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Influence of process conditions on the physicochemical properties of jussara pulp (*Euterpe edulis*) powder produced by spray drying

Influência das condições de processo nas propriedades físico-químicas de pó de polpa de juçara (Euterpe edulis) produzido por atomização

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Abstract

The objective of this work was to optimize the spray drying of jussara pulp using mixtures of modified starch (MS) with whey protein concentrate (WPC) or soy protein isolate (SPI) as the carrier agents. Two central composite rotatable designs were used to evaluate the effect of the independent variables of inlet air temperature (140 °C to 200 °C), carrier agent concentration - CAC (0.5 to 2 g carrier agent/g jussara pulp solids) and the proportions of MS:WPC or MS:SPI (5 to 30 g WPC or SPI/100 g carrier agent) on the following responses for powders formulated with MS:WPC and MS:SPI, respectively: moisture content (0.3% to 1.4% and 0.6% to 1.2%), solubility (78.0% to 92.9% and 78.9% to 83.8%), retention of total anthocyanins (49.2% to 82.9% and 34.1% to 96.9%), encapsulation efficiency (98.5% to 99.7% and 98.5% to 99.5%), hue angle (9.1 to 44.0 and 3.7 to 42.6), chroma (10.0 to 15.3 and 9.2 to 14.3) and process yield (33.2% to 55.5% and 49.9% to 78.5%). The inlet air temperature 170 °C, CAC of 1.25 and 2 g/g jussara pulp solids and proportion of MS:WPC or MS:SPI of 17.5 and 30 g/100 g were recommended as the selected conditions.

Keywords: *Euterpe edulis Martius; Whey protein concentrate; Modified starch; Soy protein isolate; Response surface methodology.*

Resumo

O objetivo deste trabalho foi otimizar a secagem por atomização da polpa de juçara, utilizando-se misturas de amido modificado (AM), concentrado proteico de soro de leite (CPL) ou isolado proteico de soja (IPS), como agentes carreadores. Dois delineamentos compostos centrais rotacionais foram realizados para avaliar o efeito das variáveis independentes: temperatura do ar de entrada (140 °C a 200 °C), concentração de agente carreador – CAC (0,5 a 2 g de carreador/g de sólidos de polpa juçara) e proporções de AM:CPL ou AM:IPS (5 a 30 g de CPL ou IPS/100 g carreador) sobre as seguintes respostas para pós formulados com AM:CPL e AM:IPS, respectivamente: teor de umidade (0,3% a 1,4% e 0,6% a 1,2%), solubilidade (78,0% a 92,9% e 78,9% a 83,8%), retenção de antocianinas totais (49,2% a 82,9% e 34,1% a 96,9%), eficiência de encapsulação (98,5% a 99,7% e 98,5 a 99,5%), ângulo de tonalidade (9,1 a 44,0 e 3,7 a 42,6), croma (10,0 a 15,3 e 9,2 a 14,3) e rendimento do processo (33,2% a 55,5% e 49,9% a 78,5%). A temperatura do ar de entrada de 170 °C, CAC de 1,25 e 2, e a proporção de AM:CPL ou AM:IPS de 17,5 e 30 foram recomendadas como condições selecionadas.

Palavras-chave: *Martius Euterpe edulis; Concentrado proteico de soro de leite; Amido modificado; Isolado proteico de soja; Metodologia de superfície de resposta.*

1 Introduction

Epidemiological studies have consistently shown there is a clear, significant, positive association between the regular consumption of fruits, nuts and vegetables and a reduced incidence of chronic diseases (CARVALHO et al., 2010). In this context, phenolic compounds, specifically anthocyanins, have attracted attention due to their antioxidant properties and their great potential as natural food colorants (MELO et al., 2009; VIEIRA et al., 2013). The anthocyanins, which belong to the flavonoid group, represent an attractive source of pigments that are responsible for the cyanic colours, ranging from salmon pink to red and from violet to dark blue, that are observed in most of the flowers, fruits and leaves of the angiosperms commonly found in nature (CAVALCANTI et al., 2011).

The two major anthocyanins in jussara (*Euterpe edulis* Martius) fruits were identified as cyanidin 3-glucoside and cyanidin 3-rutinoside (BRITO et al., 2007), which are the same anthocyanins present in assai fruit (*Euterpe oleracea*) (SCHAUSS et al., 2006).

The jussara palm (*Euterpe edulis*) is widely distributed throughout the Brazilian Atlantic Forest and produces edible palm hearts and spherical fruits known as jussara. These fruits contain only one light brown seed that is covered by a thin, dry skin that is shiny and dark purple and, due to its high anthocyanin content, appears almost black in colour when ripe (BORGES et al., 2011).

The jussara fruit is most frequently used as pulp, which is consumed as such or further used in different kinds of beverages, ice creams or sweets. The dehydration of jussara is an important alternative to extend the shelf-life of this fruit, in addition to facilitating the transport, storage and handling of the final product.

Spray drying transforms a liquid or pasty food into a powder by the atomization of the pasty material into small particles of a high-pressure spray after contact with hot air. Compared with other drying methods, this process is noted for its applicability to heat-sensitive products such as foods, due to the rapid evaporation of the water, which reduces the temperature of the product and keeps the drying time very short, reducing thermal damage and ensuring that good quality food is produced (MASTER, 1979; OSORIO et al., 2014).

Despite all the advantages related to this drying process, spray-dried fruit pulp tends to present some problems, such as handling stickiness and high hygroscopicity. This is due to the presence of low molecular weight sugars and organic acids in fruits, resulting in low glass transition temperatures and, consequently, considerable stickiness. Thus, they can stick on the dryer chamber wall during the drying process, leading to low product recovery and operational problems. In this context, the use of high molecular weight carrier agents, such as starches and derivatives, gums, lipids and proteins, is recommended during spray drying (BHANDARI et al., 1997). These carrier agents are usually used as encapsulating agents in the microencapsulation process.

Modified starch (MS) is the result of pyroconversion, in which the starch is usually heated in the presence of acid or alkali. Partial hydrolysis of the starch takes place as well as repolymerization to form more highly branched polymers (KENYON, 1995). Modified starch provides excellent protection to anthocyanins during spray drying and can be used in high concentrations in which there is an increase in viscosity (SANTANA et al., 2013).

Whey proteins (WP) are by-products of cheese manufacturing with significant commercial potential (FLORES et al., 2014a). Soy proteins (SP) represent an important component of soybean seeds (35% and 40%, w/w). They possess high gelling and emulsification properties (BERNARD et al., 2011; CAILLARD et al., 2009; NESTERENKO et al., 2014), and an amino acid profile suitable for protein fortification in beverages (FLORES et al., 2014b; NESTERENKO et al., 2014). Other health benefits associated with whey proteins include antimicrobial activity, inhibition of the angiotensin-converting enzyme and anticarcinogenic activity, among others. They can also be processed into pH-sensitive hydrogels or nanoparticles for the controlled release of bioactive compounds such as anthocyanins (FLORES et al., 2014a; MOSER et al., 2017). Some papers can be found in the literature reporting that whey proteins and soy protein isolate can be used as alternatives to polysaccharide-based wall materials with relatively greater functionality (BETZ; KULOZIK, 2011; BETZ et al., 2012; OIDTMANN et al., 2012; FLORES et al., 2014a, b; MOSER et al., 2017).

Spray drying technology requires well-adjusted operating conditions as well as an adequate composition of the solution containing the active compounds. The former include factors such as the inlet air temperature, atomization airflow, liquid flow rate, aspirator suction velocity and solids concentration, amongst others (GALLO et al., 2011; ROCCIA et al., 2014). Response surface methodology (RSM) is a statistical analytical tool that predicts the appropriate levels of the independent variables for optimizing the response variables. Factorial designs are frequently used in experiments involving several factors, where it is necessary to study the joint effect of the factors on a response.

In this study, RSM was applied to evaluate the influence of the concentrations of mixed carrier agents (modified starch, whey protein concentrate or soy protein isolate) and the inlet air temperature on the process yield, moisture content, solubility, total anthocyanin retention, encapsulation efficiency and colour of the spray dried jussara pulp.

2 Material and methods

2.1 Material

Jussara fruit were supplied by farmers from rural communities in Ubatuba, São Paulo, Brazil. During pulping, water was added (pulp:water ratio 1:2, w/w) and the jussara pulp then packed into plastic bottles (1 L) and immediately frozen. The use of frozen pulp was necessary because these fruits are extremely perishable. Table 1 shows the composition of the jussara pulp used in the trials.

The carrier agents used were: modified starch (Hi-Cap 100, National Starch, São Paulo, Brazil), whey protein concentrate (WPC 80, Alibra, Campinas, Brazil) and soy protein isolate (SPI SUPRO® 783, Solae, Barueri, Brazil).

2.2 Mixture viscosity

The viscosity of the feed mixtures was determined from the steady-shear flow curves (shear stress X shear rate) using a controlled stress R-2000EX rheometer (TA Instruments, Delaware, USA) at 25 °C, with a geometry of serrated parallel plates (cone diameter - Ø40 mm) and a gap of 500 µm. These conditions were adopted based on preliminary results. Samples of 2.0 mL were introduced into the rheometer with the aid of an automatic pipette. All tests were carried out in duplicate and a new sample was used for each replicate. The solvent trap rheometer accessory was used during measurements in order to prevent solvent evaporation. Flow curves were obtained from three shear rate ramps carried out in the following order: increasing-decreasing-increasing shear rate in the range from 0.1 s^{-1} to 250 s^{-1} (CANO-CHAUCA et al., 2005). The rheograms were analysed according to an empirical model of the flow curves, corresponding to the second (decreasing) shear rate ramp, and the apparent viscosity was calculated as the correlation between shear stress (σ) to shear rate (γ).

2.3 Sample preparation and spray drying process

Before the spray drying process, the jussara pulp was vacuum filtered through porous fabric using a Büchner funnel. This procedure was carried out with the purpose of removing suspended solids to avoid obstruction of the atomizer nozzle of the spray dryer. The mixed carrier agents (MS:WPC or MS:SPI) were added directly to the filtrate to obtain a desirable concentration according to Table 2. Dissolution of the mixture was achieved using a homogenizer-type 'Ultra-Turrax' (IKA T25 D, Staufen, Germany) at room temperature and 14,000 rpm for 10 minutes (complete dissolution) and kept under magnetic agitation during the passage of the mixture through the atomizer nozzle.

The process was carried out using a laboratory-scale spray dryer (Büchi model B290, Flawil, Switzerland). The equipment was operated concurrently using a spray nozzle with an orifice of 0.7 mm in diameter. The mixture was fed into the drying chamber using a peristaltic pump, and the feed mass flow rate and compressed air flow rate were 5 mL/min and 500 L/h, respectively. The tests were carried out under different processing conditions according to the experimental design as explained in the following section. The independent variables were: inlet air temperature (140°C to 200 °C), carrier agent concentration - CAC (0.5 to 2 g carrier/g jussara pulp solids) and ratio of MS:WPC or MS:SPI (5 to 30 g WPC or SPI/100 g carrier). These parameter conditions were obtained in a previous test. The feed mass flow rate was also obtained in preliminary tests. The drying time was about 30 minutes. The powders obtained were placed in hermetic containers and stored in desiccators containing silica gel until analysed.

2.4 Experimental design

For this study, Response surface methodology was used twice to evaluate the effect of three independent process variables on seven response variables, mainly related to the profitability of the process and the quality of

Table 1. Composition of the jussara pulp.

Analysis	Mean ± SD[a]	Method
Moisture (%)	76.59 ± 0.08	Horwitz (2006)
Total protein (%)	3.49 ± 0.05	Horwitz (2006)
Fat (%)	19.19 ± 0.12	Horwitz (2006)
Ash (%)	1.03 ± 0.01	Horwitz (2006)
Total sugar (%)	4.62 ± 1.46	By difference
Reducing sugar (%)	2.20 ± 0.56	Horwitz (2006)
Total acidity (g citric acid/100 g)	0.23 ± 0.02	Horwitz (2006)
pH	4.99 ± 0.07	pH Meter at 25 °C
Total soluble solids (°Brix)	10.15 ± 0.21	Bench refractometer
Vitamin C content (mg vitamin C/100 g)	30.59 ± 0.98	Horwitz (2006)
Anthocyanins (mg/g)	8.35 ± 0.36	Cinquanta et al. (2002)

[a] Percentage on a wet weight basis; All data presented as the mean ± standard deviation (SD) of three replicates (n = 3).

Table 2. Levels of the process variables.

Independent variables	Coded variables	Levels				
		-1.68	-1	0	1	1.68
inlet air temperature (°C)	x_1	140	152	170	188	200
carrier agent concentration (g/g)	x_2	0.5	0.8	1.25	1.7	2
MS:WPC or MS:SPI ratio	x_3	5	10	17.5	25	30

Table 3 shows the experimental design for the response variables studied.

the powder. A 2^3 central composite rotatable design with eight factorial points, six axial points and three central points was used to obtain a second-order prediction model. The independent variables evaluated were inlet air temperature, CAC and the MS:WPC or MS:SPI ratio. Table 2 shows the levels of the process variables. The response variables taken into consideration were process yield (A), moisture content (B), solubility (C), retention of total anthocyanins (D), encapsulation efficiency (E) and colour (Hue - F and chroma - G).

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 \quad (1)$$

The experimental data were fitted to a second-order polynomial Equation 1 as follows:

where y is the response (dependent variable); β_0 is the constant regression coefficient; β_1, β_2 and β_3 are the linear regression coefficients; β_{11}, β_{22} and β_{33} are the quadratic regression coefficients; β_{12}, β_{13} and β_{23} are the interaction regression coefficients; and x_1, x_2 and x_3 represent the coded values of the independent variables.

In order to obtain the regression coefficients, an analysis of variance (ANOVA) was carried out using the Statistica 9.0 (Statsoft, Tulsa, U.S.A.) software package. Only variables with a confidence level above 90% ($p \leq 0.1$) were considered as significant. Response surface methodology was applied to the optimize spray drying of the jussara pulp.

2.5 Analytical methods

2.5.1 Process yield

The drying yield was determined as the ratio of the mass of total solids in the powder to the mass of total solids in the feed solution. The measurements were made in triplicate.

2.5.2 Moisture content

The powder moisture content was determined gravimetrically using a vacuum oven (Memmert, Germany) at 70 °C to constant weight (HORWITZ, 2006), weighing on an analytical balance (250 L, Adam, Co. UK) with a precision of ±0.0001 g. The measurements were made in triplicate.

2.5.3 Solubility

Solubility was determined according to the Eastman and Moore method (1984) as cited by Cano-Chauca et al. (2005), with some modifications. 100 mL of distilled H_2O were transferred to a blender jar. The powder sample (1 g, dry weight basis) was carefully added to the blender operating at high velocity for 5 min. The solution was then placed in a tube and centrifuged at 3000×g for 5 min. An aliquot of 25 mL of the supernatant was transferred to pre-weighed Petri dishes and immediately oven-dried at 105 °C to constant weight. The solubility (%) was calculated from the weight difference and the measurements were made in triplicate.

2.5.4 Anthocyanin content

The total anthocyanin content (TA) was determined using a spectrophotometric method (FRANCIS, 1982). Approximately 10 mg (m) of powder were extracted twice with 10 ml (V) of a HCl/water/ethanol solution (1/29/70) and the extract centrifuged for 10 min at 10,000×g and 25 °C and recorded using a Beckman DU-7-B340 spectrophotometer (Beckman, Krefeld, Germany). The total anthocyanin content was expressed as cyanidin-3-rutinoside, which was previously identified as the major anthocyanin present in jussara (Brito et al., 2007). The molar absortivity (MM) used for cyanidin-3-rutinoside was 32,800 at the λ_{max} absorbance (abs about 534 nm), in a HCl/water/ethanol solution (1/29/70) at 20 °C (CINQUANTA et al., 2002) (Equation 2). The measurements were made in triplicate.

$$\text{Anthocyanins}(mg/g) = \frac{\text{Abs} \times \text{MM} \times \text{V} \times 10^3}{E_M \times L \times m} \quad (2)$$

where: E_M is the molar absorbency (32800 l/mol.cm), L is the light path.

The retention of total anthocyanins (RTA) obtained after processing, was calculated according to Equation 3:

$$RTA(\%) = \frac{TA_{powder}}{TA_{pulp}} \times 100 \quad (3)$$

2.5.5 Encapsulation efficiency

In order to evaluate the effectiveness of microencapsulation, the TA content and surface anthocyanins of the microcapsules were determined and the ratio of TA to surface anthocyanins calculated. For the determination of TA, the extracts were prepared in accordance with that described in the item above (anthocyanin content).

For the determination of surface anthocyanins (SA), 100 mg of sample were directly extracted with 10 mL ethanol in a vortex for 30 s, followed by centrifugation at 3,000×g rpm for 10 min at 20 °C. After phase separation, the clear supernatant was collected and filtered through a 0.45-mm pore-sized Millipore membrane (IDHAM et al., 2012).

The TA and SA contents were determined using the method of Cinquanta et al. (2002) and the encapsulation efficiencies calculated according to the modified equation of Barbosa et al. (2005) as shown in Equation 4.

$$EE(\%) = \frac{(TA - SA)}{TA} \times 100 \quad (4)$$

2.5.6 Colour

The colour of the jussara powder was measured using a colorimeter (model CR400, Konica Minolta, Osaka, Japan) with a CIELAB scale (L^*, a^* and b^*), with D65 as the illuminant and a 10° observer angle as the reference system. The colour measurements were expressed in terms of lightness L^* (L^* = 0 for black and L^* = 100 for white) and the chromaticity parameters a^* (green [-] to red [+]) and b^* (blue [-] to yellow [+]). The cylindrical coordinate H^o (hue angle) and C^* (chroma) were determined from these parameters using Equations 5 and 6. The measurements were made in triplicate.

$$H^o = \arctan\left(\frac{b^*}{a^*}\right) \quad (5)$$

$$C^* = \left(a^{*2} + b^{*2}\right)^{1/2} \quad (6)$$

3 Results and discussion

3.1 Mixture viscosity

As stated before, the viscosity of the feed mixtures was determined from the steady-shear flow curves. The most appropriate mathematical model for describing the flow characteristics was the Power Law, with flow behaviour indexes (n) below 1 for all the MS:WPC or MS:SPI concentrations.

Thus the mixture "filtered jussara pulp + MS:WPC or MS:SPI" could be characterized as a fluid with shear-thinning behaviour, which is typical of most food materials, especially fruit pulps (TONON et al., 2008).

The apparent viscosity was calculated as the ratio between shear stress and shear rate. At low shear rates, the apparent viscosity of all the samples decreased with increasing shear rate, whereas above 60 s^{-1}, the viscosity of the samples was almost constant. Furthermore, the apparent viscosity of the samples increased with increasing concentration of MS:WPC or MS:SPI (Figure 1a and 1b and Table 3).

3.2 Statistical evaluation of the experimental design

3.2.1 Process yield

The process yield corresponds to the product recovery and is mainly determined from the powder collection efficiency. The yield of the microencapsulation process could be improved by changing the spray drying conditions in order to decrease the sticking of particles to the surfaces of the dryer chamber. In a spray-drying system, the loss of material is mostly due to the sticking of sprayed droplets and powder to the apparatus wall, and to inefficient cyclones with poor efficiency in collecting fine particles (WANG; LANGRISH, 2009). Retention of the product on the chamber wall is undesirable. Firstly, it is not cost effective due to more frequent shutdowns of the dryer for cleaning and secondly, it affects product quality, because the deposits can be scorched, and when dislodged, mix in with and contaminate the entire product. Accumulated product receiving more intense heat treatment may have different properties, such as moisture content, colour and solubility and cannot be considered as product. Furthermore, the deposits influence the drying volume and heat transfer processes between the chamber walls and the moving fluids.

According to Table 3, the experimental values of the process yield for the spray drying of jussara pulp using MS:WPC and MS:SPI ranged from 33.22% to 55.45% and from 49.93% to 78.50%, respectively. These values were similar to those obtained for the spray drying of oregano essential oil with maltodextrin, Arabic gum and modified starch (59.7%), açai with maltodextrin (34.3% to 55.7%) and pequi pulp with Arabic gum (29.6% to 56.1%) (BOTREL et al., 2012; TONON et al., 2008; SANTANA et al., 2013).

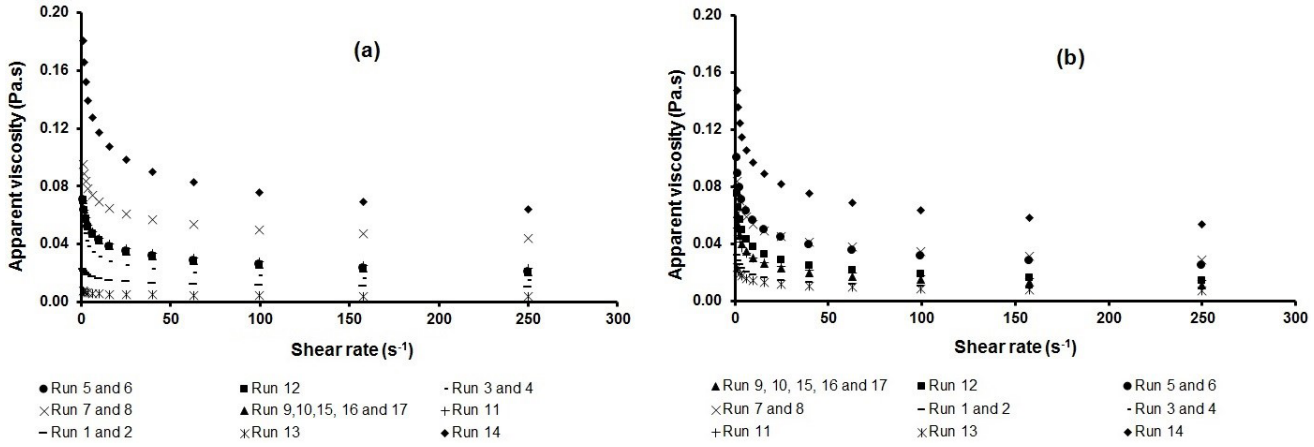

Figure 1. Apparent viscosity versus shear rate for the different concentrations: (a) MS:WPC and (b) MS:SPI.

Table 3. Matrixes of the central composite designs (x_i: independent variables) and experimental data obtained for the response variables studied (A_{1i} and A_{2i}).

Run	x_1	x_2	x_3	A_1	A_2	B_1	B_2	C_1	C_2	D_1	D_2	E_1	E_2	F_1	F_2	G_1	G_2
1	152	0.8	10	43.70	58.30	0.81	0.85	85.80	82.26	52.78	57.07	98.54	99.15	39.50	24.50	15.19	13.33
2	188	0.8	10	41.41	55.11	0.71	1.02	84.79	81.69	49.18	55.59	98.55	99.05	33.10	19.16	14.58	12.41
3	152	1.7	10	45.31	59.67	1.40	1.10	83.48	81.57	54.14	65.14	98.77	99.46	26.65	8.49	13.39	13.48
4	188	1.7	10	43.96	60.68	1.01	0.91	81.55	81.39	54.90	85.90	98.71	99.36	25.39	29.79	12.68	13.40
5	152	0.8	25	45.92	63.04	1.06	0.75	79.36	80.64	58.22	50.93	98.70	99.04	16.84	17.58	11.37	11.51
6	188	0.8	25	45.86	59.25	0.51	0.94	79.92	81.16	55.23	34.10	98.69	98.98	13.89	13.58	11.46	10.16
7	152	1.7	25	54.16	65.51	0.65	0.63	77.99	79.85	81.43	75.42	99.36	99.36	14.72	15.30	10.93	9.55
8	188	1.7	25	53.21	69.33	0.83	0.88	78.36	80.04	82.30	76.25	99.41	99.29	9.12	14.17	10.03	9.22
9	140	1.25	17.5	52.88	63.60	0.96	1.16	79.99	81.36	78.49	40.10	99.22	98.47	20.33	42.60	11.87	11.44
10	200	1.25	17.5	51.49	63.73	0.25	0.78	80.22	81.33	80.31	39.33	99.19	98.64	17.39	5.08	11.56	11.03
11	170	0.5	17.5	39.66	65.86	0.67	0.90	90.04	83.78	52.36	55.14	98.51	98.97	12.50	3.70	14.19	14.34
12	170	2	17.5	54.88	73.77	0.50	0.72	78.68	78.87	82.85	91.32	99.46	99.43	43.96	50.89	12.21	10.63
13	170	1.25	5	33.22	49.93	0.45	0.73	92.86	83.17	52.59	49.83	98.52	98.93	39.93	33.35	15.32	13.67
14	170	1.25	30	55.45	78.50	0.99	0.93	78.51	79.67	82.37	96.87	99.74	99.26	24.33	3.81	10.01	10.17
15*	170	1.25	17.5	51.61	68.53	0.73	0.74	80.51	80.17	60.74	37.01	99.19	98.87	10.09	32.06	11.72	11.28
16*	170	1.25	17.5	49.57	68.53	0.73	0.73	80.58	80.59	61.71	36.03	98.86	98.87	9.73	29.43	11.80	10.51
17*	170	1.25	17.5	49.66	68.98	0.77	0.77	80.47	80.74	60.75	37.76	98.98	98.75	9.45	23.66	11.94	10.37

A_1 to G_1 and A_2 to G_2: response variables for the process yield (%), moisture content (%), solubility (%), total anthocyanin retention (%), encapsulation efficiency (%), and hue and chroma for the jussara powders obtained with MS:WPC and MS:SPI, respectively; x_1, x_2 and x_3: independent variables for the inlet air temperature (°C), CAC (g carrier/g jussara pulp solids) and MS:WPC or MS:SPI ratios (g WPC or SPI/100 g carrier), respectively; * Central point.

Table 4. Regression coefficients and values for R^2 for the final reduced models.

Regression coefficient	A_1	A_2	B_1	B_2	C_1	C_2	D_1	D_2	E_1	E_2	F_1	F_2	G_1	G_2
Constant														
β_0	50.90	68.96	0.73	0.74	79.86	81.08	64.73	38.94	98.96	98.76	12.77	28.63	11.75	11.20
Linear														
β_1	-	-	-	-	-	-	-	-	-	-	-	-	-	-
β_2	3.32	2.40	-	-	-2.02	-0.82	7.95	14.77	0.25	0.15	-	-	-0.65	-0.59
β_3	4.55	5.22	-	-	-3.23	-0.81	8.51	-	0.27	-	-7.05	-	-1.54	-1.32
Square														
β_1^2	-	-2.57	-	-	-	-	-	-	-	-	-	-	-	-
β_2^2	-1.43	-	-	-	1.06	-	-	13.07	-	0.20	4.79	-	0.48	0.45
β_3^2	-2.47	-2.38	-	-	1.53	-	-	13.12	-	0.16	6.17	-	0.29	-
Interactions														
β_{12}	-	-	-	-	-	-	-	-	-	-	-	-	-	-
β_{13}	-	-	-	-	-	-	-	-	-	-	-	-	-	-
β_{23}	-	-	-	-	-	-	5.40	-	-	-	-	-	0.23	-0.51
$F_{calculated}$	14.15	9.02	5.37	9.15	13.82	19.22	14.15	13.31	21.65	12.53	6.67	5.89	136.38	14.16
$F_{tabulated}$	2.46	2.56	2.46	2.46	2.56	2.56	2.46	2.56	2.46	2.56	2.46	2.46	2.56	2.56
R^2	0.90	0.78	0.43	0.43	0.85	0.79	0.85	0.77	0.83	0.76	0.61	0.50	0.99	0.89

A_1 to G_1 and A_2 to G_2: response variables for the process yield (%), moisture content (%), solubility (%), total anthocyanin retention (%), encapsulation efficiency (%), and hue and chroma for the jussara powders obtained with MS:WPC and MS:SPI, respectively; - No significant effect at a level <10%; R^2: adjusted square coefficient of the fitted model (indicates the percentage of variability accounted for by the model); β_i: the estimated regression coefficient for the main linear effects; β_i^2: the estimated regression coefficient for the quadratic effects; β_{ij}: the estimated regression coefficient for the interaction effects; i = 1: inlet air temperature; i = 2: carrier agent concentration - CAC; i = 3: MS:WPC or MS:SPI ratio.

The coefficients of determination (R^2) for the fitted models were 90% and 78% and Table 4 shows all the significant linear and quadratic effects and two-factor interactions on A_1 and A_2. On the one hand, CAC and the ratios of MS:WPC and MS: SPI had positive linear effects on A, as shown by the significant coefficients (Table 4), whilst the opposite effect was obtained for the quadratic effects. The addition of high molecular weight solutes, such as the mixtures of MS:WPC and MS:SPI, prior to spray drying, was necessary in order to obtain powders. The yield was positively affected by the CAC and MS:WPC and MS: SPI ratios. Despite the negative quadratic effect of these solutes, an increase in the concentration of jussara pulp in the solute improved the product yield.

Figure 2a and b shows the response surfaces for the process yields in the drying of jussara pulp using MS:WPC and MS:SPI. Analysing this figure, a trend can be seen between CAC and the ratio of MS:WPC or MS:SPI with respect to process yield, showing an increase in the response for the highest values of CAC and MS:WPC or MS:SPI ratio. This possibly occurred because there was an increase in the glass transition temperature of the powder due to the addition of a high molecular weight carrier (MS or WPC or SPI). This causes a decrease in powder stickiness, and consequently increases product recovery during the spray drying process.

3.2.2 Moisture content

Moisture content is an important property of a powder, and is related to the drying efficiency. Furthermore, lower moisture contents limit the ability of water to act as a plasticizer, which reduces the glass transition temperature. The moisture contents of the jussara pulp powders produced with MS:WPC and MS:SPI varied from 0.25% to 1.40% and from 0.63% to 1.16%, respectively (Table 3). All the experiments showed low values for MC in the microcapsules. Similar moisture content ranges were obtained for spray dried açai juice, black mulberry juice and blackberry juice by Tonon et al. (2011), Fazaeli et al. (2012) and Ferrari et al. (2012), respectively.

The MC parameter did not change as a consequence of the combination of independent variables (Table 4). In other words, no variable studied showed significance at the level of 10% with respect to the response of moisture content, indicating that MC_1 is 0.73% and MC_2 is 0.74% for any value of the inlet air temperature, CAC and MS:WPC and MS:SPI ratios within the range studied.

3.2.3 Solubility

According to Table 3, the experimental values for the solubility of the spray dried jussara pulp made using MS:WPC and MS:SPI ranged from 77.99% to 92.86% and from 78.87% to 83.78%, respectively. These values were similar to those found for spray dried açai and mango juices (TONON et al., 2009; Cano-CHAUCA ET al., 2005) and higher than those found for spray dried pomegranate juice (VARDIN; YASAR, 2012).

The coefficients of determination (R^2) for the fitted models were 85% and 79%. CAC and the MS:WPC and MS:SPI ratios had negative linear effects on C, as shown by the significant coefficients (Table 4), while the opposite effect was obtained for the quadratic effects. The addition of high molecular weight solutes, such as MS:WPC and MS:SPI mixtures, prior to spray drying, was necessary in order to obtain powders. The solubility was negatively affected by the CAC and MS:WPC and MS: SPI ratios. Despite the negative quadratic effect of these solutes, an

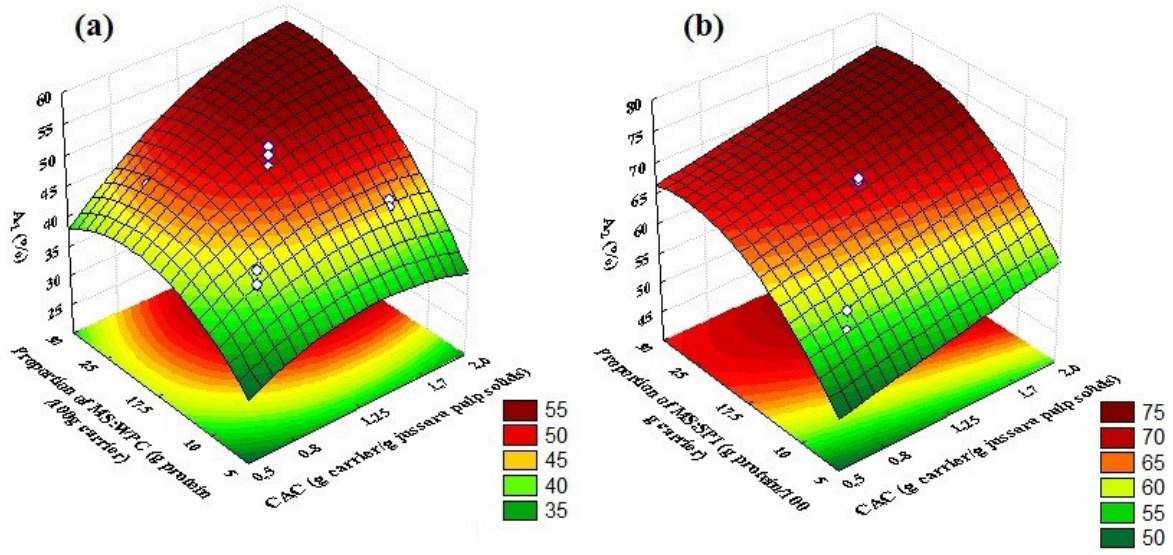

Figure 2. Response surface plots for process yield (A). (a) jussara powders with MS:WPC and (b) jussara powders with MS:SPI.

increase in the concentration of jussara pulp in the solute decreased the solubility after spray drying.

Figure 3a and b shows the solubility of powdered jussara pulp as a function of CAC and the MS:WPC or MS:SPI ratios. It was shown that the solubility of the powdered jussara pulp decreased as a function of increases in CAC and in the MS:WPC or MS:SPI ratios. This figure shows that low initial protein contents resulted in a high degree of solubility, whereas high protein concentrations led to a decrease in solubility, which was not expected since it is understood that at high carrier and protein concentrations the solubility would tend to increase rather than decrease. Moser et al. (2016) and Kha et al. (2010) reported that powder solubility was not influenced by CAC when studying the production of Gac (*Momordica cochinchinensis*) and grape juice powder using maltodextrin, WP and SPI, which was not shown in the present research. However, Yousefi et al. (2011) reported that solubility was strongly affected by the carrier type and, in some cases, by carrier concentration. Nevertheless, the reduction in solubility was small (Table 3) and all systems showed good rehydration characteristics. The functional properties of proteins are frequently limited by their relatively poor solubility, particularly close to the isoelectric point (pI) (MOSER et al., 2016). The pI of whey protein is at a pH near to 5.0 (Duongthingoc et al., 2013) and that of soy protein near to 4.5 (Wang et al., 2010).

3.2.4 Total anthocyanin retention

For the whole experimental design the values for anthocyanin retention ranged from 49.18 to 82.85% and from 34.10 to 96.87%, respectively (Table 3). Similar values were obtained by Tonon et al. (2008) and Ferrari et al. (2012) for the spray drying of blackberry and açai juices using maltodextrin with carrier agents. Charve and Reineccius (2009) evaluated the effect of different proteins on the encapsulation of flavours by spray drying using whey protein isolate, and the authors obtained 87% retention of the volatiles.

The coefficients of determination (R^2) for the fitted models were 85% and 77%. Table 4 shows that the CAC and MS:WPC and MS:SPI ratios presented significant linear (D_1 and D_2) and quadratic (D_2) effects and two-factor interactions for D_1, presenting positive effects. According to Figure 4a, D was higher for higher CAC and carrier ratios, which may explain the good encapsulation by these carrier mixtures. This is important in terms of the industrial production of the pigment, considering that jussara pulp has been shown to be a stable anthocyanin source. Conversely, according to Figure 4b, greater pigment retention was obtained with the highest CAC and highest and lowest MS:SPI ratios.

3.2.5 Encapsulation efficiency

The experimental values obtained for the encapsulation efficiency of the jussara powders with MS:WPC and MS:SPI varied, respectively, from 98.51% to 99.74% and from 98.47% to 99.46% (Table 3).

Figure 5 shows the encapsulation efficiencies obtained for the spray-drying process. Encapsulation efficiencies refer to the potential of the wall material to encapsulate or hold the core material inside the microcapsule. Encapsulation efficiencies are also related to the shelf life of the anthocyanin content of the powder.

The coefficients of determination (R^2) for the fitted models were 83% and 76%. The carrier concentration and MS:WPC and MS:SPI ratios had positive effects on encapsulation efficiency (Figure 5); the highest CAC and highest MS:WPC and MS:SPI ratios resulting in more

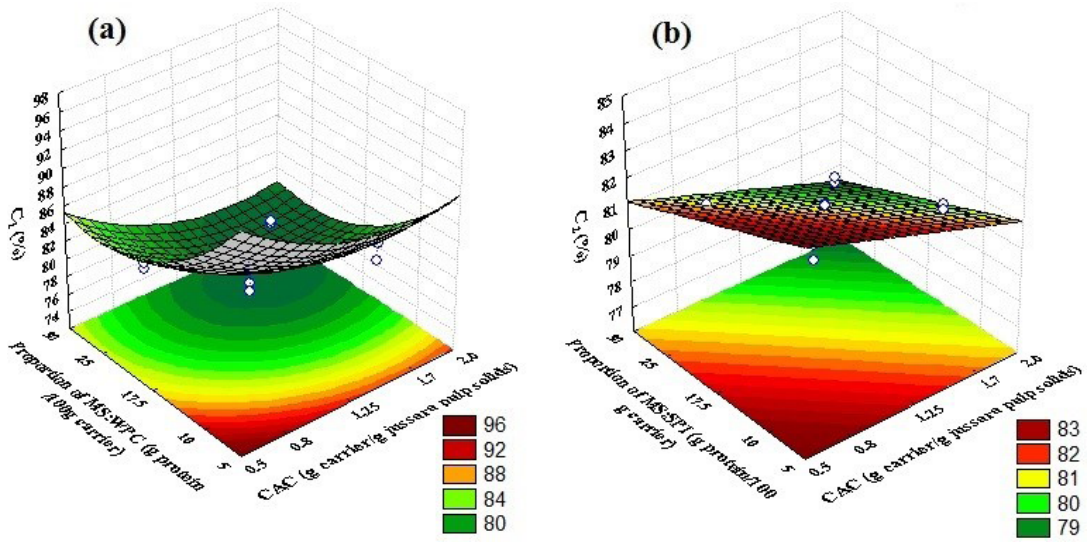

Figure 3. Response surface plots for solubility (C). (a) jussara powders with MS:WPC and (b) jussara powders with MS:SPI.

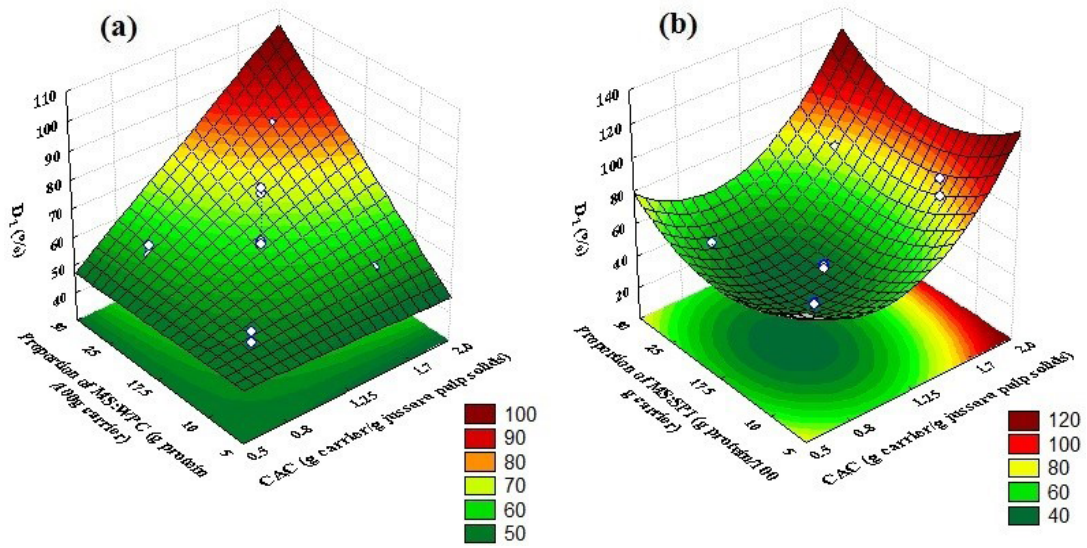

Figure 4. Response surface plots for the retention of total anthocyanins (D). (a) jussara powders with MS:WPC and (b) jussara powders with MS:SPI.

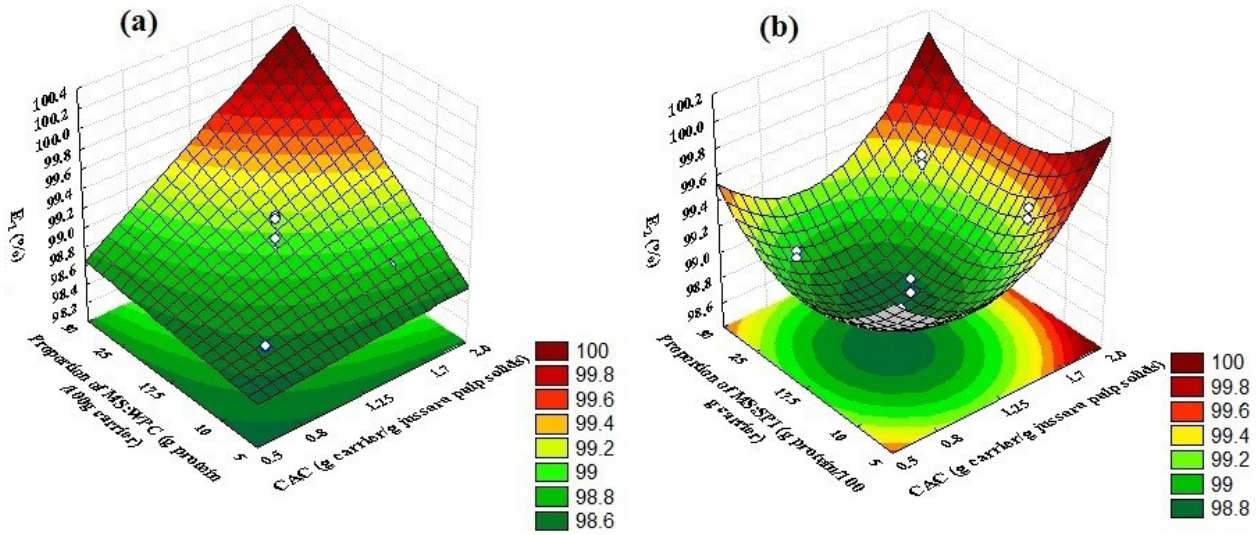

Figure 5. Response surface plots for encapsulation efficiency (E). (a) jussara powders with MS:WPC and (b) jussara powders with MS:SPI.

efficient mixing of the WPC with the MS or SPI with the MS, to form microcapsules. This information is consistent with the results presented in section 3.2.4 for the retention of total anthocyanins, where it was observed that the highest CAC and MS:WPC and MS:SPI ratios resulted in better preservation of the anthocyanins present in in the jussara powder.

The powders produced with MS: WPC or MS: SPI showed high E values, greater than 98%, demonstrating that the blending of WPC with MS or of SPI with MS is a good alternative to encapsulate anthocyanins (Table 3).

Young et al. (1993) also observed increased encapsulation efficiency with an increase in carrier concentration. According to the authors, high solids concentrations in the wall material may be associated with less migration of core material to the surface in the early stages of drying. Furthermore, the increase in E can be attributed to the formation of thick matrices around the encapsulated material.

According to Idham et al. (2012), the carrier combination giving the greatest encapsulation efficiency and the effectiveness of the interactions depended on the chemical and physical structure of each support material. Therefore, the use of whey protein concentrate or soy protein isolate combined with modified starch in the formulation has a greater potential to encapsulate the anthocyanins than the carrier agents alone. Robert et al. (2010) reported greater E values for anthocyanins using soy protein isolate

(35.8% to 100%) and maltodextrin (89.4% to 100%) as the wall materials. Using soy protein isolate, the authors observed that E reached higher values for anthocyanins than for polyphenols, showing the ability of this carrier to bind anthocyanins, which could be related to the flavylium cation of the anthocyanins. Deng et al. (2014), using only soy protein or a soy protein/ starch blend, also found that E varied considerably with the composition of the encapsulating materials.

3.2.6 Colour

The second-order polynomial model obtained for the hue angle was not suitable for the prediction of this colour parameter, due to the low coefficients of determination obtained for the jussara pulp powders with MS:WPC and MS:SPI (R^2 = 0.61 and R^2 = 0.50, respectively), as shown in Table 4.

According to Table 3, the hue angle for the spray drying of jussara pulp using MS:WPC and MS:SPI varied from 9.12 to 43.96 and from 3.70 to 42.60, respectively, which is associated with the red colour characteristic of the jussara flesh. These values indicated that the colour parameters of jussara powders were located in the first quadrant of the CIELAB colour diagram (+a^* and +b^*), corresponding to the region of red and yellow colours, where 0° is pure red and 90° is pure yellow.

The linear and quadratic terms obtained for the MS:WPC ratio and the quadratic term obtained for CAC were statistically significant at $p < 0.10$ (Table 4).The MS:WPC ratio was the independent variable that most affected the hue angle. As the proportion of MS:WPC increased, so the hue angle values decreased and enhanced the purple colour of the jussara powders. However, for the powders made with MS:SPI, this parameter did not change as a consequence of changes in the ratio. In general, the colour of the jussara powder became lighter and less red as the MS:WPC ratio increased, which must be taken into consideration depending on the application of the final product.

The experimental values obtained for the chroma of the spray dried jussara pulp made using MS:WPC and MS:SPI ranged from 10.01 to 15.32 and from 9.22 to 14.34, respectively (Table 3).

The coefficients of determination (R^2) obtained for the fitted models were 99% and 89% and Table 4 shows the significant linear and quadratic effects and two-factor interactions obtained on G_1 and G_2. On one hand, CAC and the MS:WPC and MS: SPI ratios had negative linear effects on G, as shown by the significant coefficients (Table 4), while the opposite effect was obtained for the quadratic effects.

The chroma represents the intensity and purity of the colour, regardless of how light or dark it is. A colour with a high chroma value appears to be bright or concentrated; whereas a low chroma colour looks pale, greyish or diluted. Higher carrier concentrations and MS:WPC and MS: SPI ratios decreased the colour intensity, i.e., the powders showed less saturated staining (Figure 6).

The chroma followed the same trend as the values for a*, indicating that the parameter a* was more important in determining the chroma of the jussara juice powder.

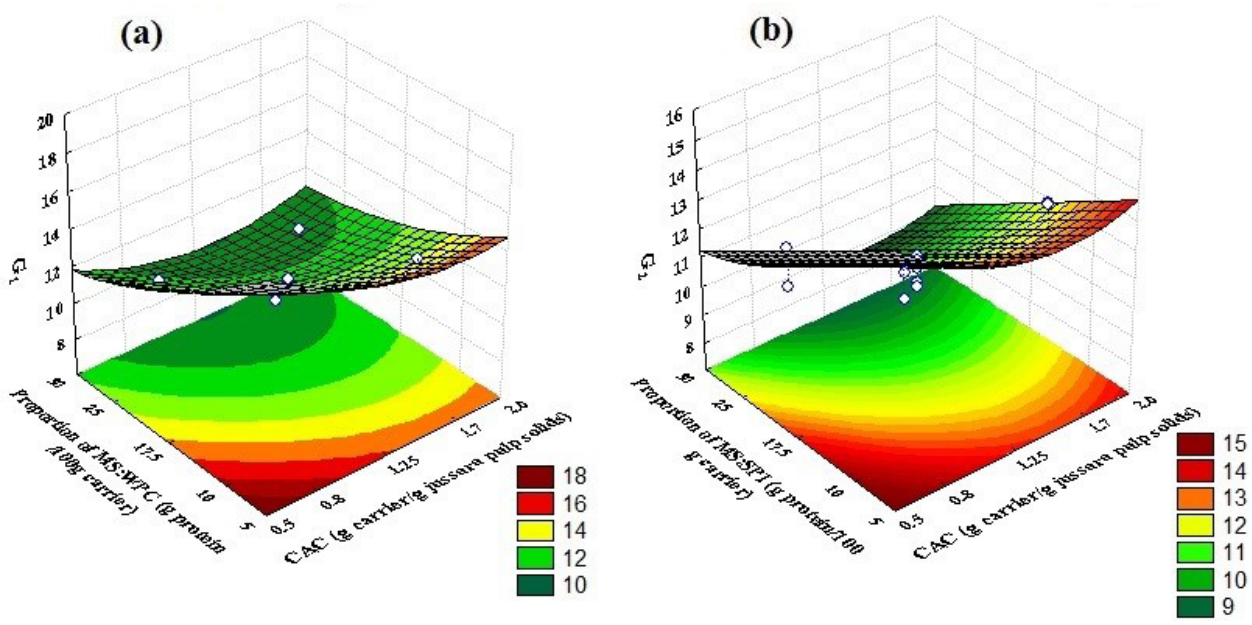

Figure 6. Response surface plots for chroma (G). (a) jussara powders with MS:WPC and (b) jussara powders with MS:SPI.

3.3 Selecting the best process conditions

The best conditions for the spray drying microencapsulation of jussara pulp using mixtures of modified starch with whey protein concentrate or soy protein isolate as the carrier agents were determined in order to obtain higher values for process yield, anthocyanin retention and encapsulation efficiency when using the two experimental designs. Thus an inlet air temperature of 170 °C, CAC of 1.25 and 2 g/g and proportion of MS:WPC or MS:SPI of 17.5 and 30 g/100 g were recommended as the selected conditions.

4 Conclusions

The response surface methodology was adequate and it was possible to select the best spray drying conditions. An inlet air temperature of 170 °C, CACs of 1.25 and 2 g/g and MS:WPC or MS:SPI ratios of 17.5 and 30 g/100 g can be recommended as the selected conditions to obtain spray-dried products with maximum values for process yield, anthocyanin retention and encapsulation efficiency. In general, the results obtained in this study indicated that good quality powders with high anthocyanin contents could be produced by spray drying. The results are of interest to the food sector, to obtain, for example, novel and inexpensive sources of natural jussara flavourings for use in the production of dry mixes, beverages, desserts and other products, as well as in the reconstitution of the juice itself. The cosmetics companies are also looking for novel and inexpensive sources of natural pigments, for use as colorants and flavourings.

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