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Preservation of vitamin C and phenolic compounds from Umbu(spondias tuberosa arr. Cam.) via spray drying

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Abstract

Umbu is a tropical fruit with high content of bioactive compounds. However, maturation causes significant losses on nutrient density reducing the nutritional value of this highly appreciated fruit. Thus, the objective of the present work was to encapsulate the Umbu fruit bioactive compounds using spray drying and identify the variables affecting the biomolecules preservation. A Box-Behnken experimental design with 3 factors was set varying inlet temperature, atomization flow rate, and maltodextrin concentration for process otimization. Then the powder physicochemical and chemical properties were characterized, and results were modeled using a polynomial equation. Results revealed that the droplet size and maltodextrin concentration had a significant influence on the conservation of the biomolecules. Drying kinetics favoring fast formation of a particle crust increases encapsulation efficiency. Bioactive compounds retention was achieved by increasing maltodextrin even at high temperatures, where a matrix is formed hindering chemical degradation. Process optimization was validated and revealed low inlet temperatures (122ºC), high atomization flow rate (5kg/h) and high maltodextrin concentration (20%) to be the most desirable conditions for bioactive compounds retention.

Keywords: Spray Drying; Umbu; Preservation; Modeling; Optimization

1. Introduction

Recently there has been an increase in interest in bioactive compounds and their presence on human diet, mostly because these compounds help in maintaining the cellular metabolic homeostasis on different tissues[1]. The functionality and preservation of these compounds on foods have influenced the individual choice for healthyl eating. Additionally, properties such as antioxidants, anti-inflammatory, and hypoglycemic present in foods that contain bioactive compounds contribute to preventive health care [2].

Fruits are a food category that is rich in bioactive compounds, principally compounds with antioxidant activity such as polyphenols and vitamins C, along with carotenoids and flavonoids with less extent. Exotic tropical and subtropical fruits respectively present high content bioactive compounds as well[3]. One of those tropical fruits is Umbu (*Spondias tuberosa* Arr. Cam.) which is a typical fruit from the north and northeast Brazil[4]. However, it still is underexploited and has low consumption because of high perishability, which presents a significant challenge that hinders its full potential.

Umbu is highly appreciated mostly because of refreshing and acidic flavors[5]. After harvest, and under ambient temperatures the fruit is conserved for three days, while under refrigeration at 5 ºC lasts 15 days. Nutritional values of Umbu fruit derives from a combination of sugars, phenolic compounds, carotenoids, and vitamin C[6]. Therefore, depending of maturation period the concentration of these bioactive compounds can range from 10-40 mg/100g for vitamin C, 10-90 mg/ 100g for phenolic compounds, 1.5-10μg/g of carotenoids and 7- 40 mg/100g of flavonoids[6,7].

Market expansion and increasing interest in tropical fruits represent an opportunity for the small farmers business to explore tropical fruit economically [8]. However, fruits like Umbu present a difficult challenge for mass distribution and processing due to short shelf-life. In this terms, an excellent alternative to improve consumption and distribution of fruit such as Umbu is the use of industrial drying methods, as the spray-drying process that is known principally due to the efficiency [9]. The advantages of the dried powder over the conventional fruit are the lower storage costs, lower transportation cost, higher concentration and the increased stability of active substances[10].

Spray drying is already widely used for fruit preservation and production of fruit powders from juices[11]. In this process, a product stream is atomized inside a drying chamber where it meets a passing drying gas at high temperatures that convert the product droplets into powder[12]. Typically, the produced powders present low residual moisture and water activity both consider critical for preservation and storage[13]. Because of the high amount of low molecular weight molecules such as sugars, it is often required the use of encapsulation additives to ensure higher yields and lower powder hygroscopic. Also noteworthy is the use of polysaccharides additives to create an encapsulation matrix to increase bioactive compounds retention. Encapsulation additives like maltodextrin have already proven to be beneficial for higher retention of bioactive compounds within the powder by forming a protective matrix or crust around the droplet [11,14].

Over the recent years many studies have focused on spray drying process of common fruit juice such as oranges[15], apples[16], and fruit berries[17]. Reports on spray drying of exotic tropical fruits juice comprisejussara pulp[18], soursop[19], cashew apple[20], cupuassu[21]. However, no work has been studied on umbu (*Spondias tuberosa* Arr. Cam.) spray drying.

Careful selection of spray drying conditions is crucial to obtain high quality products. Therefore the objective of the present work was to spray drying umbu fruit pulp with maltodextrin and assess the influence of the spray drying conditions on the physicochemical, functional and chemical properties of the umbu powder. Additionally, optimization using response surface methodology was used to determine which were the best spray drying parameters to increase bioactive compounds content.

2. Materials and Methods

2.1 Materials

The Umbu (*Spondias tuberosa* Arr. Cam.) was purchased between January and March 2018 from a local market on Campina Grande – Paraiba - Brazil, at a stage of green maturation, which is suitable for pulp preparation. The fruits were hand-picked and visually selected using as standard a similar size, shape, color, and degree of maturation. Maltodextrin DE10 (ingredion, food grade) was used as encapsulation agent. Chemicals used to assay the composition of the blends and powders were purchased from Sigma-Aldrich Chemical Co. (St. Louis, MO, USA).

2.2 Umbu pulp preparation

The Umbus were washed in running water to remove the coarse soils, then sanitized with sodium hypochlorite solution at a concentration of 20 ppm, and then rinsed. Umbu pulp was cooked for 5 min to increase pulp extraction and then homogenized at 500 rpm for 10 min. The resultant pulp was finally sieved using a 1 mm mesh. Maltodextrin was also added accordingly to the design of experiments.

2.3 Spray Drying Process

Spray drying was performed using a LABMAQ model SD-10, with a 2.0 mm two-fluid nozzle and cyclone for powder collection on open loop. Atmospheric air with 0.014 g H_2O / g air was withdrawn without any treatment and blown at a constant drying gas flow rate of 500 kg/h. Umbu pulp, with 13%wt solids content, was pumped at a constant feed flow rate of 4 kg/h. Drying experiments followed a 3^3 Box-Behnken design of experiments with a random sequence. The selected factors were the inlet temperature, atomization flow rate, and maltodextrin concentration. All factors had three levels, where, inlet temperature varied between 110, 140 and 170 ºC, the atomization flow rate varied between 2, 3, and 4 kg/h, and maltodextrin concentration varied between 5%wt, 10%wt and 15 %.

2.4 Analytical Methods

2.4.1 Process yield and moisture content

Process yield was determined by the ratio between the amount of powder collected with the mass of solids in the feed as given by equation 1.

$$Process\ Yield\ (\%) = \frac{Powder\ Collected\ in\ Cyclone}{Total\ Solids \cdot Sample\ volume} \cdot 100$$

(Eq.1)

Solids content and powder moisture content were determined using gravimetric methodology according to the Association of Official Analytical Chemists [22].

2.4.2 Color

Sample color was determined using a MiniScan XE Plus colorimeter with the function COLOR PLOT D65/10º. Browning index was determined by equation 2 [23] .

$$Browning\ Index = \frac{100(x-0.31)}{0.172}$$

(Eq.2)

Where x, is

$$x = \frac{(a^*+1.75L)}{(5.645L+a^*-3.012b^*}$$

(Eq.3)

2.4.3 Particle Size Distribution and Particle Morphology

Scanning electron microscopy(SEM) was used to evaluate both particle size distribution and particle morphology. A TESCAN VEGA 3, operated at 5kV was used without samples coating. SEM images were analyzed using ImageJ to determine the particle size distribution. Bulk density was determined by pouring 30 samples into an empty 100 mL graduated measurement cylinder. The volume was registered, and the ratio of mass with occupied volume cylinder gives the bulk density. Tap density was determined similarly, but the powder samples where tapped 100 times until the new volume was registered.

2.4.5 Chemical composition

The total phenolic content was determined using an adapted Folin-Ciocoateu method [24] where tannic acid is used as a standard and results are expressed as mg tannic acid equivalent / g of samples. Vitamin C content was determined using the method proposed by Freebairn [25] where results are expressed as ascorbic acid equivalents.

2.5 Process modeling

Results were modeled by non-linear regression using a second order polynomial equation. All models were assessed using ANOVA for lack of fit.

$y = \beta_0 + \beta_1 \cdot Ti + \beta_2 \cdot Ti^2 + \beta_3 \cdot F_a + \beta_4 \cdot F_a^2 + \beta_5 \cdot C_m + \beta_6 \cdot C_m^2 + \beta_7 \cdot Ti \cdot F_a + \beta_8 \cdot Ti \cdot C_m + \beta_9 \cdot F_a \cdot C_m$ (Eq.4)

Where, Ti is the inlet temperature, F_a the atomization flow rate and C_m the maltodextrin concentration.

3. Results and Discussion

A Box-Behnken design of experiments was used to establish and optimize microencapsulation of umbu fruit bioactive compounds using spray drying. The rationale for choosing this type of design is related with its proven efficiency when compared to other experimental designs[26]. Table 1 presents the results of the powder characterization.

(insert table 1)

Results were modeled using a second-order polynomial equation to understand the influence of each process parameter and the combination of parameters on powder properties. The results of the fitting are presented in table 2.

(insert Table 2)

3.1 Process Yield

Process yield is calculated by the ratio of the mass of powder collected with the mass of total solids fed into the spray. Yield is an crucial process response because it indicates the process performance. Process yield varied between from 27.63 % to 75.33 % depending on process conditions. The results are satisfactory for a process in developing and in a range that corroborates with other fruit dehydration processes. Low process yield reflects the powder accumulation inside the drying chamber. Considering the results presented in table 2, process yield is significantly influenced by both linear and quadratic atomization flow rate ($p < 0.05$).

Both parameters influence the droplet size because increasing atomization flow rate produces smaller droplets then lower atomization flow rate. Authors have related that powder accumulation can occur due to the deposit of wet droplets, which are not sufficiently dry when hit the wall, or because of the material properties at drying temperatures . The size of the droplet influences yields suggesting that larger droplets hit the walls still wet and stick, while smaller droplets do not. Thus, when higher atomization flow rates are used, droplets have higher velocity forming a smaller angle and thus hit the wall at a lower region with less moisture. Similar results were obtained by [27] when spray drying black mulberry juice and [28] when drying xylooligosaccharides.

$$yield(\%) = 75.04 * F_a - 1482 * F_a^2$$

(eq.5)

3.2 Residual moisture

Residual moisture has a direct influence on water activity and thus on powder chemical and microbiological stability. The residual moisture varies between 6.62 % to 3.41 % depending on process inlet temperature. These results corroborate with the range observed in other works with fruit juices [29]. After fitting the polynomial equation, our results revealed that linear inlet temperature and quadratic inlet temperature have a significant effect on residual moisture ($p <$ 0.05). Quadratic coefficients reveal a peak within the range of temperatures used. A minimum moisture content is obtained for 140 ºC while for 110 ºC, and 170 ºC results reveal higher values. Higher moisture contents at 110 ºC inlet temperature are due to higher relative humidity on drying gas resulting in a lower mass transfer from the droplet into the drying medium. As for 170 ºC, a possible justification may be the fast formation of a crust on the droplet surface that hinders water diffusion, and thus the powder retains more water. This factor mostly occurs when maltodextrin concentration is also high because both parameters favor the formation of a thicker wall .

$$Residual\ Moisture\ (\%) = 52.43 - 0.67 * T_i + 0.002 * T_i^2$$

(eq.6)

3.3 Color and Browning index

Color is an important factor for acceptability because it rules visual attractiveness[30]. After initial processing, umbu fruit pulp was light greenish with a* =1.85, b* = 22.49 and L = 52.15. Similar values were reported by [31] After processing, powder samples revealed a* values varying from 0.59 to 1.44, b* values from 10.57 to 19.36 and L values varying from 79.46 to 82.22. From modeling, linear and quadratic maltodextrin concentration had a significant influence on L values. For b* values, quadratic temperature, linear and quadratic atomization flow rate and linear and quadratic maltodextrin concentration had a significant influence on response. For a* values, no independent variable had significant influence. An increase in L is expected with higher maltodextrin concentration because maltodextrin is white and because of Umbu concentration decreases. Similar results were found for purple sweet potato[32]. b* values on powder samples are smaller than the unprocessed pulp reflecting a loss on yellowness due to processing. However, high inlet temperature(170 ºC) do not reduce powder yellowness as much as it occurs at lower temperatures(110 ºC). Temperature influences b* values possibly due to chemical degradation reactions that turn food brown. Browning index reveals the purity of color brown and is considered an important parameter in processes where enzymatic or non-enzymatic browning reactions might occur. Model analyses returned that almost every parameter was significant on the browning index. Higher inlet temperature reveals higher browning index possibly because of higher browning reaction kinetics. Green pigments (chlorophyll) are thermally unstable and form a brown pheophytin due to loss of Mg under acidic conditions. However, when maltodextrin concentration is also high, less browning is verified. A possible justification can be the protection from temperature and oxygen offered by the maltodextrin crust at the droplet surface[33]. When atomization flow rate is assessed, smaller particles become less brown at higher temperatures, while at lower temperatures the browning index is similar for any atomization flow rate. This increase in browning index can be a result from higher moisture content or water activity on larger droplets that increases browning reactions kinetics.

$$L = 61.92 + 0.08 * Cm - 0.02 * Cm^2$$

(eq.7)

$$b = 0.001 * T_i^2 + 10.16 * F_a - 1.17 * F_a^2 - 1.07 * Cm + 0.07 * Cm^2$$

(eq.8)

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$Browning\ Index = 0.001 * T_i^2 + 13.25 * F_a - 1.46 * F_a^2 - 1.5 * Cm + 0.12 * Cm^2 - 0.01 * T * Cm - 0.17 * F_a * Cm$ (eq.9)

3.4 Particle size and morphology

Properties like reconstitution, handling or chemical stability can be influenced by smaller particles sizes[34]. The diameter of one hundred particles was determined directly from SEM micrographs of each sample and repeated three times. Particle size varied from 5.2 µm to 9.3 µm mostly influenced by atomization flow rate as expected. The range of particle size is similar to other works using a two-fluid nozzle which results in smaller particles then hydraulic nozzles[35]. The atomization flow rate parameter was initially chosen to influence droplet size during drying. Thus, linear and quadratic coefficients have a significant influence on particle diameter as expected. Droplet formation results from an energy balance between atomization gas kinetic energy and the cohesive liquid energy like viscosity. Therefore, if liquid viscosity is the same, then higher atomization flow rate creates smaller droplets. Inlet temperature has some influence on particle size distribution due to the ballooning effect. From our results maltodextrin concentration has no evident influence on particle size. Analyzing figure 1 some degree of agglomeration is verified especially at lower temperatures. The agglomeration at lower temperatures can be related to higher moisture content which leads to particle bridging due to hydrogen bonds between water hydroxyl groups[36]. No cracks or fissures were detected at particles surface suggesting that water diffusion was never catastrophic to the particle surface.

$D_N 50(\mu m) = -8.2 * F_a + 1.6 * F_a^2$ (eq. 10)

3.6 Vitamin C content

Bioactive compounds preservation during a thermal process is a critical factor when assessing the impact of process parameters. Vitamin C is one of the most important nutrients present on umbu fruit pulp and one of the most important on human diet because humans are unable to synthesize Vitamin C. Values ranging between 10-40 mg of vitamin C per 100 g of pulp are often found and in our work an initial value of 25.2 ± 0.1 mg /100g was found. After spray drying, the powder presented vitamin C content ranging from 9.0 to 22.8 mg/ 100g meaning that retention ranged from 36% to a maximum of 90%. Modeling revealed the linear inlet temperature and maltodextrin concentration and the linear interaction between the inlet temperature and maltodextrin concentration to influence vitamin c content significantly. Three response surfaces were generated from the fitted polynomial equation and are presented on figures 2a-c. The negative influence of temperature can be attributed to increased degradation kinetics that results in vitamin C loss. Similar results were found for mandarin (*Citrus unshiu*)[37] Influence of maltodextrin can be attributed to its higher molecular weight and hydrogen bonding interactions that reduces vitamin C diffusion into the droplet surface thus reducing the exposure to air and temperature[38]. Then, the interaction between temperature and maltodextrin can be attributed to the formation of a crust on the droplet surface that is favored by high drying temperatures and maltodextrin concentration. This barrier becomes impermeable to labile molecules thus increasing its retention. Therefore, according to our results, it can be expected higher retention when maltodextrin concentration is high. Considering the atomization flow rate, it was found that droplet size has a low impact on vitamin C retention.

$$Vitamin\ C\ content\ (mg/100mg) = -121.74 + 1.26 * T + 2.76 * Cm \quad \text{(eq.12)}$$

3.8 Phenolic compounds content

Phenolic compounds are another category of bioactive compounds important on food quality and human diet thus maintaining high content levels is critical for product quality. Umbu pulp typically presents values ranging from 10-90 mg/100g while in our work value of 48 mg/100g was found for the unprocessed pulp. A higher decrease of phenolic compounds was found, with values ranging from 4.66 to 10.25 mg/ 100g. Analyzing the regression coefficients almost every factor was significant ($p < 0.05$) for phenolic compounds content. The relation between factors influencing phenolic compounds content is depicted at figures 2d-f. Similarly to vitamin C, higher inlet temperatures cause chemical degradation of phenolic compounds. [39] using microwave-assisted extraction found that temperatures above 150 ºC cause a significant loss on most phenolic compounds, while at temperatures ranging 100° to 125 ºC the loss was irrelevant. Higher atomization flow rate reduces phenolic compounds possibly because of increased surface area contacting heated drying air [40]. However, an interaction between atomization flow rate and maltodextrin concentration was also determined by regression meaning that larger droplets do not require as much maltodextrin as a smaller droplet to maintain high contents of phenolic compounds. This result suggests molecular diffusion can be a very relevant factor on maintaining high phenolic compounds content. Higher phenolic contents also suggests that intermolecular interactions with maltodextrin can be beneficial on stabilizing phenolic compounds by encapsulation as propose by [14].

$$Phenolic\ Content\ (mg/100mg) = 42.12 - 0.34 * Ti + 0.001 * T^2 - 8.26 * F_a + 0.99 * F_a^2 - 0.03 * Cm^2 \quad \text{(eq.13)}$$

3.9 Process optimization

On our work, the desirability approach was used for three optimizations each focusing three different goals. First, process yield was maximized, then a second run was performed where powder properties were maximized, and finally, on a third run, the bioactive compounds retention were maximized. The results for all objectives are presented in table 3. All goals reached desirability's above 0.9. Analyzing results, it is clear that mid inlet temperatures favor all objectives, while the atomization flow rate has different behaviors depending on the maximization goal. Maltodextrin concentrations above 10% also maximize all goals. The results obtained after process optimization reflect the analyses provided on process assessment. Validation experiments were preformed to confirm the optimization results and are summarized in table 3. Results from validation experiments are similar to the obtained values when using the optimization function for each of the objectives. Therefore, the optimization can be trusted.

4. Conclusions

Umbu fruit pulp was successfully spray dried in the presence of maltodextrin to maintain high bioactive compounds content by encapsulation. By analyzing the influence of the process parameters through mathematical modeling, it was possible to identify the most influencing factors for powder physicochemical, sensory, and functional properties. Results also indicate that maltodextrin creates a matrix of molecular interactions that hinder chemical degradation of vitamin C and phenolic compounds. Process optimization was successfully validated and

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revealed that mid inlet temperatures (122 ºC) along with high atomization flow rate (5 kg/h) and high maltodextrin concentration (20 %) provide the highest bioactive compounds retention possible.

Conflict of interest

The authors have no conflict of interest to declare.

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Table 1 – Summary of spray drying process conditions and powder properties

Inlet Temperature (° C)	Atomization Flow Rate (kg/h)	Maltodextrin Concentration (%)	Yield (%)	Residual Moisture Content (%)	L	a*	b*	Browning Index	D_N50 (µm)	Vitamin C* (mg/100g)	Total Phenolic compounds (mg/100g)

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110 ± 2	2.0 ± 0.1	10.0	53.9	6.62 ±0.01	79.5 ± 0.3	0.59 ±0.05	10.6± 0.2	14.49	7.35	9.02 ± 0.11	8.63 ± 0.63
170 ± 3	2.0 ± 0.1	10.0	30.8	4.20 ± 0.02	80.0 ± 0.4	1.18± 0.01	15.3 ± 0.1	21.79	9.27	13.98 ± 0.24	10.25 ± 0.39
110 ± 2	4.0 ± 0.2	10.0	56.8	6.66 ± 0.01	81.9 ± 0.2	0.49 ± 0.05	12.5 ± 0.1	16.65	5.71	17.55 ± 0.28	8.02 ± 0.12
170 ± 3	4.0 ± 0.2	10.0	49.5	4.16 ± 0.05	79.9 ± 0.3	0.89 ± 0.05	15.6 ± 0.1	21.97	6.04	16.04 ± 0.37	8.91 ± 0.25
110 ± 2	3.0 ± 0.1	5.0	72.7	6.25 ± 0.06	79.4 ± 0.2	0.69 ± 0.01	15.6 ± 0.2	21.94	4.87	11.15 ± 0.33	6.67 ± 0.32
170 ± 3	3.0 ± 0.1	5.0	47.5	5.55 ± 0.01	77.3 ± 0.5	1.49 ± 0.05	19.4 ± 0.5	29.62	6.08	18.19 ± 0.34	7.44 ± 0.18
110 ± 2	3.0 ± 0.1	15.0	68.9	5.86 ± 0.04	83.6 ± 0.2	0.33 ± 0.11	14.0 ± 0.7	18.19	5.22	22.84 ± 0.69	7.64 ± 0.10
170 ± 3	3.0 ± 0.1	15.0	39.4	5.48 ± 0.03	83.2 ± 0.3	0.95 ± 0.06	16.6 ± 0.2	22.54	5.27	17.26 ± 0.18	7.14 ± 0.29
140 ± 2	2.0 ± 0.1	5.0	27.6	3.65 ± 0.01	79.1 ± 0.2	0.84 ± 0.02	15.4 ± 0.1	21.86	7.12	17.01 ± 0.25	8.89 ± 0.19
140 ± 2	4.0 ± 0.2	5.0	56.4	3.64 ± 0.09	78.0 ± 0.1	1.25 ± 0.06	16.4 ± 0.1	24.27	5.95	15.53 ± 0.67	4.67 ± 0.14
140 ± 2	2.0 ± 0.1	15.0	32.5	3.53 ± 0.04	81.1 ± 0.3	1.37 ± 0.02	13.7 ± 0.2	19.27	8.33	20.17 ± 0.28	6.70 ± 0.11
140 ± 2	4.0 ± 0.2	15.0	75.3	3.44 ± 0.02	82.0 ± 0.1	1.44 ± 0.03	13.0 ± 0.1	18.23	5.98	18.29 ± 0.18	7.54 ± 0.34
140 ± 2	3.0 ± 0.1	10.0	68.4	3.41 ± 0.02	81.2 ± 0.1	1.06 ± 0.01	14.1 ± 0.1	19.58	5.39	18.88 ± 0.72	6.72 ± 0.10
140 ± 2	3.0 ± 0.1	10.0	66.3	3.54 ± 0.05	81.3 ± 0.1	1.07 ± 0.01	14.1 ± 0.1	19.56	5.42	19.82 ± 0.22	6.69 ± 0.09
140 ± 2	3.0 ± 0.1	10.0	69.9	3.45 ± 0.02	82.1 ± 0.2	1.06 ± 0.01	14.0 ± 0.1	19.30	5.39	19.22 ± 0.13	6.72 ± 0.09

***(Ascorbic Acid Equivalents)**

Table 2 – Responses coefficients of data fitting by non-linear regression

	Yield	Residual Moisture Content	L	a*	b*	Browning Index	D_N50	Vitamin C*	Total Phenolic compounds

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intercept	-131.97	**52.425 ***	**61.923 ***	-7 992	4 908	2 474	11 992	**-121.739 ***	**42.115 ***
T	1 084	**-0.674 ***	0.079	0.117	-0.059	-0.012	0.046	**1.256 ***	**-0.340 ***
T^2	-0.006	**0.002 ***	-0.0002	-0.0004	**0.001 ***	**0.001 ***	0.0001	-0.003	**0.001 ***
F	**75.037 ***	0.991	8 334	0.150	**10.164 ***	**13.248 ***	**-8.204 ***	22 967	**-8.262 ***
F^2	**-1482 ***	-0.140	-1 009	0.042	**-1.173 ***	**-1.458 ***	**1.600 ***	-2 385	**0.985 ***
Cm	3 525	-0.273	**0.077 ***	-0.007	**-1.065 ***	**-1.502 ***	0.601	**2.760 ***	0.165
Cm^2	-0.218	0.009	**-0.018 ***	0.005	**0.069 ***	**0.115 ***	-0.006	0.033	**-0.029 ***
T*F	0.132	-0.0008	-0.022	-0.002	-0.014	-0.017	-0.013	-0.054	-0.00607
T*Cm	-0.007	0.0005	0.003	-0.0003	-0.002	**-0.006 ***	-0.002	**-0.021 ***	-0.002
F*Cm	0.705	-0.004	0.099	-0.017	-0.084	**-0.172 ***	-0.059	-0.020	**0.253 ***
R^2	86.46	91.92	92.20	73.54	98.77	99.53	96.55	83.86	96.35
Adj R^2	57.18	77.38	78.18	25.91	96.58	98.68	90.36	54.82	89 772
f	31.14	68.73	9890	12.76	1700.3	1942.3	2896.9	57.95	354.46
p	0.002	0.0005	<0.00001	0.01	<0.00001	<0.00001	<0.00001	0.0006	0.00002
SSE	909.17	1.81	8.18	0.502	4.34	209.1	2.71	1.30	0.009
RMSE	12.3	0.54	1.21	0.28	0.85	5.90	0.67	0.47	0.039
P (%)	13.4	5.07	0.69	19.29	2.24	7.11	4.15	15.86	2.82

T – Temperature; F- Atomization Flow rate; Cm- Maltodextrin concentration; * – significant for $p < 0.05$

Table 3 - Criteria for optimization of Umbu powder properties and process, most desirable solutions and validation

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Factor	Lower Limit	Upper Limit	Desirability Lower Limit	Desirability Upper Limit	Factor settings / Predicted responses	Factor settings / Predicted responses	Factor settings / Predicted responses	Validation Experiment Settings	Validation Experiment Settings	Validation Experiment Settings
Inlet temperature (ºC)	100	200	-	-	114	132	122	115	130	120
Atomization Flow Rate (kg/h)	1	5	-	-	3.32	1.24	5	3.3	1.2	5
Maltodextrin Concentration (%)	0	20	-	-	11.64	14.68	20	11.5	15	20
Responses										
Yield (%)	27.6	75.3	0.0	1.0	74.86	-	-	**70.3**	26.01	44.1
Residual Moisture Content (%)	3.4	6.6	1.0	0.0	-	3.51	-	5.64 ± 0.05	**3.9** ± **0.04**	4.87 ±0.07
Browning Index (%)	14.49	29.61	1.0	0.0	-	15.02	-	19.2	**15.23**	18.87
Vitamin C Content (mg/100g)	9.02	22.84	0.0	1.0	-	-	23.04	19.87 ± 0.43	20.3 ± 0.22	**22.33** ± **0.12**
Phenolic Comp. Content (mg/100g)	4.66	10.25	0.0	1.0	-	-	12.58	7.34 ± 0.34	6.6 ± 0.45	**9.83** ± **0.67**
Desirability	-	-	-	-	0.99	0.90	1.0	-	-	-

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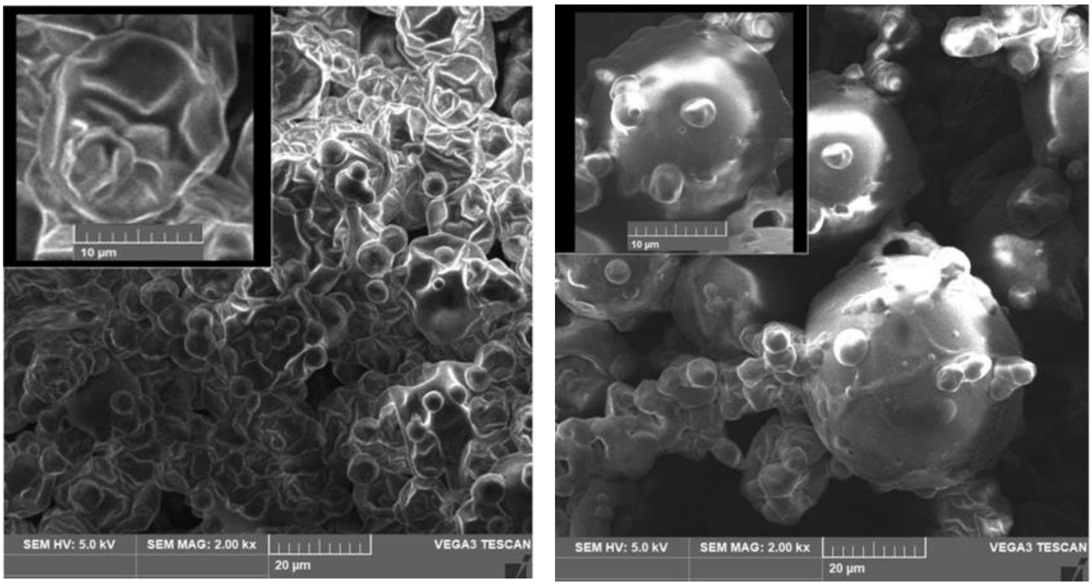

(A) (B)

Figure 1 – SEM micrographs of (A) sample 1: 110ºC, 2kg/h and (B) sample 2: 170ºC, 2kg/h

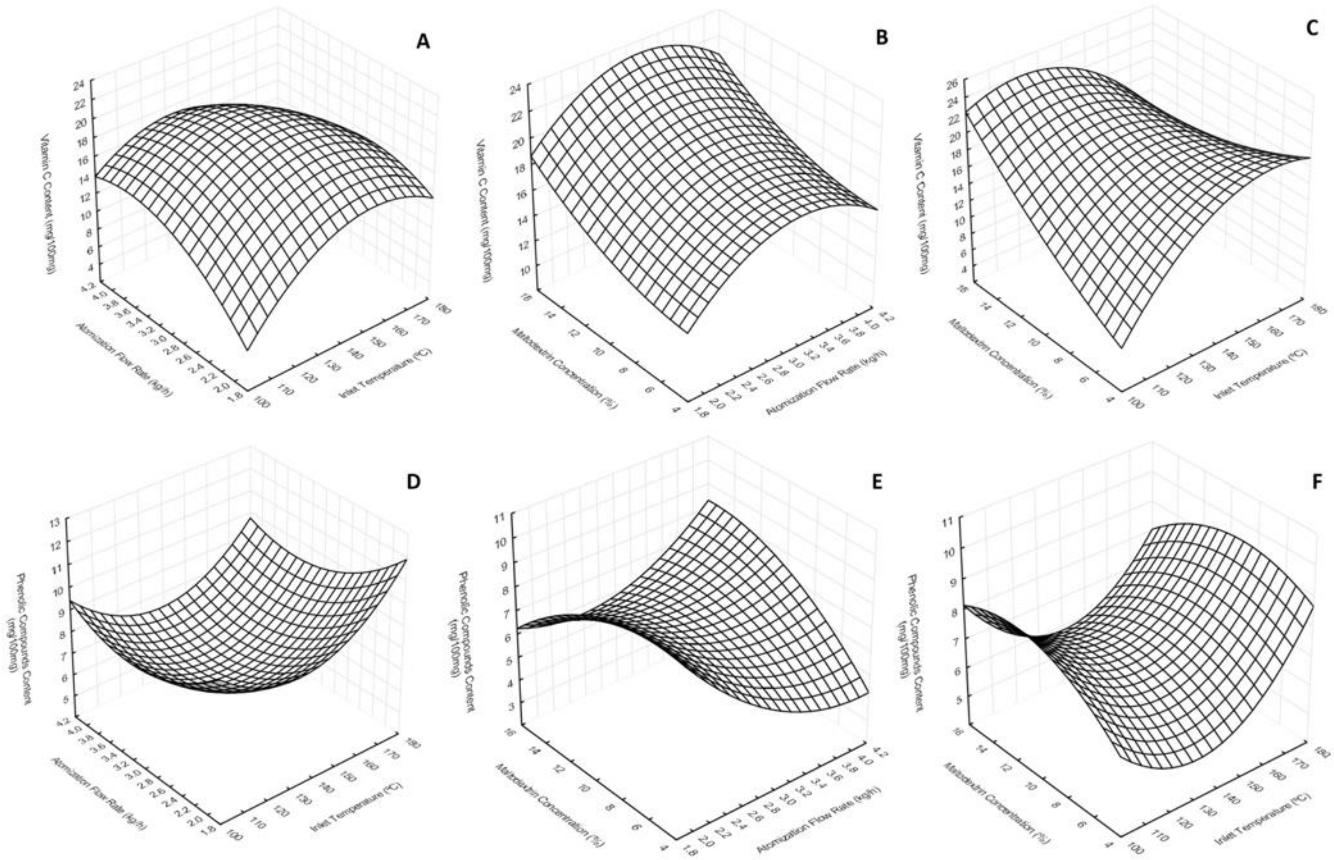

Figure 2 – Response surface curves for vitamin C content for (A) 10% Maltodextrin (B) 140ºC Inlet temperature (C) 3kg/h atomization flow rate and for phenolic compounds contents for (D) 10% Maltodextrin (E) 140ºC Inlet temperature (F) 3kg/h atomization flow rate