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Influence of the feed composition and the spray drying process on the quality of a powdered mixture of blackberry (*Rubus glaucus* Benth)

Influencia de la composición del alimento y el proceso de secado por aspersión en la calidad de una mezcla de mora en polvo (*Rubus glaucus* Benth)

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Abstract

The Castile blackberry (*Rubus glaucus* Benth) is a fruit rich in active components, which is framed within the context of functional foods, to provide well-being to the human being. The objective of the research was to evaluate the effect of the feed composition and the spray drying process (SD) on the quality of the blackberry powdered mixture (BPM). The response surface methodology was used with a central composite face-centered design (α=1), considering the following independent variables: inlet air temperature (IAT) (140 - 160 °C), outlet air temperature (OAT) (80 - 90 °C), atomizer disk speed (ADS) (20000 - 24000 rpm) and maltodextrin (MD) (5 - 9 %). The dependent variables were water activity (a_w), humidity (X_w), solubility (S), wettability (W_e), hygroscopicity (H), angle of repose (AR), particle size (Span), total phenols (TP), antioxidant activity (ABTS and DPPH), anthocyanins (A), ellagic acid (EA) and product yield (Y). In general, W_e is the most critical variable in BPM, being statistically affected by most of the independent variables and by their linear and quadratic interactions. On the other hand, the experimental optimization of multiple responses defined the independent variables as IAT (159.3 °C), OAT (89.3 °C), ADS (24000 rpm), MD (5.5 %), and the dependent variables as a_w (0.166±0.002), X_w (7.10±0.05 %), S (88.01±0.17 %), W_e (54.4±1.3 min), AR (7.4±3.7°), Span (1.69±1.04), TP (4513.1±23.5 mg AGE/100g dry base (db), ABTS (4012.7±34.0 mg TE/100g db), DPPH (5359.4±39.1 mg TE/100g db), A (281.2±29.4 mg C_3G/100g db), EA (2653.0±155.0 mg/100g db) and Y (46.4±13.2 %). The spray drying (SD) process provides effective protection for the active compounds of the blackberry extract. In addition, it guarantees physicochemical stability for storage.

Keywords: active compounds, physicochemical stability, antioxidant capacity.

Resumen

La mora de Castilla (*Rubus glaucus* Benth) es una fruta rica en componentes activos, que se enmarca dentro del contexto de los alimentos funcionales, para aportar bienestar al ser humano. El objetivo de la investigación fue evaluar el efecto de la composición del alimento y el proceso de secado por aspersión (SD) sobre la calidad de la mezcla de polvo de mora (BPM). Se utilizó la metodología de superficie de respuesta con un diseño central compuesto centrado en las caras (α=1), considerando las siguientes variables independientes: temperatura del aire de entrada (IAT) (140 - 160 °C), temperatura del aire de salida (OAT) (80 - 90 °C), velocidad del disco atomizador (ADS) (20000 - 24000 rpm) y maltodextrina (MD) (5 - 9 %). Las variables dependientes fueron actividad de agua (a_w), humedad (X_w), solubilidad (S), humectabilidad (W_e), higroscopicidad (H), ángulo de reposo (AR), tamaño de partícula (Span), fenoles totales (TP), actividad antioxidante (ABTS y DPPH), antocianinas (A), ácido elágico (EA) y rendimiento de producto (Y). En general, W_e es la variable más crítica en BPM, estando estadísticamente afectada por la mayoría de las variables independientes y por sus interacciones lineales y cuadráticas. Por otro lado, la optimización experimental de respuestas múltiples definió las variables independientes como IAT (159.3 °C), OAT (89.3 °C), ADS (24000 rpm) y MD (5.5 %) y las variables dependientes como a_w (0.166±0.002), X_w (7.10±0.05 %), S (88.01±0.17 %), W_e (54.4±1.3 min), AR (7.4±3.7°), Span (1.69±1.04), TP (4513.1±23.5 mg AGE/100g base seca (db), ABTS (4012.7±34.0 mg TE/100g db), DPPH (5359.4±39.1 mg TE/100g db), A (281.2±29.4 mg C_3G/100g db), EA (2653.0± 155.0 mg/100g db) y Y (46.4±13.2 %). El proceso de secado por aspersión (SD) proporciona protección efectiva a los compuestos activos del extracto de mora. Además, garantiza estabilidad fisicoquímica para el almacenamiento.

Palabras clave: compuestos activos, estabilidad fisicoquímica, capacidad antioxidante.

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1 Introduction

The World Health Organization (WHO) has identified fruits and vegetables as essential components of a healthy diet, as they can reduce the risk of some non-communicable diseases, such as heart disease and certain types of cancer. Therefore, worldwide, consumer trends point to foods that contain beneficial components that provide consumers with both physical and mental well-being, as well as emotional well-being, which is why some natural ingredients from fruits, vegetables, grains, seeds and botanicals (Majumder & Annegowda, 2021).

The blackberry (*Rubus glaucus* Benth) is a fruit belonging to the Rosaceae family, genus Rubus, commonly known as Castile blackberry or Andean blackberry, is part of the berries, whose term is commonly used to describe any small fruit, with a sweet flavour and rounded shape (Toledo-Martín it et al., 2018). Berries are considered the primary sources of phenolic compounds, and in general, the level of antioxidants is four times higher than other fruits, ten times higher than vegetables and 40 times higher than cereals (Gonçalves da Rosa it et al., 2014). The blackberry is characterized by its nutritional qualities, standing out as a source of minerals, vitamins (A, B, C, E, among others), calcium, phenolic compounds and carotenoids (Morales it et al., 2021). Within the group of fruits, the blackberry is one of the essential antioxidants. It is considered a functional fruit due to the presence of various bioactive components to which antioxidant properties, anti-inflammatory, chemopreventive and antimicrobial activity, effects on blood lipids and atherosclerosis, resulting in the prevention of chronic diseases such as cancer, cardiovascular diseases, and neurodegenerative diseases (Schulz & Chim, 2019).

Spray drying (SD) is a one-step procedure in which the fluid feed is sprayed into droplets, which come into contact with hot air and are converted directly into dry encapsulated powder particles (Martínez-Preciado it et al., 2022). This technique allows the microencapsulation of active components, which are protected in a matrix or wall system against possible adverse reactions (Albarrán-Mondragón it et al., 2022), against undesirable effects of light, humidity and O_2, thus contributing to increasing the useful life of the product and promoting a controlled release of the encapsulated (Jafari it et al., 2021).

The blackberry is considered a promising crop in the process of diversification of Colombian agriculture and being part of the list of fruits with the highest consumption share at the national level. The physicochemical, morphological, and physiological characteristics make this fruit one of the most labile in the Colombian fruit and vegetable chain, being highly perishable and susceptible to mechanical damage, which represents a limiting factor during post-harvest. Therefore, it is necessary to look for alternatives that allow this problem to be corrected and that, in addition, provide added value to blackberry in order to strengthen the agro-chain. The present investigation contributes to the generation of a novel product, with greater use of the total solids (TS) provided by the fruit since most of the investigations show that the TS provided by the drying additives are higher than those of the fruit, giving the product low sensory characteristics.

In this context, the research objective was to evaluate the influence of the feed composition and the SD process on the quality of a blackberry powdered mixture (BPM).

2 Materials and methods

2.1 Materials

Blackberry concentrate (BC) (Nutrium SAS Colombia. °Brix at 20ºC: 13 - 15 %. citric acidity; 3.1 - 5.5 %. pH at 20 °C: 2.5 - 3.5), maltodextrin (MD) with dextrose equivalent 18 -20 (Ingredion Colombia SA) and gum arabic (GA) (TIC Pretested® Gum Arabic FT Powder. TIC Gums. USA). The BPM consisted of BC extract and the additives MD and GA.

2.2 Spray drying

Batches of 2000 g of feed to the spray dryer were prepared, which consisted of a mixture of a diluted suspension of BC with 10.45 % TS, AG (0.4%) and MD according to the experimental design. A Silverson L5M series homogenizer (Chesman, England) operated at 10,000 rpm, 10 min and controlled temperature in a thermostatic bath at 25 °C was used. A pilot SD (Vibrasec PASALAB 1.5, co-current flow) operating at subatmospheric pressure (81.6 kPa) and a circulating airflow (0.6 m^3/h) was used. For the evaluation of the SD process, the response surface methodology was used, with a central design composed of a centred face (α=1);

the independent variables were: MD (5 - 9 %), inlet air temperature (IAT) (120 - 160 °C), outlet air temperature (OAT) (70 - 90 °C) and atomizer disk speed (ADS) (20000 - 24000 rpm); and the dependent variables: moisture (X_w), water activity (a_w), solubility (S), wettability (W_e), hygroscopicity (H), angle of repose (AR), particle size (Span), total phenols (TP), antioxidant activity (ABTS and DPPH), anthocyanins (A), ellagic acid (EA) and product yield (Y).

2.3 Characterization of materials

a_w: Association of Analytical Communities (AOAC) method 978.18, using a dew-point hygrometer at 25 °C (Aqualab 3TE, Decagon, WA, USA). X_w: AOAC 925.09 method, using a drying oven (UFE 400, Memmeert GmbH, Germany). S: modified method proposed by Al-Maqtari it et al. (2021) weighing 0.5 g sample in a falcon tube and adding 50 mL of water; then it is stirred in a vortex at 24,000 rpm for 2 min and centrifuged (Hettich Universal, Model 320R, Germany) at 3,100 rpm for 10 min (Al-Maqtari it et al., 2021). Subsequently, a 25 mL aliquot of the supernatant was taken and dried in an oven at 105 °C for 1 h, recording the weight after heating and expressing it as (%) by weight difference. W_e: it was determined according to the methodology described by (Sarabandi it et al., 2019). H: was determined according to the static sorption isotherm method using 2 g of sample in a relative humidity environment controlled by a saturated solution of potassium iodide (KI) (a_w KI 25 °C = 0.689). AR: it was determined according to the methodology described in Saini it et al. (2021), using 10 g of sample and a height of 10 cm. Span: based on the 10, 50 and 90 % percentiles (Span = (D90 -D10) / D50), the Mastersizer 3000 particle analyzer (Malvern Instruments Ltd, Worcestershire, UK), Aero S cell, 0.5 absorption index and 1.45 refractive index.

2.4 BPM functional activity

0.1 g of BPM was extracted with 40 mL of a methanol/water mixture (70:30); the hydroalcoholic extract obtained was used in the following analyses. TP: it was determined by the Folin-Ciocalteu colourimetric method described by da Silva it et al. (2018). A mixture of the hydroalcoholic extract (100 μL) and 400 μL of an aqueous solution of Na_2CO_3 (0.07 M) was stirred at 2000 rpm for 1 min and allowed to stand for 5 min; subsequently, 500 μL of Folin-Ciocalteu reagent (0.2 N) was added, stirred and left in the dark for two h. The absorbance of the mixture was read in a UV/VIS spectrophotometer (Thermo Scientific Evolution 60) at 760 nm. The results were expressed as mg gallic acid equivalent (GAE)/100g BPM db.

2.4.1 Antioxidant activity. ABTS method

A solution of ABTS (2,2-azinobis (3-ethylbenzothiazoline-6-sulfonic acid) 7 mM was mixed with a solution of potassium persulfate (2.45 mM) in a 1:1 ratio; the mixture was allowed to react at room temperature, and in the absence of light for 16 h until the formation of the radical cation ABTS The solution was diluted in ethanol until a working solution with the absorbance of 0.70 ± 0.2 at 754 nm was obtained Subsequently, 50 μL of the BPM hydroalcoholic extract were added to an aliquot of 950 μL of the ABTS solution, stirred in a vortex at 2000 rpm for 1 min and left in the dark for 7 min at room temperature. At 734 nm, the percentage of radical inhibition was calculated by comparing the absorbance of a reagent blank. The results were expressed as mg Trolox equivalent (TE)/100g BPM db (da Silva it et al., 2018).

2.4.2 Antioxidant activity. DPPH method

A methanolic solution of the radical DPPH (6-hydroxy-2, 5,7, 8-tetramethylchromane-2-carboxylic acid) 0.025 mM the absorbance of the solution was adjusted to 0.70 ± 0.2 at 517 nm. In Eppendorf tubes, 950 μL of DPPH solution and 50 μL of the hydroalcoholic extract of BPM were added, stirred at 2000 rpm for 30 s and allowed to react for 30 min the dark at room temperature. After the time, the absorbance of the solution at 517 nm was determined, and the percentage of radical inhibition was calculated by comparing the absorbance with a reagent blank. The results were expressed as mg TE/100g BPM db (Rodríguez-Gutiérrez it et al., 2019).

2.5 Anthocyanins and ellagic acid

In a Falcon tube (50 mL), 0.250 g of BPM were mixed with 25 mL of acidified methanol (1 % HCl). The extraction was carried out in an ultrasonic bath for 15 min; then, it was centrifuged at 8000 rpm for 15 min at 25 ºC and filtered through a 0.45 μm membrane. The procedure was repeated (15 and 10 mL, respectively), and the extracts obtained were combined and diluted to 50 mL in a volumetric flask. Finally, a volume of the extract was filtered through a 0.22 μm membrane into an amber vial and analyzed by HPLC. The contents of Anthocyanins (A) expressed as mg Cyanidin-3-glucoside (C_3G)/100g BPM db and ellagic acid (EA) (mg/100g BPM db) were determined by HPLC (Shimadzu, USA Manufacturing INC.), detector diode array (SPD-20A), with pumping system (LC-20CE), C18 column (Phenomenex Luna ® 5 μm C18(2) 100A 250x4.6 mm) and mobile phase acetonitrile/water/formic acid (80: 18:2), in gradient mode at a constant flow (0.5 mL/min). Chromatograms were processed with Chromeleon 7.2 software. (Dionex, Thermo Scientific, USA) Furthermore, C_3G and EA quantification was performed at a wavelength of 517 and 284 nm and a retention time of 40.83 and 59.67 min, respectively.

2.6 *Product yield*

The Y of the process was expressed as a percentage from the relationship between the TS of the BPM obtained in the cyclone and the TS of the feed to the SD.

2.7 *Statistic analysis*

The results were analyzed from an analysis of variance (ANOVA), with a significance level of 95 % (α=0.05), using the Statgraphics Centurion XVII software (version 17.2.00 /2016). The dependent variables (evaluated in triplicate) were modelled according to a second-order equation (equation 1): Y (dependent variable), β_0 (constant), β_A, β_B, and β_C (coefficients of the independent variables A, B, and C, respectively); β_{AB}, β_{AC}, and β_{BC} (coefficients of linear variable interactions), and β_A^2, β_B^2, and β_C^2 (coefficients of quadratic interactions). The models were fitted according to the lack of fit method and the regression coefficient (R^2). The experimental optimization was carried out by setting weights, impacts, and criteria to obtain BPMs with the best quality attributes. The validation of the models was carried out from the relative mean error (RME) (eq. 1), comparing the values of the dependent variable predicted by the model and the experimental one at the optimal condition (3 replicates) (eq. 2).

$$Y = \beta_0 + \beta_A A + \beta_B B + \beta_C C + \beta_{AB} AB + \beta_{AC} AC + \beta_{BC} BC + \beta_A^2 A^2 + \beta_B^2 B^2 + \beta_C^2 C^2 \quad (1)$$

$$RME = \left| \frac{\text{Model value-Experimental value}}{\text{Model value}} \right| \times 100 \quad (2)$$

Table 1. Experimental results of the blackberry powder mixtures (BPM) properties obtained by spray drying (SD).

Run	**IAT** (°C)	**OAT** (°C)	**ADS** (rpm)	**MD** (%)	a_w (%)	**Xw** (%)	**S** (%)	W_e (min)	**H** (%)	**AR** (°)
1	140	80	24000	5	0.231 ± 0.004	9.7 ± 0.16	87.6 ± 0.3	28.6 ± 1.3	15.8 ± 0.1	9.3 ± 0.3
2	150	85	22000	7	0.228 ± 0.013	7.75 ± 0.11	90.0 ± 0.1	22.5 ± 1.3	16.4 ± 0.2	4.2 ± 0.3
3	160	90	20000	5	0.203 ± 0.006	8.04 ± 0.12	89.1 ± 0.5	50.6 ± 3.3	17.4 ± 0.1	7.4 ± 1.5
4	140	90	24000	9	0.203 ± 0.007	4.23 ± 0.09	89.0 ± 0.8	41.9 ± 4.1	16.2 ± 0.0	5.5 ± 1.6
5	150	85	24000	7	0.187 ± 0.003	4.79 ± 0.15	87.2 ± 1.5	30.8 ± 4.3	16.6 ± 0.1	6.8 ± 0.3
6	140	80	20000	5	0.216 ± 0.001	8.63 ± 0.07	84.0 ± 1.7	11.6 ± 2.2	18.1 ± 0.1	6.1 ± 0.6
7	150	85	22000	5	0.198 ± 0.003	7.26 ± 0.14	86.7 ± 0.2	23.3 ± 3.5	17.0 ± 0.0	10.4 ± 0.7
8	150	80	22000	7	0.212 ± 0.003	6.75 ± 0.07	88.9 ± 0.4	16.8 ± 7.4	16.4 ± 0.1	7.0 ± 0.7
9	140	90	20000	9	0.165 ± 0.009	5.53 ± 0.04	89.6 ± 0.5	19.5 ± 3.4	16.8 ± 0.0	6.1 ± 0.5
10	160	80	20000	9	0.205 ± 0.005	5.28 ± 0.05	88.5 ± 1.6	23.1 ± 5.0	16.8 ± 0.1	6.7 ± 0.6
11	150	85	22000	7	0.196 ± 0.009	5.99 ± 0.08	87.3 ± 0.6	27.6 ± 1.6	16.5 ± 0.2	7.6 ± 0.0
12	150	85	22000	9	0.210 ± 0.012	7.25 ± 0.16	88.2 ± 0.3	35.7 ± 4.3	16.1 ± 0.1	5.2 ± 0.7
13	160	90	24000	5	0.186 ± 0.005	8.95 ± 0.07	85.2 ± 0.8	31.4 ± 5.5	17.2 ± 0.0	9.2 ± 0.7
14	160	85	22000	7	0.231 ± 0.006	8.93 ± 0.03	84.5 ± 1.6	35.2 ± 3.9	15.5 ± 0.0	5.9 ± 0.3
15	160	80	24000	9	0.236 ± 0.010	6.25 ± 0.04	89.5 ± 0.3	19.7 ± 6.1	14.7 ± 0.1	11.1 ± 0.4
16	150	90	22000	7	0.141 ± 0.006	5.69 ± 0.10	87.9 ± 0.6	10.9 ± 2.2	17.9 ± 0.1	5.4 ± 0.1
17	150	85	22000	7	0.202 ± 0.003	7.17 ± 0.06	87.0 ± 0.7	24.2 ± 2.6	17.0 ± 0.0	5.6 ± 0.2
18	150	85	22000	7	0.233 ± 0.001	7.43 ± 0.07	86.7 ± 0.8	20.3 ± 1.7	13.5 ± 0.3	13.2 ± 1.6
19	150	85	20000	7	0.193 ± 0.008	7.08 ± 0.09	86.5 ± 1.1	14.4 ± 0.4	15.7 ± 0.1	5.8 ± 0.3
20	140	85	22000	7	0.189 ± 0.008	7.28 ± 0.10	87.3 ± 0.6	14.6 ± 1.2	17.0 ± 0.0	4.1 ± 1.1
21	150	85	22000	7	0.224 ± 0.006	8.08 ± 0.07	83.9 ± 0.9	27.8 ± 6.5	16.1 ± 0.1	6.0 ± 0.3

BPM: blackberry powdered mixture; SD: spray drying; IAT: inlet air temperature; OAT: outlet air temperature; ADS: atomizer disk speed; MD: maltodextrin; a_w: water activity; X_w: moisture; S: solubility; W_e: wettability; H: hygroscopicity; AR: angle of repose.

Table 2. Experimental results of the blackberry powder mixtures (BPM) properties obtained by spray drying (SD).

Run	TP (mg GAE/100g db)	ABTS (mg TE/100g db)	DPPH (mg TE/100g db)	A (mg C3G/100g db)	EA (mg/100g db)	Y (%)
1	4407.1 ± 71.2	4112.0 ± 66.8	4257.3 ± 231.4	305.6 ± 5.7	2756.7 ± 16.9	26.2
2	4086.2 ± 17.0	3910.0 ± 23.5	4004 ± 227.3	293.7 ± 1.7	2865.3 ± 5.8	20.6
3	4724.5 ± 124.5	4614.5 ± 99.6	5086.5 ± 51.7	244.4 ± 15.9	2462.3 ± 45.2	19.5
4	3961.2 ± 31.0	3812.6 ± 47.1	3728.1 ± 10.4	281.0 ± 10.4	3085.8 ± 24.4	53.7
5	4157.2 ± 14.3	4293.7 ± 160.7	4651.9 ± 255.7	275.6 ± 4.9	3022.0 ± 33.2	49
6	4830.5 ± 24.2	2306.3 ± 55.7	4834.9 ± 225.1	291.3 ± 2.0	2694.8 ± 38.9	51.9
7	5186.9 ± 10.1	3124.8 ± 126.4	4488.7 ± 133.2	269.3 ± 9.3	2308.8 ± 56.7	43.4
8	4492.4 ± 35.0	2690.0 ± 408.6	3915.5 ± 188	264.7 ± 3.3	2266.3 ± 5.3	44.2
9	4321.5 ± 15.4	4079.6 ± 128.0	5025.2 ± 129.9	225.0 ± 6.1	2273.9 ± 41.2	62.9
10	4131.7 ± 89.7	3923.4 ± 228.7	4442.0 ± 187.8	239.0 ± 2.3	2125.0 ± 34.1	53.4
11	4489.7 ± 83.5	4883.1 ± 46.9	5445.7 ± 209.1	238 ± 13.6	2359.8 ± 14.9	45.6
12	4129.2 ± 49.4	3681.9 ± 78.9	4383.8 ± 187.2	239.4 ± 6.4	2650.0 ± 44.9	54.1
13	4632.3 ± 21.9	4975.7 ± 206.7	5640.6 ± 204.0	288.5 ± 9.3	2954.1 ± 5.2	63.6
14	4336.2 ± 0.3	5383.4 ± 207.3	4961.8 ± 75.1	262.5 ± 8.0	2777.9 ± 18.1	43
15	4045.1 ± 52.3	3440.4 ± 151.7	4011.5 ± 150.2	226.6 ± 4.5	1848.6 ± 23.7	53.4
16	4652.2 ± 36.9	4155.5 ± 233.5	4981.4 ± 47.4	246.8 ± 7.8	2387.4 ± 31.5	27.2
17	4506.5 ± 17.3	4581.4 ± 49.6	4950.7 ± 122.6	272.8 ± 1.2	2404.5 ± 25.4	48.5
18	4655.5 ± 16.6	3615.1 ± 149.1	4480.8 ± 117.5	252.1 ± 3.8	2721.8 ± 36.2	58
19	4681.3 ± 6.4	4428.9 ± 63.4	5019.8 ± 115.0	263.4 ± 6.1	2507.7 ± 19.0	56
20	4716.2 ± 25.9	4151.6 ± 184.9	5024.6 ± 122.2	263.2 ± 5.1	2635.0 ± 0.7	60.7
21	4557.4 ± 18.5	4102.4 ± 221.2	4777.3 ± 95.8	231.1 ± 49.9	2816.2 ± 26.1	39.5

BPM: blackberry powdered mixture; SD: spray drying; TP: total phenols; ABTS and DPPH: antioxidant activity methods; A: anthocyanins; EA: ellagic acid; Y: yield.

Table 3. ANOVA of the blackberry powder mixtures (BPM) obtained by spray drying (SD).

Variable	Major effects				Quadratic effects				Lineal interactions					
	IAT	OAT	ADS	MD	IAT^2	OAT^2	ADS^2	MD^2	IAT-OAT	IAT-ADS	IAT-MD	OAT-ADS	OAT-MD	ADS-MD
a_w	0.1466	0.0384	0.3077	0.6345	0.2361	0.1383	0.6167	0.4621	0.5125	0.4508	0.2169	0.6209	0.2169	0.2032
X_w	0.2184	0.4018	0.8127	0.9934	0.0719	0.2531	0.1297	0.5041	0.0506	0.4038	0.8342	0.3433	0.2896	0.365
S	0.412	0.755	0.9153	0.6435	0.4871	0.3412	0.9553	0.7239	0.762	0.3993	0.6212	0.2168	0.3863	0.9235
W_e	0.011	0.2711	0.0315	0.0546	0.1627	0.0203	0.59	0.0164	0.0304	0.0025	0.015	0.3214	0.0459	0.0818
H	0.4788	0.498	0.3735	0.6513	0.8616	0.4363	0.7902	0.8577	0.9871	0.853	0.7045	0.3995	0.5834	0.9596
Span	0.6877	0.9908	0.8538	0.8235	0.8669	0.7232	0.9918	0.8986	0.9366	0.8124	0.9748	0.7781	0.6872	0.8933
AR	0.7394	0.7606	0.4203	0.3522	0.6443	0.9703	0.9326	0.8963	0.4575	0.7415	0.9494	0.5538	0.9881	0.8786
TP	0.2857	0.632	0.0975	0.0266	0.7343	0.9804	0.3152	0.5799	0.2034	0.3825	0.7792	0.9303	0.3289	0.9166
ABTS	0.1638	0.1127	0.4724	0.4841	0.0878	0.1225	0.383	0.1143	0.4089	0.315	0.5405	0.4436	0.5191	0.1139
DPPH	0.938	0.2329	0.28	0.8967	0.4334	0.494	0.7087	0.4747	0.5535	0.2584	0.5303	0.8699	0.6646	0.3245
A	0.9862	0.6482	0.2335	0.4591	0.8478	0.8251	0.5703	0.7631	0.8235	0.6244	0.7825	0.2488	0.5663	0.8491
EA	0.6902	0.7348	0.0978	0.3638	0.4811	0.1467	0.3064	0.4878	0.1237	0.3793	0.5923	0.0851	0.2523	0.9794
Y	0.4191	0.4356	0.963	0.6135	0.552	0.2942	0.5067	0.7846	0.8361	0.1146	0.3986	0.198	0.4948	0.5224

BPM: blackberry powdered mixture; SD: spray drying; IAT: inlet air temperature; OAT: outlet air temperature; ADS: atomizer disk speed; MD: maltodextrin; a_w: water activity; X_w: moisture; S: solubility; W_e: wettability; H: hygroscopicity; AR: angle of repose; TP: total phenols; ABTS and DPPH: antioxidant activity methods; A: anthocyanins; EA: ellagic acid; Y: yield.

3 Results and discussion

According to the experimental design, Tables 1, 2 and 3 present the mean values plus the standard deviation of the BPM obtained by SD and ANOVA, respectively.

3.1 Humidity and water activity

Figure 1 presents the response volume graphs of the X_w and a_w of the BPM obtained by SD, where the mean values fluctuated between 0.141 - 0.236 and 4.2 - 9.7%, respectively. These values guarantee microbiological and physicochemical stability, fundamental for determining useful life. The a_w presented a significant difference ($p<0.05$) to the linear effect of OAT, while the X_w did not present significant differences ($p>0.05$) for the independent variables, nor with their linear or quadratic interactions. However, due to the hygroscopic nature of dry materials, BPMs require packaging with low permeability to water vapor and even O_2 for storage to minimize possible

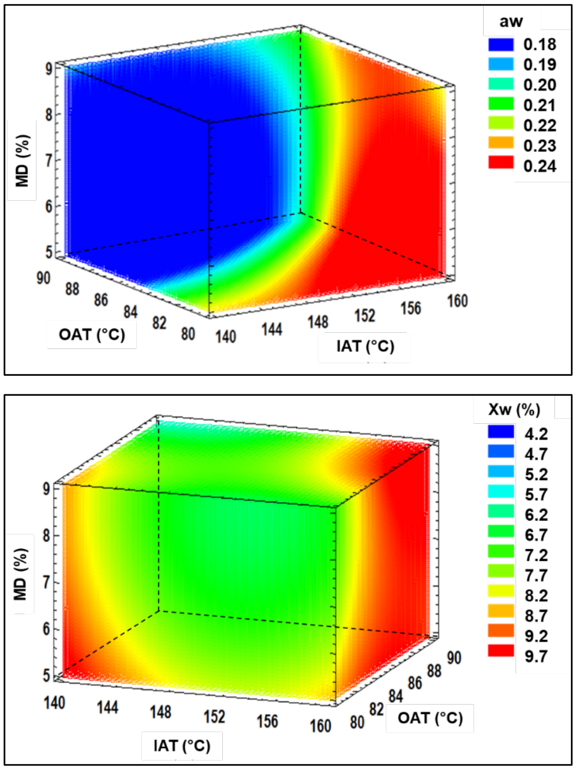

Figure 1. Response volume graphs of the water content (X_w) and water activity (a_w) of the blackberry powdered mixture (BPM) obtained by spray drying (SD).

chemical and enzymatic deterioration reactions. A study of freeze-and spray-drying of blackberry juice with carrier agents reported that the moisture content in the final product was 6-6.11 % and 3.4-3.7 %, respectively (Bhatta it et al., 2020).

The response volume graph showed a decrease in a_w with increasing OAT, where BPM experiences a higher vapour pressure that contributes to reducing X_w and a_w (Rybak it et al., 2020). On the other hand, this behaviour was consistent with the greater heat transfer given to the microdroplets, where the increase in OAT revalidates a lower a_w and X_w. a_w values in the range found have been reported in blackberry powders of different varieties: 0.15-0.29 in Rubus fruticosus (Ferreira it et al., 2018) and 0.163 in Rubus spp (Franceschinis it et al., 2014).

3.2 *Wettability*

Figure 2 presents the response surface and volume graphs of the W_e of the BPM obtained by SD. The W_e was the most critical variable of the BPM, fluctuating its average values in a wide range (10.9 - 50.6 min), which denotes the difficulty of the diffusion or penetrability of the water inside the particles; therefore, its instantaneous characteristics are not considered the most favourable in conditions free of external agitation or mixing. This situation is evidenced in the ANOVA, which showed that W_e presented significant differences ($p<0.05$) for the main effects of IAT and ADS, the quadratic OAT and MD effects, and the linear interactions IAT-OAT, IAT-ADS, IAT-MD and OAT-MD.

The response of the W_e is high to the operating conditions imposed on the SD process, mainly by the IAT and its linear interactions. A decrease in W_e is observed at low IAT and with the interaction IAT (140 - 146 °C) - ADS (20000 - 21000 rpm) or IAT (140 - 146 °C) - MD (5 - 6 %), which could be attributed to the lower thermal efficiency caused by low IAT, increasing the moisture content of the BPM and, at the same time, the agglomeration of the particles due to the binding bridges that the water forms with the MD. This situation contributes to an agglomeration of particles, whose predominance of the particle size distribution was reflected in the Span between values of 1 - 2, allowing a more excellent penetrability of the water inside the agglomerated particles (Ferrari it et al., 2012). Therefore, the ADS is conditioned the drying surface area and surface tension, which translates into how the particles were individually wetted (Ermis, 2021) and how they contributed to the binding bridges between particles. The response volume plot illustrates the SD operating conditions that make W_e more favourable (blue region): IAT (140 - 146 °C), MD (5 - 8 %), ADS (20,000 - 22,000 rpm) and in the entire OAT range. Some authors have reported better times for W_e (116.2 s) in blackberry powders encapsulated with various carrier agents and using fresh blackberry as raw material instead of blackberry concentrate (Ferrari it et al., 2012).

3.3 *Solubility, hygroscopicity, particle size and angle of repose*

Figure 3 presents the response surface and volume graphs of the S, H, Span and AR of the BPM, with values between (83.9 - 90.0 %), (13.5 - 18.1 %), (0.39 - 5.83) and (4 .1 - 13.2°) respectively). However, the ANOVA did not present significant differences ($p>0.05$) of these dependent variables for the independent variables, nor with the linear or quadratic interactions. In general, the values of S were greater than 83 %, indicating that the BPM have good

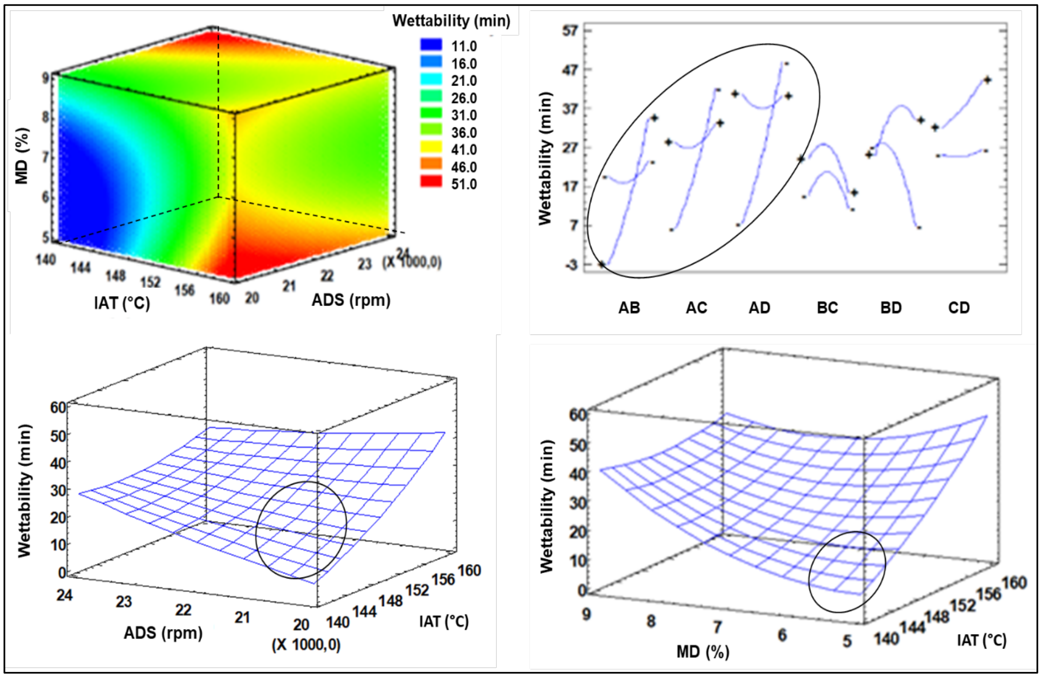

Figure 2. Volume and response surface plots of the blackberry powdered mixture (BPM) wettability (We).

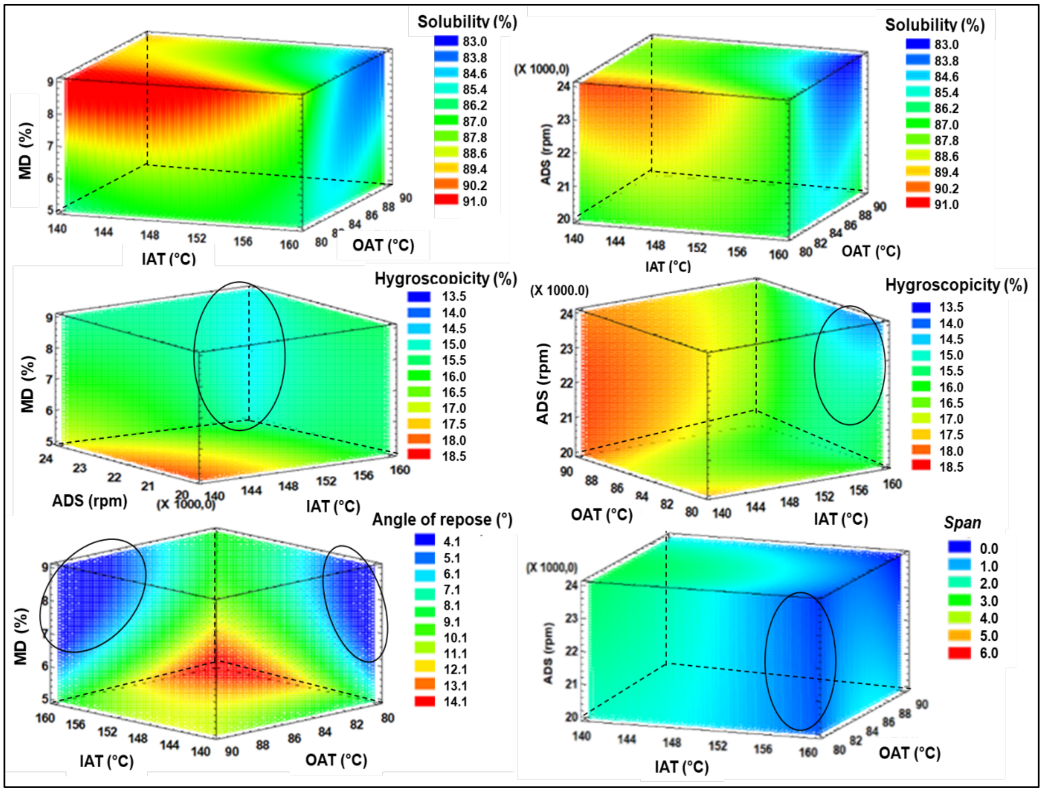

Figure 3. Response volume graphs of the solubility (S), hygroscopicity (H), angle of response (AR) and Span of the blackberry powdered mixture (BPM).

reconstitution properties when subjected to external forces. In addition, these results were consistent with the values found for H. The response volume graphs show that the most favourable conditions for S were IAT: 140 - 152 °C, OAT: 80 - 84 °C, MD: 7 - 9 % and ADS: 23,000 24,000 rpm. Compared to other berry powders, some research has reported the following S results: raspberry powder (95.4 %) (Gagneten it et al., 2019), strawberry powder (88.57 - 99.97 %) (Balcitorun & Ozdemir, 2021), Rhodomyrtus berry powder (27.57-31.31 %), and raspberry powder (38.53-45.26 %).

On the other hand, it is observed that BPM presents significant levels of water absorption, initially caused by the formation of a monolayer of water molecules on the surface, due to the affinity of polar compounds (anthocyanins, organic acids, vitamins), among others, with the surrounding water molecules. The formation of multilayers of water molecules due to the Van der Waals forces between them is presented (Bastías-Montes it et al., 2018). Finally, the water condenses due to the energetic interactions of the sorption system and diffuses into the BPM (Liu it et al., 2019). This situation contributes to the possible loss of the glassy amorphous state reached by the BPM in the SD process due to the high rate of water evaporation, configuring greater mobility of water inside the structure, and an increase in the rate of deterioration of the product or loss of available quality when reaching a rubbery amorphous state.

Response volume graphs show that the most favourable conditions for H were as follows: IAT: 156 - 160 °C, OAT: 80 - 84 °C, MD: full range, and ADS: 22,000 - 24,000 rpm. Some H values in berries obtained by SD have been reported: chokeberry powder, 14.3 - 18.2 % and maqui powder, 21.1 - 24.0 % (Bastías-Montes it et al., 2018).

The AR fluctuated between 4.1 and 13.2°, which denotes a good fluidity of the BPM, a situation that could be favored by the presence of the MD, which in general reduces the Van der Waals forces and the cohesiveness of the dust particles. In the response volume graph, two regions are observed where the operating conditions favour AR: 1) IAT (156-160 °C), OAT (88-90 °C), MD (6.5 - 9.0 %) and ADS (over the entire range); and 2) IAT (144-140 °C), OAT (82-80 °C), MD (6.5-9.0 %) and ADS (over the entire range). In blackberry powders, they found variations for AR, according to the wall material used: MD (28.07°) and GA (44.98°) (Díaz it et al., 2015), in blackberry juice powders with different modifications. Physical values for potato starch (18.76 - 35.16°) and maqui berry (*Aristotelia chilensis*) atomized powders, values of 22.3 ± 1.6° have been reported.

The response volume graphs show that the most favourable conditions for the Span were IAT: 152 - 160 °C, OAT, ADS and MD: in the entire range. The fluctuation of particle sizes is related to the humidity reached the OAT of the SD, where the formation of agglomerates is evident with the affinity of its components for water molecules. Various investigations in berry powder matrices have reported the following results: raspberry, blackcurrant and elderberry powders, 1.23, 1.17 and 1.12, respectively (Gagneten it et al., 2019), strawberry powder, 2.11 (Pellicer it et al., 2019); and maqui powder, 1.06 - 2.0 (Makinistian it et al., 2020).

3.4 Total phenols and antioxidant activity

Figure 4 presents the volume response graphs of TP, A, EA content and antioxidant activity (ABTS and DPPH) of BPM obtained by SD. The active components content and the antioxidant activity of the BC used during the investigation were as follows: TP (4274.2 mg AGE/100g db), ABTS (5284.8 mg TE/100g db), DPPH (5580.7 mg TE/ 100g db), A (301.50 mg C_3G/100g db) and EA (607.26 mg/100g db). The bibliography mainly reports active components values associated with blackberries in the fresh state; while in blackberry transformed products, TP values and antioxidant activity are highlighted in bayberry suspension (TP: 14.7 mg GAE/100g and A: 37.9 mg C_3G/100g) (Samborska it et al., 2019). On the other hand, during SD, high process temperatures can adversely affect the structure of phenolic compounds, causing structural changes and the formation of different compounds, thus reducing antioxidant activity (Khalifa it et al., 2019). The variability of these active components in the BC is affected by various factors, some of which are typical of primary production: genetics, agronomic practices, soils, environmental conditions, rainfall, state of maturation, among others; in addition, other factors associated with the vacuum concentration process during its manufacture (Madrigal-Gamboa it et al., 2021).

For BPM, TP content and antioxidant capacity (ABTS and DPPH) presented mean values that fluctuated between (3961.2 - 5186.9 mg GAE/100g BPM db), (2306.3 - 5383.3 mg TE/100g BPM db) and (3728.1 - 5640.6 mg TE/100g BPM db), respectively. The ANOVA showed significant differences of the TP ($p<0.05$) to the content of MD, while ABTS and

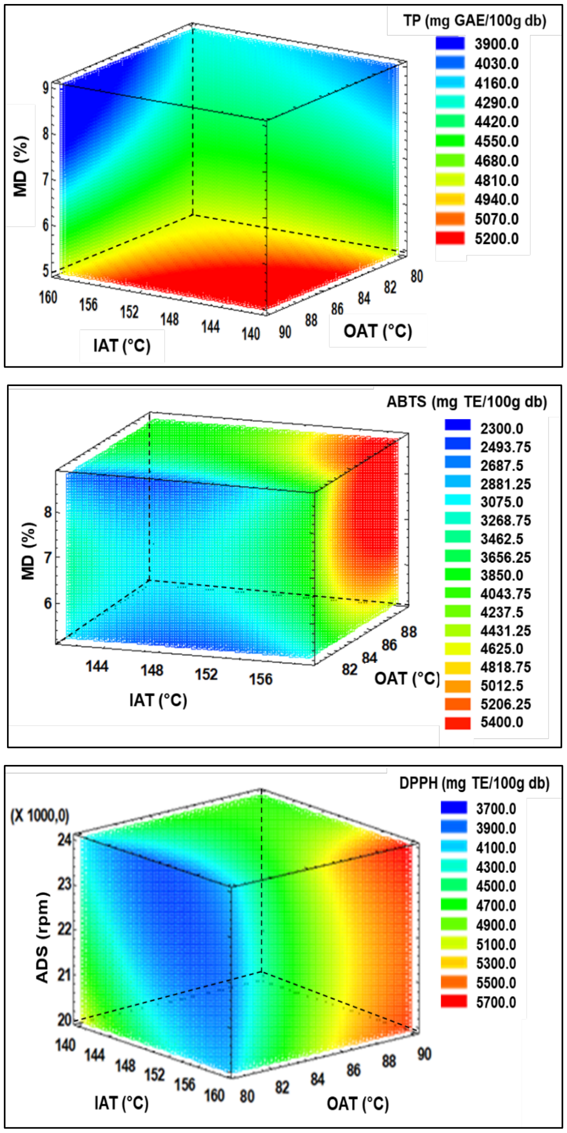

Figure 4. Response volume graphs of total phenols (TP) and blackberry powdered mixture (BPM) antioxidant activity.

DPPH did not present significant statistical differences (p>0.05) with any of the independent variables, nor with their linear or quadratic interactions. The response volume graph identifies that the content of TP increased when the formulations fed to the SD contained a lower concentration of MD, which suggests a more significant recovery by extraction of the TP when less encapsulation is present in the BPM. In this context, the SD operating conditions favour a higher TP content was: 140 °C, OAT: 90 °C, ADS: 21127 rpm and MD: 5.0 %.

On the other hand, ABTS and DPPH presented similar behaviours, where the most favourable conditions tend to be located throughout the MD (ABTS) and ADS (DPPH) range and at higher operating temperatures (IAT: 156 - 160 °C and OAT: 85 - 90 °C), where the drier particles could be allowing a better extraction [48]. Under these conditions, there is evidence of an effective encapsulation action by the MD and GA, forming a thin film around the droplet, protecting the BPM from thermal degradation and oxidation (Nogueira it et al., 2020). Additionally, it is highlighted that the GA contains hydroxyl groups (OH) in its molecular structure, which enables the formation of hydrogen bonds with the phenols, the anthocyanins and the water of the feed suspension to the SD; in this way, the OH groups promote the interaction between the wall material and the encapsulated compounds to form the coating matrix (Colín-Cruz it et al., 51). Farias-Cervantes it et al. (2018) reported DPPH values of 6.2 and 4.4 mg TE/100g, respectively, for blackberry *Rubus adenotrichos* L. and *Rubus fruticosus* L, and ABTS of 406.2 and 379.8 mg TE/100g, respectively. A similar study reported antioxidant activity values of 2.5 mg/mL ± 0.01 for *Rubus fruticosus* extract from Beni Messaouar.

3.5 *Anthocyanins and ellagic acid*

Figure 5 presents the response volume graphs of the A and AE contents of the BPM obtained by SD; the values fluctuated in ranges of 225.0 - 305.6 mg C_3G/100g BPM db and 1848.6 - 3085.8 mg/100g BPM db, respectively. The ANOVA did not show significant statistical differences (p>0.05) of the A and the AE for the independent variables, nor with the linear or quadratic interactions; however, it is observed that the region with the highest content of A (reddish-yellow zone) corresponds to the operating conditions: IAT (156 - 160 °C), OAT (80 - 86°C), MD (5 - 6 %) and ADS (23,000 - 24,000 rpm); while the EA content is favoured when the system operates at IAT (156 - 160 °C), OAT (85 - 90 °C), MD (7 - 9 %), ADS (22000 - 24000 rpm).

The A's are sensitive and unstable compounds during processing. Process factors such as intensity and duration of heating, presence of O_2 and light, pH of the medium, initial concentration in the substrate (juice or concentrate), and the presence of drying agents influence the stability of these compounds. In general, it was observed that the increase in temperature affected anthocyanins to a greater extent compared to EA; however, for both compounds, a

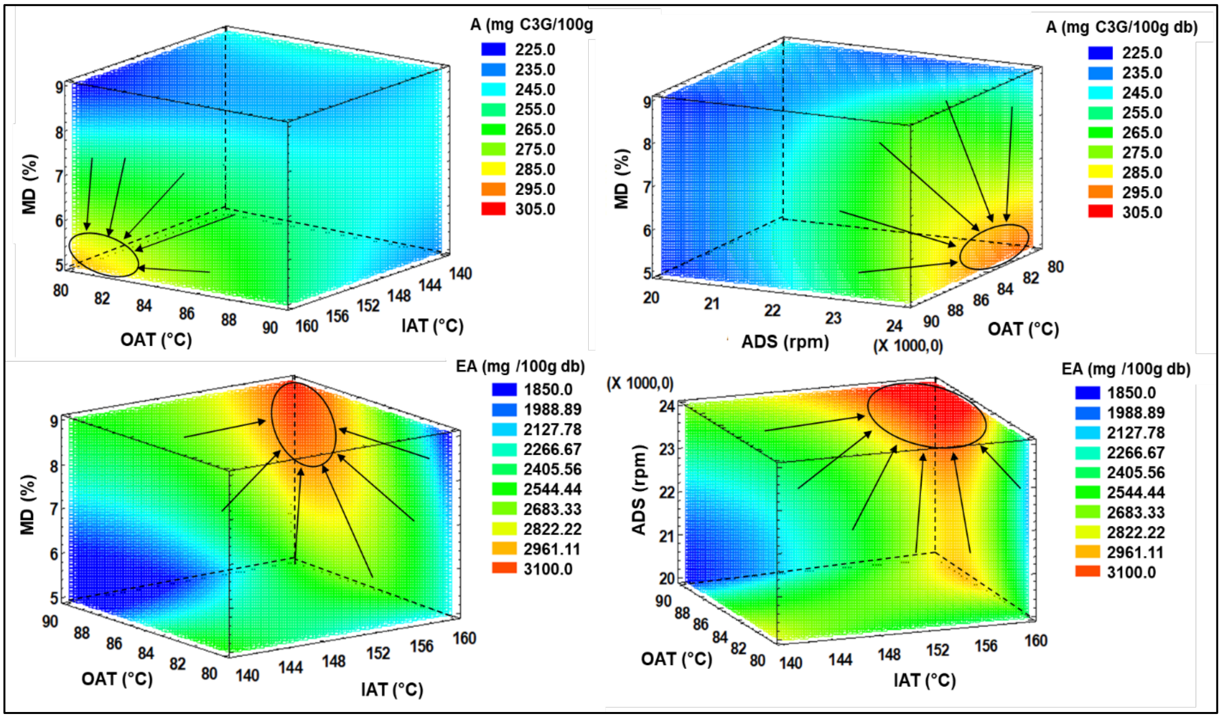

Figure 5. Response volume graphs of blackberry powdered mixture (BPM) anthocyanins (A) and ellagic acid (EA).

protective effect of the MD and GA microencapsulated was presented from the same feeding suspension to the SD, providing good stability in the AC against the initial thermal stress imposed by the IAT and in the drying chamber (Mudalip it et al., 2021). Additionally, it is considered that there is a synergy with the acid pH of the blackberry feeding suspension (3.0), contributing to the more excellent stability of the A in the BPM (Soldatkina it et al., 2017). In this context, Santos it et al. (2019) reported a positive effect on the stability of the total monomeric A of *Rubus fruticosus* at low pH values (2.0 and 3.5). On the other hand, in a study of *R. fruticosus* it was found as phenolic acids, it mainly contains gallic acid, protocatechuic acid, p-hydroxybenzoic acid, caffeic acid, p-coumaric acid, and ellagic acid (Vega it et al., 2021).

Compared to other Rubus berries (blackberries and raspberries), the fresh fruits of R. glaucus are characterized by having high EA contents, with values of the order of 393 mg/kg fresh (Schulz & Chim, 2019). Furthermore, free EA levels have increased during thermal processes, probably due to the release of hexahydroxydiphenic acid from ellagitannins and its transformation into EA (Laaksonen it et al., 2020).

In general, the content of the active compounds in the BPM presents variability that does not denote a well-defined behaviour, given various phenomena: 1) the effect of the presence of the TS provided by the drying additives ≈ 5.9 %), which reduces its value in the BPM, 2) the effect of degradation due to thermal stress during the SD process (Silva-Espinoza it et al., 2020), 3) the variability of AC in the BC of each batch, 4) to the different MD-GA interactions with the active components, which generates different structural complexes, which protect the metabolite to a greater or lesser extent and affect the diffusion of the solvent inside the matrix during its extraction. The contribution of phenolic compounds in Castile blackberry is variable and complex and includes anthocyanins, flavonoids, phenolic acids, and ellagic acid, among others. Phenolic compounds and ascorbic acid contribute significantly to the antioxidant activity of BPM; however, these compounds are labile under different processing and storage conditions. In particular, anthocyanins are degraded by cleavage, derivatization, and polymerization, while ascorbic acid is degraded by oxidation-reduction reactions to generate dihydroxy-ascorbic acid and 2,3-deketogulonic acid under certain conditions. Both degradations may be related to changes in the antioxidant capacity of the final product obtained under different SD conditions.

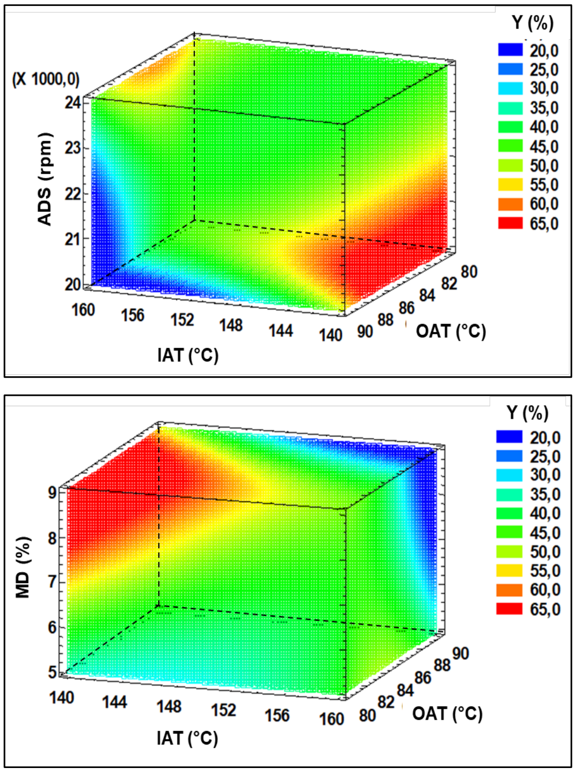

Figure 6. Response volume plots of blackberry powdered mixture (BPM) product yield (Y).

3.6 *Yield*

The mean values of Y fluctuated between 19.5 and 63.6 %; however, the ANOVA did not present significant statistical differences (p>0.05) for the independent variables, linear and quadratic interactions. Figure 6 presents the Y response volume graph of the BPM, observing that the operating conditions that favour the highest Y are presented in the red regions or zones: IAT (146 - 140 °C), OAT (80 - 88 °C), ADS (20,000 - 21,000 rpm) and MD (7 - 9 %). It is observed that when the SD feed colloidal system formulation contains more MD, the Y immediately increases. Similarly, increasing the IAT favours the adhesiveness of the material and tends to reduce the Y.

The Y in the BPM spray drying process is an essential parameter directly related to the composition of the feed. Stickiness phenomena were observed in the contact walls of the preparation and feeding tanks to the SD and in the pipes that reach the atomization system. The thermal stress imposed favoured the adhesiveness of the microparticles with the internal metal wall, forming a deposit or ring in the drying chamber (Machado it et al., 2018). On the other hand, the operation of the SD process in sub-atmospheric conditions generates a draft induced by the fan, which could favour the removal of fine particles in the cyclone (Rybak it et al., 2020).

The literature reports the positive and negative effects of IAT, highlighting that the variations presented could be related to the composition of the SD feed and specific conditions imposed during the SD process (Pellicer it et al., 2018). Farías-Cervantes it et al. (2020), during the obtaining of blackberry powder, reported a decrease in Y with increasing IAT and the concentration of the encapsulating agent (agave fructans), associating this with a problem of stickiness, which meant that the temperature of the particles was above the glass transition temperature (Tg). Quintero-Castaño it et al. (2020) reported 31.56-65.48 % in BC (*Rubus glaucus* Benth) and 64.3 - 78.3 % in blackberry juice (Farías-Cervantes it et al., 2020).

3.7 *Modelling and experimental optimization of the SD process*

Table 4 presents the regression coefficients and the R^2 of the second-order polynomial models for each dependent variable evaluated. The regression adjustment of the mathematical models allowed us to identify that approximately 70 % of the dependent variables (a_w, X_w, W_e, TP, ABTS, DPPH, A, EA and Y) presented adjustments to the experimental data with R^2 values > 75 %; while in 30 % (S, H, AR and Span) the fit was less than 75 %. These values may be due to the high number of variables (Al-Maqtari it et al., 2021) evaluated, where variables such as Span reached an R^2 of the order of 15.9 %. However, the lack of fit test did not show statistically significant differences (p> 0.05). All the dependent variables presented a random distribution of the residuals, ensuring that the data could be parameterized according to a normal distribution. Under this context, these mathematical models can be considered acceptable to explain the experimental data (Taofiq it et al., 2019).

Table 5 presents the experimental optimization of multiple responses of the BPM SD process, which illustrates the criteria defined in order to obtain the independent variables that best fit the desired BPM quality; additionally, the impacts were set according to the importance of the variable and the weights taking into account the results of the ANOVA and the permissibility that the variable moves away from or approaches the defined criterion.

Table 4. Modeling and R^2 coefficients of the 2-order polynomial models for the dependent variables of the blackberry powder mixtures (BPM).

Coefficient	aw	Xw	S	*We*	H	AR	Span	TP	ABTS	DPPH	A	EA	Y
B_0	-0.972853	434.431	-322.136	-695.021	263.38	-843.926	-285.634	-82018.3	40506.7	11707	4234.34	90199.4	596.275
IAT	-0.0449755	-6.78227	5.05888	-11.471	0.1116	6.79145	0.632826	474.262	-2596.96	-1722.54	-10.7695	-823.989	-20.9085
OAT	0.0881555	-0.681951	-4.78217	23.3803	-5.531	7.83195	4.26371	944.684	3417.61	1067.49	-26.3992	135.711	65.9722
ADS	0.0001123	0.0115593	0.0162393	0.053231	-0.00201	0.0021295	0.00386	1.00596	0.116764	-3.37149	-0.168807	-2.81299	-0.20192
MD	-0.0943308	-5.19537	19.2783	-23.5116	1.51641	-4.48961	6.2646	641.633	-52.0259	1573.89	-42.0378	-812.912	160.442
IAT2	0.000144	0.0121707	-0.010429	0.034671	-0.0016	-0.010885	-0.002524	-0.49698	7.19604	2.92257	0.0330134	1.14466	0.0563611
IAT-OAT	0.0001875	0.034975	-0.011175	0.168325	0.000375	-0.045325	0.00302	-5.24058	7.45252	5.48184	0.096975	7.24895	-0.048465
IAT-ADS	-2.44E-07	0.0000132	-0.000036	-0.00039	0.000005	0.0000218	-0.00001	0.003781	-0.010377	0.0124899	-0.000241	-0.004114	0.0004938
IAT-MD	-0.000956	-0.007062	-0.046062	-0.52406	0.02219	-0.0093125	0.003	2.58696	13.5093	14.5568	-0.300688	-5.41535	-0.518212
OAT2	-0.000764	-0.026717	0.0588841	-0.30292	0.029791	0.0034599	-0.021474	-0.14282	-25.0054	-10.1016	-0.152146	-10.6048	-0.419356
OAT-ADS	-3.13E-07	-0.00003	-0.000113	-0.00013	0.000046	-0.0000796	-0.000024	0.000718	-0.015356	0.003314	0.0012284	0.0189775	0.000757
OAT-MD	0.0019125	0.077125	-0.167375	0.733375	-0.064875	-0.004375	-0.077275	-19.164	28.5427	-19.8295	1.27137	24.9094	-0.8236
ADS^2	-1.40E-09	-2.38E-07	-2.95E-08	2.97E-07	-6.13E-08	4.91E-08	-3.84E-09	-0.00004	0.0000783	0.0000337	0.0000025	0.0000432	0.0000016
ADS-MD	0.0000022	-0.0000722	0.0000197	0.000663	-0.0000066	-0.0000428	-0.000028	0.00215	-0.091151	-0.0532543	-0.0004622	-0.000572	-0.000859
MD^2	0.0020995	0.0917675	0.129275	2.02052	0.0411943	0.421624	0.0479124	20.5374	-161.135	-66.1821	-1.30092	-28.1561	0.634903
R^2	75.4	85.6	70.3	93.5	51.9	50.2	15.9	85.8	88.4	77.3	75.1	87.3	75.6
Lack of Fick (p-value)	0.1599	0.1826	0.979	0.1016	0.5392	0.8126	0.6281	0.5093	0.7161	0.9914	0.9403	0.7823	0.8207

R^2: regression coefficient; BPM: blackberry powdered mixture; ?0: constant; IAT: inlet air temperature; OAT: outlet air temperature; ADS: atomizer disk speed; MD: maltodextrin; a_w: water activity; X_w: moisture; S: solubility; H: hygroscopicity; AR: angle of repose; TP: total phenols; ABTS and DPPH: antioxidant activity methods; A: anthocyanins; EA: ellagic acid; Y: yield.

Table 5. Experimental optimization of blackberry powder mixtures (BPM).

Variable	Criterion	Impact	Weight	Experimental value	Model value	|RME| (%)
a_w	0.188	4	Medium	0.166 ± 0.002	0.188	11.7
X_w (%)	6.9	3	Medium	7.10 ± 0.05	8.32	14.7
S (%)	Maximize	3	Low	88.0 ± 0.2	84.61	4
W_e (min)	Minimize	4	Medium	54.4 ±1.3	30.54	78.1
H (%)	Minimize	3	Low	17.25 ± 0.21	16.87	2.2
AR	Minimize	3	Low	7.4 ± 3.7	8.12	8.9
Span	Minimize	4	Medium	1.69 ± 1.04	0.62	172.6
TP (mg GAE/100g db)	Maximize	5	Medium	4513.1 ± 23.5	4443.17	1.6
ABTS (mg TE/100g db)	Maximize	4	Medium	4012.7 ± 34.0	5252.1	23.6
DPPH (mg TE/100g db)	Maximize	4	Medium	5359.4 ± 39.1	5587.52	4.1
A (mg C_3G/100g db)	Maximize	4	Medium	281.2 ± 29.4	287.57	2.2
EA (mg/100g db)	Maximize	4	Medium	2653.0 ± 155.0	3073.63	13.7
Y (%)	Maximize	5	Medium	46.4±13.2	58.6	20.8

BPM: blackberry powdered mixture; RME: relative mean error; a_w: water activity; X_w: moisture; S: solubility; H: hygroscopicity; AR: angle of repose; TP: total phenols; ABTS and DPPH: antioxidant activity methods; A: anthocyanins; EA: ellagic acid; Y: yield

In this context, the experimental optimization reflected desirability of 66.5 % and defined the optimal conditions of the independent variables: IAT: 159.3 °C, OAT: 89.3 °C, ADS: 24000 rpm and MD: 5,5 %. The values determined by the second order polynomial models and the experimental values obtained in triplicate at the optimal condition are also illustrated. The determination of the RME for each dependent variable identified that approximately 85 % of these (a_w, X_w, S, H, AR, TP, ABTS, DPPH, A, EA and Y) presented values less than 24 %, which is considered acceptable, given the number of independent variables evaluated. On the other hand, the variables W_e and Span presented values higher than 70 %, which is consistent with what was previously stated, being the variables more sensitive to the process and the water-particle interactions of the BPM.

In this context, the experimental optimization reflected desirability of 66.5 % and set the optimal conditions obtained from the independent variables: IAT: 159.3 °C, OAT: 89.3 °C, ADS: 24000 rpm and MD: 5,5 %, which provided theoretical values provided by the second order polynomial models, which, compared with the experimental values obtained in triplicate at the optimal condition, produced the values of the relative mean error (RME), where 75 % of the dependent variables (a_w, X_w, S, H, TP, DPPH, A, EA and Y) presented values lower than 20 %, which is considered acceptable, given the number of independent variables evaluated. On the other hand, the variables W_e, Span and DPPH presented values higher than 50 %, which is consistent with what was mentioned above, being the variables more sensitive to the process and the water-particle interactions of the BPM.

Conclusions

The process of obtaining BPM using SD technology allowed the development of a microbiologically stable product with potential multipurpose applications (gastronomic or as raw material for the food or pharmaceutical industry), contributing to improving the competitiveness of the agro-chain. The use of BC as raw material led to practical technological development and effective control during the preparation of the SD feed suspension, which was designed with a ratio $TS_{BC}/TS_{MD+GA} = 1.771$ and represents a contribution of the blackberry of 63.9% of TS in the microcapsules obtained. The research team considers that this value should be informed to the client or consumer.

MD and GA as encapsulating agents provided adequate protection in the active compounds present in the blackberry extract, conferring a potential antioxidant effect. The W_e was the most critical variable of the BPM, reflecting the difficulty of water diffusion inside the particles; therefore, its instantaneous characteristics are not considered the most favourable in conditions free from agitation or external mixing. The response surface methodology represented an effective statistical tool for optimising multiple responses (13 dependent variables), setting the process conditions as follows: IAT: 159.3 °C, OAT: 89.3 °C and ADS: 24000 rpm.

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References

Albarrán-Mondragón, F., Orozco-Villafuerte, J., Mulia-Rodríguez, J., Hernández-Jaimes, C., Cruz-Sosa, F., and Buendía-González, L. (2022). Total phenolic content in fruits and in in vitro cultures of *Bromelia karatas* L. *Revista Mexicana de Ingeniería Quimica 21*(1), Bio2685. https://doi.org/10.24275/rmiq/Bio2685

Al-Maqtari, Q. A., Mohammed, J. K., Mahdi, A. A., Al-Ansi, W., Zhang, M., Al-Adeeb, A., and Yao, W. (2021). Physicochemical properties, microstructure, and storage stability of *Pulicaria jaubertii* extract microencapsulated with different protein biopolymers and gum arabic as wall materials. *International Journal of Biological Macromolecules 187*, 939-954. https://doi.org/10.1016/j.ijbiomac.2021.07.180

Arabpoor, B., Yousefi, S., Weisany, W., and Ghasemlou, M. (2021). Multifunctional coating composed of *Eryngium campestre* L. essential oil encapsulated in nano-chitosan to prolong the shelf-life of fresh cherry fruits. *Food Hydrocolloids 111*, 106394. https://doi.org/10.1016/j.foodhyd.2020.106394

Bhatta, S., Stevanovic, T., and Ratti, C. (2020) Freeze-drying of maple syrup: Efficient protocol formulation and evaluation of powder physicochemical properties. *Drying Technology 38*, 9, 1138-1150. https://doi.org/10.1080/07373937.2019.1616751

Balci-torun, F., and Ozdemir, F. (2021). Encapsulation of strawberry flavour and physicochemical characterization of the encapsulated powders. *Powder Technology 380*, 602-612. https://doi.org/10.1016/j.powtec.2020.11.060

Bastías-Montes, J. M., Choque-Chávez, M. C., Alarcón-Enos, J., Quevedo-León, R., Muñoz-Fariña, O., and Vidal-San-Martín, C. (2018). Effect of spray drying at 150, 160, and 170 °C on the physical and chemical properties of maqui extract (*Aristotelia chilensis* (Molina) Stuntz). *Chilean Journal of Agricultural Research 79*, 144-152. http://dx.doi.org/10.4067/S0718-58392019000100144

Colín-Cruz, M. A., Pimentel-González, D. J., Carrillo-Navas, H., Alvarez-Ramírez, J., and Guadarrama-Lezama, A. Y. (2019). Co-encapsulation of bioactive compounds from blackberry juice and probiotic bacteria in biopolymeric matrices. *Food Science and Technology 110*, 94-101. https://doi.org/10.1016/j.lwt.2019.04.064

Correâ-Filho, L. C., Lourenço, M. M., Moldaõ-Martins, M., and Alves, V. D. (2019). Microencapsulation of ?-Carotene by spray drying: Effect of wall material concentration and drying inlet temperature. *Journal of Agricultural and Food Chemistry 2019*, 8914852. https://doi.org/10.1155/2019/8914852

da Silva, D. F., Itoda, C., Rosa, C. I. L. F., Vital, A. C. P., Yamamoto, L. N., Yamamoto, L. Y., and Matumoto-Pintro, P. T. (2018). Effects of blackberries (*Rupus sp*.; cv. *Xavante*) processing on its physicochemical properties, phenolic contents and antioxidant activity. *Food Science and Technology 55*, 11, 4642-464. https://doi.org/10.1007/s13197-018-3405-6

Díaz, D. I., Beristain, C. I., Azure, E., Luna, G., and Jimenez, M. (2015). Effect of wall material on the antioxidant activity and physicochemical properties of *Rubus fruticosus* juice microcapsules. *Journal of Microencapsulation 32*, 3, 247-254. https://doi.org/10.3109/02652048.2015.1010458

Farías-Cervantes, V. S., Salinas-Moreno, Y., Chávez-Rodríguez, A., Luna-Solano, G., Medrano-Roldan, H., and Andrade-González, I. (2020). Stickiness and agglomeration of blackberry and raspberry spray dried juices using agave fructans and maltodextrin as carrier agents. *Czech Journal of Food Science 38*, 4, 229-236. https://doi.org/10.17221/350/2018-CJFS

Farias-Cervantes, V. S., Chávez-Rodríguez, A., García-Salcedo, P. A., García-López, P. M., Casas-Solís, J., and Andrade-González, I. (2018). Antimicrobial effect and in vitro release of anthocyanins from berries and Roselle obtained via microencapsulation by spray drying. *Journal of Food Processing and Preservation 42*(10), 1-8. https://doi.org/10.1111/jfpp.13713

Ferrari, C. C., Germer, S. P. M., Alvim, I. D., Vissotto, F. Z., and de Aguirre, J. M. (2012). Influence of carrier agents on the physicochemical properties of blackberry powder produced by spray drying. *International Journal of Food Science and Technology 47*, 6, 1237-1245. https://doi.org/10.1111/j.1365-2621.2012.02964.x

Ferrari, C. C., Germer, S. P. M., and de Aguirre, J. M. (2012). Effects of spray-drying conditions on the physicochemical properties of blackberry powder. *Drying Technology 30*, 2, 154-163. https://doi.org/10.1080/07373937.2011.628429

Ferreira Nogueira, G., Matta Fakhouri, F., and Augustus de Oliveira, R. (2018). Microencapsulation of blackberry pulp with arrowroot starch and gum arabic mixture by spray drying. *Journal of Microencapsulation 35*, 5, 482-493. https://doi.org/10.1080/02652048.2018.1538264

Franceschinis, L., Salvatori, D. M., Sosa, N., and Schebor, C. (2014). Physical and functional properties of blackberry freeze- and spray-dried powders. *Drying Technology 32*, 197-207. https://doi.org/10.1080/07373937.2013.814664

Gagneten, M., Cor, R., Gómez, M., Sozzi, A., and Leiva, G. (2019). Spray-dried powders from berries extracts obtained upon several processing steps to improve the bioactive components content. *Powder Technology 342*, 1008-1015. https://doi.org/10.1016/j.powtec.2018.09.048

Gonçalves da Rosa, C., Dellinghausen Borges, C., Zambiazi, R., Kuhn, J., Rickes da Luz, Z., Döring Krumreich, F., Benvenutti, E., and Ramos Nunes, M. (2014). Encapsulation of the phenolic compounds of the blackberry (*Rubus fruticosus*). *Food Science and Technology 58*, 527-533. http://dx.doi.org/10.1016/j.lwt.2014.03.042

González, I. (2016). Effect of spray drying temperature and agave fructans concentration as carrier agent on the quality properties of blackberry powder. *International Journal of Food Engineering 12*, 5, 451-459. https://doi.org/10.1515/ijfe-2015-0287

Jafari, S. M., Arpagaus, C., Cerqueira, M. A., and Samborska, K. (2021). Nano spray drying of food ingredients; materials, processing and applications. *Trends Food Science and Technology 109*, 632-646. https://doi.org/10.1016/j.tifs.2021.01.061

Khalifa, I., Li, M., Mamet, T., and Li, C. (2019). Maltodextrin or gum arabic with whey proteins as wall-material blends increased the stability and physiochemical characteristics of mulberry microparticles. *Food Bioscience 31*, 100445. https://doi.org/10.1016/j.fbio.2019.100445

Laaksonen, O., Mäkilä, L., Jokinen, M., Metz, T., Kallio, H., and Yang, B. (2020). Impact of storage on sensory quality of blackcurrant juices prepared with or without enzymatic treatment at industrial scale. *European Journal of Food Science and Technology 246*, 12, 2611-2620. https://doi.org/10.1007/s00217-020-03601-0

Liu, W., Midya, J., Kappl, M., Butt, H. J., and Nikoubashman, A. (2019). Segregation in drying binary colloidal droplets. *ACS Nano 13*, 5, 4972-4979. https://doi.org/10.1021/acsnano.9b00459

Machado, A. P., Rezende, C. A., Rodrigues, R. A., Barbero, G. F., Rosa, P. T., and Martínez, J. (2018). Encapsulation of anthocyanin-rich extract from blackberry residues by spray-drying, freeze-drying and supercritical antisolvent. *Powder Technology 340*, 553-562. https://doi.org/10.1016/j.powtec.2018.09.063

Madrigal?Gamboa, V., Jiménez?Arias, J., Hidalgo, O., Quesada, S., Pérez, A. M., and Azofeifa, G. (2021). Membrane processing effect of blackberry (*Rubus adenotrichos*) on cytotoxic and pro?apoptotic activities against cancer cell lines. *Journal of Food Processes and Preservation 45*, e15575. https://doi.org/10.1111/jfpp.15575

Majumder, P., and Annegowda, H. V. (2021). Fruit and vegetable by-products: novel ingredients for a sustainable society. *Valorization of Agri-Food Wastes and By-Products*, 133-156. https://doi.org/10.1016/B978-0-12-824044-1.00006-4

Makinistian, F. G., Gallo, L., Sette, P., Salvatori, D., and Bucalá, V. (2020). Nutraceutical tablets from maqui berry (*Aristotelia chilensis*) spray-dried powders with high antioxidant levels. *Drying Technology 38*, 9, 1231-1242. https://doi.org/10.1080/07373937.2019.1629589

Martínez-Preciado, A., Silva-Jara, J., Flores-Nuño, B., Michel, C., Castellanos-Haro, A., Macías-Rodríguez, M. (2022). Microencapsulation by spray-drying of Manilkara zapota pulp and probiotics (*Lactobacillus fermentum* A15): Assessment of shelf-life in a food matrix. *Revista Mexicana de Ingeniería Química 20*, 2, 635-648. https://doi.org/10.24275/rmiq/Alim2166

Morales, D. P., Chim, J. F., Barin, J. S., Vizzotto, M., Farias, C. A., Ballus, C. A., and Barcia, M. T. (2021). Influence of the cultivar on the composition of blackberry (*Rubus spp.*) minerals. *Journal of Food Composition and Analysis. 100*, 103913. https://doi.org/10.1016/j.jfca.2021.103913

Mudalip, S. K. A., Khatiman, M. N., Hashim, N. A., Man, R. C., and Arshad, Z. I. M. (2021). A short review on encapsulation of bioactive compounds using different drying techniques. *Materials Today: Proceedings 42*, 288-296. https://doi.org/10.1016/j.matpr.2021.01.543

Nogueira, G. F., Soares, C. T., Martin, L. G. P., Fakhouri, F. M., and de Oliveira, R. A. (2020). Influence of spray drying on bioactive compounds of blackberry pulp microencapsulated with arrowroot starch and gum arabic mixture. *Journal of Microencapsulation 37*,1, 65-76. https://doi.org/10.1080/02652048.2019.1693646

Oancea, S. (2021). A review of the current knowledge of thermal stability of anthocyanins and approaches to their stabilization to heat. *Antioxidants 10*, 9, 1337. https://doi.org/10.3390/antiox10091337

Pellicer, J. A., Fortea, M. I., Trabal, J., Rodríguez-López, M. I., Carazo-Díaz, C., Gabaldón, J. A., and Núñez-Delicado, E. (2018). Optimization of the microencapsulation of synthetic strawberry flavour with different blends of encapsulating agents using spray drying. *Powder Technology*

338, 591-598. https://doi.org/10.1016/j.powtec.2018.07.080

Pellicer, J. A., Fortea, M. I., Trabal, J., Rodríguez-López, M. I., Gabaldón, J. A., and Núñez-Delicado, E.. (2019). Stability of microencapsulated strawberry flavour by spray drying, freeze drying and fluid bed. *Powder Technology 347*, 179-185. https://doi.org/10.1016/j.powtec.2019.03.010

Quintero-Castaño, V. D., Vasco-Leal, J. F., Cuellar-Nuñez, L., Luzardo-Ocampo, I., Castellanos-Galeano, F., Álvarez-Barreto, C., Bello-Pérez, L. A., and Cortés-Rodriguez, M. (2020). Novel OSA-modified starch from Gros Michel banana for encapsulation of Andean blackberry concentrate: production and storage stability. *Starch/Staerke 26*, 1-11. https://doi.org/10.1002/star.202000180

Rodríguez-Gutiérrez, G., Cardoso, J. C., Rubio-Senent, F., Serrano, A., Borja, R., Fernández-Bolaños, J., and Fermoso, F. G. (2019). Thermally-treated strawberry extrudate: A rich source of antioxidant phenols and sugars. *Innovative Food Science and Emerging Technologies 51*, 186-19. https://doi.org/10.1016/j.ifset.2018.05.017

Rybak, K., Samborska, K., Jedlinska, A., Parniakov, O., Nowacka, M., Witrowa-Rajchert, D., and Wiktor, A. (2020). The impact of pulsed electric field pretreatment of bell pepper on the selected properties of spray dried juice. *Innovative Food Science and Emerging Technologies 65*, 102446. https://doi.org/10.1016/j.ifset.2020.102446

Saini, A., Panwar, D., Panesar, P. S., and Bera, M. B. (2021). Encapsulation of functional ingredients in lipidic nanocarriers and antimicrobial applications: a review. *Environmental Chemistry Letters 19*, 2, 1107-1134. https://doi.org/10.1007/s10311-020-01109-3

Samborska, K., Jedli?ska, A., Wiktor, A., Derewiaka, D., Wo?osiak, R., Matwijczuk, A., and Witrowa-Rajchert, D. (2019). The effect of low-temperature spray drying with dehumidified air on phenolic compounds, antioxidant activity, and aroma compounds of rapeseed honey powders. *Food Bioprocess Technology 12*, (6), 919-932. https://doi.org/10.1007/s11947-019-02260-8

Santos, S. S., Rodrigues, L. M., Costa, S. C., & Madrona, G. S. (2019). Antioxidant compounds from blackberry (*Rubus fruticosus*) pomace: Microencapsulation by spray-dryer and pH stability evaluation. *Food Packaging and Shelf Life 20*, 100177. https://doi.org/10.1016/j.fpsl.2017.12.001

Sarabandi, K., Jafari, S. M., Mahoonak, A. S., and Mohammadi, A. (2019). Application of gum Arabic and maltodextrin for encapsulation of eggplant peel extract as a natural antioxidant and color source. *International Journal of Biological Macromolecules 140*, 59-68. https://doi.org/10.1016/j.ijbiomac.2019.08.133

Schulz, M., & Chim, J. F. (2019). Nutritional and bioactive value of Rubus berries. *Food Bioscience 31*, 100438. https://doi.org/10.1016/j.fbio.2019.100438

Soldatkina, L. M., Novotna, V. O., and Salamon, I. (2017). Degradation kinetics of anthocyanins in acidic aqueous extracts of berries. ?????? ???, 22, 61, 55-66. https://doi.org/10.18524/2304-0947.2017.1(61).94711

Silva-Espinoza, M. A., Ayed, C., Foster, T., Camacho, M. D. M., and Martínez-Navarrete, N. (2020). The impact of freeze-drying conditions on the physico-chemical properties and bioactive compounds of a freeze-dried orange puree. *Foods 9*, 1, 32. https://doi.org/10.3390/foods9010032

Taofiq, O., Corrêa, RC, Barros, L., Prieto, MA, Bracht, A., Peralta, RM, and Ferreira, IC. (2019). A comparative study between conventional and non-conventional extraction techniques for the recovery of ergosterol from *Agaricus blazei* Murrill. *Food Research International 125*, 108541. https://doi.org/10.1016/j.foodres.2019.108541

Toledo-Martín, E. M., García-García, M. D. C., Font, R., Moreno-Rojas, J. M., Salinas-Navarro, M., Gómez, P., and Río-Celestino, D. (2018). Quantification of total phenolic and carotenoid content in blackberries (*Rubus Fructicosus* L.) using near infrared spectroscopy

(NIRS) and multivariate analysis. *Molecules 23*, 12, 3191. https://doi.org/10.3390/molecules23123191

Vega, E. N., Molina, A. K., Pereira, C., Dias, M. I., Heleno, S. A., Rodrigues, P., and Barros, L. (2021). Anthocyanins from *Rubus fruticosus* L. and *Morus nigra* L. applied as food colorants: A natural alternative. *Plants 10*, 6, 1181. https://doi.org/10.3390/plants10061181