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Research Article

Effect of the Spray Drying Process on the Quality of Coconut Powder Fortified with Calcium and Vitamins C, D3 and E

[1, 2]Lucas Aguirre Juan Carlos, [2]Giraldo Giraldo German Antonio and [1]Cortés Rodríguez Misael
[1]Universidad Nacional de Colombia, -Sede Medellín. Calle 59 A N 63-20, Medellín,
[2]Universidad del Quindío, Carrera 15 Calle 12 Norte, Armenia-Colombia

Abstract: The objective of this study was to optimize the process of Spray Drying (SD) for the obtaining of coconut powder fortified with Physiologically Active Compounds (PAC), according to the dryer's operating characteristics and the product, being (SD) is one of the most used technologies in the powder industry, guaranteeing good quality attributes for various applications in the food sector; it was used a response surface design based on five independent variables:: Maltodextrin (MD), Inlet Air Temperature (IAT), Outlet Air Temperature (OAT), Atomizing Disk Velocity (ADV) and drying Chamber Vacuum Pressure (VPC) and the dependent variables: yield (*R), Deposit Formation (DF) in the drying chamber, humidity (X_w), water activity (a_w), Hygroscopicity (H), Solubility (S), wettability (Hu), color (L*, a* y b*), recovery of PAC (Ca, vitamins C, D_3 and E), Peroxide Index (PI) and particle size (D_{10}, D_{50} y D_{90}). The results were analyzed statistically from the Statgraphics XVI.I software and through analysis of variance with 5% level of significance. In general, response variables were affected by all independent variables. The experimental optimization defined the CP+PAC process conditions as follows: IAT: 170°C; OAT: 85.8°C; ADV: 26676 rpm; VPC: 1.6” H_2O; MD: 7.0%; and with quality attributes: X_w: 1.7±0.4%; a_w: 0.171±0.018; H: 8.4±0.5%; S: 58.4±2.1%; Hu: 263.0±19.8s; L*: 79.5±0.9; a*: 1.5±0.1; b*: 9.5±0.4; PI: 2.4±1.3 meq H_2O_2/kg oil; DFC: 32.4±2.3%; *R: 44.0%; D_{10}: 1.70±0.05 µm; D_{50}: 8.46±2.09 µm; D_{90}: 78.18±24.30 µm; Ca: 41.7±2.3%; Vit.C: 32.4±6.2%; Vit.D_3: 7.8±1.8%; Vit.E: 6.1±1.9%; making it a hygroscopic product, potentially sensitive to oxidative processes, which can cause changes in color, strange flavors or odors.

Keywords: Coconut, dehydration, encapsulation, vitamins, yield

INTRODUCTION

Spray Drying (SD) is the most widely used industrial process to obtain powdered products from fruits and vegetables, associated to short process times that contribute to minimum thermal deterioration, given that the formation of small drops produces high specific surface and high mass transfer. The rate and short times of the drying process permit its application even in thermosensitive products, which has generated its massive use in the development and microencapsulation of their own and/or added bioactive compounds (Mishra *et al*., 2014). In spite of these characteristics, the selection of operating parameters is paramount to achieve high nutritional quality and the best physical and physicochemical characteristics of the powders (Phisut, 2012).

In this context, many tropical fruits, fruits rich in bioactive compounds (vitamins, antioxidants, pigments, among others), extracts or other food products considered thermosensitive have been assessed under the SD technique by varying processing conditions and the wall materials or drying aides: golden berry (Cortés Rodríguez *et al*., 2017); avocado (Marulanda, 2015); guacamole (Estrada-Mesa, 2015); cane (Avila *et al*., 2014); cane + probiotic microorganism (Salazar Alzate *et al*., 2015); Amalaki or Amla (*Emblica officinalis*) (Mishra *et al*., 2014), blueberry (*Vaccinium ashei*) (Jiménez-Aguilar *et al*., 2011), aronia (*Aronia melanocarpa*) (Horszwald *et al*., 2013), açai (*Euterpe oleraceae* Mart.) (Tonon *et al*., 2010), cactus pear or nopal (*Opuntia ficus-indica*) (Saénz *et al*., 2009; Medina-Torres *et al*., 2013), casis or black currant (*Ribes nigrum* L.) (Bakowska-Barczak and Kolodziejczyk, 2011), *Garcinia cowa* (Parthasarathi *et al*., 2013), pomegranate (*Punica granatum* L.) (Robert *et al*., 2010; Goula and Adamopoulos, 2012), guava (*Psidium guajava* L.) (Osorio *et al*., 2011), mango (*Mangifera indica* L.) (Cano-Chauca *et al*., 2005; Caparino *et al*., 2012), passion fruit (*Passiflora* sp.,) (Borrmann *et al*., 2013), cashew (*Anacardium occidentale* L.) (Da Silva Bastos *et al*., 2012),

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Corresponding Author: Lucas Aguirre Juan Carlos, Universidad Nacional de Colombia, - Sede Medellín. Calle 59 A N 63-20, Medellín, Colombia

cantaloupe (*Cucumis melo*) (Solval *et al*., 2012), blackberry (*Morus nigra*) (Fazaeli *et al*., 2012b), noni or great morinda (*Morinda citrifolia* L.) (Krishnaiah *et al*., 2011), breadfruit or durian (*Durio zibethinus*) (Chin *et al*., 2010), gac (*Momordica cochinchinensis*) (Kha *et al*., 2010), lemon (Roustapour *et al*., 2006), myrtle (*Myrica* sp.,), (Fang and Bhandari, 2011), orange (Chegini and Ghobadian, 2005; Goula and Adamopoulos, 2010), tomato (Goula and Adamopoulos, 2005a; Goula and Adamopoulos, 2005b; Goula and Adamopoulos, 2008a; Goula and Adamopoulos, 2008b), pineapple (*Ananas comosus*) (Abadio *et al*., 2004), coffee oil (Frascareli *et al*., 2012), jaboticaba (*Myrciaria jaboticaba* (Silva *et al*., 2013), camu camu (*Myrciaria dubia*) (Fracassetti *et al*., 2013), acerola cherry (Moreira *et al*., 2009), among others.

Coconut is a no-climacteric tropical fruit with high nutritional value. It is a perennial plant that produces fruit continually for 60-70 years, classifying in two maturity stages: tender coconut and mature coconut. Coconut Water (CW) and Coconut Pulp (CP) are the edible portions of the fruit. Coconut water is considered a refreshing beverage low in calories, fat free and rehydrating, which contains sugars, vitamins, minerals (potassium, sodium, calcium, magnesium, iron, phosphorus, zinc, manganese, copper, sulfur, aluminum, boron, selenium and chlorine), growth promoting factors, proteins and amino acids. The CP, which is the biggest edible part of the fruit contains amino acids, minerals, antioxidants, like phenols and tocopherols and it is used mainly to prepare a liquid suspension known as Coconut Milk (CM), to extract the oil and in diverse products from the food and cosmetic industries (DebMandal and Mandal, 2011; Appaiah *et al*., 2015).

Coconut oil may be used for food and industrial applications, it contains between 50 and 60% fat, it is rich in medium chain fatty acids (59.7%) of which 92.7% are saturated fatty acids, 6.1% monounsaturated fatty acids that burn easily to produce energy instead of storing in the body and 1.2% polyunsaturated fatty acids. Lauric acid is the principal fatty acid in coconut oil (49.1%) (DebMandal and Mandal, 2011; Appaiah *et al*., 2015).

Recently, modern medical research has confirmed many health benefits in multiple coconut products (DebMandal and Mandal, 2011); which is why, within this context, it becomes necessary to use this fruit in the technologically most effective manner, contributing to the generation of the value of its agro-chain and giving it a strong boost in the diversification of new products that fit within the range of functional foods.

Calcium and vitamins C, D_3 and E are Physiologically Active Compounds (PAC) that have shown potential health benefits (Indyk *et al*., 1996; Yu *et al*., 2000; Parthasarathi and Anandharamakrishnan, 2016; Nesterenko *et al*., 2014); however, some of these components suffer oxidative processes during their processing and storage that degrade them rapidly (Anandharamakrishnan and Ishwarya, 2015; Yoo *et al*., 2006). Likewise, some limitations are reported in the absorption of these PAC in the gastrointestinal tract and their poor total bioavailability (Abuasal *et al*., 2012), which is why much interest exists in developing new functional foods that include them (Parthasarathi and Anandharamakrishnan, 2016).

The aim of this study was to assess the influence of the conditions of the spray drying process on the quality attributes of coconut powder fortified with PAC, like calcium and vitamins C and D_3 and E, which have been identified as deficient nutrients in the Colombian population, associated to diseases, like osteoporosis, anemia, blindness and rickets, among others.

MATERIALS AND METHODS

Coconuts (*Coconuts nucifera* L.) Enano Malayo (manila) or Alto Pacífico (typical) varieties from the Colombian Pacific region were used. Their age of flowering to harvest was approximately 12 months and the post-harvest time was between 15 and 36 days, time during which through preliminary studies it was determined that they have the acceptable quality to be used as raw matter for their processing. The whole coconuts used were initially washed with water and disinfected with a sodium hypochlorite solution (200 ppm), then the CW was removed and were scalded during 20 min in boiling water at $T \approx 96°C$ (local barometric pressure ≈ 640 mmHg); thereafter, the shell was removed from the CP. The CP selected was again washed with water and disinfected with hypochlorite, cut into pieces and ground (TM32 INOX BRAHER 3HP-16801002 mill).

Characterization of the CP+PAC properties was performed according to the following methodologies: Humidity percentage (X_w): Official method AOAC (1990) 930.15/90; water activity (a_w): determined with a spray point hygrometer at 25°C (Aqualab series 3TE, Decagon, Devices, Pullman, WA, USD) (Cortés-Rodríguez *et al*., 2007); Solubility (S): method used by Cano-Chauca *et al*. (2005) modified, described by Estrada-Mesa (2015); Hygroscopicity (H): gravimetric method to construct sorption isotherms (Martínez-Navarrete *et al*., 1998) using a saturated KI solution at 25°C ($a_w = 0.689$), expressed as humidity percentage (b.s); wettability (Hu): determined as the time needed for 1 g of powder to disappear from the surface of a 100-mL volume of water at 20°C (Fuchs *et al*., 2006). Peroxide Index (PI): conducted on the oil extracted, obtained according to the method by Bae and Lee (2008) modified, which took 4 g of powder. The PI was determined through the spectrophotometry based on the capacity of the peroxides to oxidize ferrous ions into ferric ions, which react with diverse reagents that produce colored complexes (Hornero-Méndez *et al*.,

2001) Eq. (1), where A_m and A_b correspond to the absorbance of the sample and the target, respectively, at a 500-mm wavelength; m is calibration curve slope, W = weight of the sample (g), 2 is the conversion factor to express as meqH_2O_2, 55.84 = molecular weight of iron and V_t = final volume (mL) of the reaction mixture:

$$\frac{\mathrm{meqH_2O_2}}{\mathrm{kg\ sample}} = \frac{(A_m - A_b)*1/m}{W*2*55{,}84}*V_t \quad (1)$$

Quantification of vitamins E and D_3 was carried out via High-Resolution Liquid Chromatography (HRLC) (Shimatzu Prominence 20A), using a reverse phase column (C18-5 µm 4.6×250 mm), diode array, mobile phase: Acetonitrile/methanol/water (45.3/51.2/3.5), flow: 1 mL/min, furnace temperature 40°C and wavelengths of 325 and 265 nm, respectively. Vitamin C quantification was also conducted through HRLC, using a reverse phase column (C18 RP-5 µm 4.6×250 mm), diode array, mobile phase: KH_2PO_4 0.02 M pH = 3.00 (ortho-phosphoric acid 85%), flow: 1 mL/min, furnace temperature of 35°C, 244-nm wavelength and an injection volume of 5 µL. Extraction of vitamin C was done according to the methodology by Gutiérrez *et al*. (2007), adapted by Peña Correa *et al*. (2013); while extraction of vitamins D_3 and E was done according to the methodology proposed by Cortés-Rodríguez (2004) modified through inclusion treatment through ultrasound during 20 min to the samples treated with hexane. Calcium quantification was conducted through flame atomic absorption spectrophotometry, according to NTC 4807 (2000), supported on NTC 5151 (2003). The CP+PAC fortification criteria were set within the framework of the declaration of nutritional properties established in Resolution 333 of 2011 (Colombian Ministry of Social Protection), to declare the descriptor "Rich in" or "Excellent source of" PAC in a 100-g serving.

Particle sizes were determined as percentiles D_{10}, D_{50} and D_{90}, using the Mastersizer 3000 (Malvern Instrument Ltd., Worcestershire, UK), prior dispersion of the samples in 500 mL of distilled water until obtaining a darkening value of 10±1%, considering the size distribution from Mie's theory and using the refraction index of 1.52 (Mirhosseini *et al*., 2008). Color was determined through the CIE-La*b* coordinates, using an X-Rite spectrophotometer model SP62, D_{65} illuminant, 10° observer as a reference (Cortés-Rodríguez *et al*., 2007).

Additionally, micrographs were made of the CP+PAC using scanning electron microscopy (Jeol 5910LV) at 15 Kv (Cano-Chauca *et al*., 2005), where the samples were deposited on a copper conductive tape and on a sample holder, then coated with gold in a vacuum evaporator (Dentom Vacuum, 30 mA, 5 kV, 100 millitorr).

Preparation of the feed emulsion to the drier: Sample lots were prepared of 3000 g of Feed Emulsion (FE) to the drier. Initially, a mixture of CP, CW and drinking water at a ratio of (CW+H_2O) /CP = 2.0 was homogenized in a blender (Osterizer 600 Watts) in position III during 5 min; then, the mixture was filtered in a 500-µm mesh screen, separating the fiber from the CM. The fiber was subjected to a drying process at 40°C for 48 h and then to dry milling (IKA MF 10.1 mill, USD). The CM was again homogenized in a homogenizer (Silverson series L5) using the emulsifying head at 10,000 rpm during 10 min, adding the native milled fiber (5% p/p) and the rest of the ingredients: dairy serum (instant WCP 80) as tensoactive agent (0.5%), NaCl (9 mmol/L), xanthan gum (0.5% p/p), Tert-Butylhydroquinone (TBHQ) (200 mg/kg) and the PAC in the chemical forms of powdered calcium citrate (6.0 g) (BELL CHEM), vitamin C (1.0 g) (ascorbic acid): 99.5% in powder, BELL CHEM), vitamin D_3 (0.5 g) (cholecalciferol): 512900 UI/g in powder, BELL CHEM), vitamin E (1.0 g) (DL-α-tocopherol acetate): 50% USP GRADE in powder, BELL CHEM). A cooling bath was used during the preparation to keep the FE temperature under 35°C.

Spray drying process: A spray drier flow pilot was used in co-current (Vibrasec, model PASLAB 1.5). The evaluation of the spray drying process was conducted through the response surface methodology with a composite central design (Table 1), considering the independent variables: Maltodextrin (MD) (5-15%), Inlet Air Temperature (IAT) (150-170°C), Outlet Air Temperature (OAT) (80-90°C), Atomizing Disc Velocity (ADV) (24000-28000 rpm) and Vacuum Pressure in the Chamber (DCVP) (1.0-1.88"H_2O) and the dependent variables: *R, DF, X_w, a_w, S, H, Hu, PI,

Table 1: Experimental design of the SD process

Run	IAT (°C)	OAT (°C)	ADV (rpm)	VPC ("H_2O)	MD (%)
1	160	85	26000	1.88	10
2	170	80	28000	1.00	15
3	150	80	28000	1.88	15
4	170	90	24000	1.88	5
5	170	85	26000	1.44	10
6	150	80	24000	1.00	5
7	160	85	26000	1.44	15
8	170	90	24000	1.00	15
9	160	85	26000	1.44	10
10	170	80	28000	1.88	5
11	170	90	28000	1.00	5
12	160	85	26000	1.00	10
13	160	85	26000	1.44	5
14	160	85	26000	1.44	10
15	160	80	26000	1.44	10
16	160	85	28000	1.44	10
17	160	85	24000	1.44	10
18	160	85	26000	1.44	10
19	160	85	26000	1.44	10
20	160	85	26000	1.44	10
21	150	90	28000	1.88	5
22	150	90	28000	1.00	15
23	170	80	24000	1.88	15
24	150	85	26000	1.44	10
25	150	90	24000	1.88	15
26	160	90	26000	1.44	10

Table 2: Experimental optimization results of the coconut powder SD process

Run	X_w	a_w	S (%)	H (%)	L*	Vit.C (%)	Vit.D_3 (%)	Vit.E (%)
1	1.82±0.02	0.27±0.00	39.72±2.39	8.01±0.12	76.00±1.07	40.20±2.06	18.81±8.67	11.18±4.36
2	2.69±0.05	0.28±0.00	69.45±5.51	9.48±0.17	80.33±0.60	40.03±3.08	8.25±0.98	2.57±0.12
3	1.59±0.02	0.16±0.00	74.68±5.63	9.95±0.59	78.94±0.56	39.80±1.71	5.53±0.62	2.56±0.27
4	1.38±0.09	0.26±0.01	58.72±2.18	7.91±0.11	74.63±0.93	31.13±3.56	14.70±1.14	9.35±0.81
5	1.22±0.06	0.19±0.00	59.79±2.72	8.00±0.08	73.96±1.32	56.80±0.97	32.84±5.58	7.97±1.18
6	2.38±0.08	0.21±0.01	48.69±1.63	7.95±0.15	73.96±1.32	30.54±2.44	15.01±0.87	12.50±0.79
7	1.20±0.01	0.15±0.01	63.03±4.88	9.09±0.11	80.87±0.63	33.34±7.87	17.07±0.85	1.87±0.48
8	1.67±0.01	0.18±0.01	67.18±4.42	9.73±0.07	82.20±0.67	38.55±1.57	20.18±6.52	5.12±1.73
9	2.02±0.02	0.23±0.01	47.06±2.41	7.84±0.64	77.03±1.35	37.29±9.92	17.81±0.94	3.55±0.29
10	1.50±0.06	0.24±0.00	51.88±3.07	7.74±0.18	76.29±1.00	34.28±4.48	15.85±2.64	7.94±1.51
11	2.59±0.23	0.25±0.01	58.02±5.22	9.66±0.20	76.10±0.62	32.92±6.85	15.18±0.62	6.74±0.48
12	2.10±0.09	0.21±0.00	54.77±0.85	8.67±0.04	76.73±0.29	29.92±6.08	7.52±0.89	2.88±0.19
13	1.83±0.02	0.25±0.01	45.22±3.79	6.44±0.10	68.44±0.25	28.63±2.48	5.95±0.29	5.26±0.13
14	1.44±0.08	0.18±0.01	49.19±1.08	8.57±0.08	77.50±0.68	43.13±2.98	20.01±0.52	3.31±0.06
15	2.36±0.01	0.28±0.00	59.38±4.44	9.56±0.10	77.13±1.10	44.07±3.65	27.78±0.76	6.65±0.10
16	2.74±0.07	0.22±0.00	59.03±2.99	7.73±0.05	76.94±0.67	22.92±6.78	7.49±2.71	3.26±0.68
17	1.30±0.22	0.24±0.01	79.86±5.68	7.31±0.09	78.61±0.54	39.17±1.69	15.19±2.99	8.31±0.99
18	1.40±0.09	0.24±0.01	42.55±0.66	7.60±0.21	77.24±1.01	49.14±1.20	16.22±4.47	5.42±1.01
19	1.59±0.03	0.23±0.00	38.93±3.32	7.99±0.11	78.52±0.39	40.38±15.43	22.26±3.87	3.27±0.41
20	0.83±0.09	0.18±0.01	51.70±4.53	7.35±0.17	77.71±0.75	46.03±7.80	17.94±0.69	2.86±0.09
21	0.91±0.06	0.19±0.01	48.87±0.32	7.47±0.25	76.46±0.50	38.05±2.49	19.00±1.74	9.11±0.70
22	1.45±0.12	0.15±0.01	72.33±3.98	9.83±0.09	82.25±0.64	35.89±9.86	10.77±0.35	8.01±1.13
23	0.99±0.06	0.11±0.00	56.66±4.44	10.02±0.05	80.89±0.36	32.19±5.43	9.03±2.71	3.34±0.73
24	1.95±0.06	0.23±0.00	62.43±5.26	8.66±0.11	78.93±1.34	40.73±6.44	24.33±0.42	6.93±0.03
25	1.74±0.03	0.18±0.00	67.67±2.38	10.32±0.99	80.49±0.22	25.96±6.83	5.80±0.75	2.70±0.24
26	0.69±0.09	0.19±0.00	49.76±2.92	7.47±0.13	78.64±1.32	40.31±3.40	18.20±3.90	8.61±1.75

Run	Ca (%)	Hu (s)	ÍP (meqH_2O_2/kg)	FD (%)	*R (%)	D_{50} (µm)	D_{90} (µm)
1	51.81	243.33±23.35	4.69±0.17	42.18	45.86	8.40±0.13	46.33±2.96
2	52.01	296.67±12.58	3.36±0.56	42.91	38.54	12.87±1.05	244.60±161.00
3	48.41	305.33±11.24	2.50±0.48	42.27	38.34	17.95±6.48	498.70±174.10
4	56.54	139.67±13.05	5.93±0.09	30.85	45.50	19.49±3.38	396.21±80.43
5	61.61	157.67±11.55	1.53±0.39	36.48	52.63	58.61±17.70	1153.20±208.28
6	38.90	124.00±2.65	4.60±0.79	43.92	35.89	48.68±23.67	678.77±64.95
7	43.39	281.67±17.56	3.03±0.02	44.20	45.51	39.64±11.48	809.24±143.55
8	55.41	224.67±36.30	3.61±0.73	43.18	46.98	12.97±1.63	487.46±72.76
9	57.17	236.33±8.50	3.48±0.17	42.61	43.19	17.77±3.67	499.39±142.93
10	63.26	180.67±18.93	3.32±0.35	27.54	51.99	20.99±15.33	178.40±62.48
11	49.80	179.00±35.68	4.38±0.50	32.12	44.82	45.86±14.19	566.04±121.84
12	44.74	184.00±27.87	3.65±0.39	43.23	38.99	11.34±3.85	49.23±21.83
13	36.63	159.33±27.68	3.64±0.53	44.70	31.34	9.77±0.60	77.37±6.25
14	54.45	312.67±4.62	2.57±0.27	43.17	45.02	8.10±0.05	156.06±26.79
15	54.89	165.67±18.23	2.63±0.47	47.36	44.32	10.83±1.90	94.63±38.49
16	43.07	127.00±21.79	3.04±0.39	45.89	34.54	9.44±0.24	43.27±4.62
17	48.24	224.00±7.94	4.43±0.26	43.98	46.64	15.17±3.72	617.81±104.21
18	56.76	145.00±30.51	3.27±0.22	42.74	47.91	10.14±0.16	206.34±80.30
19	54.94	211.67±5.69	3.08±0.35	43.33	47.06	7.91±0.56	643.70±70.95
20	50.05	304.00±21.17	3.31±0.24	43.79	43.77	5.54±0.36	82.51±20.24
21	55.30	111.00±12.53	2.57±0.59	27.86	47.33	19.97±2.11	188.04±77.56
22	46.04	179.00±35.68	2.72±0.35	40.06	44.79	14.62±6.21	575.75±117.39
23	57.83	291.67±4.04	2.70±0.22	42.90	50.97	36.07±5.73	462.16±24.69
24	57.54	148.67±9.87	3.67±0.54	31.41	49.11	12.92±5.14	66.75±4.24
25	46.79	293.33±19.86	2.79±0.19	42.56	38.15	10.20±5.83	630.89±128.53
26	56.87	191.67±9.45	3.70±0.27	44.78	43.09	12.64±3.84	692.74±77.91

retention of PAC (%), D_{10}, D_{50}, D_{90}, L*, a* y b*). All tests were run in triplicate for each experiment. Additionally, process yield (*R) was determined: kg solids from the powder obtained/kg solids from the FE (Tonon *et al*., 2008) and the Deposit Formation (DF) within the drying chamber: kg adhered material/kg FE.

The experimental design matrix, the analysis of the results and the optimization procedure were performed using Statgraphics Centurion XVI.I software, with Analysis of Variance (ANOVA) and a confidence level of 95%, from the optimum operating conditions.

RESULTS AND DISCUSSION

Table 2 presents the mean values and the standard deviations of the dependent variables of CP+PAC in function of the conditions of the SD process. Note that the FE had solid contents between 23.7 and 32.3%, where the viscosity values were lower than 1000 cP, adequate conditions to operate the pilot unit used. In addition, all the formulations considered had absolute values of ζ>25 mV, which denotes a negative electric potential in the proximities of the coion layer formed on

Table 3: ANOVA (p-values) for response surface models

	Principal effects					Quadratic effects				
Variables	A	B	C	D	E	A^2	B^2	C^2	D^2	E^2
X_w	0.0004**	0.0000**	0.0000**	0.1449	0.0017**	0.2731	0.0756	0.0002**	0.0021**	0.0563
a_w	0.0104*	0.0000**	0.1111	0.0000**	0.0000**	0.0419*	0.1194	0.8866	0.0123*	0.0000**
S	0.5845	0.0499*	0.0001**	0.0027**	0.0005**	0.0118*	0.6804	0.0000**	0.0004**	0.5402
H	0.0232*	0.0000**	0.1377	0.0216*	0.0000**	0.0006**	0.0000**	0.0086**	0.0005**	0.4084
L*	0.0000**	0.0669	0.0441*	0.3728	0.0000**	0.9163	0.0002**	0.0004**	0.9126	0.0000**
%R-Vit C	0.0030**	0.6094	0.0027**	0.0518	0.3668	0.0001**	0.1689	0.0013**	0.0959	0.0012**
%R-Vit D_3	0.0014*	0.0004**	0.0035**	0.0000**	0.0000**	0.0000**	0.0004**	0.0000**	0.0000**	0.0000**
%R-Vit E	0.4114	0.1238	0.0002**	0.0000**	0.0087**	0.0079**	0.0032**	0.8472	0.0487*	0.0001**
%R-Ca	0.4758	0.7245	0.3691	0.2314	0.2504	0.0062**	0.0515	0.1063	0.4626	0.0042**
Hu (s)	0.7989	0.4627	0.0078**	0.0970	0.0010**	0.0231*	0.4677	0.3561	0.1467	0.0623
PI	0.0000**	0.0035**	0.0002**	0.0049**	0.0871	0.0000**	0.2965	0.0140*	0.0000**	0.9779
DF (%)	0.0000**	0.0059**	0.0106*	0.0034**	0.0000**	0.0000**	0.0003**	0.0043**	0.0717	0.0201*
Yield (%)	0.3576	0.7370	0.0175*	0.1044	0.0095**	0.0051**	0.8585	0.1318	0.5558	0.0245*
D_{50}	0.0000**	0.7948	0.4116	0.6734	0.0001**	0.0000**	0.1397	0.1935	0.0406*	0.0105*
D_{90}	0.0000**	0.0000**	0.0000**	0.9805	0.0000**	0.0000**	0.2980	0.8945	0.0000**	0.0525

	Effects of the interaction									
Variables	AB	AC	AD	AE	BC	BD	BE	CD	CE	DE
X_w	0.0000**	0.0120*	0.1431	0.0029**	0.0179*	0.0053**	0.0000**	0.0000**	0.0000**	0.3513
a_w	0.0000**	0.0032**	0.0000**	0.0697	0.2783	0.0046**	0.0021**	0.0000**	0.0727	0.0000**
S	0.2446	0.7430	0.1018	0.0000**	0.1408	0.8528	0.0101*	0.0411*	0.9917	0.0181*
H	0.0000**	0.1381	0.9221	0.0000**	0.0088**	0.0002**	0.5999	0.0002**	0.0000**	0.0044**
L*	0.0001**	0.0000**	0.0000**	0.1224	0.0904	0.4112	0.0000**	0.0194*	0.0000**	0.0000**
%R-Vit C	0.1765	0.3132	0.0004**	0.0603	0.0003**	0.2947	0.8498	0.0266*	0.0012**	0.0493*
%R-Vit D_3	0.0000**	0.0208*	0.0006**	0.0002**	0.0000**	0.0381*	0.0875	0.1018	0.4201	0.0000**
%R-Vit E	0.3126	0.0001**	0.0018**	0.2412	0.0000**	0.0005**	0.0362*	0.7419	0.0000**	0.0001**
%R-Ca	0.9916	0.2194	0.6610	0.6616	0.5718	0.5174	0.1975	0.3341	0.6464	0.1100
Hu (s)	0.2528	0.0043**	0.2622	0.7884	0.6701	0.0248*	0.0508	0.2616	0.0770	0.5927
PI	0.0000**	0.0000**	0.0086**	0.0003**	0.0904	0.0000**	0.0001**	0.0032**	0.3655	0.0000**
DF (%)	0.0238*	0.0001**	0.0005**	0.0005**	0.0274	0.3042	0.0000**	0.0015**	0.0002**	0.0000**
Yield (%)	0.4669	0.0270*	0.8507	0.0338*	0.0071**	0.4961	0.0507	0.0494*	0.3693	0.0052**
D_{50}	0.9108	0.3992	0.7367	0.0001**	0.0000**	0.0003**	0.5298	0.0000**	0.4899	0.1481
D_{90}	0.0001**	0.0375*	0.1098	0.0000**	0.0000**	0.0232*	0.2932	0.0000**	0.0000**	0.0089**

the particle interphase and which contribute to increasing the repulsive forces among the particles (Estevinho *et al*., 2014; Mirhosseini *et al*., 2008; Rezvani *et al*., 2012). Table 3 presents the ANOVA results for the response surface models. In general, the ANOVA results showed that the response surface models were significant ($p<0.05$) with 95% CI for all the dependent variables.

Table 2 presents the mean values and the standard deviations of the dependent variables of CP+PAC in function of the conditions of the SD process. Note that the FE had solid contents between 23.7 and 32.3%, where the viscosity values were lower than 1000 cP, adequate conditions to operate the pilot unit used. In addition, all the formulations considered had absolute values of ζ>25 mV, which denotes a negative electric potential in the proximities of the coion layer formed on the particle interphase and which contribute to increasing the repulsive forces among the particles (Estevinho *et al*., 2014; Mirhosseini *et al*., 2008; Rezvani *et al*., 2012). Table 3 presents the ANOVA results for the response surface models. In general, the ANOVA results showed that the response surface models were significant ($p<0.05$) with 95% CI for all the dependent variables.

Humidity and water activity: The ANOVA showed that the X_w of the CP+PAC presented significant differences ($p<0.05$) with respect to variables IAT, OAT, ADV and MD; with the interactions IAT-OAT, IAT-MD, OAT-VPC, OAT-MD percentage, ADV-VPC, ADV-MD, IAT-ADV, OAT-ADV and with the quadratic interactions AVD^2 y VPC^2, which is evidenced in the curvature of the response surface graphics (Fig. 1). In spite of the statistically significant effects, the changes observed in X_w were relatively small, like the variability coefficients, fluctuating between 0.69±0.09% and 2.69±0.05%, which denotes that at the operating conditions assessed, the heat transfer rates from hot air to the particle generates good mass transfer until reaching values of X_w that could be associated to the monolayer's humidity values (Rodríguez-Bernal *et al*., 2015; Goula *et al*., 2008a, 2008b; Moreira *et al*., 2009; Ávila *et al*., 2014) and complemented with the a_w values that fluctuated between 0.11±0.00 and 0.28±0.00, which provides good microbiological stability or microbiologically safe products (Fennema, 2010; Tontul and Topuz, 2017). Similar values of a_w were obtained in powdered watermelon (Quek *et al*., 2007), acai (Tonon *et al*., 2010), Jamun (*Syzygium cumini*) (Santhalakshmy *et al*., 2015). The a_w had significant differences ($p<0.05$) with respect to variables IAT, OAT, MD and VPC; with the interactions IAT-OAT, IAT-ADV, IAT-MD, OAT-MD, OAT-VPC, ADV-MD, MD-VPC and the quadratic interactions IAT^2, MD^2 and VPC^2.

A trend is noted to diminished the X_w content of CP+PAC with increased IAT (greater heat transfer to the particle) and principally when the system operates at high ADV and OAT (Da Silva *et al*., 2013; Ávila *et al*., 2015; Santhalakshmy *et al*., 2015; Tontul and Topuz, 2017) and with increased MD content in the FE.

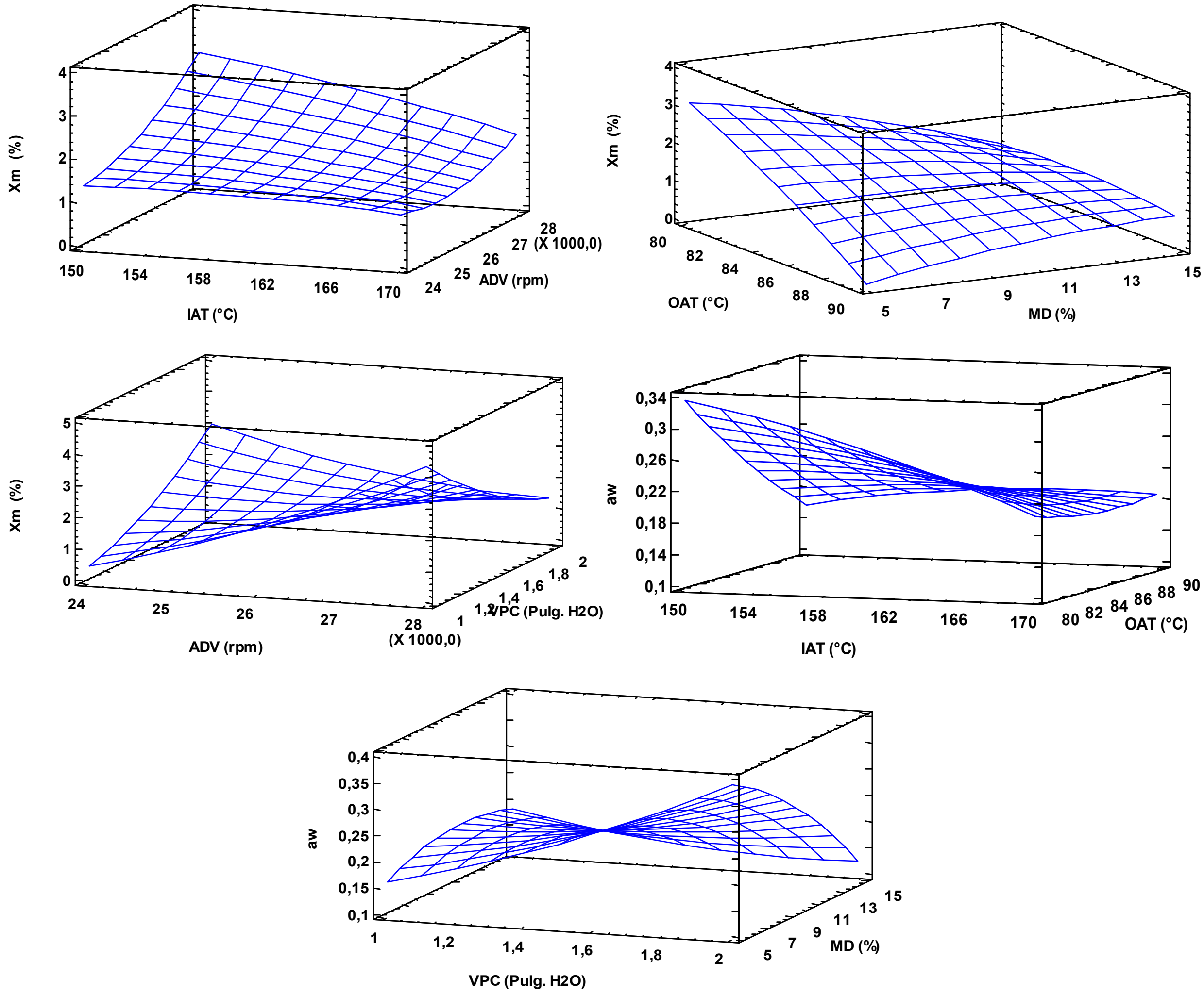


Fig. 1: Response surface graphics of X_w percentage and a_w in function of the independent variables

This situation is coherent, given that the smaller particle sizes obtained at high ADV have a greater surface area, which increases heat and mass transfer and shortens the trajectory of the water diffusion in the drops (Tontul and Topuz, 2017). All this favors water evaporation; in addition, increased OAT implies a decrease in feed flow and greater energy use of the drop formed, which finally diminishes its humidity. The OAT is an important process parameter related to powder quality and energy consumption of the drier; if it is superior to the vitreous transition temperature (T_g) of the powder obtained, it can be sticky and agglomerate (Santana *et al.*, 2017).

Low values of X_w limit the capacity of water to act as a plasticizer and also produces increased T_g, which could favor some properties of flow and dispersion of powders, like fluidity, stickiness, agglomeration and stability of the PAC and other quality attributes during storage (Roos, 2010; Da Silva *et al.*, 2013; Santhalakshmy *et al.*, 2015; Daza *et al.*, 2016; Khuenpet *et al.*, 2016; Santana *et al.*, 2017). Generally, the X_w of powdered foods (juices, fruit and vegetable extracts, among others) obtained through SD <5%, obtaining high useful life times (Henríquez *et al.*, 2013; Shishir *et al.*, 2017).

The increase of MD in FE increased the total content of solids, reducing the amount of free water to evaporate, which implies lower energy levels to reach low values of X_w in the CP+PAC; a similar behavior was reported for several products: tamarind (Bhusari *et al.*, 2014), *Morus nigra* (Fazaeli *et al.*, 2012b), *Rhamnus purshiana* (Gallo *et al.*, 2011), gac (Kha *et al.*, 2010), watermelon juice (Quek *et al.*, 2007) and pineapple juice (Abadio *et al.*, 2004). Further, MD exerts upon the CP+PAC matrix a depressant effect of a_w as a polar solute, a situation reported in numerous publications (Quek *et al.*, 2007; Bakar *et al.*, 2013; Oberoi and Sogi, 2015; Tontul and Topuz, 2017); however, in other products, like sapodilla powder (Chong and Wong, 2015) and gac powder (Kha *et al.*, 2010) no important a_w change is reported.

The a_w of CP+PAC had a tendency to increase with diminished IAT and principally at low OAT where the feed flow to the drier is lower (Tontul and Topuz, 2017). This increase of a_w is enhanced when the system operates at low IAT (150°C), which confers lower driving force to the heat transfer and with low ADV (24000 rpm) that increases the size of drops; in both circumstances, a_w tends to values of 0.320 and 0.280, respectively. In addition, low water diffusion from inside through the matrix is further hindered if a crust appears on the particle surface (Goula and Adamopoulos, 2010; Daza *et al*., 2016). Similar behaviors have been observed in pomegranate (Watson *et al*., 2017) and mountain tea (Nadeem *et al*., 2011).

The effect of the VPC on the a_w and X_w is not clearly defined. It depends on their interactions with the other operating variables, observing mainly in the a_w a strong interaction with MD, reaching its maximum value at high VPC and low MD contents, which is coherent because at these conditions it implies that the particles have a low time of residence that favors lower rates of heat and mass transfer and the consequent higher content of X_w and a_w. For X_w, interactions VPC-OAT and VPC-ADV are considered the most important, identifying their highest values when the system operates at low VPC and high ADV or low VPC and high OAT.

Solubility: S had significant linear effects ($p<0.05$) with respect to the independent variables OAT, ADV, VPC and MD; additionally, with the interactions OAT-MD, ADV-MD, VPC-MD and with the quadratic interactions IAT^2, ADV^2, MD^2, with their mean values and standard deviation fluctuating between 38.93±3.32% and 79.86±5.68% (Fig. 2).

It is noted that MD has the greatest effect on the S of CP+PAC, with a synergistic effect when the FE contain high levels of MD and the system operates at low OAT, ADV and VPC conditions. This situation is attributed to MD, which is a non-crystalline amorphous material, highly soluble in water

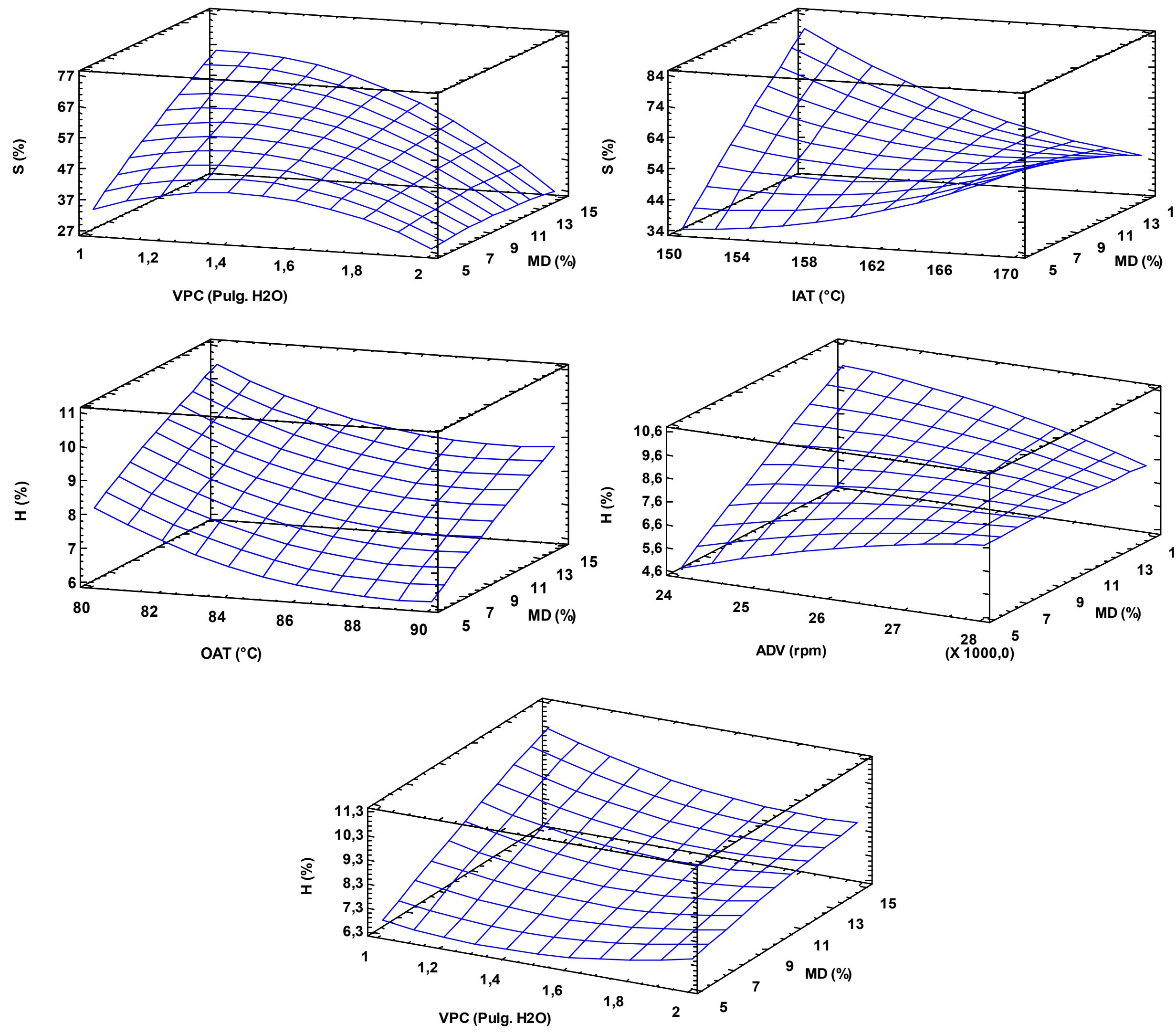


Fig. 2: Response surface graphics of S and H in function of the independent variables

(Santhalakshmy *et al*., 2015) and to its encapsulating role (Cano-Chauca *et al*. 2005; Wang and Zhou, 2012); which favors reconstitution processes or the availability of the encapsulated compounds in a food system (Daza *et al*., 2016; Jafari *et al*., 2017); furthermore, S is influenced by the FE properties or raw materials used and by the properties of the powder, like particle size, surface area and their physical state, where a rough surface and an amorphous state (non-thermodynamic state) is more favorable (Cano-Chauca *et al*., 2005; Caparino *et al*., 2012; Du *et al*., 2014; Bicudo *et al*., 2015; Avila *et al*., 2015; Cortés-Rojas *et al*., 2015; Jafari *et al*., 2017; Tontul and Topuz, 2017). Similar results to those obtained in this research have been reported in diverse powder matrices (Grabowski *et al*., 2006; De Oliveira *et al*., 2009; Bakar *et al*., 2013; Avila *et al*., 2015; Avila *et al*., 2015; Moghaddam *et al*., 2017); however, some investigations have reported diminished S of pineapple powder (Abadio *et al*., 2004; Tontul and Topuz, 2017) and in avocado powder (Marulanda, 2015) who reports the formation of complexes from the structural interactions of the fat with the MD.

The effect of the interaction of high IAT and OAT on diminished S is also highlighted, which could be attributed to the oily composition of the powder matrix or to the formation of a surface layer, given the high heat transfer conditions, which reduce the diffusion and wettability of the particle during reconstitution (Jafari *et al*., 2017), which was reported by Chegini and Ghobadian (2005) in orange powder and by Quek *et al*. (2007) in watermelon powder; while a contrary effect has been reported for tomato (Goula and Adamopoulos, 2005a, 2005b); mountain tea (Nadeem *et al*., 2011); pomegranate (Vardin and Yasar, 2012); pitahaya (Bakar *et al*., 2013); tamarind (Muzaffar and Kumar, 2015); sour cherry (Moghaddam *et al*., 2017).

Generally, the S values obtained in this study are relatively lower with respect to those obtained in other investigations. Daza *et al*., (2016) report in powder from Cagaita+gum Arabic extracts (S: 94.4-97.8%) and powder from Cagaita+inulin extract (S: 87.7-95.9%); Santhalakshmy *et al*. (2015) in powder from jamun+MD (S: 87.67±0.5% and 99.67±0.58%). Avila *et al*. (2015) in powder from sugar cane+MD (S: 98%).

This behavior could be because the extracts from the fruits mentioned are very rich in sugars (saccharose, glucose disaccharide and fructose, among others) soluble in water and with a vast amount of Hydroxyl groups (OH) in the molecule (King *et al*., 1984; Dib Taxi *et al*., 2003). The CP+PAC has dietary fiber content equivalent to 23.9±1.6 of which close to 93% is insoluble in water (Trinidad *et al*., 2006; Raghavendra *et al*., 2006; Yalegama *et al*., 2013).

Hygroscopicity: Hygroscopicity had significant linear effects ($p<0.05$) with respect to the independent variables IAT, OAT, VPC and MD, with the interactions IAT-OAT, IAT-MD, OAT-ADV, OAT-VPC, ADV-VPC, ADV-MD and VPC-MD and with the quadratic interactions IAT, OAT, ADV and VPC, which is why their fluctuations were between 6.4±0.11.0% and 10.3±1.0% (Fig. 2). Hygroscopicity tended to increase with diminished IAT, principally when it interacts with high MD contents and low OAT, reaching the maximum water absorption values approximately with 10%. Although the CP+PAC has fat content equivalent to 0.31 g/g SS, which confers it hydrophobic characteristics, the behavior of H is affected by the other components and amounts present in the powder (Mishra *et al*., 2014) and, for this case, MD has good affinity with H_20, which favors water adsorption. A synergistic effect is also noted with the X_w with which the product leaves the drier (>> at low OAT), forming hydrogen bridges in hydrophilic points of the soluble and insoluble material, which could also favor the product's stickiness or cohesiveness (Tonon *et al*., 2008; Tontul and Topuz, 2017).

Some authors report a similar behavior of MD in different foods: sugar cane powder (Ávila *et al*., 2015), red pitahaya (Bakar *et al*., 2013) and tomato powder (Goula and Adamopoulos, 2008a), as well as in other products (Bakar *et al*., 2013; Goula and Adamopoulos, 2008b; Igual *et al*., 2014), which increases the molecular mobility of water and diminishes its T_g (Bakar *et al*., 2013). However, opposite results were obtained in other powdered products: acai (Tonon *et al*., 2008), jujube (Chen *et al*., 2014), sapodilla (Chong and Wong, 2015), blackberry (Ferrari *et al*., 2013), amla (Mishra *et al*., 2014), cherries (Moghaddam *et al*., 2017) and cactus pears (Rodríguez-Hernández *et al*., 2005), as when other wall materials were used, like gum Arabic, buttermilk protein concentrate and isolated soybean protein (Bhusari *et al*., 2014; Igual *et al*., 2014; Muzaffar and Kumar, 2015).

Other authors report that increased IAT increases water absorption (Castro-Muñoz *et al*., 2015; Chen *et al*., 2014; Moghaddam *et al*., 2017) because it reduces the X_w from the product and contributes to the formation of more porous particles and is consequential with the particle surface area (De Souza *et al*., 2015; Daza *et al*., 2016).

The relationship of H with respect to ADV and VPC was similar, without setting a defined trend, given that it depends highly on the interaction with MD; H decreases with increased VPC when FE has a high content of MD (15%), while the effect of ADV is more favorable on H (<<<) when the system operates at low ADV and MD.

Wettability: Hu may be defined as the capacity of a porous agglomerate system (powder) to be penetrated by a liquid due to the capillary forces (Hogekamp and Schubert, 2003). The Hu is inversely related to particle size, where the larger and more irregular particles show more spaces between them, making them more easily

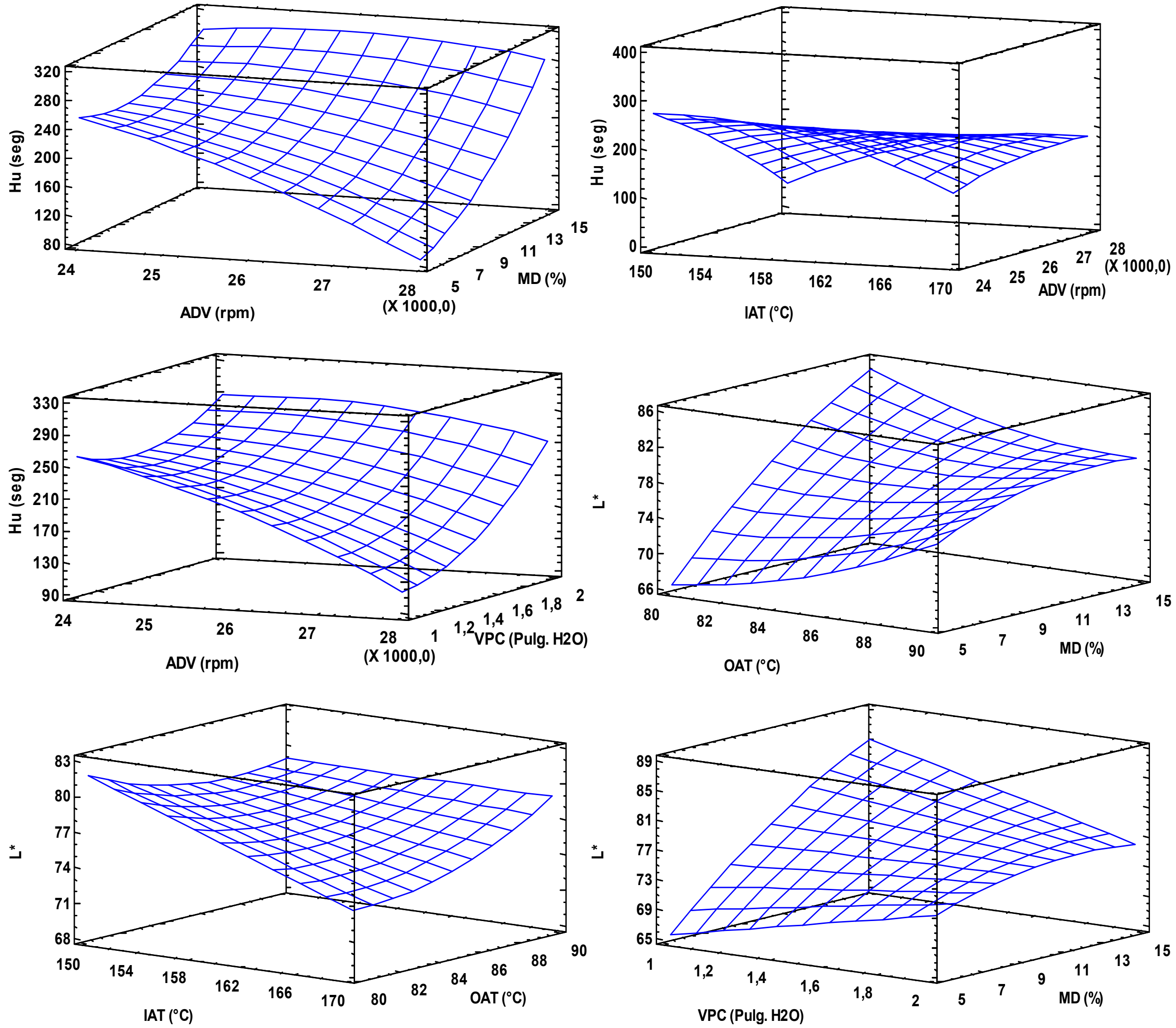


Fig. 3: Response surface graphics of Hu and L* in function of the independent variables

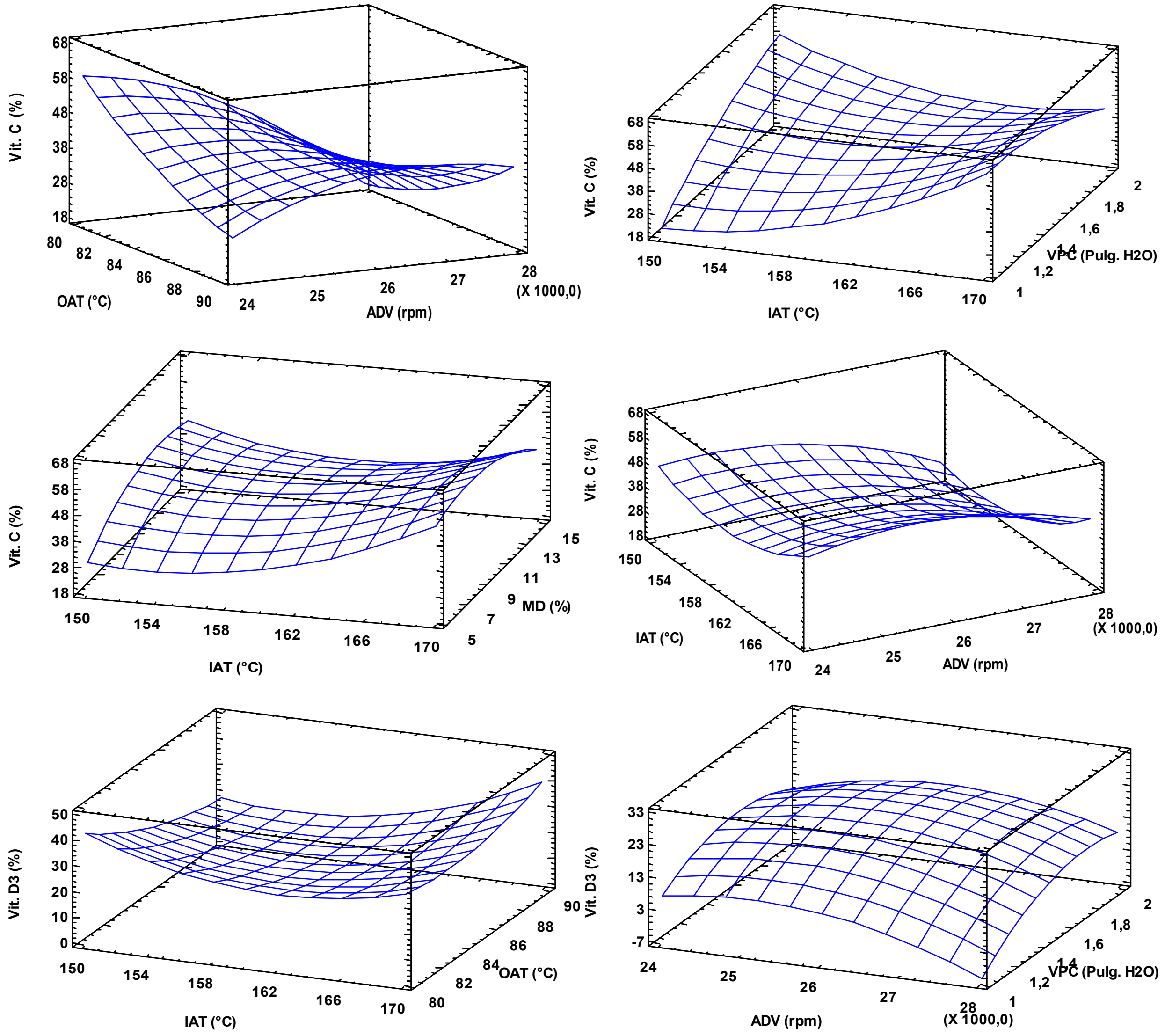
penetrated by water, with the opposite occurring in the smallest particles that have reduced interstitial space hindering the liquid's penetration, resulting in poor reconstitution properties (Cynthia *et al*., 2014). A Hu time of a few seconds is desirable for powders with good reconstitution properties; hence, CP+PAC showed a negative aspect in relation to this property (varying between 124.00±2.65 and 312.67±4.62 sec) (Fig. 3) due to the high concentration of fat, being a hydrophobic material that does not absorb water; while Santana *et al*. (2017) with powdered babassu milk 12.8±0.1 min on average and Santhalakshmy *et al*. (2015), with jamun powder varied between 82.67 and 116 sec) as shown by the sample.

Color: From the color parameters, L* of CP+PAC is mainly highlighted, which had significant differences due to the linear effects of the independent variables IAT, ADV and MD percentage, with the linear interactions IAT-OAT, IAT-ADV, IAT-VPC, OAT-MD, ADV-MD, VPC-MD, ADV-VPC and with the quadratic interactions OAT^2, ADV^2 and MD^2, varying the averages within the ranges 68.4±0.3-82.2±0.6 (Fig. 3). Now, a* and b* chromaticity, although having statistical differences with respect to the independent variables and their interactions, had quite subtle changes: a* (1.4±0.1 → 3.0±0.2) and b* (8.29±0.30-11.28±0.53), which are not perceptible to the human eye and place them on the a*b* chromatic plane in the achromatic zone (grey zone) (Gilabert, 1998; Alvarado and Aguilera, 2001; Santhalakshmy *et al*., 2015).

L* present a tendency to diminish with increased IAT, observing greater darkening when the system operates at low OAT, ADV and VPC. This situation is attributed principally to a set of non-enzymatic reactions that may be present: Maillard (fructose, glucose) (Siriphanich *et al*., 2011); oxidation of the ascorbic acid present, which is very reactive and whose degradation permits the formation of dycarbonil intermediaries (Solval *et al*., 2012); peroxidation of fatty acids present, especially those unsaturated that react with O_2 and its reagent species, producing aldehydes and ketones, which react with the amino acids, forming brown pigments (Kha *et al*., 2014; Luna-Guevara *et al*., 2017); finally, caramelization reactions of the sugars present (Cano-Chauca *et al*., 2005; Goula and Adamopoulos, 2005a; Quek *et al*., 2007; Fazaeli *et al*., 2012a; Daza *et al*., 2016; Bazaria and Kumar, 2016). All these reactions are favored at high IAT (Chen *et al*., 2014; Horuz *et al*., 2012; Jiménez-Aguilar *et al*., 2011; Kha *et al*., 2010). Another very important aspect that must be considered is the correlation of the oxidative processes with the CP+PAC a_w, where its increase favors a higher rate of darkening (Henríquez *et al*., 2013; Shishir *et al*., 2017).

An important effect is observed on the CP+PAC color with increased MD in the FE, which produces greater clarity (>L*) due to the natural whiteness as wall material and its concentration (Tontul and Topuz, 2017; Comunian *et al*., 2011; Santhalakshmy *et al*., 2015). Rodríguez-Hernández *et al*. (2005) found a direct correlation between the concentration of the carrier material and the total color difference (ΔE) of cactus pear powder; while other researchers highlight the same effect of the MD or wall material used: Ahmed *et al*. (2010) for potato powder; Yousefi *et al*. (2011) and Jafari *et al*. (2017) with pomegranate powder; Jiménez-Aguilar *et al*. (2011) with blueberry powder; Fazaeli *et al*. (2012b) with *Morus nigra* powder; Bazaria and Kumar (2016) with beet powder.

The effect of the VPC is observed principally with the interaction with the MD, where the clarity of CP+PAC is greater with lower VPC, which implies a greater time of residence in the drying chamber; likewise favoring non-enzymatic reactions.



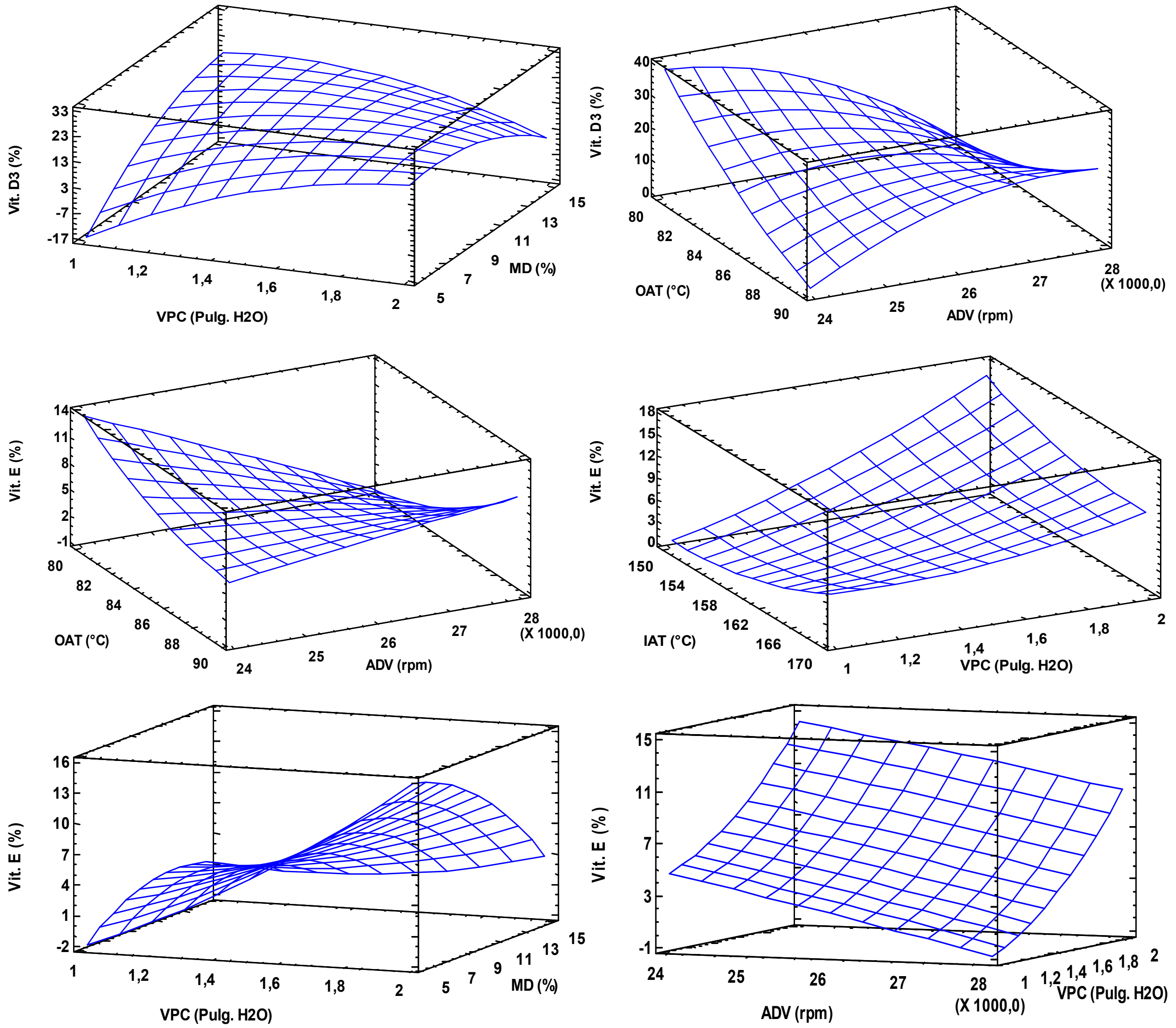


Fig. 4: Response surface graphics of PAC in function of the independent variables

Physiologically Active Components (PAC): Calcium content had no significant effects ($p>0.05$) with respect to the independent variables assessed, or with their interactions; while ANOVA had statistical differences ($p<0.05$) in Vit.D$_3$ (Cholecalciferol) with respect to the linear and quadratic effects of all the independent variables; while Vit.C (ascorbic acid) with respect to IAT and ADV and Vit.E (DL-α-Tocopherol acetate) with respect to ADV, VPC and MD. Significant differences ($p<0.05$) were noted with respect to quadratic interactions IAT^2, ADV^2, MD^2 (Vit.C) and IAT, OAT, VDC and MD (Vit.E) and with respect to interactions IAT-ADV, IAT-VPC, OAT-ADV, OAT-VPC, ADV-MD, VPC-MD, OAT-MD (Vit.D$_3$); IAT-OAT, IAT-ADV, IAT-VPC, IAT-MD, OAT-ADV, OAT-VPC, VPC-MD (Vit.C) and IAT-VPC, ADV-OAT, ADV-MD, ADV-VPC, VPC-MD (Vit.E) (Fig. 4). In this context, vitamin retention is strongly influenced by the operating variables, varying Vit.D$_3$ between 5.5±0.6% and 27.8±0.8%; Vit.C between 22.9±6.8% and 56.8±1.0% and Vit.E between 2.6±0.3% and 12.0±0.7%.

SD has been used as a technique that permits encapsulating and preserving the nutritional value of some vitamin groups (Hartman *et al*., 1967; Gharsallaoui *et al*., 2007; Ray *et al*., 2016); however, the properties of the powder matrix and of its PAC present, depend on the combination of multiple factors, highlighting IAT, the total content of FE solids and additives used and the degree of protection provided to the nucleus material, pulverization and drying, among others (Bimbenet *et al*., 2002; Pérez-Alonso *et al*., 2003). The greatest protection effect of the MD as an encapsulating agent of vitamin C in CP+PAC was observed when its content ranged between 10 and 12%.

Vitamin C is a water-soluble vitamin and sensitive to heat; however, the IAT effect on CP+PAC did not have a well-defined behavior, given that it depends on its interaction with other independent variables; for example, increased IAT favored retention of Vit.C when the system operates at low VPC (> time of residence) and ADV (> particle size), which could be generating surface darkening with lower temperature profiles within the structure. Higher retention of Vit.C is observed when the system operates at low IAT (< thermal stress) and high VPC (< time of residence), which is more coherent.

Many researchers have reported diverse effects on Vit.C during SD: Cortés Rodríguez *et al.* (2017) reported retention levels of 69.7±0.7% in golden berry powder by using MD as encapsulating agent; Goula and Adamopoulos (2006) reported higher losses through thermal degradation (>IAT) and oxidation; Solval *et al.* (2012) reported higher losses between 9.8 and 49.2% with increased IAT (170 to 190°C) in cantaloupe powder; Islam *et al.* (2016) reported losses between 29 and 35%, being higher with increased MD in orange powder; Thankitsunthorn *et al.* (2009) reported losses of 62.1% with IAT at 140°C in currant powder; Rodríguez-Hernández *et al.* (2005) reported losses between 72 and 49% in cactus pear powder with IAT between 205 and 225°C; Kaya *et al.* (2010) reported losses of 72.5% in kiwi powder without using encapsulating agent; Angel *et al.* (2009) reported losses between 60.3 and 43.1% in passion fruit powder using MD as encapsulating agent and IAT between 180 and 190°C; Estevinho *et al.* (2016) reported losses of 56.4, 55.5 and 54.6% in Vit. C, using as an encapsulating agents sodium alginate, chitosan and modified chitosan, respectively; and Nesterenko *et al.* (2014) reported recovery levels of 77 to 87% of ascorbic acid microencapsulated with isolated soy protein modified through acylation and cationization.

Vitamin D_3 had a similar behavior as Vit.C with its retention greatest when the system operates at high IAT (explained previously) and with low OAT (particles with < thermal stress). The protection effect of MD is not well defined. A dependency exists with respect to other variables, like VPC and ADV, observing greater Vit D_3 retentions when its content in the FE is high and VPC between 1.2 and 1.4 "H_20, or vice versa, when MD is low and high VPC (1.8-2.0 "H_20), that is, a central zone exists, highlighted in the MD-VPC graphic that favors its retention. The major effect of ADV is noted because of the interaction with OAT, with its greatest retention at low OAT and ADV. Under this situation, the particles have < thermal stress and higher particle sizes, which favors retention of this PAC.

Few research works report on the effects of the operating variables of the SD process on the Vit.D_3 retention. Fortification of powdered milk is highlighted, considering equivalent losses to 30% (Indyk *et al.*, 1996).

Vitamin E tends to increase retention levels, particularly when the system operates at high VPC and low MD contents in the FE and high VPC and high IAT, identifying the time of residence of the particle in the drier as key for its retention. The highest retention of Vit.E is observed with increased IAT, as already explained. Another important variable in Vit.E retention is reached when the system operates at low ADV and OAT, a similar effect observed in Vit.D_3 retention.

Some investigations have reported the effects of the operating variables of the SD process on the retention of some chemical forms of Vit.E: Hategekimana *et al.* (2015) reported losses between 20.8 and 28.5% in nanocapsules obtained through SD, using starch capsule as wall material; Pierucci *et al.* (2006), used MD and gum Arabic obtaining losses of 73 and 87%, respectively; Faria *et al.* (2010), using green pea protein and carboxymethyl cellulose in the microencapsulation of α-tocopherol, reached retention levels between 73 and 87%; Nesterenko *et al.*, (2014) reported recovery levels from 61 to 68% of α-tocopherol microencapsulated with isolated soy protein modified through acylation and cationization; and (Parthasarathi and Anandharamakrishnan, 2016), using whey protein obtained an encapsulation yield of α-tocopherol at 89.6%.

Peroxide index: Coconut is a product with an important content of saturated and unsaturated fatty acids, which is why the CP+PAC is a product vulnerable to lipid oxidation, giving way to peroxide formation and resulting in undesirable rancid odors and flavors, hence, PI is a quality and freshness indicator of these types of products used to evaluate the initial stages of their oxidative process (Kha *et al.*, 2014; Luna-Guevara *et al.*, 2017).

The PI had significant linear effects ($p<0.05$) with respect to the independent variables IAT, OAT, ADV and VPC, with respect to interactions IAT-OAT, IAT-ADV, IAT-VPC, IAT-MD, OAT-VPC, OAT-MD, ADV-VPC and VPC-MD and with the quadratic interactions IAT^2, ADV^2 and VPC^2, which permitted obtaining a variation between 1.5±0.4 and 5.9±0.1 meq H_2O_2/kg oil, corresponding to 1.1±0.1 and 3.32±0.17 meq H_2O_2/kg powder, respectively (Fig. 5). Although the CP+PAC structure is a powder matrix restructured with a fat content of 30.54±0.90%, the results obtained from the PI are below the maximum value permitted, according to the norm established for pressed vegetable oils in the codex standard 210-1999 (15 meq O_2/kg oil or 7.5 meq H_2O_2/kg oil) (Codex Alimentarius Commission, 1994, Codex standard for grated desiccated coconut-CODEX STAN 177-1991).

The effect of IAT and the VPC was not as expected, given that the response surface graphics showed a trend to increasing PI with diminished IAT and with increased VPC (< time of residence). Some authors have reported diverse effects of IAT, for example, Kha *et al.* (2014), reported a variation of PI between 3.4 and 7.9 meq/kg oil in the

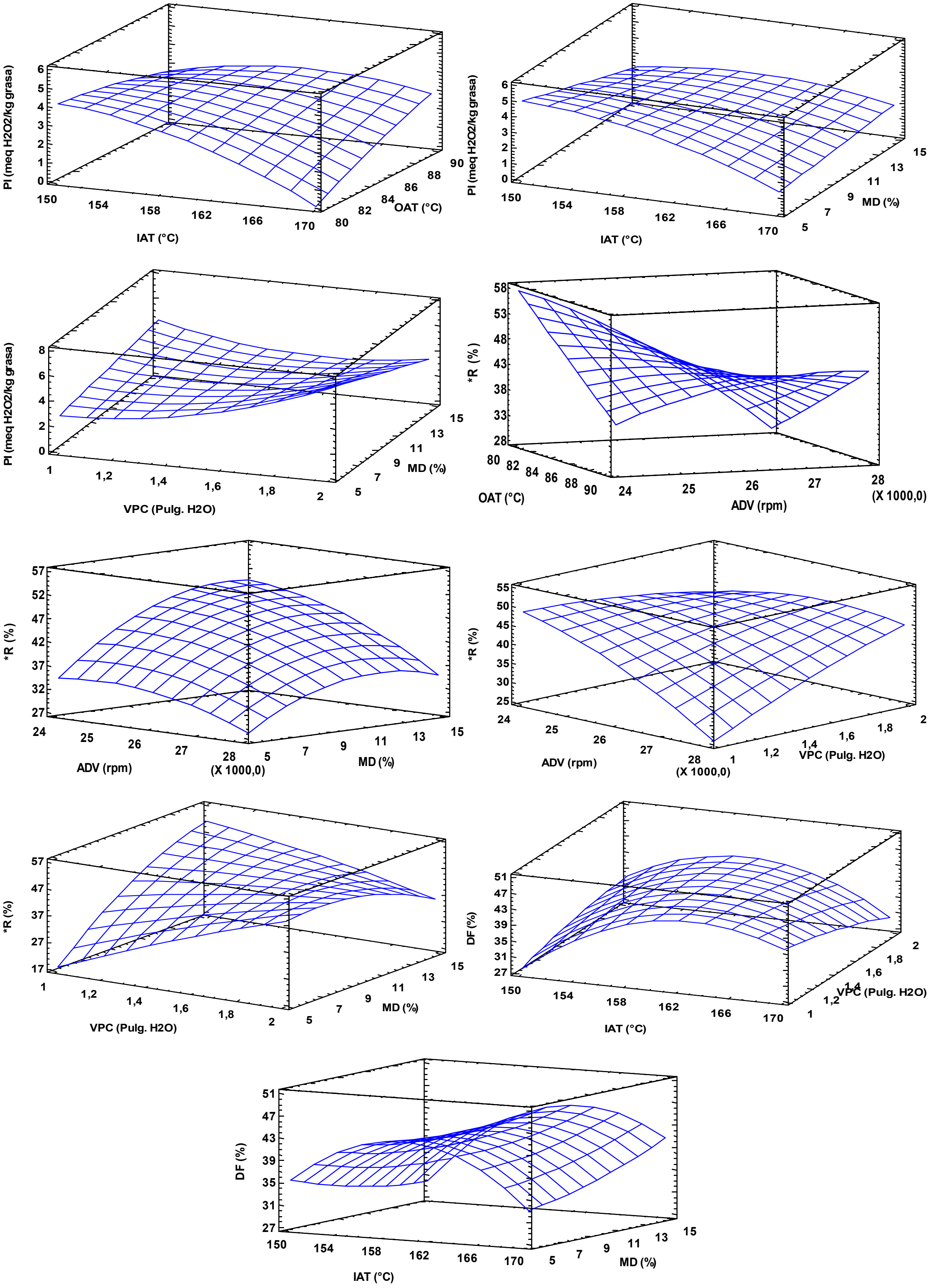


Fig. 5: Response surface graphics of the PI, *R and DF in function of the independent variables

microencapsulation of gac oil, increasing significantly with increased IAT and OAT; while Frascareli *et al.* (2012) reported no significant differences of the PI in the microencapsulation of coffee oil due to the effect of the IAT (150 and 190°C), reaching mean PI value of 0.96 meq H_20_2/kg oil.

For the CP+PAC, it is considered that favorability exists for peroxide formation of the CP+PAC when the FE has low MD contents and high OAT. This effect of the MD-OAT interaction on the stability of the CP+PAC to oxidative processes favors in part the wall material used and its combination, which form a dense and continuous restructured matrix, which could hinder O_2 transfer through the structure and, thus, delay oxidation of the fat content (Hogan *et al.*, 2003; Kagami *et al.*, 2003; Luna-Guevara *et al.*, 2017). Some researchers have reported an effect similar to that found in this research, during processing through SD: Orlien *et al.* (2000), reported values of PI<15 meq O_2/kg oil in the microencapsulation of fish oil with an MD matrix, saccharose and gelatin; Mohammed *et al.* (2017), reported values of PI: 3.43±0.05 meq O_2/kg oil in the microencapsulation of the *Nigella sativa* L. oil, using as wall material sodium caseinate and MD (dextrose equivalent = 10) in a 1:9 ratio (p/p) and with IAT between 150 and 190°C; and Santana *et al.* (2017) reported in the powdered babassu milk effective protection of MD against lipid oxidation.

Yield and deposit formation: The *R is an important indicator in industrial productivity and profitability of powdered products obtained through SD, with adhesiveness/stickiness being one of the principal causes of its decrease (Can Karaca *et al.*, 2016). Likewise, the DF within the drying chamber represents a process problem, which is formed due to semi-humid deposits of drops that are not dry enough before hitting the wall and by sticky deposits caused by the nature of the product at the drying temperature; in any case, both variables are affected in an inversely proportional manner (Masters, 1985; León-Martínez *et al.*, 2010; Fazaeli *et al.*, 2012a).

The *R had significant linear effects ($p<0.05$) with respect to ADV and MD, with respect to the interactions IAT-ADV, IAT-MD, OAT-ADV, ADV-VPC and VPC-MD and the quadratic interactions IAT^2, MD^2, with the results obtained (31.3-52.6%) lower than those recommended by Bhandari *et al.* (1997) (>50%). Additionally, the DF had significant linear effects ($p<0.05$) with respect to the IAT and OAT factors, with respect to interactions IAT-MD, OAT-MD, ADV-MD, VPC-MD, IAT-ADV, IAT-VPC and ADV-VPC and with the quadratic interactions IAT^2, OAT^2 and ADV^2, which produced a variation in the processes between 27.5 and 47.4% (Fig. 5). These low values of *R and high DF are attributed to small lots of FE (3 kg) prepared, from the losses due to the material adhered in tanks and piping, which affects its value percentage wise; however, given that it is carried out equally for all the experiments, it makes them comparable within the optimization process.

The *R of the CP+PAC had a tendency to increase with an increased percentage of MD and with decreased ADV, which is attributed mainly to the role of the MD on the vitreous transition temperature (T_g), reducing the product's stickiness (Adhikari *et al.*, 2009; Tonon *et al.*, 2010; Osorio *et al.*, 2011; Jayasundera *et al.*, 2011a, 2011b, 2011c; Ferrari *et al.*, 2012; Roustapour *et al.*, 2012; Muzaffar and Kumar, 2015; Tontul and Topuz, 2017). Additionally, higher content of MD produces an FE with higher content of total solids and higher density and viscosity, which produces lower radial velocity and less collisions of the drops against the walls of the drying chamber.

The DF did not have well-defined trends, highlighting principally the MD interactions with the rest of the independent variables. The conditions that favor most the lower DF occur at IAT = 150°C in the entire MD range (35-31%) and with low VPC (27-31%), which is mainly attributed to the lower stickiness experienced by the structure at low IAT; however, a similar condition is reached when the system operates at high IAT and VPC, suggesting that the particles do not reach high temperatures during low time of residence. Santana *et al.* (2017), reported that high contents of MD or starch (25 and 20%, respectively) increased DF significantly, given that this implies increased input viscosity, generating higher availability and probability that the solids adhere to the chamber walls. Avila *et al.* (2015), report high DF values (6.1-86.5%) and low *R (10.2-91.3%) in cane powder, caused mainly by the effect of IAT on the fusion of the carbohydrates present. Other similar results were reported by Bhandari *et al.* (1997) and Jayasundera *et al.* (2011a, 2011b, 2011c) for saccharose solutions.

Other investigations highlight different effects on *R and DF: Chegini and Ghobadian (2005) evaluated the use of MD on the *R and DF in orange powder and with IAT between 130 and 150°C, finding *R between 18 and 35% and that formulations without MD generate a glassy film on the walls; while increased IAT caused the material fusion, greater adhesion to the wall (>DF), lower *R and the formation of a dry layer on the drop's surface, obstructing the water diffusion. Other similar results have been reported for tamarind powder (Cynthia *et al.*, 2014); orange powder (Goula and Adamopoulos, 2010); pomegranate powder (Vardin and Yasar, 2012); black mulberry powder (Fazaeli *et al.*, 2012a); caqui powder (Du *et al.*, 2014); sugar cane powder (Avila *et al.*, 2015); beet powder (Bazaria and Kumar, 2016) and in saccharose solutions (Jayasundera *et al.*, 2011a, 2011b, 2011c).

Increased *R due to the effect of lower ADV is not clear, nor is it evident with the interaction found at low

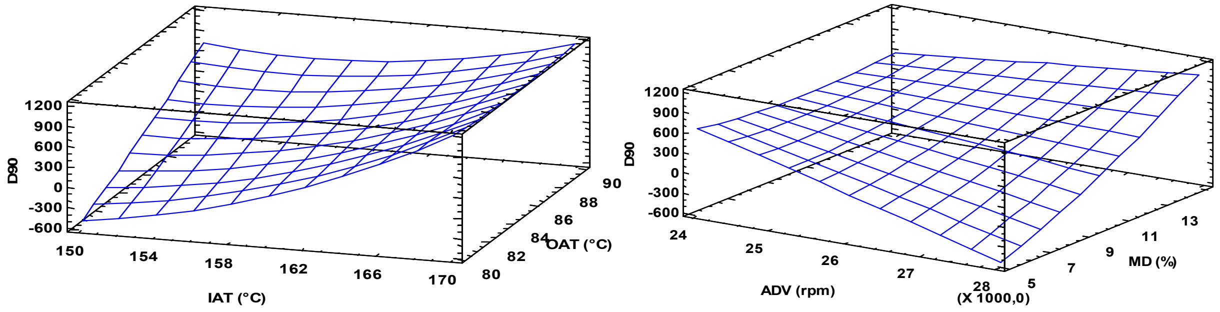


Fig. 6: Response surface graphics of particle size D_{90} in function of the independent variables

OAT, where the bigger particles undergo lower thermal stress that affects negatively the heat and mass transfer and, hence, the elimination of water during drying, which should favor its stickiness and greater DF or adhesiveness of the product to the metal of the drying chamber). In addition, the interaction with VPC guarantees high *R in any range of ADV.

Particle sizes: In general, particle sizes in percentiles D_{10}, D_{50} and D_{90} are affected statistically ($p<0.05$) one way or another by the independent variables and their linear and quadratic interactions. It is highlighted that percentiles D_{10} and D_{50} had fluctuations of 1.6±0.0-3.0±0.8 and 5.5±0.4-58.6±17.7 µm, respectively, which are not considered critical variables; rather, they are acceptable within the behavior of powdered products obtained through SD; while percentile D_{90} was the most relevant parameter because of its variability (46.3±3.0-1153.2±208.3 µm), presenting significant linear effects ($p<0.05$) with respect to IAT, OAT, ADV and MD, with respect to the interactions IAT-OAT, IAT-ADV, IAT-MD, OAT-ADV, OAT-VPC, ADV-VPC, ADV-MD and VPC-MD and the quadratic interactions of ADV^2 (Fig. 6).

These results showed that the CP+PAC represent a non-homogenous particulate system or with high variability, which was evidenced due to the agglomeration observed. This situation supposes the existence of cohesive phenomena among particles, given the high composition of fat content present and principally from the free oil on the particle surface that would be contributing in the formation of irreversible link bridges (Frascareli *et al.*, 2012; Zotarelli *et al.*, 2017). In addition, the spaces between large particles could be occupied by smaller particles, increasing the apparent density and their rehydration properties (Santana *et al.*, 2017). According to Hogekamp and Schubert (2003), the presence of bigger particulate material can favor the solubility or instantaneous properties, given that increased interstices favor the powder's water penetration, wettability and dispersibility.

The D_{90} percentile had a tendency to increase at high IAT and OAT, potentiating at 170 and 90°C, respectively, which could be related to the greater cohesion of the particles with the formation of aggregates, as already mentioned. The behavior of the D_{90} percentile with respect to the ADV variables is not well defined, rather, it interacts principally with MD, potentiating at high ADV and MD, where the latter produces a higher viscosity of the FE that overlaps the ADV effect, resulting in bigger particle sizes (Goula and Adamopoulos, 2004; Adhikari *et al.*, 2009; Ferrari *et al.*, 2012; Tonon *et al.*, 2008; Tontul and Topuz, 2017). At low MD contents, the ADV effect is coherent, diminishing the D_{90} at high ADV (Jumah *et al.*, 2000; Chegini and Ghobadian, 2005; Cortés-Rojas *et al.*, 2015).

Optimization of the spray drying process: According to the results obtained from the dependent variables and from the ANOVA performed, the experimental optimization was planned by bearing in mind the most important variables of the process, maximizing S, L*, R*, D_{90} and PAC; minimizing H, Hu, PI and DF; in addition, a medium value was set for X_w and a_w, given that their fluctuations were not very large. Under this context, the optimal conditions obtained were: IAT: 170°C; OAT: 85.8°C; ADV: 26676 rpm; VPC: 1.6" H_2O; MD: 7.0%; while the dependent variables obtained from three replicates at the optimal process conditions were the following: X_w: 1.7±0.4%; a_w: 0.171±0.018; S: 58.4±2.1%; H: 8.4±0.5%; L*: 79.5±0.9; a*: 1.5±0.1; b*: 9.5±0.4; Vit. C: 32.4±6.2%; Vit. E: 6.1±1.9%; Vit. D_3: 7.8±1.8; Ca: 41.7±2.3%; Hu: 263.0±19.8 s; PI: 2.4±1.3 meq H_2O_2/kg; DF: 32.4±2.3%; *R: 44.0%; D_{10}: 1.70±0.05 µm; D_{50}: 8.46±2.09 µm; D_{90}: 78.18±24.30 µm. Furthermore, the proximal composition of CP+PAC was: fat: 30.5±0.9%, protein: 4.1±0.5%, total dietary fiber: 23.9±1.6%, ashes: 2.3±0.0%, highlighting the dietary fiber, which confers health benefits to the consumer, besides its PAC present.

Powder morphology: Figure 7 presents CP+PAC micrographs obtained at the optimal conditions selected, which mostly exhibited spherical shapes with particle sizes fluctuating between 20 and 65 µm, smooth surfaces and some rough, some particles show collapsed walls and structurally agglomerated. In

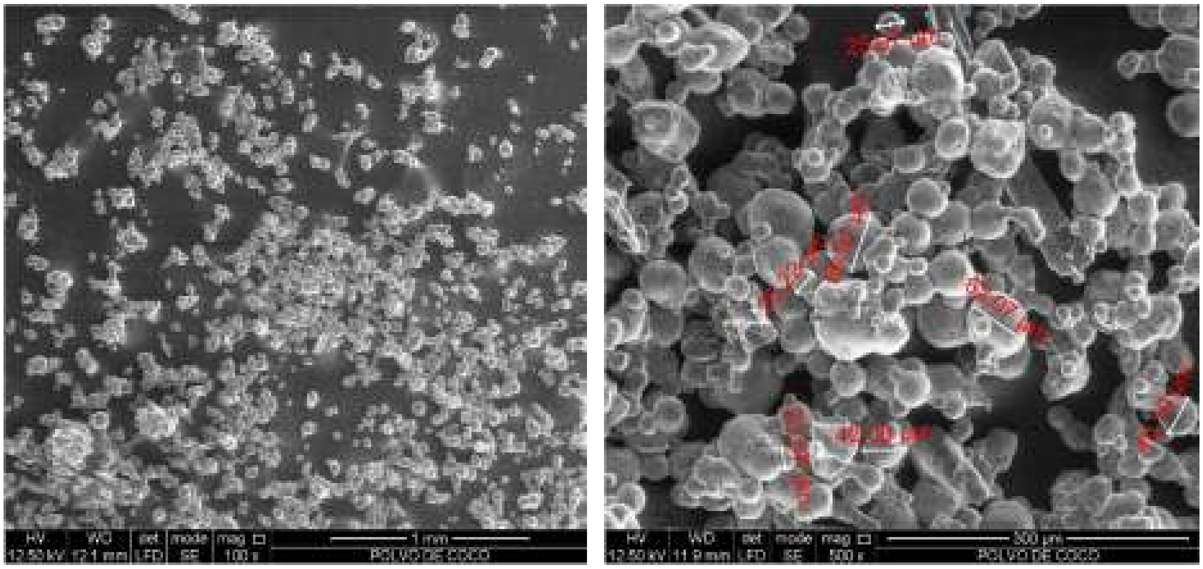


Fig. 7: CP+PAC micrographs obtained through SD at 100 and 500 X

addition, fibrous-type particles exist, without apparent fissures or sharp edges or surfaces. This microstructure observed in the CP+PAC is considered characteristic of powders dried through pulverization (Frascareli *et al*., 2012; Jafari *et al*., 2017; Mohammed *et al*., 2017). The Surface structure and the fibrous material without fracturing or low-porosity matrix indicates the effective role of MD as encapsulating agent providing coverage over the nucleus, which-likewise-acts as a thermal defense against oxidation and any unwanted physical and chemical change (Cortés-Rojas *et al*., 2015).

León-Martínez *et al*. (2010) have correlated these characteristics as consequence of electric and static effects and of the van der Waals forces; while Frascareli *et al*. (2012) suggest that the microstructural collapse experienced by the particles is formed due to their contraction during drying and subsequent cooling. Some authors have reported similar morphologies of products obtained through SD and by using gum Arabic as wall material: monoterpene microcapsules (Bertolini *et al*., 2001), cardamom oleoresin (Krishnan *et al*., 2005), cumin oleoresin (Kanakdande *et al*., 2007) and coffee extract (Rodrigues and Grosso, 2008), among others. Some investigations have defined as the most favorable microstructures those where the particles are spherical and smooth, given the greater protection and retention of the ingredients (<surface/volume ratio), higher apparent density (better packaging) and good fluidity (Santana *et al*., 2017).

CONCLUSION

The coconut matrix represents a complex food system for its transformation into powder, given its high-fat content and insoluble fiber with high hardness mechanical characteristics; however, a technological development was fine-tuned to confer coconut powder good physicochemical and physical quality attributes; besides improved nutritional composition by incorporating Ca and vitamins C, D_3 and E; however, the influence of SD is quite notable on the vitamin degradation.

Due to the multiple effects of the independent variables IAT, OAT, ADV, VPC and MD on the dependent variables assessed: X_w, a_w, S, H, L*, a*, b*, Hu, PI, Calcium, Vit.C, Vit.D_3, Vit.E, *R and DF the experimental optimization carried out by using statistical tools represents an effective path to define the most suitable conditions of the SD process, while representing significant progress for its subsequent industrial scaling and its potential generation of added value to the coconut agro chain.

The experimental optimization defined the CP+PAC processing conditions, thus: IAT: IAT: 170°C; OAT: 85.8°C; ADV: 26676 rpm; VPC: 1.6 ”H_2O; MD: 7.0%; and with quality attributes: X_w: 1.7±0.4%; a_w: 0.171±0.018; S: 58.4±2.1%; H: 8.4±0.5%; L*: 79.5±0.9; a*: 1.5±0.1; b*: 9.5±0.4; Hu: 263.0±19.8 s; PI: 2.4±1.3 meq H_2O_2/kg oil; DF: 32.4±2.3%; *R: 44.0%; D_{10}: 1.70±0.05 μm; D_{50}: 8.46±2.09 μm; D_{90}: 78.18±24.30 μm; Vit. C: 32.4±6.2%; Vit. E: 6.1±1.9%; Vit. D_3: 7.8±1.8; Ca: 41.7±2.3%, making it a hygroscopic product, potentially sensitive to oxidative processes during storage that could derive into changes in color, flavor, or strange odors, which is why it will require a package with high permeability to water vapor and O_2 to minimize these changes.

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