Research Article

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

1,2Lucas Aguirre Juan Carlos, 2Giraldo Giraldo German Antonio and 3Cortés Rodríguez Misael
1Universidad Nacional de Colombia, -Sede Medellín. Calle 59 A N 63-20, Medellín,
2Universidad del Quindío, Carrera 15 Calle 12 Norte, Armenia-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 (Xₕ), water activity (aₕ), Hygroscopicity (H), Solubility (S), wettability (Hu), color (L*, a* y b*), recovery of PAC (Ca, vitamins C, D₃ and E), Peroxide Index (PI) and particle size (D₉₀, D₃₀ y D₁₀). 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" H₂O; MD: 7.0%; and with quality attributes: Xₕ: 1.7±0.4%; aₕ: 0.17±0.018; H: 8.4±0.5%; S: 58.4±2.1%; Hu: 263.0±19.8 s; L*: 79.5±0.9; a*: 1.5±0.1; b*: 9.5±0.4; PI: 2.4±1.3 meq H₂O/kg oil; DFC: 32.4±2.3%; *R: 44.0%; D₉₀: 1.70±0.05 μm; D₃₀: 8.46±2.09 μm; D₁₀: 78.18±24.30 μm; Ca: 41.7±2.3%; Vit.C: 32.4±6.2%; Vit.D₃: 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, dehydation, 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., 2013); Annalaki 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 oleraceaef Mart.) (Tonon et al., 2010), cactus pear or nopal (Opuntia ficus-indica) (Saénz et al., 2009; Medina-Torres et al., 2013), cassis 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.) (Can-Ochaue et al., 2005; Caparino et al., 2012), passion fruit (Passiflora sp. ) (Bornmann et al., 2013), cashew (Anacardium occidentale L.) (Da Silva Bastos et al., 2012).
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; Gaula and Adamopoulos, 2005b; Gaula and Adamopoulos, 2008a; Gaula 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, D3 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 D3 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 Pacifico (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 ≈ 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 (Xw): Official method AOAC (1990) 930.15/90; water activity (aw): 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 (Martinez-Navarrete et al., 1998) using a saturated KI solution at 25°C (aw = 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., 2018).
Quantification of vitamins E and D₃ 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 4.0×250 mm), diode array, mobile phase: KH₂PO₄ 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 D₃ and E was done according to the methodology proposed by Cortés-Rodríguez (2004) modified through inclusion treatment of PAC in a 100-g serving.

Particle sizes were determined as percentiles D₁₀, D₅₀, and D₉₀, 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 refractive index of 1.52 (Mirhosseini et al., 2008). Color was determined through the CIE-L*a*b* coordinates, using an X-Rite spectrophotometer model SP62, D₆₅ 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+H₂O)/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 (IKAF M10.1 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 D₃ (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”H₂O) and the dependent variables: *R, DF, X, S, H, Hu, PI, *VPC ("H₂O"), MD (%)

### Table 1: Experimental design of the SD process

<table>
<thead>
<tr>
<th>Run</th>
<th>IAT (°C)</th>
<th>OAT (°C)</th>
<th>ADV (rpm)</th>
<th>VPC (&quot;H₂O&quot;)</th>
<th>MD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.88</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>170</td>
<td>80</td>
<td>28000</td>
<td>1.00</td>
<td>15</td>
</tr>
<tr>
<td>3</td>
<td>150</td>
<td>80</td>
<td>28000</td>
<td>1.88</td>
<td>15</td>
</tr>
<tr>
<td>4</td>
<td>170</td>
<td>90</td>
<td>24000</td>
<td>1.88</td>
<td>5</td>
</tr>
<tr>
<td>5</td>
<td>170</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>6</td>
<td>150</td>
<td>80</td>
<td>24000</td>
<td>1.00</td>
<td>5</td>
</tr>
<tr>
<td>7</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>15</td>
</tr>
<tr>
<td>8</td>
<td>170</td>
<td>90</td>
<td>24000</td>
<td>1.00</td>
<td>15</td>
</tr>
<tr>
<td>9</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>10</td>
<td>170</td>
<td>80</td>
<td>28000</td>
<td>1.88</td>
<td>5</td>
</tr>
<tr>
<td>11</td>
<td>170</td>
<td>90</td>
<td>28000</td>
<td>1.00</td>
<td>5</td>
</tr>
<tr>
<td>12</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.00</td>
<td>10</td>
</tr>
<tr>
<td>13</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>5</td>
</tr>
<tr>
<td>14</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>15</td>
<td>160</td>
<td>80</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>16</td>
<td>160</td>
<td>85</td>
<td>28000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>17</td>
<td>160</td>
<td>85</td>
<td>24000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>18</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>19</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>20</td>
<td>160</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>21</td>
<td>150</td>
<td>90</td>
<td>28000</td>
<td>1.88</td>
<td>5</td>
</tr>
<tr>
<td>22</td>
<td>150</td>
<td>90</td>
<td>28000</td>
<td>1.00</td>
<td>15</td>
</tr>
<tr>
<td>23</td>
<td>170</td>
<td>80</td>
<td>24000</td>
<td>1.88</td>
<td>15</td>
</tr>
<tr>
<td>24</td>
<td>150</td>
<td>85</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
<tr>
<td>25</td>
<td>150</td>
<td>90</td>
<td>24000</td>
<td>1.88</td>
<td>15</td>
</tr>
<tr>
<td>26</td>
<td>160</td>
<td>90</td>
<td>26000</td>
<td>1.44</td>
<td>10</td>
</tr>
</tbody>
</table>
### Table 2: Experimental optimization results of the coconut powder SD process

<table>
<thead>
<tr>
<th>Run</th>
<th>Xc (%)</th>
<th>a (%)</th>
<th>S (%)</th>
<th>H (%)</th>
<th>L* (%)</th>
<th>Vit.C (%)</th>
<th>Vit.D (%)</th>
<th>Vit.E (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.82±0.02</td>
<td>0.27±0.00</td>
<td>39.72±2.39</td>
<td>8.01±0.12</td>
<td>76.01±0.17</td>
<td>20.42±0.26</td>
<td>18.81±0.67</td>
<td>11.18±0.36</td>
</tr>
<tr>
<td>2</td>
<td>2.69±0.05</td>
<td>0.28±0.00</td>
<td>69.45±5.51</td>
<td>9.48±0.17</td>
<td>80.33±0.60</td>
<td>40.03±3.08</td>
<td>8.25±0.98</td>
<td>2.57±0.12</td>
</tr>
<tr>
<td>3</td>
<td>1.59±0.02</td>
<td>0.16±0.00</td>
<td>74.68±5.63</td>
<td>9.95±0.59</td>
<td>78.94±0.56</td>
<td>39.80±1.71</td>
<td>5.53±0.62</td>
<td>2.56±0.27</td>
</tr>
<tr>
<td>4</td>
<td>1.38±0.09</td>
<td>0.26±0.01</td>
<td>58.72±2.18</td>
<td>7.91±0.11</td>
<td>74.63±0.93</td>
<td>31.13±3.56</td>
<td>14.70±1.14</td>
<td>9.35±0.81</td>
</tr>
<tr>
<td>5</td>
<td>1.22±0.06</td>
<td>0.19±0.00</td>
<td>59.79±2.72</td>
<td>8.00±0.08</td>
<td>73.96±1.32</td>
<td>56.80±0.97</td>
<td>32.84±5.58</td>
<td>7.97±1.18</td>
</tr>
<tr>
<td>6</td>
<td>2.38±0.08</td>
<td>0.21±0.01</td>
<td>48.69±1.63</td>
<td>7.95±0.15</td>
<td>73.96±1.32</td>
<td>30.54±2.44</td>
<td>15.01±0.87</td>
<td>12.50±0.79</td>
</tr>
<tr>
<td>7</td>
<td>1.20±0.01</td>
<td>0.15±0.01</td>
<td>63.03±4.88</td>
<td>9.09±0.11</td>
<td>80.87±0.63</td>
<td>33.34±7.87</td>
<td>17.07±0.85</td>
<td>1.87±0.48</td>
</tr>
<tr>
<td>8</td>
<td>1.67±0.01</td>
<td>0.18±0.01</td>
<td>67.18±4.42</td>
<td>9.73±0.07</td>
<td>82.70±0.67</td>
<td>38.55±1.57</td>
<td>20.18±6.52</td>
<td>5.12±1.73</td>
</tr>
<tr>
<td>9</td>
<td>2.02±0.02</td>
<td>0.23±0.01</td>
<td>47.06±2.41</td>
<td>7.84±0.64</td>
<td>77.03±1.35</td>
<td>37.29±9.52</td>
<td>17.81±0.90</td>
<td>3.55±0.29</td>
</tr>
</tbody>
</table>

Additionally, process yield (R) was determined: kg solids from the powder obtained/kg solids from the FE 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...
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 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 3: ANOVA (p-values) for response surface models

<table>
<thead>
<tr>
<th>Variables</th>
<th>Principal effects</th>
<th>Quadratic effects</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$X_w$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$a_w$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S</td>
<td></td>
<td></td>
</tr>
<tr>
<td>H</td>
<td></td>
<td></td>
</tr>
<tr>
<td>L</td>
<td></td>
<td></td>
</tr>
<tr>
<td>%R-Vit C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>%R-Vit D</td>
<td></td>
<td></td>
</tr>
<tr>
<td>%R-Vit E</td>
<td></td>
<td></td>
</tr>
<tr>
<td>%R-Ca</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hu (s)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PI</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DF (%)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Yield (%)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ds</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ds</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Effects of the interaction</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

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 $\zeta$<25 mV, which denotes a negative electric potential in the proximities of the colloid layer formed on the particle interphase and which contribute to increasing the repulsive forces among the particles (Esteinvo et al., 2014; Mirmosseini 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 (Rodriguez-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) (Santhalashmy 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; Santhalashmy 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](image-url)
(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 Ghabadian (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; Yagleama 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% 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 H2O, which favors water adsorption. A synergistic effect is also noted with the Xw 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 Tg (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 (Rodriguez-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 Xw 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
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 OAT², ADV² and MD², 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 Adamopoulou, 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, 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 ($AE$) 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.
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; Rodriguez-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 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; Rodriguez-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 by 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 D₃ 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 D₃ retentions when its content in the FE is high and VPC between 1.2 and 1.4 “H₂O, or vice versa, when MD is low and high VPC (1.8-2.0 “H₂O), 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.D₃ 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.D₃ 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 IAT², ADV² and VPC², which permitted obtaining a variation between 1.5±0.4 and 5.9±0.1 meq H₂O₂/kg oil, corresponding to 1.1±0.1 and 3.32±0.17 meq H₂O₂/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 O₂/kg oil or 7.5 meq H₂O₂/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 Frascaerci 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 H₂O₂/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 O₂ 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: Orljen et al. (2000), reported values of PI<15 meq O₂/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 O₂/kg oil in the microencapsulation of the Nigella sativa L. oil, using as wall material sodium caseinate and MD (dextrrose 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; Fazaéli 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 IAT², MD², 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 IAT², OAT² and ADV², 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 (Tg), 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 Gholbadian (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 (Fazaéli 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
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_{90} 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 ADV² (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 Ghoabadian, 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_0 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: 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_{90}: 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.

---

*Fig. 6: Response surface graphics of particle size D_{90} in function of the independent variables*
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, D3 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: Xw, aw, S, H, L*, a*, b*, Hu, PI, Calcium, Vit.C, Vit.D3, 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 "H2O; MD: 7.0%; and with quality attributes: Xw: 1.7±0.4%; aw: 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 H2O2/kg oil; DF: 32.4±2.3%; *R: 44.0%; D10: 1.70±0.05 µm; D50: 8.46±2.09 µm; D90: 78.18±24.30 µm; Vit. C: 32.4±6.2%; Vit. E: 6.1±1.9%; Vit. D3: 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 O2 to minimize these changes.

REFERENCES


