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Pineapple powder quality: Effect of the feedstock solution composition, homogenization and spray drying process

Calidad del polvo de piña: Efecto de la composición de la solución alimentadora, la homogenización y el proceso del secado por aspersión

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Abstract

The objective of this research was to evaluate concentration of compounds in the feedstock solution and the conditions of the spray drying process and process conditions for high-pressure homogenization and spray drying on powder pineapple (pulp, core, and peel) quality. The independent variables were considered: maltodextrin (8.0-12.0%), homogenization pressure of the suspension fed to the dryer (30-50 MPa), rotary atomizer speed (18000-20000 rpm), inlet air temperature (125-155°C), and outlet air temperature (80-90°C). The experimental optimization of multiple responses defined the independent variables as homogenization pressure (44.4 MPa), maltodextrin (10.2%), inlet air temperature (134.9°C), outlet air temperature (80°C), and rotary atomizer speed (18296 rpm). The dependent variables were: a_w (0.215±0.006), solubility (81.9±1.3%), hygroscopicity (12.6±0.9%), wettability (312.41±21.9 s), angle repose (32.18±2.7°), particle size as D_[4,3] (22.6±1.5 μ m), total polyphenols (301.98±13.8 mg GAE/100g_{db}), total flavonoids (199.85±11.3 mg QE/100g_{db}), ABTS (179.14±10.3 mg TE/100g_{db}), DPPH (308.49±8.1 mg TE/100g_{db}), and Vitamin C (33.2±0.3 mg AA/100g_{db}).

Keywords: Ananas comosus; antioxidant activity; fruit powder; physicochemical properties, Response surface methodology.

Resumen

Este estudio investigó el efecto de la composición y las condiciones de proceso para la homogenización de alta presión y el secado por aspersión sobre la calidad del polvo de piña (pulpa, núcleo y extracto de cáscara). Las variables independientes consideradas fueron maltodextrina (8,0-12,0%), presión de homogenización de la suspensión de alimentación al secador (30-50 MPa), velocidad del disco atomizador (18000-20000 rpm), temperatura del aire a la entrada (125-155°C) y temperatura del aire a la salida (80-90°C). La optimización experimental de múltiples respuestas definió las variables independientes como maltodextrina (10,2%), presión de homogenización de la suspensión de alimentación al secador (44,4 MPa), velocidad del disco atomizador (18296-20000 rpm), temperatura del aire a la entrada (134.9°C) y temperatura del aire a la salida (80°C). Las variables dependientes a estas condiciones fueron a_w (0,215±0,006), solubilidad (81,9±1,3%), higroscopicidad (12,6±0,9%), humectabilidad (312,41±21,9 s), ángulo de reposo (32,18±2,7°), tamaño de partícula D_[4,3] (22,6±1,5 μ m), polifenoles totales (301,98±13,8 mg GAE/100g_{db}), flavonoides totales (199,85±11,3 mg QE/100g_{db}), ABTS (179,14±10,3 mg TE/100g_{db}), DPPH (308,49±8,1 mg TE/100g_{db}), and Vitamina C (33,2±0,3 mg AA/100g_{db}).

Palabras clave: Ananas comosus; actividad antioxidante; polvo de fruta; propiedades fisicoquímicas, metodología de superficie de respuesta.

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1 Introduction

Pineapple (*Ananas comosus*) is one of the most consumed tropical fruits in the world and represents one of the most important commodities within the tropical fruit market. It has a total global production of approximately 28 million tons for the year 2019, and Costa Rica, the Philippines, and Brazil were the main producing countries (FAOSTAT, 2019). Pineapple pulp has a low caloric content and is desired for its pleasant sweet taste. Furthermore, it is an important source of dietary fiber, Mg, K, Fe, and antioxidant compounds, such as vitamin C, polyphenols, and flavonoids among others, thus allowing for its classification in the context of functional foods (Ancos *et al.*, 2016; Lobo & Yahia, 2016).

The pineapple agribusiness produces a large amount of waste (crown, peel, and core). While this presents a problem for the environment and its management generates higher costs for the producer, these residues can be used as a source of compounds of biological interest. Pineapple peel contains phenolic compounds, such as gallic acid, catechin, epicatechin, hydroxymenzoic acid, chlorogenic acid, and ferulic acid; while, pineapple core contains Vitamin C and proteolytic enzymes, such as bromelain (Azizan et al., 2020; Sepúlveda et al., 2018). In the framework of the circular economy, pineapple waste is being used as a raw material for the production of alcohol, vinegar, biogas and as a source of cellulose in the paper industry, as well as a source of active compounds for the food and cosmetics industry (Azizan et al., 2020; Roda & Lambri, 2019; Sepúlveda et al., 2018). In this sense, the present research contributes to the utilization of valuable compounds from pineapple residues by integrating them into a new food matrix that could provide a higher nutritional content and influence the characteristics of the final product.

Spray drying (SD) processes have been widely used in recent years by the food and pharmaceutical industry because of the way it protects the active compounds and because it is a simple, fast, and cost-effective process (Tontul & Topuz, 2017). The SD process depends on several factors associated with the formulation, such as the type and amount of encapsulant and the operating conditions, including flow rate, temperature and total solids of the feed, atomizing disk type and speed, nozzle type, and pressure, air inlet and outlet temperature, residence time or vacuum pressure in the drying chamber, or air flow (Ferreira *et al.*, 2019; Lacerda *et al.*, 2016; Moghaddam *et al.*, 2017; Zhang *et al.*, 2020).

In general, fruit powders contain a high percentage of low molecular weight sugars (glucose, sucrose, fructose, among others), which leads to problems of stickiness, agglomeration, high hygroscopicity, and low glass transition temperature (Tg). Drying additives with high molecular weight and high Tg contribute to solving these problems (Goula, 2017). Common additives include carbohydrates (maltodextrins (MD) with different dextrose equivalents, starches, and cyclodextrins), proteins (whey, soy, casein, and gelatin), and gums (Arabic, xanthan, and guar) (Labuschagne, 2018). Different studies have evaluated the effect of the concentration of compounds in the feedstock solution and the conditions of the SD process on fruit powders and their active compounds, highlighting the following: (1) Pineapple peel extract (IAT: 150 Y 190°C)(Lourenço, Moldão-Martins, *et al.*, 2020), (2) pineapple-mint juice (MD: 0, 3, and 15%) (Braga *et al.*, 2020), (3) sour cherry juice (IAT 100-140°C); (4) carrot (MD and GA (1:1, 2:1), rotary atomizer speed: 28000 and 39000 rpm) (Janiszewska-Turak & Witrowa-Rajchert, 2020), (5) sapota (MD 15% and GA 15%) (Araujo *et al.*, 2020), (6) Blackberry (IAT: 140-160°C, OAT: 80-90°C, MD: 5-9%, rotary atomizer speed: 20000 18000 rpm (Cortés-Rodríguez *et al.*, 2022).

The objective of this research was to evaluate concentration of compounds in the feedstock solution and the conditions of the SD process and process conditions for high-pressure homogenization and spray drying on powder pineapple (pulp, core, and peel) quality.

2 Materials and methods

2.1 Materials

Fresh and ripe pineapples (Golden Sweet), were collected from the department of Valle del Cauca - Colombia. In addition, gum Arabic (Tic Pretested gum Arabic FT Powder, Tic Gums, USA), maltodextrin (MD) with dextrose equivalent 18-20 (Ingredion, Colombia), and $Ca_3(PO_4)_2$ were used as wall material (HRA uniquímica, Colombia).

2.2 Feedstock suspension

Initially, whole pineapples were washed with pressurized water and sanitized in a 2 mL/L of Citrosan® solution. The pulp and core were processed in a homogenizer (UTL 50- Ultraturrax; IKA, Marietta, GA, USA) for 5 minutes at 10000 rpm. Part of the liquid fraction of the previous homogenate was used to disintegrate the separated peel, using a blender (3V 4655 / Oster, Hilliard, Ohio, USA) for 5 minutes at high speed (position 3). subsequently, the resulting product was filtered through a 500 mesh sieve and the extract obtained was mixed again with the pulp and core homogenate. Batches of 2500 g of pineapple feedstock suspension were formulated, containing, GA (Tg = 126 °C) (1.0%) (Cardona et al., 2022), MD (Tg = 198 °C) (8-12%) and Ca₃(PO₄)₂ (0.8%). The preliminary suspension obtained was again homogenized in a colloid mill (MK 2000/5, Colloid mill, IKA, Marietta, GA, USA), GAP (minimum), and with a 1 min of recirculation time. Finally, it was passed through a high-pressure homogenizer (SRH60-70, Shanghai Samro Homogenizer Co. Ltd., China) with a homogenization pressure (HP) (30-50 MPa) and 3 minutes of recirculation time.

2.3 Spray drying process

The drying process was carried out using an atomizing disk spray dryer, with co-current flow (Vibrasec, model PASLAB 1.5), and operating at sub-atmospheric conditions (85.9 Pa). 26 experiments were carried out using response surface methodology (RSM) through a face-centered central composite design ($\alpha = 1$), considering the independent variables: MD (8.0-12.0%). The ranges of MD used in the experimental design were defined in order to guarantee a powder ratio of pineapple solids/rest of solids (S pineapple/S rest)> 1, homogenization pressure (HP) (30-50 MPa), rotary atomizer speed (RAS) (18000-20000 rpm) , inlet air temperature (IAT) (125-155°C), and outlet air temperature (OAT) (80-90°C). The ranges of the values of the independent variables were determined in previous spray-drying tests. The dependent variables were as follows: water activity (a_w) , solubility, wettability, angle of repose, mean diameter of equivalent volume $(D_{[4 3]})$, total phenols, total flavonoids, antioxidant capacity (ABTS and DPPH), and vitamin C (table 1).

2.4 Characterization of the properties of pineapple powder

The a_w was determined according to the AOAC 978.18/2012 method, using a 25°C dew point hygrometer (3TE / Aqualab, Decagon, Devices, Pullman, WA, USA). The solubility was determined according to the methodology of Lucas *et al.* (2018) with modifications: dilution ratio = 50 mL distilled water/1 g of product. Hygroscopicity was determined as the water absorption level of 2 g of pineapple powder (Marulanda *et al.*, 2018). The wettability was performed by pouring 1 g pineapple powder from a height of 10 cm over 150 mL of distilled water at 25°C, it was determined as the time in which the product was completely submerged. The angle of repose was determined according to the methodology described by Geldart *et al.* (2006) using 10 g of pineapple powder in a funnel located at a height of 10 cm. The D_[4,3] was determined in a laser diffraction particle analyzer (3000/Mastersizer; Malvern Instruments Ltd., Worcestershire, UK), using the Aero S.

The determination of total polyphenols, total flavonoids and antioxidant capacity (ABTS, DPPH) was performed on a methanolic extract obtained initially from the mixture of 1 g of pineapple powder and 10 mL of methanol/water (70/30), treated for 10 minutes in an ultrasonic bath, and then centrifuged at 8000 rpm (15 min, 4°C). Finally, the supernatant was separated, filtered, and diluted with the methanol/water solvent to achieve a final volume of 25 mL in a volumetric flask. The total polyphenols were determined by the Folin-Ciocalteu colorimetric method according to the methodology described by Gallón et al. (2020). A standard calibration curve was constructed with gallic acid (20- 300 µg/mL), absorbance was read at 760 nm (Evolution 60S UV-vis / spectrophotometer; Thermo Fisher Scientific, Waltham, MA USA), and the results were expressed as mg gallic acid equivalent (GAE)/100gdh. Total flavonoids were determined according to the colorimetric method described by Sharma et al. (2016) with modifications. 500 μ L of the extract was mixed with 150 µL of NaNO₂ (5% w/v), 150 µL of AlCl₃ (10% w/v), and 700 µL of NaOH (1M). A standard calibration curve was made with quercetin (10-300 μ g/mL), absorbance was read at 510 nm, and the results were expressed as mg quercetin equivalents (QE)/100 g_{db} .

D	HP	MD		OAT (CO)	RAS	_	S	Н	We	AR	D[4,3]	ТР	TF	DPPH	ABTS	VitC
Kun	(MPa)	(%)	IAI (C)	UAI (C)	(rpm)	\mathbf{a}_{w}	(%)	(%)	(s)	(°)	(mm)	(mg GAE/100 g)	(mg QE/100 g)	(mg TE/100 g)	(mg TE/100 g)	(mg/100g)
1	40	10	125	85	20000	0.192	79.8	15.8	158.8	33.2	42	199.8	153.3	274.3	158.2	5.9
2	40	10	140	85	20000	0.198	75	16.3	367	37.6	42.1	230.6	193	317.9	183.8	12
3	50	8	125	90	22000	0.187	77.5	17.3	194.7	31.5	82.7	185.6	199.8	336.3	235	26
4	40	10	140	85	20000	0.218	74.5	16.8	305.3	33.3	42.3	238.1	147.5	348.8	219.1	7.1
5	50	10	140	85	20000	0.21	73.9	16.5	257.7	39.7	42.5	235	205.6	292.3	225.6	39.1
6	50	8	155	90	18000	0.212	78.6	19.8	152.7	36.3	70.8	161.3	124.7	356.1	252	26.7
7	40	12	140	85	20000	0.196	81	18.8	308.3	37.2	42.6	204.9	117.3	254.7	153	8.2
8	40	10	140	90	20000	0.16	77.1	21.4	325.7	36.9	42.9	200.8	133.3	289.8	194.3	8.3
9	30	8	155	90	22000	0.168	77.2	16.6	281.3	38.8	75.3	212.3	136.1	324.9	209.1	9.9
10	40	10	155	85	20000	0.211	77.4	19.6	281.3	33.7	82.3	192.3	158.8	320.9	227.5	13.2
11	30	12	155	90	18000	0.195	77.5	15.6	263.3	33.1	33	193	152.8	331.6	154.6	18.6
12	50	12	125	90	18000	0.252	75.4	16.6	213.3	39.3	63.8	201.5	188.8	279.6	192	10.2
13	30	12	125	90	22000	0.206	76.8	16.6	331	23.5	68.9	189.4	186.5	242.2	204.1	6.7
14	40	10	140	85	20000	0.197	75	15.8	362.7	39.5	47.3	229.4	194.9	318.9	133.3	12.7
15	40	10	140	85	22000	0.249	75.5	15.3	321.7	32	61	176.5	151.3	318.5	108.6	10.8
16	50	12	155	80	18000	0.227	76.7	15.8	331	38.3	37.9	180.7	161.2	331.3	221.8	33.7
17	40	10	140	80	20000	0.231	75.7	13.7	312	38.4	46.8	240.5	144.8	344.8	237.7	33.4
18	50	12	125	80	22000	0.206	75.7	14.8	434.3	31.7	57.9	255.4	203.2	300.7	252.4	8.3
19	40	10	140	85	20000	0.206	75.1	16.3	359.3	36.9	44.7	229.9	188.3	315.9	232	11.5
20	50	8	155	80	22000	0.18	75.1	16.3	333	30.4	82.4	230.2	166.7	346.3	236.3	16
21	40	10	140	85	18000	0.25	78.7	14	437.7	31.7	47.7	237.2	200.4	359.5	188.2	11.6
22	40	10	140	85	20000	0.203	75	15.9	378.7	38.1	48	227.9	190.9	316.9	225.1	11.4
23	30	10	140	85	20000	0.243	75.6	16.3	268.7	40	50.2	198	144	324.8	238.6	22.2
24	40	8	140	85	20000	0.234	74.9	17.7	346	35.8	57.3	230	210.6	361.8	229.8	12.6
25	30	8	125	80	18000	0.208	78.7	17.1	175.7	39.6	57.3	289.7	199.2	309.6	160.6	16.4
26	30	12	155	80	22000	0.154	78.3	14.8	312.3	33	67.3	280.4	129.1	289.5	169.2	16.1

Table 1. Experimental design and mean values of the dependent variables.

S: solubility, H:hygroscopicity, We: Wettability, AR: angle repose, D[4,3]: particle size, TP: total polyphenols, TF: total flavonoids, VitC: vitamin C.

ABTS and DPPH were determined according to the methodology described by Gallón *et al.* (2020), performing calibration curves with Trolox (40-240 μ M for ABTS and 0.02-0.12 mg/mL for DPPH) and were expressed as mg Trolox equivalent (TE)/100g_{db}. Vitamin C extraction was carried out with 2 g of pineapple powder and 10 mL of KH₂PO₄ (0.02 M, pH: 3.00 adjusted with 85% ortho-phosphoric acid). The quantification was carried out by HPLC (Shimadzu Prominence) according to the methodology described by Lucas *et al.* (2018) and the results were expressed as mg ascorbic acid (AA)/100g_{db}.

2.5 Statistical analysis, experimental modeling, and optimization

The analysis of variance (ANOVA) was performed with a confidence level of 95%, expressing the main effects and the linear and quadratic interactions of the independent variables. The software Statgraphics Centurion XVII. II was used, and the experimental data were fitted to a second order polynomial model (equation 1). In the equation, y represents the independent variable, B_0 , B_i , B_{ii} , B_{ij} indicate the regression coefficients, x_i and x_j indicate the linear effect, x_i^2 the quadratic effect, and x_{ij} refers to the interactive effect between two independent variables. The adequacy of the models was determined using the regression coefficient R^2 ,

while the optimization of multiple experimental responses was carried out according to the desirable characteristics in the final product. The validation of the models was carried out from the relative mean error (RME) (equation 2) between the value of the experimental response variable to the optimal condition (3 replicates) and the value predicted by the model.

$$y = B_0 + \sum_{i=1}^n B_i x_i + \sum_{i=1}^n B_{ii} x_i^2 + \sum_{i=1}^n \sum_{i=1}^n B_{ij} x_{ij}$$
(1)

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

3 Results and discussion

The mean values of the evaluated dependent variables and the response volume plots are shown in Table 1, while the p-values corresponding to the analysis of variance (ANOVA) are presented in Table 2. In general, the ANOVA showed significant differences (p < 0.05) for all dependent variables, except for the ABTS variable. This variable shows a significant effect of the independent variables on the process responses, mainly for the variables MD, IAT and OAT.

Table 2.	ANOVA	(n-value)) for PP	response	surface	models.
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		\mathbf{a}_{w}	S	We	AR	D _[4,3]	ТР	TF	DPPH	ABTS	VitC
	HP	0.182	0.469	0.858	0.931	0.205	0.185	0.139	0.172	0.812	0.044*
	MD	0.135	0.034*	0.549	0.672	0.039*	0.346	0.045*	0.003*	0.201	0.514
Main effects	IAT	0.413	0.306	0.09	0.878	0.001*	0.77	0.88	0.072	0.241	0.307
	OAT	0.021*	0.549	0.825	0.646	0.497	0.161	0.756	0.043*	0.443	0.011*
	RAS	0.964	0.186	0.105	0.912	0.054	0.053	0.22	0.101	0.187	0.899
	HP-HP	0.537	0.094	0.107	0.051	0.445	0.64	0.462	0.511	0.171	0.004*
	MD-MD	0.617	0.236	0.668	0.855	0.514	0.585	0.914	0.491	0.901	0.116
Quadratic effects	IAT-IAT	0.111	0.098	0.016*	0.106	0.002*	0.226	0.716	0.116	0.947	0.079
	OAT-OAT	0.051	0.747	0.894	0.35	0.213	0.42	0.204	0.819	0.402	0.141
	RAS-RAS	0.028*	0.723	0.059	0.027*	0.052	0.715	0.426	0.049*	0.108	0.169
	HP-MD	0.09	0.089	0.33	0.351	0.037*	0.224	0.616	0.23	0.684	0.106
	HP-IAT	0.243	0.155	0.074	0.352	0.022*	0.028*	0.033*	0.047*	0.169	0.112
	HP-OAT	0.043	0.672	0.054	0.195	0.002*	0.062*	0.129	0.846	0.939	0.242
	HP-RAS	0.21	0.171	0.096	0.694	0.012	0.724	0.778	0.895	0.407	0.051
Interaction offects	MD-IAT	0.109	0.401	0.263	0.86	0.001*	0.718	0.602	0.266	0.119	0.764
interaction enects	MD-OAT	0.033	0.034*	0.678	0.707	0.043*	0.787	0.111	0.454	0.934	0.067
	MD-RAS	0.226	0.396	0.024*	0.119	0.091	0.020*	0.043*	0.899	0.302	0.356
	IAT-OAT	0.506	0.333	0.019*	0.21	0.005*	0.577	0.155	0.087	0.124	0.09
	IAT-OAT	0.005	0.068	0.967	0.646	0.010*	0.041*	0.578	0.014*	0.246	0.13
	OAT-RAS	0.833	0.341	0.553	0.71	0.017*	0.184	0.437	0.452	0.507	0.068
	\mathbf{R}^2	93.39	86.23	99.67	93.33	97.54	95.07	85.92	95.59	82.5	95.07

* Significant Difference (p<0.05)

S: solubility, H:hygroscopicity, We: Wettability, AR: angle repose, $D_{[4,3]}$: particle size, TP: total polyphenols, TF: total flavonoids, VitC: vitamin C.



Figure 1. Pineapple powder a_w area and volume response graphs.

3.1 Water activity

The mean values of the a_w fluctuated between 0.154±0.006 and 0.252±0.004. These reflect significant differences (p <0.05) with respect to OAT and its linear interactions OAT-HP and OAT-MD, with the linear interaction IAT-RAS and the quadratic RAS-RAS; however, at these conditions the changes of a_w are very low ($\Delta a_w = 0.02$). The response volume graph (Figure 1) shows a trend to lower a_w values when the system operates mainly at high OAT, AIT and RAS (blue area), which is consistent due to the higher driving force to mass transfer experienced by the particle at high process temperatures, which is also reflected in the low moisture values of the particles (0.87 - 2.64%). (Lucas et al., 2018). furthermore, the effect of high RAS values produces higher tangential velocities at the disc exit and at the same time smaller particle sizes, which favors higher water evaporation and lower a_w (Shishir *et al.*, 2014). On the other hand, lower a_w values are found at low OAT, RAS, and IAT, which is not consistent with the phenomenology. This could be attributed to the variation found in the viscosity of the dryer feedstock, caused by the total solids of the pineapple, the additives (MD and GA), and the synergistic effects of MD-GA (Cardona et al., 2022; Janiszewska-Turak et al., 2017; Navarrete-Solis et al., 2019).

In general, pineapple powder presented low values $(a_w < 0.255)$, and this contributes to better product stability due to the lower availability of free water in the powder, which is responsible for the biochemical, oxidative, and chemical reactions, as well as microbiological growth (Caliskan & Dirim, 2016; Daza *et al.*, 2016). Similar a_w results have been reported in fruit powders: Cagaita (0.13-0.26) (Daza *et al.*, 2016), Jussara (0.216-0.314) (Da Silva *et al.*, 2016), Watermelon (0.153-0.289) (Oberoi & Sogi, 2015), and Jamun (0.18-0.25) (Santhalakshmy *et al.*, 2015).

3.2 Solubility

The mean values of solubility fluctuated between 73.9 ± 0.6 and $81.0\pm0.5\%$, which reflected significant effects (p <0.05)



Figure 2. Pineapple powder solubility response volume.

with respect to the MD and the linear interaction MD-OAT. These results are considered good because the solubility is affected by the final particle composition. An important component of the particles is the insoluble fiber contributed to the suspension by the core and the fruit pulp (24.14 ± 0.01) - $47.6 \pm 0.00 \text{ g/100}$). The response volume graph (Figure 2) shows a trend of increasing solubility with an increase in MD (less solids soluble/solids insoluble ratio in the feedstock suspension). This is mainly attributed to its non-crystalline structure and amorphous nature, as it has a hydrophilic or polar character very similar to the water molecule (Fazaeli et al., 2012)(Mahdi et al., 2017). This same influence of MD on solubility has been reported in powder fruits obtained by spray drying, including the following: pomegranate, black mulberry [31], and extract of Murraya koenigii (Linn) leaves (Sablania & Bosco, 2018).

On the other hand, it is observed that the linear interaction MD-OAT was negative. This could suggest an increase in solubility at low OAT, considering that the particles could be presenting a high specific and irregular surface. It is suggested that higher process temperatures result in the formation of a possible surface layer in the particles (Chegini & Ghobadian, 2005), which is made up of structural complexes of MD with sugars, celluloses, hemicellulose, and pectin, among others. These complexes result from the pineapple structure, which reduces the solubility of the particles (less diffusion of water in the microstructure). Additionally, the effect of high temperatures in products rich in sugars such as pineapple produces caramelization reactions that trigger compounds that are less soluble (Braga *et al.*, 2020).

3.3 Hygroscopicity

Hygroscopicity is an important parameter in food powders, as it represents the water adsorption capacity of the environment and influences the final quality during storage [7]. The mean values of hygroscopicity fluctuated between 13.7 ± 0.2 - $21.4\pm0.2\%$, which characterizes pineapple powder as slightly hygroscopic (Lacerda et al., 2016). Similar results have been reported for other powder fruits, such as: cagaita (13.8 - 19.4%) (Daza et al., 2016); satureja montaña (18.3 - 20.0%) (Vidović et al., 2014), and uchuva (12.8 - 18.1) (Cortés et al., 2017). The ANOVA showed significant differences (p<0.05) between the variables IAT and OAT and both the linear interactions HP-MD, HP-IAT, HP-RAS, and MD-RAS and the quadratic effects MD-MD and RAS -RAS. In general, Hygroscopicity is considered to be affected by formulation and process conditions. This suggests that there is a relationship between the adsorption forces at the pineapple powder interface with respect to the random distribution of the aforementioned components or complexes, in addition to the microstructure and surface area conferred by the SD process. (Moghaddam et al., 2017; Souza et al., 2018). On the other hand, the moisture and a_w present in the particles interact with the MD, GA and the compounds present in the pineapple, generating liquid bridges between the particles that reconfigure the structure of the particles, thus generating greater capillarity phenomena, which allows greater adsorption of the surrounding moisture (Moghaddam *et al.*, 2017; Souza *et al.*, 2018).

The surface area and volume response graph (Figure 3) show the growth of hygroscopicity with increasing process temperatures (IAT and OAT). This could be directly related to the higher evaporation rate of water, which contributes to the formation of microcapsules with smaller particle sizes and larger surface areas (Moghaddam et al., 2017). On the other hand, the rapid formation of the particles, due to a high driving force, favors a non-thermodynamic amorphous glassy state, higher porosity, and larger surface area, which favors higher adsorption of water from the surrounding air (Daza et al., 2016; Gallo et al., 2011; Moghaddam et al., 2017). Some authors have reported this tendency relating it to the humidity of powdered fruits (more IAT and OAT less humidity and more hygroscopicity), as is the case of jamun (Santhalakshmy et al., 2015) and coconut (Santhalakshmy et al., 2015).

The effect of all the linear interactions of HP with IAT, MD, and RAS was positive, which favored a higher hygroscopicity at high values of HP, IAT, and MD in the RAS range between 19000 - 21000 rpm. This situation is attributed to the fact that a higher HP in the suspension reduces particle sizes, mainly when the contribution of total solids from the MD is lower. This is consistent with that which was presented in powders of other matrices: sugar cane (Largo *et al.*, 2015), coconut (Lucas *et al.*, 2018), and red pitaya shell (Bakar *et al.*, 2013).



Figure 3. Pineapple powder hygroscopicity response area and volume graph.



Figure 4. Pineapple powder wettability main effects and response volume.



Figure 5. Pineapple powder $D_{[4,3]}$ response volume.

3.4 Wettability

The mean wettability values fluctuated between 158.8 - 437.7 s, indicating that the pineapple powder exhibits low instantaneous properties (Lucas *et al.*, 2018). The slow inward diffusion of water is the result of mass transfer mechanisms, initially by capillary forces and then by the chemical potential difference between the liquid water and the pineapple powder particle (Martínez-Navarrete *et al.*, 1998). In addition, the affinity of the functional groups of the polymeric segments of the pineapple powder interface to the water molecule could be a contributing factor (Islam *et al.*, 2016).

The main effects graph (Figure 4) shows a greater weight in wettability changes with respect to the IAT and RAS variables; however, the ANOVA presented significant differences (p<0.05) with respect to the RAS-MD interactions. This situation makes wettability highly dependent on specific SD conditions, identifying 2 areas in the response volume analysis where water penetration times are lower (blue color): 1) IAT (150-160°C), OAT (86-90°C), MD (11-12%) and RAS throughout the range; and 2) IAT (120-130°C), OAT (80-88°C), and RAS and MD throughout the range. These conditions are consistent with the quadratic interaction of IAT where a curvilinear behavior maximizes wettability between 140 and 150°C. In the 1st condition, the decrease in wettability with IAT and OAT corresponded to the highest achieved values of D_[4,3], which generates a larger free space between the interstices of the pineapple powder and a lower resistance to water diffusion (Santhalakshmy et al., 2015). On the other hand, the effect of the higher IAT on the pineapple powder could be generating a more porous and fractured structure, which favors water diffusion towards the interior of the particles and increases the wetting rate. This situation has been described in orange powder (Chegini & Ghobadian, 2005) and in a mixture of carrot and watermelon (Mestry et al., 2011).

3.5 Particle size

The size distribution of the pineapple powder presented a monomodal behavior, fluctuating the values of the diameter equivalent to the mean volume $(D_{[4,3]})$ between 33.0±0.1-

82.7±0.1 μ m. Some research has reported the effect of SD process conditions on particle size and especially RAS (Chegini & Ghobadian, 2005; Janiszewska-Turak & Witrowa-Rajchert, 2020). In addition, its relationship with other characteristics of the particles, such as fluidity, rehydration capacity, solubility, dispersibility, compaction capacity, apparent density, compacted density, among others, has made it possible to define their handling, packaging, and storage conditions (Da Silva *et al.*, 2016; Gallo *et al.*, 2011; Mestry *et al.*, 2011).

The D_[4,3] presented significant differences (p<0.05) with respect to the MD and IAT variables, the linear interactions (HP-MD, HP-IAT, HP-OAT, HP-RAS, MD-IAT, MD-OAT, IAT-OAT, IAT-RAS, and OAT-RAS), and the quadratic interaction (IAT-IAT). The response volume graph (Figure 5) identified an important effect on the higher $D_{[4,3]}$ (red zone) when the system operates at high IAT (155 - 145°C), RAS (throughout the range), low MD contents (8-10%), low OAT (80-85°C), and low HP (30-42 MPa). This situation demonstrates an important effect of the high process temperatures on the phase transitions that occur in the formation of pineapple powder. Therefore, it is assumed that microstructural complexes in a rubbery state could be forming through hydrogen bonds, due to the affinity between MD and carbohydrates (glucose, fructose, among others) and soluble and insoluble fibers present in pineapple, (Pereira, 2019; Roos & Drusch, 2016). Furthermore, this contributes to a greater ring formation inside the SD chamber and a lower process performance (Tontul & Topuz, 2017). The effect of high IAT on particle size has been reported in other investigations: jamun fruit (Santhalakshmy et al., 2015), coconut (Lucas et al., 2018), guava (Shishir et al., 2014), and orange (Fazaeli et al., 2012), among others.

3.6 Angle of repose

Angle of repose is an indirect measure of the fluidity of powdered products and is a critical quality parameter in the industrial processing and storage sector (Chaul *et al.*, 2017). The mean values of angle of repose for the pineapple powder fluctuated between $23.5\pm0.5 - 40.0\pm2.0^{\circ}$, which reflects an important variability in its flow characteristics



Figure 6. Pineapple powder angle of repose response surface.

and cohesiveness. The USP 30-NF 25 regulation (2007) establishes flowability values of $<30^{\circ}$ (excellent flowability), $31^{\circ}-35^{\circ}$ (good flowability), and >36° (between fair and poor flowability). The ANOVA presented significant differences (p<0.05) with respect to the RAS-RAS interaction, where the response surface graph denotes a curvilinear shape, identifying better fluidity with values between 20000 and 20500 rpm. However, the response surface graph (Figure 6) shows that the behavior of angle of repose is enhanced by the presence of MD, identifying two favorable zones: (1) RAS (22000 rpm) and MD (12%); and (2) RAS (18000 rpm) and MD (8%). Phenomenologically, these situations are not so clear because microcapsules showed an unexpected behavior, presenting smaller particle sizes (D_[4,3]) at lower RAS. This behavior assumes that fluidity could be associated with the surface area/MD ratio that modifies both the lubrication characteristics between the particles and the particle-particle interactions. On the other hand, microstructural complexes could be forming at these process temperatures, given the affinity through hydrogen bonds between MD and carbohydrates present in pineapple. This behavior was reported in other food powders: cascara sagrada extract (Gallo *et al.*, 2011) and fermented mixed juice of carrot and watermelon (Mestry *et al.*, 2011). Other works reported a positive effect on fluidity at smaller particle sizes (more RAS) for rosemary (Chaul *et al.*, 2017) and sumac extract (Caliskan & Dirim, 2016).

3.7 Total polyphenols and flavonoids

The mean values of total polyphenols fluctuated between $161.3 \pm 6.7 - 289.7 \pm 3.2 \text{ mg GAE}/100 g_{db}$, presenting significant differences (p<0.05) with respect to the linear interactions HP-IAT, MD-RAS, and IAT-RAS. On the other hand, the mean total flavonoid values fluctuated between $117.3 \pm 0.2 - 210.6 \pm 3.8 \text{ mg QE}/100 g_{db}$, presenting significant differences (p<0.05) with respect to the MD and the linear interactions HP-IAT and MD-RAS. The response volume graph (Figure 7) shows an increase in total polyphenols and total flavonoids with increasing HP and decreasing IAT and MD. On the one hand, this behavior could be related to the effect that high HP exerts on pineapple tissues and cells, and this allows for greater breakdown of its structure and, thus, greater extraction and solubilization of polyphenolic molecules, including the total flavonoids. Additionally, the higher HP results in greater inactivation of the polyphenoloxidase enzyme, which contributes to the preservation of the total polyphenols and total flavonoids in the feed suspension (Cardona et al., 2022). On the other hand, a positive effect is observed at low process temperatures and an apparently negative effect of MD in the protection of the structure; however, the addition of MD modifies the solids pineapple/solids MD ratio. Thus, the higher MD (more solid_{*MD*}) results in both lesser contribution of solid pineapple and decreasing concentration of active compounds. Some authors reported a similar effect of MD in other matrices, such as: Satureja montana (Vidović et al., 2014), Amla (Mishra et al., 2014), and Murraya koenigii (Sablania & Bosco, 2018).



Figure 7. Pineapple powder total phenols and flavonoids response volume.



Figure 8. Pineapple powder DPPH and ABTS response volume and surface.

3.8 Antioxidant activity

The mean values of DPPH fluctuated between 242.2±2.2 - 361.8 ± 0.9 mg TE/100g_{db}. The ANOVA showed significant differences (p<0.05) in the DPPH with respect to the MD and OAT variables, the linear interactions HP -IAT and IAT-RAS, and the quadratic effect RAS-RAS. It is observed in the response surface graph (Figure 8) that the DPPH presents better levels when OAT and MD were lower, which is enhanced when the system operates with high IAT (130 - 160 °C), low HP (30 - 38 MPa), and low RAS (18000 - 20000 rpm). This behavior was not expected because the highest heat flux absorbed by the pineapple powder occurs when the driving force (IAT - OAT) is greater, which was not consistent with a lower inhibition of the radical DPPH (Mishra et al., 2014). This leads us to consider that there are other factors from the raw material (variability in the maturity of the pulp and peel, among others) and from the process control (residence time) that could be affecting the result. The effect of MD with respect to antioxidant capacity was similar to that observed in total and total flavonoids explained previously.

On the other hand, the mean ABTS values fluctuated between $108.6\pm3.2 - 252.4\pm6.3$ mg TE/ $100g_{db}$ without presenting significant differences (p<0.05) with respect to the independent variables. The response volume graph did not present a defined behavior with respect to the HP, IAT, and MD variables, which indicates that its value depends on the individual conditions imposed on the process. The DPPH and ABTS values were similar to those previously reported for pineapple peel extract microcapsules (Lourenço, Fraqueza, *et al.*, 2020).

3.9 Vitamin C

The mean vitamin C values in the pineapple powder fluctuated between $5.9 \pm 0.2 - 39.1 \pm 0.8$ mg AA / 100g bs, which reflected significant effects (p <0.05) for HP and OAT, as well as the HP-HP interaction. The response volume (Figure 9) illustrates the most favorable conditions for vitamin C: lower IAT (125-145°C), lower OAT (80-85°C), and higher HP (46-50 MPa). In general, fresh pineapple



Figure 9. Pineapple powder Vitamin C response volume.

is a fruit with a significant content of vitamin C (134 mg AA/100bs) (Azizan et al., 2020); However, its content is affected by different factors during the processing of pineapple powder, such as exposure to high temperatures, the presence of humidity, oxygen, and light. These effects are probably due to the triggering of biochemical reactions that involve the oxidation of AA to the dehydroascorbic form (DHAA), the subsequent hydrolysis of DHAA to 2,3diketogulonic acid, and the generation by polymerization of nutritionally inactive products (Estevinho et al., 2016). Other studies have reported a similar behavior of vitamin C with respect to the process temperatures on powder fruit obtained by SD in: seabuckthorn fruit (Selvamuthukumaran & Khanum, 2014), orange juice (Islam et al., 2016), acerola and seriguela juice mix (Carneiro Maranhão Ribeiro et al., 2019) and mango (Mendoza-Corvis et al., 2016). On the other hand, the positive effect at higher HP could be associated with greater vitamin C leaching from inside the structure. This is due to the greater shear imposed on the homogenization process, which reached a maximum temperature of 40°C.

3.10 Experimental modeling and optimization

It is observed that the R^2 values (Table 2) fluctuated between 82.5 and 97.5%, which indicates that the

Table 3. Regression coefficients											
Coefficient	\mathbf{a}_{W}	S	Н	We	AR	D _[4,3]	ТР	TF	DPPH	ABTS	VitC
B ₀	-10.04	635.48	-8.01	62.5	293.45	728.81	7260.34	-6859.2	-8495.6	-8481.7	3031.89
HP	-0.05	-2.22	-6.71	-0.56	-9.84	-39.32	105.65	119.33	19.76	-14.24	1.06
MD	-0.16	43.79	-13.15	0.97	18	-42.07	-310.11	-758.81	-48.89	88.81	-22.06
IAT	0.09	-6.21	-0.25	-0.55	-0.94	20.07	11.68	70.24	99.28	133.92	5.07
OAT	0.12	0.29	0.86	-0.65	-15.24	4.27	-74	176.67	37.67	-80.9	-82.76
RAS	0	-0.03	0.02	0	0.06	-0.14	-0.49	-0.38	0.05	0.29	0.03
HP-HP	0	-0.02	-0.01	0	0.04	-0.02	0.05	0.13	-0.07	0.38	0.15
HP-MD	0	-0.14	0.12	0	0.1	0.46	-1.03	0.58	0.86	0.69	-0.38
HP-IAT	0	0.01	0.01	0	0.01	-0.07	-0.31	-0.42	-0.22	-0.34	-0.05
HP-OAT	0	0.01	0.01	0	0.06	0.39	-0.71	-0.79	0.05	-0.05	0.1
HP-RAS	0,00	0	0	0	0	0	0	0	0	0	0
MD-MD	0	0.32	0.33	0	0.07	0.42	1.59	0.45	-1.71	-0.77	-1.35
MD-IAT	0	-0.04	0.01	0	0.01	-0.7	0.19	0.4	-0.53	-2.01	0.04
MD-OAT	0	-0.37	-0.09	0	-0.08	0.88	0.43	4.18	1.03	-0.28	0.91
MD-RAS	0	0	0	0	0	0	0.01	0.01	0	0.01	0
IAT-IAT	0	0.01	0	0	-0.01	0.06	-0.07	-0.03	-0.08	-0.01	-0.03
IAT- OAT	0	0.02	-0.02	0	0.04	-0.21	-0.12	-0.48	-0.36	-0.79	0.11

S: solubility, H:hygroscopicity, We: Wettability, AR: angle repose, D[4,3]: particle size, TP: total polyphenols, TF: total flavonoids, VitC: vitamin C.

Table 4. Multi-variable optimization.

Response	Goal	Impact	Weight	Theoretical value	Experimental value	RME (%)
Water activity	0.203	0.5	4	0.203	0.215 ± 0.006	5.9
Solubility (%)	Maximize	0.8	4.6	78.5	81.9 ± 1.3	4.2
Hygroscopicity (%)	Minimize	0.8	4.5	8.3	12.6 ± 0.9	51.3
Wettability (s)	Minimize	0.8	4.5	372.7	312.4 ± 21.9	16.1
D[4.3] (µ m)	Minimize	1	5	24.7	22.6 ± 1.5	8.6
Angle of repose (°)	Minimize	1	4	35.6	32.2 ± 2.7	9.5
Total polyphenols (mg GAE/100g db)	Maximize	1.1	5	320.3	302.0 ± 13.8	5.7
Total flavonoids (mg QE/100g db)	Maximize	1.1	5	210.6	199.9 ± 11.3	5.1
DPPH (mg TE/100g db)	Maximize	1.1	5	330.1	308.5 ± 8.1	6.5
ABTS (mg TE/100g db)	Maximize	1.1	5	212.4	179.1 ± 10.3	15.7
Vitamin C (mg AA/100g db)	Maximize	1.2	5	42.6	33.2 ± 0.3	19.9

quadratic regression models used were adequate to express the relationship between the dependent and independent variables. Table 3 presents the regression coefficient and Table 4 presents a summary of the multiple response optimization process, based on the models provided by RSM, which considered the following criteria: minimize variables hygroscopicity, wettability, angle repose, and D_[4,3]; and maximize variables solubility, total polyphenols, total flavonoids, ABTS, DPPH, and vitamin C; These criteria were established according to the desired quality characteristics for the pineapple powder. Additionally, a_W was set at a medium value, since its values were low and its fluctuations were not very large. Each variable was given a weight and impact according to the ANOVA results. The optimization of multiple responses reached a desirability of 72.7% and defined the independent variables as follows: HP = 44.4 MPa, MD = 10.2%, IAT = 134.9°C, OAT = 80°C, and RAS = 18296 rpm. Finding that the optimal values of MD and IAT were established in the middle range, while OAT and RAS in the lower range. The validation of the response surface model to predict the independent variables was carried out through 3 experiments under the optimal conditions established by the model, where 91% of the dependent variables presented the RME <20%, representing an acceptable value for the model.

3.11 Microstructure

Morphology affects some key quality characteristics of the products obtained by SD, such as particle size

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Figure 10. SEM micrographs (100×, 300×, 800×, and 1000×) of pineapple powder.

distribution, fluidity, moisture content and bulk density. Figure 10 presents the micrographs obtained by SEM at 100×, 300×, 800× and 1000× respectively. On the one hand, pineapple powder presented individual particles as individual structures with defined almost spherical shapes, smooth surface, non-porous, visible and without bonding bridges between them (Figure 10-A, 10-B, 10-D); on the other hand, particles were observed within larger particles (Figure 10-C), however, the particle size distribution was homogeneous (monomodal distribution) and coherent with the particle sizes found (D_[4,3] between 33. 0±0.1 and 82.7±0.1 μ m), a typical characteristic of fruit or vegetable powders obtained by SD using MD as a drying additive.

Conclusions

The application of SD process for pineapple extract was an effective technology, which allowed obtaining pineapple powder with good quality attributes, conferring added value in the pineapple agro-chain. The obtained results demonstrated greater use of the pineapple structure (pulp, core, and extract of the peel) and important contents of these components of biological interest (vitamin C, polyphenols, flavonoids), highlighting good capacity antioxidant.

The RSM allowed for analysis and modelling the behavior of the dependent variables as a function of the independent variables. It was concluded that the dependent variables evaluated are affected by the formulation of the feed suspension and by the SD process. The greater influence on pineapple powder was notable based of the effects of MD, IAT, OAT, and HP, which presented the following: good solubility, wettability, fluency, and D_{4.3}. In addition, the products presented important antioxidant capacity, supported by the high contents of total polyphenols, total flavonoid, and vitamin C. The experimental optimization, defined the processing conditions, thus: HP: 44.4 MPa, MD: 10.2%, inlet air temperature: 134.9 °C, out air temperature: 80 °C, and rotary atomizer speed: 18296 rpm and with quality attributes: a_W : 0.215±0.006, solubility: 81.89±1.3%, wettability: 312.41±21.9 s, repose angle: 32.18±2.7°, D_[4,3]: 22.6±1.5 µm, total polyphenols: 301.98±13.8 mg GAE/100g db, total flavonois: 199.85±11.3 mg QE/100g db, ABTS: 179.14±10.3 mg TE/100g db, DPPH: 308.49±8.1 mg TE/100g db, and vitamin C: 33.2±0.3 mg AA/100g db. making it, in general, a pineapple powder with characteristics appropriate for the market of fruit powders.

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