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Optimization of the spray drying process for the obtaining of coconut powder (*Cocos nucifera* L.) fortified with functionally active compounds

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Abstract

The objective of this work is to contribute to the generation of a significant advance of the coconut agroindustry in Colombia, for which the process of spray drying was optimized to obtain coconut powder added with functionally active components (CP+PAC) (calcium and vitamins C, D_3 and E), food that is framed in the context of functional foods. Initially, the behavior of the physicochemical properties of the coconut during storage at a temperature of 25ºC was evaluated. Then the base emulsion was designed, determining the influence of the composition of emulsions based on coconut milk, on its physicochemical stability, the answer surface methodology was used with a central composite design, considering the independent variables: water/coconut ratio; xantan gum; coconut fiber; terbutilhidroquinona. Subsequently, it was experimentally optimized according to the operating characteristics of the dryer and the product, using a response surface design based on five independent variables: Maltodextrin, air inlet temperature, air outlet temperature, atomizer disk speed and vacuum pressure in the drying chamber. Finally, the stability of the PC+PAC properties was evaluated, using a factorial design based on the independent variables: storage temperature, storage time and packaging.

Keywords: *coconut, colloidal system, deposit formation, yield, vitamins.*

1. Introduction

The coconut is a crop of great importance both economically and subsistence, directly dependent on more than 80 million people in more than 90 countries, in addition to the importance for its nutritional and medicinal values [1]. One of the most used techniques for the production of powders from liquid solutions and suspensions of fruit juices and pulp is spray drying (SD), with powdered food being valuable matrixs in terms of transport, packaging, storage and life useful [2], it is also one of the most effective techniques, which protects, stabilizes, releases the compounds and at the same time allows its solubility in an aqueous medium [3].

The coconut chain is identified as one of the most interesting for the Pacific and Atlantic coasts of Colombia, due to the impact it has on the population, seen from the families that depend on primary production to its commercialization and consumption. For which it is intended to provide the agroindustrial sector, the technological basis for obtaining a variety of dehydrated powders based on coconut and excellent quality attributes, which would represent new alternatives for diversification, in addition to contributing to the reduction of nutritional deficiencies in the population. In this context, this research aims to contribute in the medium to long term to increase the consumption of coconut drinks after reconstitution and to encourage the production of powdered coconut milk (CM) as a raw material for multidominios of the sector of the food industry and national and international markets

The objective of this work is to contribute to the generation of a significant advance of the Colombian agroindustry from the research, which allows the optimization of the process of spray drying (SD) for obtaining coconut powder added with physiologically active components (calcium and vitamins C, D_3 and E) (CP+PAC), food that is part of the context of functional foods.

2. Materials and Methods

Cocos (*Cocos nucifera* L.) variety Malayan Dwarf (manila) or Alto Pacifico (typical) from the Colombian Pacific region were used, with a flowering age at harvest of approximately 12 months and a post-harvest time between 15 and 36 days, time in which through preliminary studies was shown to have acceptable quality to be used as raw material for processing. The whole coconuts to be used were initially washed with water and disinfected with a 200 ppm sodium hypochlorite solution, then the coconut water (CW) was removed and blanched for 20 min in boiling water at T≈ 96°C (local barometric pressure≈ 640 mmHg), subsequently the shell of the coconut pulp (CP) was removed. The selected CP was again subjected to a water washing process and disinfection with hypochlorite, cutting into pieces and grinding (mill TM32 INOX BRAHER 3HP - 16801002).

The characterization of the PC+PAC properties were carried out according to the following methodologies: humidity (%) (X_w): official method AOAC 930.15 [4]; Water activity (a_w): determined with a dew point hygrometer at 25ºC (Aqualab 3TE series, Decagon, Devices,

Pullman, WA, USA) [5]; Solubility (S): method used by Cano-Chauca et al., (2005) modified [6]; Peroxide Index (PI): was performed on the extracted oil, obtained according to the method of Bae and Lee (2008) modified [7], where 4 g of powder was taken. The PI was determined by the spectrophotometric method based on the ability of peroxides to oxidize ferrous ions to ferric ions, which react with various reagents that produce colored complexes [8].

The quantification of vitamins E and D_3 was performed by high performance liquid chromatography (HPLC) (Shimatzu Prominence 20A), using a reverse phase column (C18 - 5 μm 4.6 mm x 250 mm), diode array, mobile phase: acetonitrile/methanol/water (45.3/51.2/3.5), flow: 1 mL/min, oven temperature 40°C and wavelengths of 325 and 265 nm, respectively. The quantification of vitamin C was also performed by HPLC, using a column in reverse phase (C18 RP-5 μm 4.6 mm x 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, wavelength of 244 nm and an injection volume of 5 μL. The extraction of vitamin C was carried out according to the methodology adapted by Peña-Correa et al., (2013) [9]; while the extraction of vitamins D_3 and E was carried out according to the methodology proposed by Cortés (2004) modified [10] by inclusion by ultrasound treatment for 20 min to the samples treated with hexane. Calcium quantification was performed by the spectrophotometry method by flame atomic absorption, according to NTC 4807 (2000), supported by ISO 5151 (2003). The CP+PAC fortification criteria were set within the framework of the declaration of nutritional properties established in Resolution 333 of 2011 (Ministry of Social Protection Colombia), for the purpose of declaring the descriptor "Rich in" or "Excellent source of " CFA in a 100 g serving.

The particle sizes were determined as percentiles D_{10}, D_{50} and D_{90}, using the Mastersizer 3000 (Malvern Instrument Ltd., Worcestershire, UK), after dispersing the samples in 500 mL of distilled water until obtaining a darkening value of 10±1%, considering the size distribution from the theory of Mie and using the refractive index of 1.52 [11]. The color was determined by means of the CIE-La*b* coordinates, using an X-Rite spectrophotometer model SP62, illuminant D65, observer of 10° as reference [5]. Additionally, micrographs of the CP+PAC were made using a scanning electron microscope (Jeol 5910LV) at 15 Kv [6], where the samples were deposited on a copper conductive tape and on a sample holder, then they were coated with gold in a vacuum evaporator (Dentom Vacumm, 30 mA, 5 kV, 100 millitorr).

Preparation of the feed emulsion to the dryer: Batches of 3000 g of dryer feed emulsion (DFE) were prepared, initially a mixture of CP, CW and drinking water with a ratio ((CW+ H_2O)/CP) was homogenized in an Osterizer 600 Watts blender (position III) for 5 min, then the mixture was filtered on a 500 μm mesh screen, separating the fiber from the LC. The fiber was subjected to a drying process at 40°C for 48 hours and then to a dry milling (IKA MF 10.1 mill, USA). The CM was homogenized again in a Silverson series L5

homogenizer using the emulsifying head at 10000 rpm for 10 min, adding the native milled fiber (5-15% w/w) and the rest of the ingredients: milk whey (Instant WPC 80) as surfactant (0.5%), NaCl (9 mMol/L), xanthan gum (0.5-1.0% w/w), terbutilhydroquinone (TBHQ) (100-200 mg/kg) and CFA in the chemical forms of Calcium Citrate powder (6.0 g) (BELL CHEM), Vitamin C (1.0 g) (Ascorbic acid): 99.5% powder, BELL CHEM), Vitamin D_3 (0.5 g) (Colecalciferol): 512900 IU/g powder, BELL CHEM), Vitamin E (1.0 g) (DL-α-Tocopherol Acetate): 50% USP GRADE powder, BELL CHEM). A cooling bath was used during the preparation to control that the temperature of the DFE will not exceed 35°C

Spray drying process: A co-current flow pilot spray dryer was used (Vibrasec, model PASLAB 1.5). The evaluation of the spray drying process was carried out using the response surface methodology with a central composite design, considering the independent variables: Maltodextrin (MD) (5-15%), air entry temperature (AET) (150-170°C), air outlet temperature (AOT) (80-90°C), atomizer disk speed (ADS) (24000-28000 rpm) and vacuum pressure in the chamber (VPC) (1.0-1.88 "H_2O) and the dependent variables: Process performance (R*) kg solids of the powder obtained/kg solids of the DFE; Deposit formation (DF) kg adhered material/kg DFE; Xw; a_w; solubility (S); Retention of the PAC (%); Particle size (D_{10}, D_{50}, D_{90}); PI, color (L*, a*, b*).

Process for assessing the stability of coconut powder during storage: The product was packed in multicoated bags made by Alico SA, laminated film thickness (PET): 12 μm, aluminum foil: 8 μm polyethylene sealant layer: 100 μm, weight of 136.54 g/m^2, with water vapor barrier <1 cc/m_2 x 24h x atm) and O_2 <1 cc/m^2 x 24h x atm). The samples were stored in climatic chambers conditioned at a relative humidity of 65%, with the treatments applied: 15-N_2, 15-Amb, 25-N_2, 25-Amb, 35-N_2 and 35-Amb, at storage times (tA): 0, 30, 60, 90, 120, 150 and 180 days. The dependent variables evaluated were: Xw, a_w, S, color (L*, a*, b*), PI, %R-VC, %R-VD_3, %R-VE, %R-ABTS, %R-DPPH, %R-FT, %R-Ca, particle size (D_{10}, D_{50}, D_{90}) and microstructural analysis.

For both the process of optimization of the emulsion and the drying process, the effect of the independent variables was determined using the multiple regression method for the prediction of the linear, quadratic coefficients and the interaction of the independent variables in the surface models In response, a polynomial model of order 2 (equation 1) was used:

$$\mathbf{Y} = \beta_0 + \beta_A A + \beta_B B + \beta_C C + \beta_D D + \beta_A^2 A^2 + \beta_B^2 B^2 + \beta_C^2 C^2 + \beta_D^2 D^2 + \beta_{AB} AB + \beta_{AC} AC + \beta_{AD} AD + \beta_{BC} BC + \beta_{BD} BD + \beta_{CD} CD \quad (1)$$

Where β_o is a constant, β_A, β_B, β_C y β_D is the linear coefficient of each factor; β_A^2, β_B^2, β_C^2 y β_D^2 is the quadratic coefficient of each factor; β_{AB}, β_{AC}, β_{AD}, β_{BC}, β_{BD} y β_{CD} is the product coefficient of the interactions of the factors. The adequacy of the models was carried out using the lack of fit test and the regression coefficient (R^2); In addition, the analysis of

variance (ANOVA) was performed with a confidence level of 95%. The matrix of the experimental design, the analysis of the results and the optimization procedure were carried out using the software Statgraphics Centurión XVI.I. From the conditions of optimal operation, three additional experiments were carried out to verify the accuracy of the model with respect to the real response variables, in order to verify the regression models and for stability in the storage a design was applied. with a factorial arrangement of the order 6*7 with two independent variables: treatment and time, where the treatment is defined as the combinations (temperature-packaging) and packaging was carried out in N_2 atmosphere and under environmental conditions.

3. Results and discussion

In the 1st phase of physicochemical characterization of the raw material (coconut) to identify the ideal conditions and determine the adequate time for its transformation as a raw material, the results showed a general deterioration of the CP and CW after 36 days of storage, mainly due to the increase in acidity, fermented odor, moisture loss (X_w), lipid oxidation, softening and discoloration of the CP, among others.

In the 2nd phase of design and optimization of the base emulsion, the optimal conditions were: water/coconut ratio (($CW+H_2O$)/CP): 2.0; xanthan gum: 0.5%; Coconut fiber (CF): 5.0%; antioxidant (TBHQ): 200 mg/kg, reaching a potential-ζ: -45.6±2.5 mV; viscosity: 741.7±25.5 cP; color (L*: 67.5±0.7, a*: 3.2±0.2 and b*: 8.6±0.5); Peroxide value (PI): 0.14±0.04 meqH_2O_2/kg; particle size (D_{10}: 4.3±0.8 μm, D_{50}: 323.7±43.6 μm and D_{90}: 743.0±65.1 μm) and total solids (TS): 20.0±0.3%.

The numerical experimental optimization was carried out in order to obtain optimal values for independent variables, thus determining the desirable response parameters that allow obtaining a final product with the appropriate quality attributes. For this case and according to the statistical results found, the criteria were the following: approximate viscosity of 1000 cP (condition of maximum viscosity allowed by the dryer), higher percentage of TS, higher L*, less enzymatic browning and oxidation of the lipids (<IP and <potential ζ). These response variables were significantly influenced by the xanthan gum and %FC, being the variables that tend for a better emulsion behavior, and where the antioxidant becomes a supporting factor that tends to the good performance of the xanthan gum interaction and %FC [12, 13, 14].

In the 3rd phase of optimization of the SD process according to the operational characteristics of the dryer and the product, where the results obtained from the dependent variables and the ANOVA performed, were planned taking into account the most important variables of the process, maximizing S, L*, R*, D_{90} and PAC; minimizing H, Hu, PI and DF, it was also set at an average value of X_w and a_w, since its fluctuations were not very large, where the optimal conditions were: air inlet temperature (AIT): 170°C; Air outlet

temperature (AOT): 85.8°C; Atomizer disk speed (ADS): 26.676 rpm; Vacuum pressure in the chamber (VPC): 1.6 "H2O; percentage of maltodextrin (%MD): 7.0.

Under these process conditions, the experimental values of the response variables were as follows: humidity: 1.7±0.4%; a_w: 0.170±0.020; solubility (S): 58.4±2.1%; hygroscopicity (H): 8.4±0.6%; L*: 79.5±0.9; a*: 1.5 ± 0.1; b*: 9.5 ± 0.5; % vitamin C retention (%R-VC): 32.4±6.2; %R-VE: 6.1±1.9; %R-VD_3: 7.8±1.8; %R-Ca: 41.7±2.9; Wettability (Hu): 263.0±19.8 s; peroxide index (PI): 2.4±1.3 meqH_2O_2/kg fat; Deposit formation (DF): 32.4±2.3%; yield (*R): 44.0±1.0%, and particle size distribution D_{10}: 1.7±0.1 µm; D_{50}: 8.5±2.1 µm; D_{90}: 78.2±24.3 µm; obtaining a product with good quality attributes; Additionally, the proximal composition of PC+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 benefits on consumer health, in addition to its PAC present [15, 16, 17].

And in the 4th phase, it was determined that the best treatment for PC+PAC storage, with respect to all the response variables, was 15°C-N_2, where the humidity gain and the increase in aw were low for this type of products and the percentages of retention of vitamins and antioxidants were the highest, reaching values at 180 days of: X_w: 2.97±0.09%; a_w: 0.342±0.009; color: (L*: 77.96±0.04, a*: 1.44±0.06 and b*: 8.59±0.13); S: 51.48±2.30%; IP: 0.225±0.19 meqH_2O_2/kg fat; %R-VC: 62.56±5.70; %R-VD3: 51.00±0.99; %R-VE: 57.18±3.23; %R-FT: 50.89±4.78; %R-DPPH: 91.88±1.79; %R-ABTS: 42.14±2.18; D_{10}: 2.44±0.12 µm; D_{50}: 51.49±1.48 µm; D_{90}: 153.80±14.0 µm [18, 19] (see figure 1).

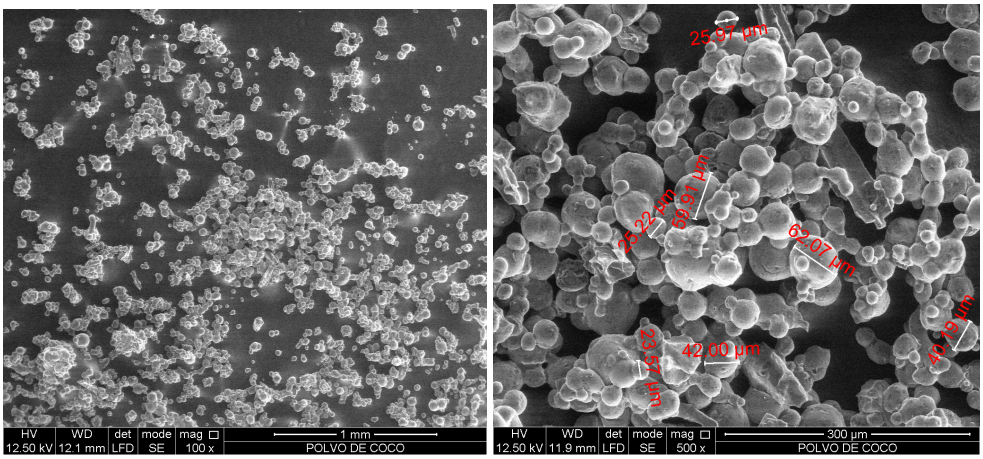

Fig. 1. Micrographs of PC+PAC obtained by SA at 100X and 500X.

4. Conclusions

In general, the intermediate levels of the hydrocolloid and fiber added are those that tend for a better emulsion behavior, the ratio ((CW+H_2O)/CP) although it influences in some

specific cases, it is not very determinant, while the antioxidant becomes in a support factor that tends to the good performance of the hydrocolloid and fiber interaction.

The experimental optimization carried out using statistical tools represents an effective way to define the most appropriate conditions of the SD process, and at the same time represents a significant advance for its subsequent industrial scaling and its potential generation of added value to the coconut agro chain.

According to the behavior of the PC+PAC during the storage this is a hygroscopic product, potentially sensitive to oxidative processes during storage, which could lead to changes in color, flavors or strange odors, so it will require a packing with low permeability to water vapor and O_2, to minimize these changes.

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