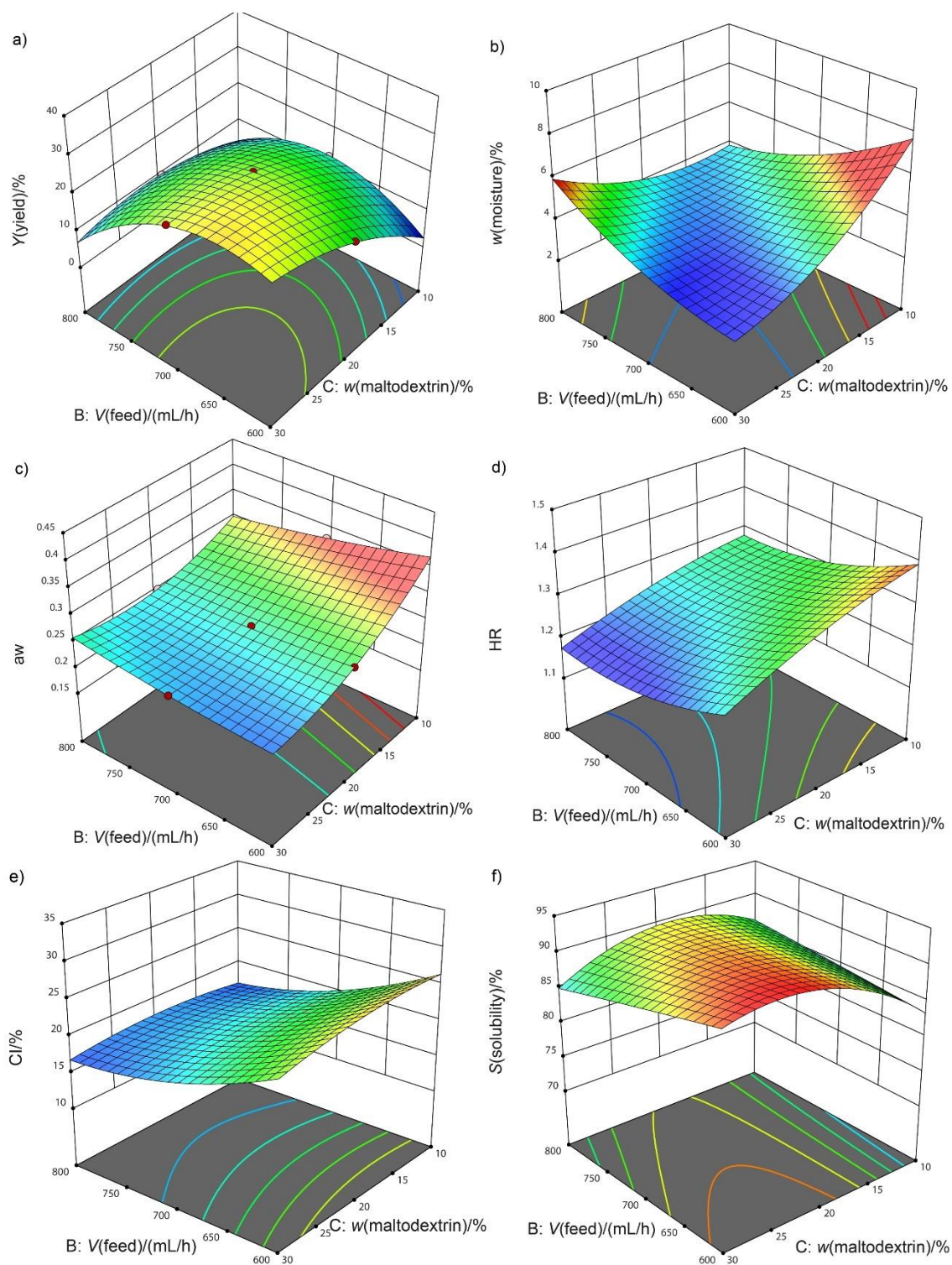


Fig. S2. Additional three-dimensional response surface plots showing the effects of feed flow rate and maltodextrin mass per volume ratio on: a) yield, b) moisture content, c) water activity, d) Hauser ratio, e) Carr's index and f) solubility of spray-dried pitahaya powder

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and maltodextrin mass per volume ratio, and B×C for the interaction between feed flow rate and maltodextrin mass per volume ratio

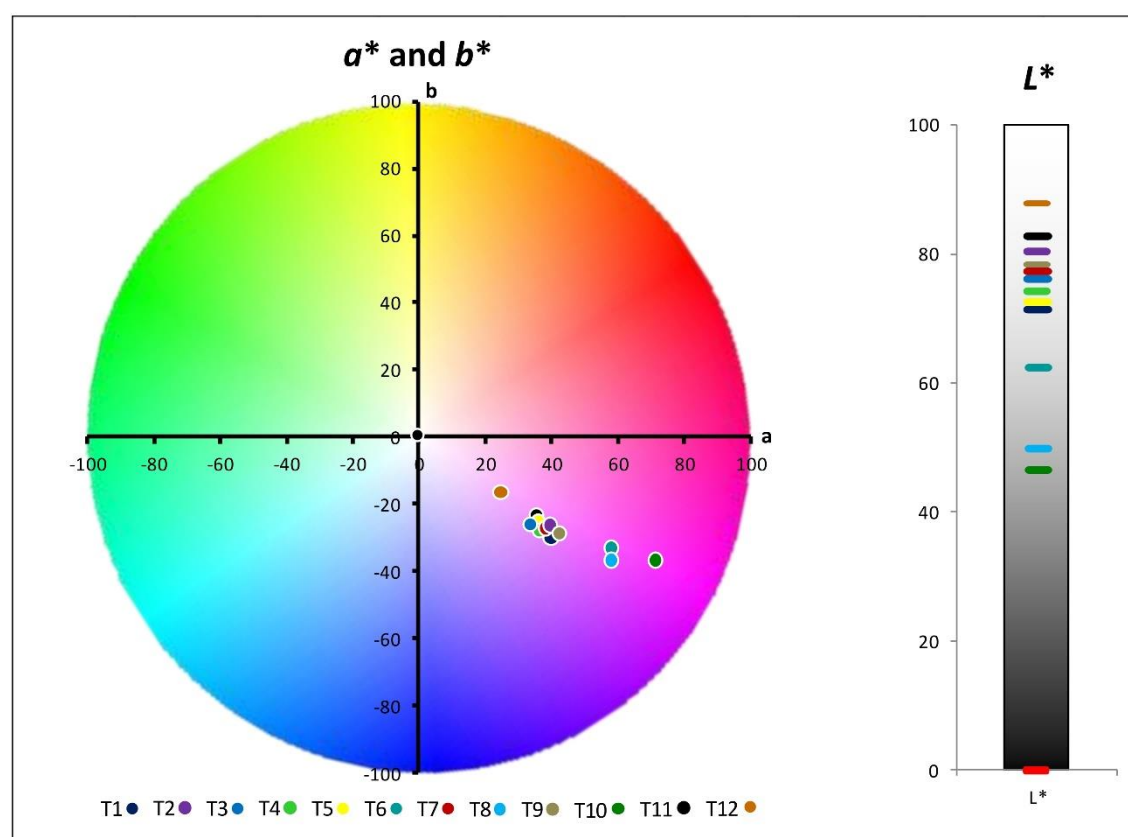


Fig. S3. CIE $L^*a^*b^*$ colour space diagram showing the distribution of spray-dried red pitahaya powder treatments. L^* represents lightness, a^* the green-red axis, and b^* the blue-yellow axis. Negative a^* and b^* values indicate green and blue tendencies, respectively, while low L^* values indicate a darker appearance

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Table S1. Experimental design parameters and responses for spray-dried red pitahaya (*Hylocereus polyrhizus*) powder

Treatment	A: <i>T</i> (inlet)/°C	B: <i>V</i> (feed)/(mL/h)	C: <i>w</i> (maltodextrin)/%	<i>Y</i> /%	<i>w</i> (moisture)/%	<i>a_w</i>	HR	CI/%	S/%	<i>L</i> *	<i>a</i> *	<i>b</i> *
T1	100	700	10	12.27	5.47	0.33	1.333	25.00	82.79	71.65	37.58	-29.67
T2	100	700	20	23.10	3.69	0.26	1.321	24.29	89.59	80.46	37.18	-25.41
T3	90	600	20	13.66	5.02	0.26	1.345	25.64	85.09	76.35	31.47	-25.42
T4	100	600	20	18.81	4.41	0.27	1.409	29.03	89.08	74.38	34.62	-27.45
T5	90	700	30	25.34	3.82	0.29	1.222	18.18	88.10	72.81	33.73	-24.83
T6	100	700	30	26.01	3.64	0.24	1.266	21.03	88.40	62.46	55.61	-32.53
T7	100	800	20	9.67	4.20	0.25	1.288	22.39	90.19	77.41	36.36	-26.20
T8	110	800	10	11.69	3.27	0.28	1.261	20.75	86.33	49.99	55.40	-36.31
T9	100	700	20	23.40	3.63	0.25	1.308	23.53	89.70	78.50	40.14	-27.94
T10	90	800	10	3.36	5.81	0.33	1.191	16.06	85.38	46.84	68.66	-36.16
T11	110	600	30	35.19	3.28	0.22	1.211	17.45	92.15	82.83	33.29	-23.26
T12	110	700	20	31.84	3.48	0.24	1.233	18.92	91.24	87.93	22.52	-15.54

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Table S2. Analysis of variance (p-values) of models describing physicochemical responses of red pitahaya powder

Parameter	Y/%	w(moisture)/%	a_w	HR	CI/%	S/%	L^*	a^*	b^*
Regression (p value)	0.002	0.0012	0.0174	0.016	0.0096	0.0053	0.4819	0.477	0.2208
A: $T(\text{inlet})/^{\circ}\text{C}$	0.0023	0.0013	0.0181	0.258	0.2304	0.0062	0.3389	0.2711	0.132
B: $V(\text{feed})/(\text{mL/h})$	0.0041	0.0277	0.0794	0.0083	0.0056	0.0347	0.538	0.3838	0.6369
C: $w(\text{maltodextrin})/\%$	0.0018	0.0004	0.0041	0.0192	0.0133	0.0023	0.8256	0.7067	0.9243
A×B	0.0331	0.0347	0.0252	0.3459	0.1302	0.0102	0.5722	0.44	0.2632
A×C	0.0738	0.0032	0.0363	0.0796	0.0654	0.0289	0.96	0.5845	0.8777
B×C	0.0307	0.0014	0.1312	0.6606	0.5836	0.0343	0.4913	0.3062	0.1694
A^2	0.0163	0.0084	0.4482	0.0094	0.0056	0.9919	0.5011	0.2817	0.0811
B^2	0.0026	0.0025	0.427	0.0668	0.0503	0.7744	0.9673	0.6199	0.8485
C^2	0.012	0.0013	0.0261	0.1965	0.147	0.0028	0.4742	0.6994	0.3588
Lack of fit (p value)	0.2029	0.7262	0.9398	0.5166	0.7304	0.1584	0.0573	0.0856	0.2657
R^2	0.9995	0.9997	0.9961	0.9964	0.9979	0.9988	0.864	0.8659	0.9461
Adjusted R^2	0.9975	0.9985	0.9786	0.9803	0.9882	0.9935	0.2522	0.2622	0.7034
Predicted R^2	0.8372	0.9808	0.9719	0.3214	0.8494	0.5585	-52.4911	-51.2542	-16.9039
Model	quadratic	quadratic	quadratic	quadratic	quadratic	quadratic	quadratic	quadratic	quadratic

The values represent the p-values obtained from the ANOVA analysis. Values less than 0.05 are considered statistically significant. A^2 indicates the quadratic effect of the inlet air temperature, B^2 corresponds to the quadratic effect of the feed flow rate, and C^2 represents the quadratic effect of maltodextrin mass per volume ratio. Interaction effects are denoted as follows: A×B for the interaction between inlet air temperature and feed flow rate, A×C for the interaction between inlet air temperature