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Physical properties of spray dried acerola pomace extract as affected by temperature and drying aids

Germano Éder Gadelha Moreira [a], Mayra Garcia Maia Costa [b], Arthur Cláudio Rodrigues de Souza [b], Edy Sousa de Brito [b], Maria de Fátima Dantas de Medeiros [a], Henriette M. C. de Azeredo [b,*]

[a] *Universidade Federal do Rio Grande do Norte, Departamento de Engenharia Química – CT, Campus Universitário UFRN, Natal/RN, CEP 59078-970, Brasil*
[b] *Embrapa Agroindústria Tropical, R. Dra. Sara Mesquita, 2270, Fortaleza/CE, CEP 60511-110, Brasil*

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ABSTRACT

The objective of this study was to assess the impact of some processing parameters on moisture content, flowability, hygroscopicity and water solubility of spray dried acerola pomace extract using maltodextrin and cashew tree gum as drying aids. The experiment was conducted according to Response Surface Methodology, with the independent variables being: inlet temperature (170–200 °C), drying aid/acerola ratio (2:1–5:1), and percent replacement of maltodextrin by cashew tree gum (0–100%). Higher inlet temperatures favored the desired physical properties of the powders, decreasing their moisture contents and hygroscopicity, and increasing flowability. The drying aids decreased the powder hygroscopicity, especially cashew tree gum (CTG), which also enhanced the powder flowability. The best processing conditions to obtain a free-flowing and least hygroscopic acerola pomace extract powder by spray drying were: inlet temperature above 194 °C; drying aid/acerola solid ratio, 4:1; percent replacement of maltodextrin by CTG, at least 80%.

1. Introduction

Acerola (*Malpighia emarginata* D.C.), also known as the Barbados cherry or West-Indian cherry, is present in South and Central America, as well as in some southern regions of North America (Johnson, 2003). It is a red round shaped fruit, with diameter varying from 1 to 4 cm, with a thin, easily bruised skin. Its demand has increased in the last decades, thanks to its high ascorbic acid contents, which ranges from 1000 to 4500 mg/100 g (Johnson, 2003; Leung & Foster, 1996; Mezadri, Fernández-Pachón, Villaño, García-Parrilla, & Troncoso, 2006). Brazil is currently the world's leading producer and exporter of acerola (Mezadri et al., 2006), especially as frozen puree and juice (Yamashita, Benassi, Tonzar, Moriya, & Fernandes, 2003). Acerola processing involves crushing and pressing of the whole fruits, generating as a byproduct a strongly red pomace, which is usually discarded despite being rich in high-value compounds (particularly vitamin C and flavonoids, both with known antioxidant properties).

Spray drying is a well-established and widely used technique to turn liquid foods into powder form. However, sugar-rich materials are difficult to spray dry, because they produce highly hygroscopic powders prone to stickiness and flowing problems. The possible consequences are impaired product stability, decreased yields (because of stickiness on the drier chamber walls), and even operational problems to the spray drier (Bhandari, Datta, & Howes, 1997). The sticky behavior is attributed to a high concentration of low molecular weight sugars and organic acids, which have low glass transition temperatures (T_g), being rubbery and thermoplastic at the temperatures of the chamber (Bhandari & Hartel, 2005; Dolinsky, Maletskaya, & Snezhkin, 2000). The fast moisture removal during spray drying results in an amorphous and highly hygroscopic product (Audu, Loncin, & Weisser, 1978; Senoussi, Dumoulin, & Berk, 1995). On the other hand, the stickiness does not occur when higher molecular weight carbohydrates such as maltodextrins are spray dried. Instead, they facilitate drying of sugar-rich foods, since their T_g increasing effect reduces hygroscopicity of powders. Thus, they are frequently used as drying aids (Bhandari & Hartel, 2005; Bhandari, Senoussi, Dumoulin, & Lebert, 1993; Bhandari et al., 1997; Silva, Sobral, & Kieckbusch, 2006). Some studies indicated that arabic gum has higher T_g values than those of maltodextrin having dextrose equivalent (DE) 10 or higher (Collares, Finzer, & Kieckbusch, 2004; Righetto & Netto, 2000), which suggests that arabic gum may be more effective than maltodextrin to reduce powder hygroscopicity. On the other hand, the high cost and availability problems associated with arabic gum (AG) have motivated researchers to look for new materials for replacing it (McNamee, O'Riordan, & O'Sullivan, 1998). Cashew tree gum (CTG), a complex water soluble heteropolysaccharide extracted from

* Corresponding author. Tel.: +55 11 85 3299 1849.
E-mail address: ette@cnpat.embrapa.br (H.M.C.de Azeredo).

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Table 1
Experimental conditions of the spray drying treatments

Treatment	Independent variables					
	IT		[DA]		CTG	
	C	UC	C	UC	C	UC
1	−1	176	−1	2.6	−1	20.2
2	1	194	−1	2.6	−1	20.2
3	−1	176	1	4.4	−1	20.2
4	1	194	1	4.4	−1	20.2
5	−1	176	−1	2.6	1	79.8
6	1	194	−1	2.6	1	79.8
7	−1	176	1	4.4	1	79.8
8	1	194	1	4.4	1	79.8
9	−1.68	170	0	3.5	0	50.0
10	1.68	200	0	3.5	0	50.0
11	0	185	−1.68	2.0	0	50.0
12	0	185	1.68	5.0	0	50.0
13	0	185	0	3.5	−1.68	0.0
14	0	185	0	3.5	1.68	100.0
15	0	185	0	3.5	0	50.0
16	0	185	0	3.5	0	50.0
17	0	185	0	3.5	0	50.0

C, coded values; UC, uncoded values; IT, inlet temperature (°C); [DA], drying aid/acerola solid ratio (g of drying aid per g of acerola solids); CTG, degree of replacement of maltodextrin by cashew tree gum (%).

cashew tree (*Anacardium occidentale*), has been pointed as a very promising material, due to its structural similarity to AG (De Paula, Heatley, & Budd, 1998; Paula, Gomes, & Paula, 2002; Zakaria & Rahman, 1996). The replacement of AG by CTG, previously suggested by some authors (Owusu, Oldham, Oduro, Ellis, & Barimah, 2005; Rosenthal, 1951), could reduce costs with AG importation and favor the cashew tree business, whose only high value added product is currently the cashew nut.

The objective of this study was to evaluate the impact of some processing conditions (inlet temperature of the spray drier, degree of replacement of maltodextrin by CTG, and drying aid/acerola ratio) on physical properties (moisture content, flowability, hygroscopicity and water solubility) of spray dried acerola pomace extract.

2. Material and methods

The acerola pomace, provided by the company *DaFruta Indústria e Comércio S.A.* (Aracati, Ceara State, Brazil), was characterized in terms of moisture content (vacuum oven method), ascorbic acid contents (redox titration with 2,6-dichloroindophenol), titratable acidity (by titration to pH 8.2 with NaOH 0.1 mol/L), total and reducing sugars (titration with Fehling reagents). Official methods (AOAC, 1995) were followed for all analyses.

The acerola pomace was diluted in water (pomace/water weight ratio, 5:1), and pressed in a Skay 93 hydraulic press (Skay, São José do Rio Preto, Brazil) against a stainless steel mesh cylinder (pore size, 3×10^{-4} m) at 5×10^{6} Pa for 30 s, producing the acerola pomace extract (APE). Cashew tree gum was extracted from a single cashew tree (Fortaleza, Ceara State, Brazil) and purified as described by Torquato et al. (2004).

A tissue homogenizer (AC 620/2, Ação Científica, Piracicaba, Brazil) was used to mix APE with maltodextrin (MD) and/or cashew tree gum (CTG), according to a central composite design (Table 1) with three variables: inlet temperature (170–200 °C), drying aid/acerola solid ratio (2:1–5:1), and percent replacement of MD by CTG (0–100%). The ranges of inlet temperatures and drying aid/acerola ratios (specially the lower limits) were based on preliminary tests, since lower temperatures and/or lower drying aid/acerola ratios led to a very sticky material. The upper limits were defined from literature data, as well as by economic reasons. The dispersion was then filtered in a stainless steel sieve (0.3 mm mesh) in order to avoid clogging of the atomizer. The spray drying was conducted in a Mini Spray Dryer Büchi B-290 (Büchi Labortechnik AG, Flawil, Switzerland) under the following operational conditions: feed rate, 0.49 kg/h; peristaltic pump rate, 1.23 kg/h; aspirator flow rate, 5.51×10^{4} kg/h.

The powders obtained by the spray drying process were packaged in sealed metallized bioriented polypropylene bags at 24 ± 2 °C before being submitted to the following analyses.

Moisture content. It was conducted by the vacuum oven method, according to AOAC (1995).

Water solubility test. The method below was described by Eastman and Moore (1984), and modified by Cano-Chauca, Stringheta, Ramos, and Cal-Vidal (2005). 1 g of the powder was carefully added to 100 mL of distilled water under agitation in a Quimis Q-221 magnetic stirrer at 385 g for 5 min. The dispersion was centrifuged at 2600 rpm for 5 min. An aliquot (25 mL) of the supernatant was transferred to a previously weighted Petri dish and vacuum-dried for 5 h at 105 °C. The final powder weight on the dish was used for determination of the water solubility of the product (g of powder per 100 g of water).

Hygroscopicity test. Hygroscopicity was defined as the moisture mass (g) absorbed by 100 g of the powder during 7 days of storage at 25 °C and 90% RH (in a desiccator with a barium chloride saturated solution), which was a modification from the method described by Callahan et al. (1982). Hygroscopicity values thus obtained are not to be considered as absolute values, since the powders were exposed to abusive conditions (unpacked in a high relative humidity environment). Instead, the objective of the authors was to evaluate variations among treatments.

Flowability test. It was based on measuring the angle of repose formed between the side surface and the base of a cone obtained when 10 g of the powder is dumped from a fixed height through a funnel on a flat horizontal surface, according to the method described by Bhandari, Datta, D'Arcy, and Rintoul (1998).

The results were analyzed by using the software Minitab 15. The models generated to represent the responses were evaluated in terms of their *F*-ratio and R^2 coefficient. The effects of the independent variables on the physical properties of the powders were studied, and conditions were established which produced a powder with minimum hygroscopicity and maximum flowability and solubility.

Table 2
Experimental responses from the spray drying treatments

Treatment	MC	ANG	HYG	WS (%)
1	4.52 ± 0.21	52.27 ± 2.77	51.16 ± 2.44	94.02 ± 4.76
2	3.70 ± 0.17	47.36 ± 2.21	43.91 ± 2.14	95.22 ± 4.24
3	4.31 ± 0.19	51.34 ± 2.96	47.12 ± 2.38	92.46 ± 4.17
4	3.62 ± 0.15	46.67 ± 2.43	38.73 ± 1.82	92.20 ± 4.68
5	4.95 ± 0.22	48.18 ± 2.76	46.45 ± 2.22	94.72 ± 5.05
6	4.22 ± 0.20	44.95 ± 2.98	41.52 ± 2.03	96.38 ± 4.91
7	3.91 ± 0.18	47.73 ± 2.74	40.31 ± 1.90	92.75 ± 4.66
8	3.09 ± 0.16	43.89 ± 2.22	34.72 ± 1.93	92.74 ± 4.82
9	5.43 ± 0.25	50.53 ± 3.01	56.44 ± 2.71	93.42 ± 4.67
10	3.96 ± 0.20	40.60 ± 2.54	40.04 ± 1.92	93.40 ± 4.78
11	5.31 ± 0.22	50.19 ± 2.87	49.10 ± 2.42	96.92 ± 4.05
12	3.8 ± 0.15	45.87 ± 2.38	34.97 ± 1.79	90.97 ± 4.11
13	4.65 ± 0.21	48.22 ± 2.63	49.36 ± 2.49	92.03 ± 4.63
14	3.48 ± 0.21	40.19 ± 1.96	38.70 ± 2.03	93.49 ± 4.25
15	4.88 ± 0.24	45.07 ± 2.56	47.34 ± 2.81	92.69 ± 4.55
16	4.35 ± 0.21	43.99 ± 2.31	46.76 ± 2.42	92.61 ± 4.16
17	5.17 ± 0.27	46.37 ± 2.46	48.53 ± 2.39	92.32 ± 4.37

MC, moisture content of the powder (g/100 g); ANG, angle of repose (in degrees); HYG, hygroscopicity (g absorbed water/100 g powder after 7-day storage at 25 °C, 90% RH); WS, water solubility.

Table 3
Regression equations (for the coded variables) and statistical parameters of the models for the physical properties of spray dried acerola pomace extracts

Equations	*F*-ratio	R^2
$MC = 4.83 - 0.41x_1 - 0.12x_1^2 - 0.37x_2 - 0.17x_2^2 - 0.14x_3 - 0.34x_3^2 - 0.01x_1x_3 - 0.24x_2x_3$	1.09	0.83
$ANG = 45.02 - 2.44x_1 + 0.55x_1^2 - 0.76x_2 + 1.43x_2^2 - 1.93x_3 + 0.07x_3^2 - 0.05x_1x_2 + 0.31x_1x_3 + 0.01x_2x_3$	1.72	0.89
$HYG = 47.65 - 3.94x_1 - 0.11x_1^2 - 3.36x_2 - 2.31x_2^2 - 2.63x_3 - 1.60x_3^2 - 0.23x_1x_2 + 0.64x_1x_3 - 0.47x_2x_3$	4.17	0.95
$WS = 92.51 + 0.19x_1 + 0.39x_1^2 - 1.48x_2 + 0.58x_2^2 + 0.38x_3 + 0.16x_3^2 - 0.39x_1x_2 + 0.09x_1x_3 - 0.13x_2x_3$	9.72	0.96

MC, moisture content of the powder; ANG, angle of repose; HYG, hygroscopicity; WS, water solubility.
x_1, inlet temperature (coded values, ranging from −1.68 to 1.68, which correspond to the uncoded values of 170 °C and 200 °C, respectively); x_2, drying aid/acerola solid ratio (coded values, ranging from −1.68 to 1.68, which correspond to 2:1 and 5:1, respectively); x_3, percent replacement of maltodextrin by cashew tree gum (coded values, ranging from −1.68 to 1.68, which correspond to 0% and 100%, respectively).

3. Results and discussion

APE presented the following basic composition: moisture content, 95.4 g/100 g; total and reducing sugars, 4.0 g/100 g and 3.6 g/100 g, respectively (expressed as glucose); titratable acidity, 0.55 g/100 g (expressed as malic acid). The ascorbic acid content was 0.16 g/100 g, corresponding to 3.56 g/100 g on a dry basis. After addition of drying aids, the extracts had total solids contents in the range of 7.8–15.6 g/100 g.

The experimental responses of the spray drying treatments of APE are presented in Table 2, while Table 3 presents the regression coefficients for each dependent variable (response) as a function of x_1 (inlet temperature), x_2 (drying aid/acerola solid ratio), and x_3 (percent replacement of MD by CTG), as well as their statistical parameters. The moisture contents of the powders were significantly reduced by increasing all variables, mainly x_1 and x_2. The angles of repose, as well as the hygroscopicity, were also reduced by increasing all variables. For the angles of repose, the decreasing order of magnitude of their effects was $x_1 > x_3 > x_2$, and for the hygroscopicity, $x_1 > x_2 > x_3$. The water solubility was negatively affected by x_2 and favored by x_1 and x_3, mainly x_1. All the models presented *F*-ratios above 1, indicating good description of the data by the models, and the R^2 coefficients were satisfactory. The best models were those for hygroscopicity and water solubility.

Figs. 1–4 present the contour plots for moisture content, flowability, hygroscopicity and water solubility of the powders, respectively. Increasing inlet temperature favored the physical properties of the product, since it resulted in decreased powder hygroscopicity and increased flowability (i.e., decreased angle of repose). The enhanced flowability of the powders produced at higher temperatures seems to be attributed to their lower moisture contents, since higher moisture contents impair flowability (Fitzpatrick, 2005). According to Scoville and Peleg (1981), this is due to the increase in liquid bridges and capillary forces acting between particles. The higher hygroscopicity of the powders produced at lower temperatures seems to be related to their higher moisture content. A major factor affecting powder stability is moisture content, since a small amount of water is able to depress the T_g enough to increase the mobility of the matrix during storage (Bhandari & Hartel, 2005; Roos, 2002; Roos & Karel, 1992).

Increasing the drying aid/acerola ratio reduced powder hygroscopicity, confirming the behavior described in previous studies (Bhandari & Hartel, 2005; Bhandari et al., 1993, 1997; Peleg & Hollenbech, 1984; Silva et al., 2006). On the other hand, high proportions of the drying aid agent tended to slightly decrease the solubility of the powders, confirming results described by Abadio, Domingues, Borges, and Oliveira (2004) and Cano-Chauca et al. (2005).

Higher cashew tree gum (CTG) proportions enhanced the powder flowability and decreased hygroscopicity, suggesting that CTG is more effective than maltodextrin (MD) to increase T_g of the powders. Although other studies about the CTG effects on T_g have

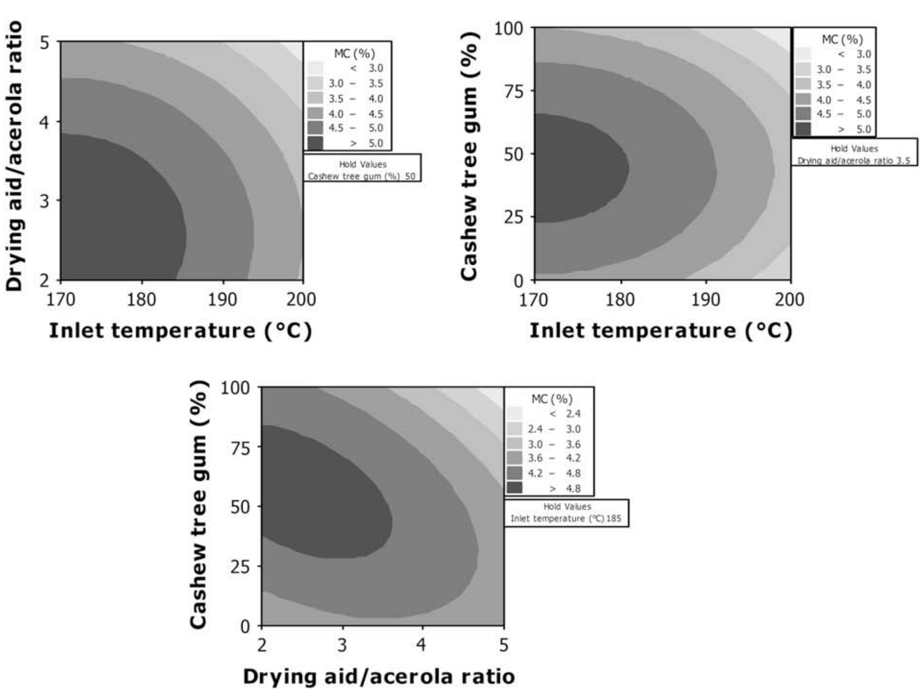

Fig. 1. Contour plots for moisture content (in g/100 g) of the powders produced by spray drying acerola pomace extract.

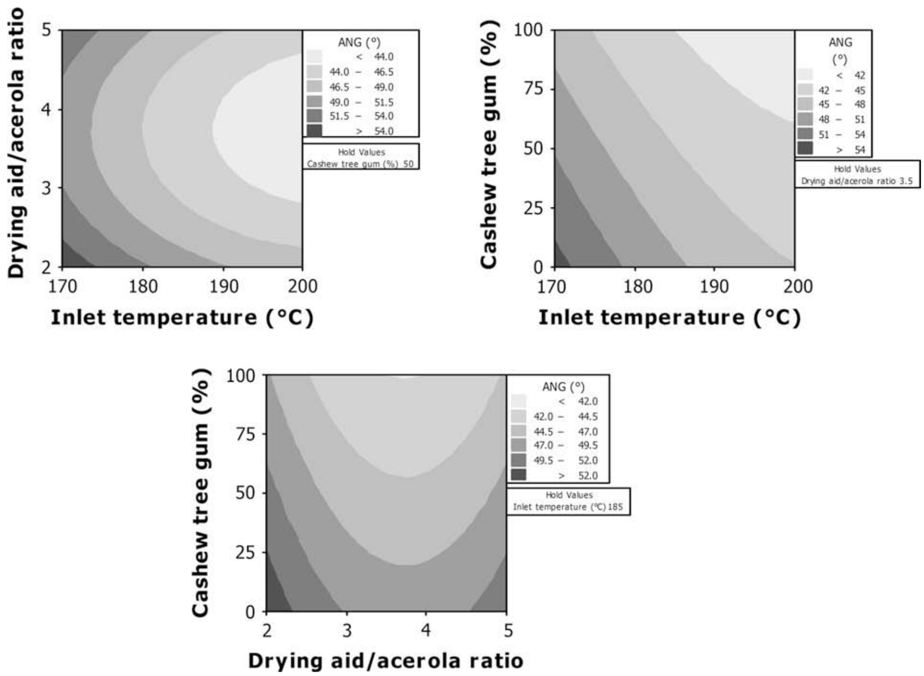

Fig. 2. Contour plots for angles of repose (in degrees) of the powders produced by spray drying acerola pomace extract.

not been found, the similarity between CTG and arabic gum structures suggest that both must have similar T_g effects. So, this result may be compared to those by Righetto and Netto (2000) and Collares et al. (2004), who reported that the T_g-increasing effect of arabic gum is higher than that of MD.

Most powders presented a tendency to flowability problems, according to the criteria suggested by Bhandari et al. (1998), who defined free-flowing powders as those having angles of repose below 45°. However, adequately free-flowing powders were produced at higher temperatures (>194 °C), with drying aid/acerola ratio around 3.5:1–4.0:1, with the drying aid consisting of at least 80% of CTG. The hygroscopicity must not be evaluated by their absolute values, since the powders were exposed to unreal abusive conditions. Anyway, the powders produced at higher temperatures, higher drying aid/acerola ratio and high degree of replacement of MD by CTG were the least hygroscopic. Since solubility was maintained above 90% in powders produced by all treatments, the negative effect of the drying aid/acerola ratio on this response was not considered to optimize the processing conditions.

So, under the ranges studied in this work, the best processing conditions to obtain a free-flowing and least hygroscopic acerola pomace extract powder by spray drying were the following: inlet

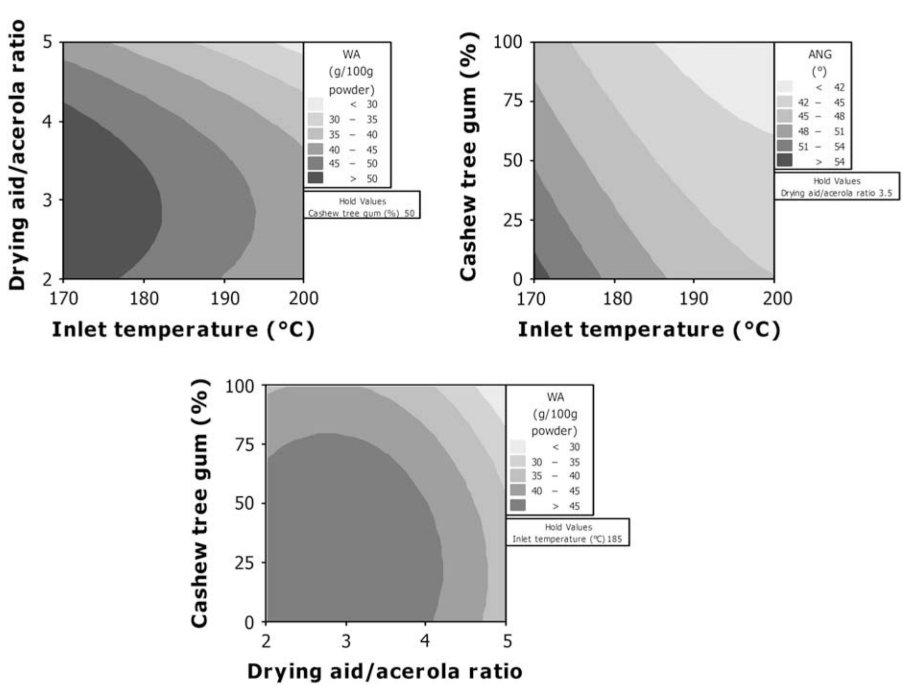

Fig. 3. Contour plots for water absorption (g water/100 g powder) by the powders produced by spray drying acerola pomace extract, after 7 days of storage at 25 °C and 90% RH.

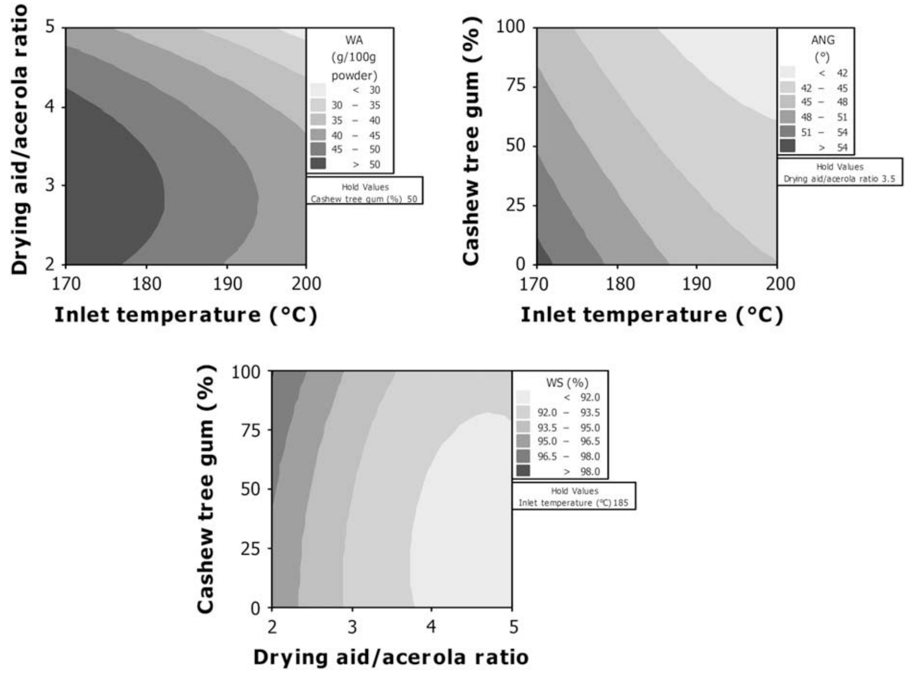

Fig. 4. Contour plots for water solubility (g of powder per 100 g of water) of the powders produced by spray drying acerola pomace extract.

temperature above 194 °C; drying aid/acerola ratio of 4.0, the drying aid being constituted by at least 80% cashew tree gum.

The cashew tree gum is abundant and inexpensive in several tropical developing countries. The assessment of new applications for this virtually unexploited polysaccharide may be important to motivate industries to recognize its potential. If properly exploited, cashew tree gum can greatly impact the cashew tree business and bring social-economical benefits to those countries.

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