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Development of dry acerola and ciriguela mixed pulp obtained by spouted bed drying and freeze-drying[1]

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ABSTRACT - During the drying process, fruit pulps may undergo changes in their quality. This study aimed to select the optimal drying conditions for acerola–ciriguela mixed pulps using spouted bed drying and freeze-drying methods. For spouted bed drying, the influence of drying temperature, feed rate, and maltodextrin concentration was investigated using a 2^3 central composite experimental design. A similar design was applied to freeze-drying, but drying temperature, vacuum chamber pressure, and maltodextrin concentration were selected as the independent variables. In both cases, moisture content, water activity, hygroscopicity, and ascorbic acid retention were used as response variables. Optimisation of both processes was performed according to the desirability function. Higher temperatures and maltodextrin concentrations resulted in lower moisture content in the final product obtained through spouted bed drying. Furthermore, increasing these variables significantly reduced the water activity of both products. Ascorbic acid retention was maximised in the spouted bed-dried product when combining low drying temperatures with a slow suspension feed rate, whereas in the freeze-dried product, higher temperature and chamber pressure achieved the best results. These findings highlight the great potential of freeze-dried mixed acerola–ciriguela powder as an ingredient for vitamin C enrichment in foods.

Keywords: Desirability function. Water activity. Moisture content. Ascorbic acid retention. Maltodextrin.

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INTRODUCTION

The food industry has been forced to create new food products, due to the growing consumer demand for healthier foods and natural products. In this context, fruits are increasingly being used as ingredients in the formulation of new products, meeting the current consumer demand (Germer *et al*., 2018). Moreover, the addition of fruits can improve the sensory aspects of new products (taste, colour, texture, aroma), increase their fibre content and enrich them in vitamins and minerals (Silva *et al*., 2016).

Although native to Central America, ciriguela (*Spondias purpurea* L.) has adapted well to the climate of the Northeast region of Brazil. Being rich in carbohydrates, pro-vitamin A, and phosphorus, its fruit has excellent sensory and nutritional qualities, for which it is widely consumed either fresh or processed (Todisco *et al*., 2014). Acerola (*Malpighia emarginata* D. C.), which is rich in antioxidant compounds such as vitamins, carotenoids, and polyphenols, stands out for the high vitamin C content of its fruit (ranging from 342.47 to 2649.72 mg/100 g), is of great economic importance, and shows agroindustrial and nutritional potential (Jaeschke; Marczak; Mercali, 2016; Ribeiro *et al*., 2018a).

The proposal to mix acerola and ciriguela aims to improve the characteristics of one pulp by complementation with nutrients and phytochemicals contained in the other. Mixed fruit juices and pulps are trends observed on the market that have some advantages compared to their separate constituents, such as improved nutritional and sensorial qualities along with new flavours and aromas (Ribeiro *et al*., 2018b). Despite all their benefits, these fruits are highly perishable due to their high moisture content, which requires some processing to reduce post-harvest losses. Drying is one of the most used techniques to increase food stability thanks to the reduction of moisture content and water activity (Ramírez; Giraldo; Orrego, 2015).

The spouted-bed dryer with inert particles is considered a promising alternative for drying fruit pulps and producing powders from liquid and paste-like foods due to its flexibility and versatility, lower costs and temperature compared to other drying techniques, along with its potential to drive granulation (Chielle *et al*., 2016; Dantas *et al*., 2019).

For processing of fruit pulps on spouted bed drying, it has been shown that the high content of low-molecular-weight sugars tends to reduce process efficiency. To minimise these problems, adjuvants are usually added to protect the active principles during drying; among them, maltodextrin with different dextrose equivalent values is widely used thanks to its high solubility (Moreno *et al*., 2016). Another technique is freeze-drying, based on the removal of water by sublimation, consisting mainly of freezing and vacuum dehydration (Harnkarnsujarit *et al*., 2016). It was developed to prevent losses of compounds responsible for food aromas, which are very sensitive to operations at high temperatures (Ramírez; Giraldo; Orrego, 2015).

Water activity, moisture content and hygroscopicity are critical parameters for quality and stability of dry foods, because water availability allows spoilage microorganisms to grow (Morais *et al*., 2020), while retention of ascorbic acid is desirable due to its beneficial effects on human health. The objective of this work was to determine the optimum processing parameters for spouted bed drying and freeze-drying of acerola-ciriguela mixed pulp, looking for a product with high ascorbic acid retention in conjunction with low water activity, moisture content and hygroscopicity.

MATERIALS AND METHODS

Materials

Acerola and ciriguela fruits were obtained at the Supply and Logistics Centre of Pernambuco (CEASA), Recife, PE, Brazil. The carrier agent was maltodextrin (MOR-REX® 1910) with 10 dextrose equivalent (DE) acquired from Corn Products (Mogi Guaçu, SP, Brazil). Inert particles of type 849 (1 kg), made of high-density polyethylene, were used to form the spout inside the drying chamber.

Acerola-ciriguela mixed pulp preparation

The fruits were purchased at the Supply and Logistics Centre of Pernambuco (CEASA-PE). Acerola and ciriguela fruits were selected based on their stage of maturation with the characteristic colouration of each one, and green and spoiled fruits were discarded. They were washed, sanitised in 2.5% sodium hypochlorite solution and pulped (Bonina Compacta - Itabuna/BA). The pulp was packed in low-density polyethylene bags, immediately frozen and stored at -18 °C (Figure 1). Acerola-ciriguela mixed pulp (ACMP) was prepared by mixing 60% (w/w) acerola and 40% (w/w) ciriguela, because this proportion had previously received the best sensory evaluation (Ribeiro *et al*., 2018b).

Physicochemical characterisation of fresh and mixed pulps

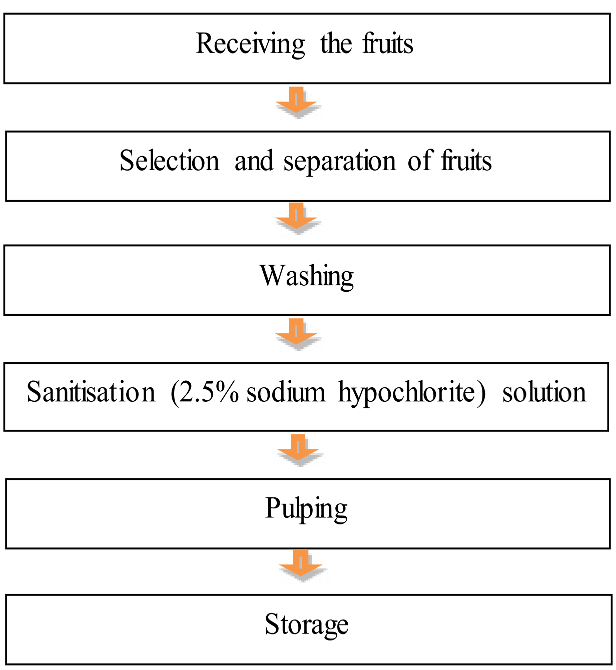
The fresh pulps of acerola, ciriguela and acerola-ciriguela mixed pulp (60% / 40%) were characterised according to the content of soluble solids (SS), titratable acidity (TA), *SS*/*TA* ratio, pH, water activity, moisture and ascorbic acid content. Soluble solids content (*SS*) was determined using a digital refractometer, and the results were expressed in °Brix. Titratable acidity (*TA*) was determined titrimetrically, and the results were expressed in g citric acid per 100 g. These parameters were analysed according to the Association of Official Analytical Chemists (2016), making it possible to calculate the *SS*/*TA* ratio. The pH was measured with a digital pH meter. Water activity (a_w) was determined using an electronic dew point water activity meter, model 4TE (Decagon, Pullman, WA, USA) at 25 °C. Moisture content was determined by drying at 105 ºC for 45 minutes using an infrared scale, model ID 50 (Marte, Piracicaba, SP, Brazil), and the results were expressed in % (w/w). Ascorbic acid content was determined by the Tillmans titration method using 2,6-dichlorophenol indophenol and analysed according to the Association of Official Analytical Chemists (2016).

Drying methods

Acerola-ciriguela mixed pulp (ACMP) was subjected to spouted bed drying and freeze-drying.

Spouted bed drying: ACMP was subjected to drying in spouted bed, model FBDJ 1.0 (Labmaq do Brasil, Riberão Preto, SP, Brazil). Experiments were conducted using temperature (70, 75 and 80 °C), suspension flow rate (4, 5, and 6 mL/min), and maltodextrin concentration (15, 20, and 25% w/w) as independent variables. On the other hand, the air pressure (4 bar) and the amount of polystyrene particles (1 kg) were kept constant in all the runs.

Figure 1 - Flowchart for obtaining acerola-ciriguela mixed pulp (ACMP)



Freeze-drying: 20 g of ACMP were placed in polyethylene Petri dishes (90 mm x 15 mm) and frozen at -22 °C. Plates were then subjected to drying in a pilot freeze dryer, model VirTis Genesis 25L (SP Scientific, Warminster, PA, USA). Experiments were conducted using temperature (-20, -30, and -40 °C), vacuum chamber pressure (300, 400, and 500 mTorr), and maltodextrin concentration (15, 20, and 25% w/w) as the independent variables.

Experimental design

A 2^3-central experimental design composed of 8 factorial points (levels ± 1) and 3 central points (level 0) was adopted. For both drying methods, the response variables were water activity, moisture content (%), hygroscopicity (g.100 g^{-1}) and ascorbic acid retention (%). The results obtained were fitted to the general Eq. (1):

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_1 X_2 + \beta_5 X_1 X_3 + \beta_6 X_2 X_3 \quad (1)$$

where Y is the response, β_0 is the constant regression coefficient, β_1, β_2 and β_3 are the linear regression coefficients, X_1, X_2, and X_3 are the independent variables for each drying method, and β_4, β_5 and β_6 are the coefficients of interaction effects.

Characterisation of ACMP powders

Water activity and moisture content: Water activity (a_w) and moisture content (*M*) were determined as previously described for pulps.

Hygroscopicity: Samples (1 g) of powders were kept for one week in wide-necked jars sealed inside a desiccator containing a NaCl saturated solution (75.29% relative humidity) and then weighed. The results were expressed as the weight of hygroscopic moisture per g of dry solid (Islam *et al*., 2016).

Ascorbic acid content and retention: Ascorbic acid retention (*AAR*) was calculated according to Eq. (2):

$$AAR(\%) = B / A \text{ X } 100 \quad (2)$$

where A is the mass of ascorbic acid present in ACMP (g dry weight) and B is the mass present in the dehydrated pulp (g dry weight).

Statistical analysis

Analysis of variance (ANOVA) and the lack of fit test (F test) were performed, and regression coefficients were estimated using Statistica 10.0 software (StatSoft, Tulsa, OK, USA) at a 5% level of significance. Optimisation of both processes was done according to the desirability function (Derringer; Suich, 1980), whose values vary between 0 and 1, with 0 representing a completely undesired fitting and 1 the most desirable one (Barros Neto; Scarmínio; Bruns, 2010).

RESULTS AND DISCUSSION

Physicochemical characterisation of fresh and mixed pulps

The soluble solids content (*SS*) was significantly different among acerola, ciriguela, and mixed pulps, with that of ciriguela pulp (17.33 ºBrix) being the highest one (Table 1). Acerola pulp showed values of pH, *SS* and acidity within the standards of identity and quality for this product (Brasil, 2002). Although there are still no established identity and quality standard for ciriguela pulp, *SS* and acidity values are close to those found by Maldonado-Astudillo *et al.* (2014). The physicochemical parameters evaluated are critical indicators of the sensory quality, stability, and consumer acceptance of fruit pulps.

Soluble solids (SS) reflect the sugar content and contribute directly to sweetness, with acerola having lower SS (7.23 ºBrix) and ciriguela significantly higher values (17.33 ºBrix), while the mixture showed an intermediate level (11.06 ºBrix), indicating dilution of sweetness from ciriguela. pH values influence flavour perception and microbial stability, with both acerola (3.33) and the mixture (3.25) being less acidic than ciriguela (2.93). Titratable acidity (TA), which affects sourness, was highest in acerola (1.05 g/100 g), lowest in ciriguela (0.72 g/100 g), and balanced in the mixture (0.93 g/100 g). The SS/TA ratio, a key index for sweetness–acidity balance, increased substantially in ciriguela (24.02) compared to acerola (6.88), with the mixture (11.85) showing a moderate flavour profile. Water activity and moisture content remained high and similar among samples, indicating the pulps' susceptibility to microbial growth and the need for appropriate preservation. Overall, mixing acerola and ciriguela pulps resulted in intermediate physicochemical characteristics, potentially enhancing sensory balance and consumer appeal.

Dehydration of ACMP by spouted bed drying

The coded levels of the independent variables and the results of quality parameters selected for ACMP dehydration by spouted bed drying are shown in Table 2, while Table 3 lists the results of ANOVA applied to each response. Non-significant variables were omitted, and the other coefficients were used in the final predictive linear equations.

The a_w values of ACMP dehydrated by spouted bed drying varied between 0.24 and 0.36 (Table 2), being lower than those of individual tropical fruits (acerola, red pitanga, and jambolan) treated in the same way (Borges *et al*., 2016) and close to those reported for acerola pulp by Dantas *et al*. (2019). According to Leong *et al.* (2011), a_w values lower than 0.6 like these highlight microbiological stability and safety of products. Moreover, such a response was shown to be positively influenced by maltodextrin concentration (X_3) and its interaction with temperature (X_1 X_3) according to Eq. (3), which showed a satisfactory coefficient of determination (R^2 = 76%) (Table 3):

$$\alpha_W = 0.316 + 0.023X_3 + 0.022X_1X_3 \quad (3)$$

As observed by Alves *et al*. (2017), an increase in inlet air temperature is responsible for a higher temperature gradient between the fed material and the drying air, hence resulting in a higher heat transfer rate, better water evaporation, and lower a_w values.

Moisture content (*M*) ranged from 4.04 to 6.26% (Table 2) in ACMP dehydrated by spouted bed drying. The influence of the independent variables on this response is described by Eq. (4):

$$M = 5.26 - 0.25X_1 + 0.26X_2 - 0.32X_3 + 0.28X_1X_2 + 0.35X_1X_3 + 0.24X_2X_3 \quad (4)$$

Although the regression coefficients of the model for *M* were significant, R^2 (Table 3) was too low (0.58); therefore, this model may only be used to get an idea of results, but not for predictive purposes. Such a response was not only negatively influenced by X_1 and X_3 and positively influenced by the suspension flow rate (X_2), but also positively influenced by interactions among all three variables, with the strongest one being that between X_1

Table 1 - Physicochemical characterisation of the pulps of acerola and ciriguela, and of the mixed pulp (60% acerola and 40% ciriguela)

Parameter	Acerola	Ciriguela	Mixed pulp
Soluble solids - *SS* (ºBrix)	7.23 c ± 0.32	17.33 a ± 0.20	11.06 b ± 0.05
pH	3.33 a ± 0.04	2.93 b ± 0.00	3.25 a ± 0.04
Titratable acidity - *TA* (g/100 g citric acid)	1.05 a ± 0.01	0.72 c ± 0.05	0.93 b ± 0.00
SS/TA ratio	6.88 c ± 0.24	24.02 a ± 1.41	11.85 b ± 0.07
Water activity	0.99 a ± 0.00	0.98 b ± 0.00	0.99 a ± 0.00
Moisture content (%)	91.61 a ± 0.67	75.59 c ± 0.07	85.48 b ± 0.54

*Mean of three replicates ± standard deviation. Mean values in the same row followed by the same superscript letter do not differ significantly from each other ($p > 0.05$) by the Tukey test and X_3. A similar decrease in the final moisture content of the product with increasing temperature was reported by Alves *et al*. (2017) for probiotic orange juice. It has been suggested that the higher the temperature, the higher the rate of mass and heat transfer to the particle, thus the stronger the conduction force for moisture evaporation (Costa *et al*., 2015; Lourenço; Moldão-Martins; Alves, 2020). The *M* values obtained in this study are not so far from those reported for açaí powders (3.22-4.79%) (Costa *et al*., 2015; Lucas; Zambiazi; Costa, 2018), as well as for acerola (8.5%), red pitanga (8.3%) and jambolan (9.7%) powders (Borges *et al*., 2016), all obtained by spouted bed drying.

The hygroscopicity (*H*) of ACMP powders obtained in the spouted bed ranged from 13.58 to 18.17 g/100 g (Table 2) and was linearly reduced by an increase in maltodextrin concentration according to Eq. (5):

$$H = 15.61 - 0.59X_2 - 1.19X_3 - 0.64X_1X_3 + 0.54X_2X_3 \quad (5)$$

which accounted for 85% (R^2 = 0.85) of its variation (Table 3).

This trend may be ascribed to the fact that maltodextrin, a material with low hygroscopicity, reduces the water adsorption capacity of dehydrated products (Ferrari; Ribeiro; Aguirre, 2012). The *H* values obtained in this study are close to those reported for cajá microencapsulated by spray drying (16.16-20.86 g/100 g) using 10 DE maltodextrin (Morais *et al*., 2020) and for powdered acerola prepared by spouted bed drying (14.10-18.88 g/100 g) (Dantas *et al*., 2019).

Table 2 - Coded levels of the independent variables and results of the responses obtained for acerola-ciriguela mixed pulp powders prepared by spouted bed drying

Run	X_1	X_2	X_3	a_w	M (%)	H (g/100 g)	AAR (%)
1	-1 (70)	-1 (4)	-1 (15)	0.31 cd ± 0.01	6.26 a ± 0.39	17.65 a ± 0.10	67.12 b ± 0.47
2	+1 (80)	-1 (4)	-1 (15)	0.24 f ± 0.01	4.46 cde ± 0.44	18.17 a ± 0.32	50.86 f ± 0.42
3	-1 (70)	+1 (6)	-1 (15)	0.30 de ± 0.00	5.72 abc ± 0.54	14.23 ef ± 0.26	62.34 c ± 0.43
4	+1 (80)	+1 (6)	-1 (15)	0.27 fe ± 0.01	5.07 abcde ± 0.51	17.03 ab ± 0.02	76.67 a ± 0.42
5	-1 (70)	-1 (4)	+1 (25)	0.30 de ± 0.00	4.39 de ± 0.30	14.58 ef ± 0.09	57.85 e ± 0.47
6	+1 (80)	-1 (4)	+1 (25)	0.36 a ± 0.00	4.04 e ± 0.78	14.26 ef ± 0.38	39.56 h ± 0.88
7	-1 (70)	+1 (6)	+1 (25)	0.32 bcd ± 0.00	4.86 bcde ± 0.38	15.06 de ± 1.01	39.12 h ± 0.49
8	+1 (80)	+1 (6)	+1 (25)	0.34 ab ± 0.01	5.62 abcd ± 0.23	13.58 f ± 0.10	44.27 g ± 0.72
9	0 (75)	0 (5)	0 (20)	0.31 cd ± 0.02	5.48 abcd ± 0.05	16.34 bc ± 0.21	59.71 d ± 0.72
10	0 (75)	0 (5)	0 (20)	0.32 bcd ± 0.00	5.97 ab ± 0.42	15.79 cd ± 0.52	59.55 d ± 0.47
11	0 (75)	0 (5)	0 (20)	0.34 abc ± 0.00	6.00 ab ± 0.45	15.07 de ± 0.28	59.86 d ± 0.54

*Mean of three replicates ± standard deviation. $X_{1=}$ temperature (ºC); $X_{2=}$ suspension flow rate (mL/min); $X_{3=}$ maltodextrin concentration (%); a_w = water activity; *M* = moisture content; *H* = hygroscopicity; *AAR* = ascorbic acid retention. Mean values in the same column followed by the same superscript letter do not differ significantly from each other (*p* > 0.05) by the Tukey test

Table 3 - Results of ANOVA applied to each response of acerola-ciriguela mixed pulp powders prepared by spouted bed drying

Source	Df	a_w	M (%)	H (g/100)	AAR (%)
Regression	6	0.026421^{ns}	12.20959*	61.35947*	3874.005*
Residual	26	0.008162	8.80423	10.60768	197.664
Lack of fit	2	0.004460^{ns}	3.83628*	4.61222*	190.066*
Pure error	24	0.003702^{ns}	4.96796^{ns}	5.99457^{ns}	7.598^{ns}
Total	32	0.034583	21.01382	71.96715	4071.669
R^2		0.76	0.58	0.85	0.95

ns: Not significant (*p* > 0.05). *Significant at *p* < 0.05. Df = degree of freedom; a_w = water activity; *M* = moisture content; *H* = hygroscopicity; *AAR* = ascorbic acid retention

Ascorbic acid retention (*AAR*) ranged from 39.12 to 76.67% (Table 2) in ACMP dehydrated by spouted bed drying, with the highest value being close to that (72.90%) reported for powdered acerola-containing formulations prepared by the same method using powdered milk as an adjuvant (Dantas *et al*., 2019).

Although the lack of fit was significant for the *AAR* model (Table 3), since the pure error value was low, i.e., very good reproducibility, there was a false result of lack of fit because the F calculated value was high due to the very low value at the denominator; therefore, the model can be considered to have statistical and predictive significance with no lack of fit.

Similarly to moisture content, *AAR* was negatively influenced by X_1 and X_3, but the interactions among variables were more complex, in that, contrary to X_1X_2 interaction, those of X_3 with the other variables had a negative impact. Such behaviour is depicted by Eq. (6), which had excellent fit accounting for no less than 95% of the variation of this response ($R^2 = 0.95$):

$$AAR = 56.14 - 1.86X_1 - 9.50X_3 + 6.77X_1X_2 - 1.38X_1X_3 - 4.36X_2X_3 \quad (6)$$

However, X_3 was by far the variable that most affected *AAR*, i.e., the higher the maltodextrin concentration, the lower the *AAR* in produced powders, confirming the negative effect of maltodextrin addition on this response. A similar effect was obtained in camu-camu pulp dehydrated by this method using maltodextrin as a protectant of bioactive compounds (Fujita *et al*., 2013). Tonon, Brabet and Hubinger (2010) stated that increasing the maltodextrin content causes some "dilution" of the product's pigments, which explains the low *AAR* values in acerola-ciriguela mixed pulp powders prepared by spouted bed drying.

Dehydration of acerola-ciriguela mixed pulp by freeze-drying

Similarly to spouted bed drying, Table 4 lists the coded levels of the independent variables and the results of quality parameters selected for ACMP freeze-drying, while Table 5 shows those of ANOVA applied to the same responses omitting non-significant variables and using the other coefficients in the final predictive linear equations.

The a_w values of freeze-dried ACMP (0.17-0.35) (Table 4) were even lower than those obtained by the spouted bed drying process (Table 2). These values are close to those reported for different freeze-dried fruit pulps (Barbosa *et al*., 2015; Caliskan; Dirim, 2016; Ramírez; Giraldo; Orrego, 2015). The influence of the independent variables on this response is described by Eq. (7), which explained 88% of its variation ($R^2 = 0.88$) (Table 5):

$$a_W = 0.244 - 0.021X_1 + 0.036X_2 - 0.038X_3 - 0.015X_2X_2 \quad (7)$$

Based on the features of this equation, the formation of particles with low a_w is expected by a decrease in X_2 as well as increases in X_1 and X_3, with only a weak negative interaction of X_2 and X_3. Increasing the maltodextrin concentration led to a reduction in the a_w of freeze-dried ACMP, as maltodextrin is a high-molecular-weight carbohydrate

Table 4 - Coded levels of the independent variables and results of the responses obtained for acerola-ciriguela mixed pulp powders prepared by freeze-drying

Run	X_1	X_2	X_3	a_w	M (%)	H (g/100 g)	AAR (%)
1	-1 (-40)	-1 (300)	-1 (15)	0.26 de ± 0.01	5.03 bc ± 0.58	16.15 ab ± 0.59	65.40 e ± 1.31
2	+1 (-20)	-1 (300)	-1 (15)	0.20 b ± 0.02	5.73 cd ± 0.21	16.08 a ± 0.10	59.93 d ± 2.01
3	-1 (-40)	+1 (500)	-1 (15)	0.35 a ± 0.00	6.97 ab ± 0.31	16.00 bc ± 0.21	62.13 c ± 2.29
4	+1 (-20)	+1 (500)	-1 (15)	0.33 a ± 0.01	6.62 a ± 0.51	14.97 ab ± 0.74	73.48 de ± 1.33
5	-1 (-40)	-1 (300)	+1 (25)	0.20 e ± 0.00	6.04 cd ± 0.60	14.03 c ± 0.03	63.98 f ± 0.91
6	+1 (-20)	-1 (300)	+1 (25)	0.17 de ± 0.00	5.29 abc ± 0.16	14.15 c ± 0.27	54.84 de ± 0.95
7	-1 (-40)	+1 (500)	+1 (25)	0.26 de ± 0.01	5.69 d ± 0.20	14.57 c ± 0.26	62.46 bc ± 1.02
8	+1 (-20)	+1 (500)	+1 (25)	0.20 bc ± 0.01	4.62 bc ± 0.24	13.95 c ± 0.35	77.20 de ± 1.64
9	0 (-30)	0 (400)	0 (20)	0.22 cd ± 0.01	5.02 cd ± 0.16	15.98 ab ± 0.31	69.10 a ± 1.91
10	0 (-30)	0 (400)	0 (20)	0.23 bcd ± 0.00	5.67 bc ± 0.15	16.56 a ± 0.39	69.00 a ± 1.68
11	0 (-30)	0 (400)	0 (20)	0.21 de ± 0.01	5.32 cd ± 0.23	16.05 ab ± 0.35	67.22 ab ± 0.97

*Mean of three replicates ± standard deviation. X_1 = freeze-drying temperature; X_2 = vacuum chamber pressure; X_3 = maltodextrin concentration; a_w = water activity; *M* = moisture; *H* = hygroscopicity; *AAR* = ascorbic acid retention. Mean values in the same column followed by the same superscript letter do not differ significantly from each other ($p > 0.05$) by the Tukey test with low hygroscopicity. Higher maltodextrin levels increase the proportion of dry solids in the formulation, thereby diluting the free water fraction and decreasing the amount of water available for microbiological and chemical reactions (Caliskan; Dirim, 2016; Igual *et al*., 2014). Similarly, Bednarska and Janiszewska-Turak (2020) and Janiszewska-Turak *et al*. (2017) observed that the greater the addition of encapsulant, the lower the a_w values of chokeberry and carrot powder juices, respectively.

The moisture content of freeze-dried ACMP, ranging from 4.62 to 6.97% (Table 4), was only a little higher than that of ACMP powders prepared by spouted bed drying and was influenced by all variables according to Eq. (8) ($R^2 = 0.73$) (Table 5):

$$M = 5.63 - 0.18X_1 + 0.22X_2 - 0.33X_3 - 0.17X_1X_2 - 0.27X_1X_3 - 0.48X_2X_3 \quad (8)$$

In addition to the negative influence of drying temperature (X_1) on this response already observed for powders prepared by spouted bed drying, X_2 had a positive effect, that is, the higher the vacuum chamber pressure, the higher the moisture content of particles produced by this drying method. On the other hand, maltodextrin concentration (X_3) had a negative effect, which has also been previously observed for sumac extract (Caliskan; Dirim, 2016).

The hygroscopicity of freeze-dried ACMP ranged from 13.95 to 16.56 g/100 g (Table 4) and was negatively influenced by X_1, X_3 and X_1X_2 interaction and positively influenced by X_2X_3 interaction, as shown by Eq. (9), which showed, however, poor fit ($R^2 = 0.59$) (Table 5):

$$H = 15.32 - 0.20X_1 - 0.81X_3 - 0.21X_1X_2 + 0.19X_2X_3 \quad (9)$$

Maltodextrin concentration had greater negative impact on hygroscopicity than freeze-drying temperature, similarly to what was observed for freeze-dried pulp of lulo (*Solanum quitoense* L.) (Igual *et al*., 2014).

The ascorbic acid retention in freeze-dried ACMP, ranging from 54.84 to 77.20% (Table 4), was, on average, higher than that ensured by the spouted bed drying process (Table 3). This response was positively influenced only by X_2 and X_1X_2 interaction, according to Eq. (10), which accounted for 91% of its variation ($R^2 = 0.91$) (Table 5):

$$AAR = 65.87 + 3.89X_2 + 5.08X_1X_2 \quad (10)$$

The Pareto charts depicted in Figures 2 and 3 illustrate the statistical effects of the independent variables that exerted statistically significant influence on water activity, moisture content, hygroscopicity, and ascorbic acid retention of dry ACMP prepared by spouted bed drying and by freeze-drying, respectively.

Optimisation of operating conditions by the desirability function

Even though the desirability function was applied to all responses, only the plots referring to water activity and ascorbic acid retention of powders obtained by both methods are illustrated in Figure 4 because they were the most influenced responses. In addition, since the objective of this study was to obtain a juice powder with the highest possible ascorbic acid content and the lowest possible water activity, maximisation of *AAR* and minimisation of a_w were imposed as constraints on the desirability function. According to Figure 4A, desirability for a_w of ACMP powder prepared by spouted bed drying can be maximised by reducing drying temperature and suspension flow rate and increasing, at the same time, maltodextrin concentration. On the other hand, the *AAR* desirability (Fig. 4B) can be increased by simultaneously raising drying temperature and suspension flow rate or by simultaneously reducing drying temperature, suspension flow rate and maltodextrin concentration.

As for ACMP powders obtained by freeze-drying, desirability for a_w can be maximised by increasing freeze-drying temperature and maltodextrin concentration but reducing, at the same time, chamber pressure (Fig. 4C), while that for *AAR* can be maximised by simultaneously increasing all three independent variables (Fig. 4D).

Table 5 - Results of ANOVA applied to each response of acerola-ciriguela mixed pulp powders prepared by freeze-drying

Source	Df	aw	M (%)	H (g/100 g)	AAR (%)
Regression	6	0.084595^{ns}	12.8554*	19.26365*	359.300*
Residual	26	0.010862	4.62730	13.38490	34.7501
Lack of fit	2	0.006490*	1.29767*	9.57621*	32.6405*
Pure error	24	0.004372^{ns}	3.32962^{ns}	3.80869^{ns}	2.1123^{ns}
Total	32	0.095457	17.48270	32.64855	394.0501
R^2		0.88	0.73	0.59	0.91

ns: Not significant ($p > 0.05$). *Significant at ($p < 0.05$). Df = degree of freedom; a_w = water activity; *M* = moisture content; *H* = hygroscopicity; *AAR* = ascorbic acid retention

Figure 2 - Statistical analysis of preparation of dry acerola-ciriguela mixed pulp by spouted bed drying. Pareto charts showing the influence of the independent variables on the responses, namely water activity (A), moisture content (B), hygroscopicity (C), and ascorbic acid retention (D)

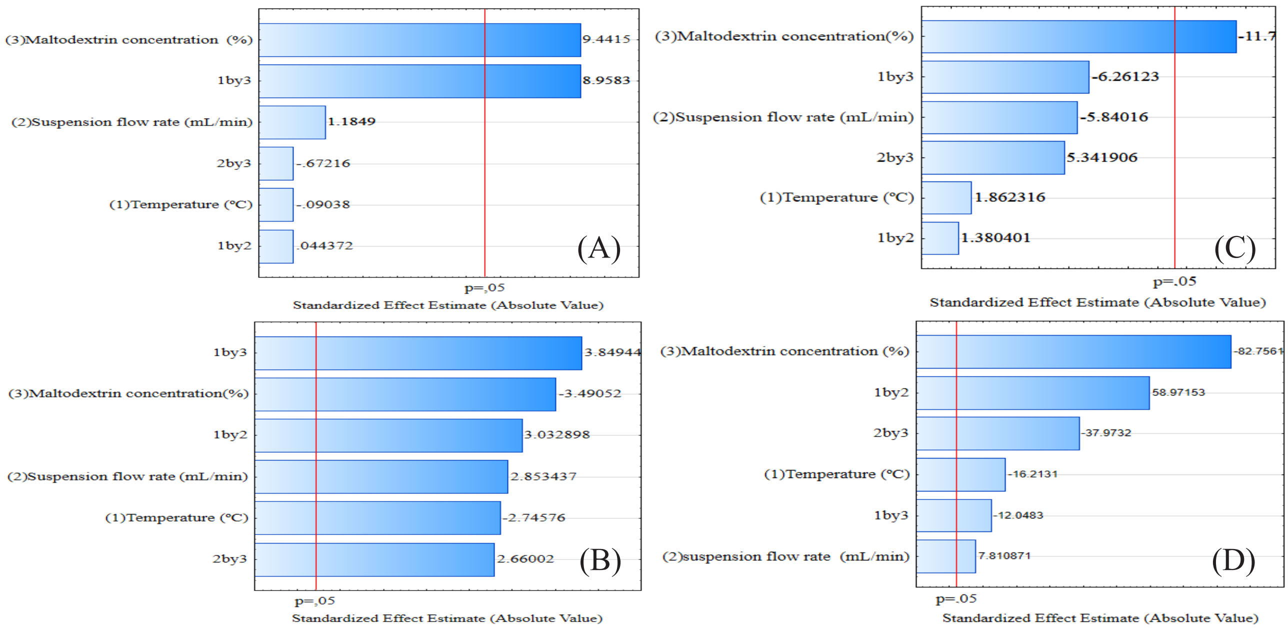
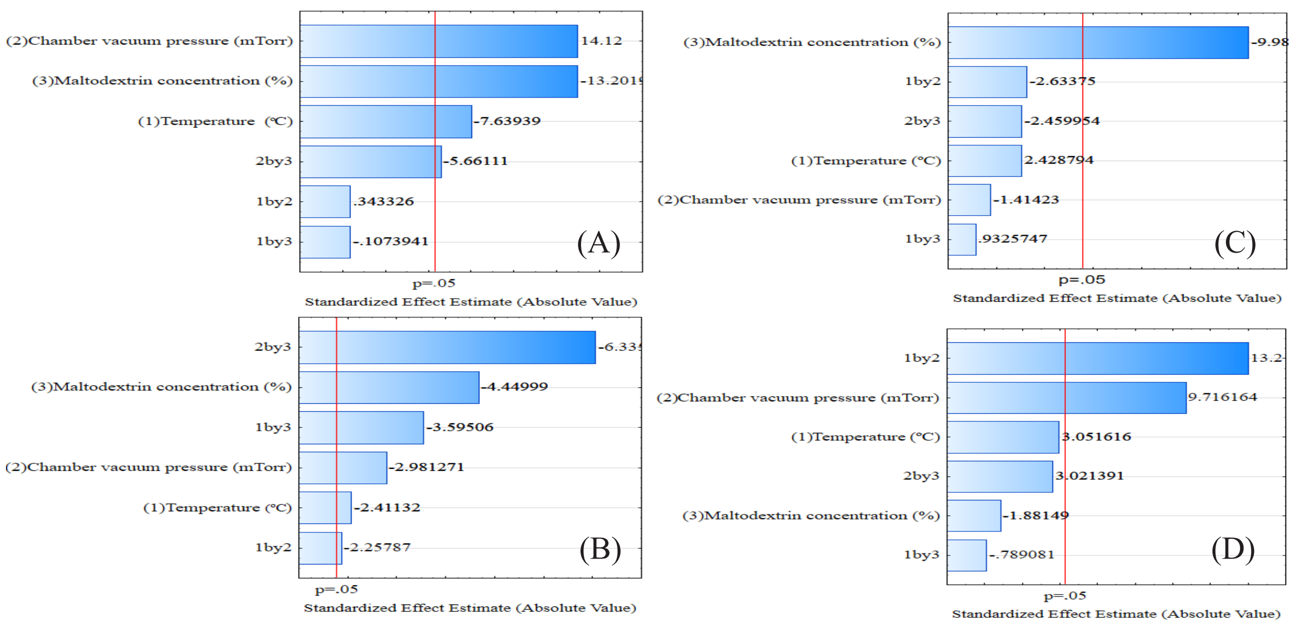
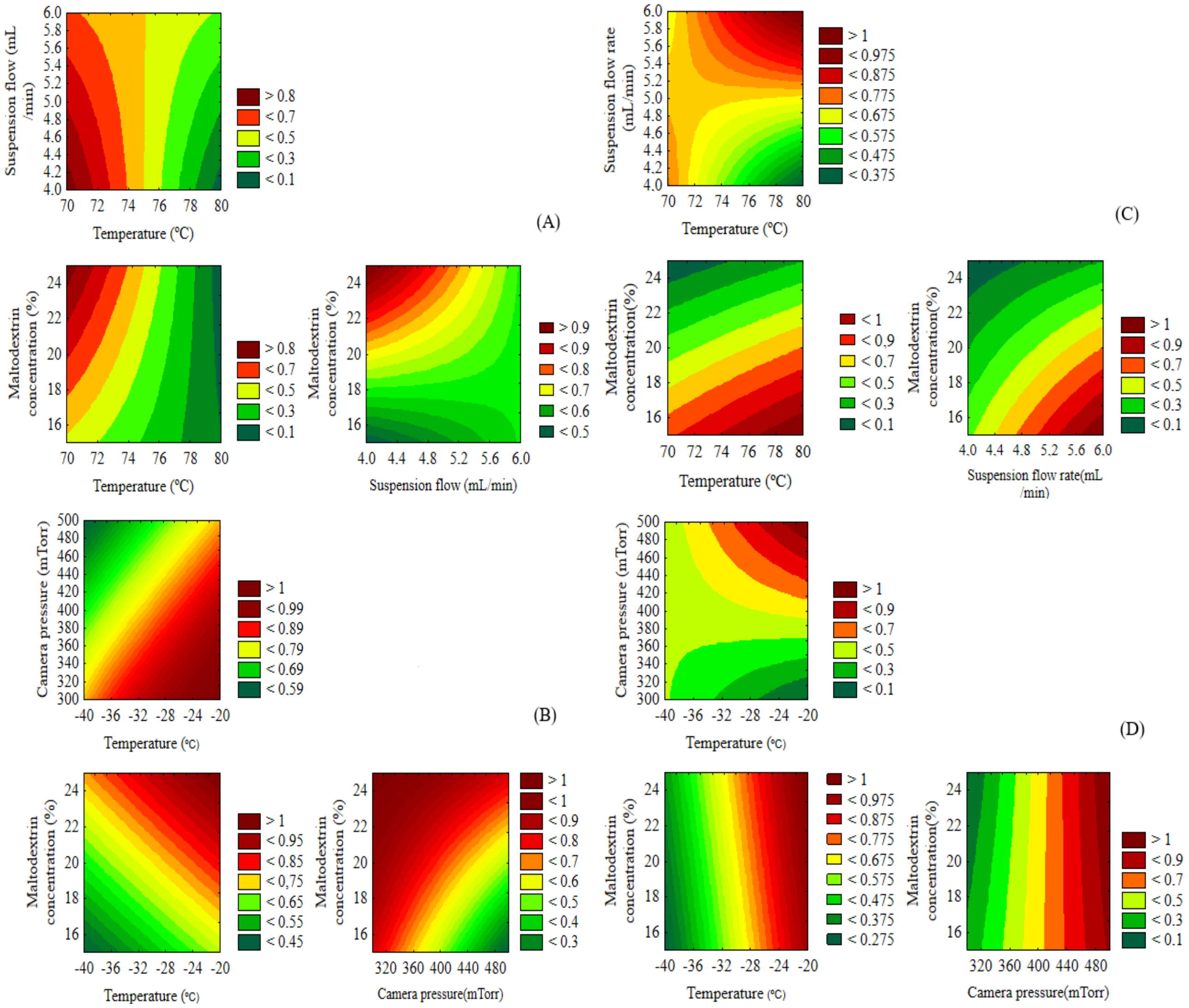


Figure 3 - Statistical analysis of preparation of dry acerola-ciriguela mixed pulp by freeze-drying. Pareto charts showing the influence of the independent variables on the responses, namely water activity (A), moisture content (B), hygroscopicity (C), and ascorbic acid retention (D), respectively



The statistical analysis of the central experimental design (11 runs) and the desirability analysis generated critical values that could be used as optimal operating conditions to obtain ACMP powders by spouted bed drying and freeze-drying (Figure 4). These conditions for spouted bed drying are drying temperature of 75 ºC, suspension flow rate of 5 mL/min and maltodextrin concentration of 15%, while those for freeze-drying are drying temperature of -20 ºC, vacuum chamber pressure of 500 mTorr and maltodextrin concentration of 25%.

Figure 4 - Plots of the desirability function applied to water activity (A and C) and ascorbic acid retention (B and D) of dry acerola-ciriguela mixed pulp prepared by spouted bed drying and freeze-drying, respectively



CONCLUSIONS

In this work, dehydration of acerola-ciriguela mixed pulp (ACMP) by spouted bed drying and freeze-drying was studied by evaluating the influence of operating conditions on moisture content, water activity, hygroscopicity, and ascorbic acid retention. The increase in temperature caused a decrease in moisture content and water activity of powders obtained by both drying methods. An increase in maltodextrin concentration caused a decrease in water activity of freeze-dried ACMP and a decrease in hygroscopicity of powders prepared by both methods. As for the ascorbic acid retention in powders, this response was reduced by an increase in maltodextrin concentration when spouted bed drying was used as dehydration method, while it increased with increasing the vacuum chamber pressure in freeze-dryer. Even though both methods ensured very good retention of ascorbic acid, freeze-drying showed better performance in this aspect; therefore, the ACMP powder produced by freeze-drying may be a good source of ascorbic acid. Nevertheless, that prepared by spouted bed drying also had satisfactory physicochemical characteristics, thus being an alternative to freeze-drying according to the circumstances. In conclusion, ACMP can be dehydrated by both methods in order to prolong the shelf life and can find several applications in the development of functional foods with high ascorbic acid content.

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DATA AVAILABILITY STATEMENT

Data-available-upon-request to the corresponding author.

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