

SPRAY DRYING OF COFFEE LEAF EXTRACT

Jefferson Luiz Gomes Corrêa¹, Kamilla Soares de Mendonça², Leonardo Reis Rodrigues³,
Mário Lúcio Vilela Resende⁴, Guilherme Eurípedes Alves⁵

(Recebido: 30 de setembro de 2015; aceito: 23 de fevereiro de 2016)

ABSTRACT: *The coffee leaf extract formulation has been used as resistance inductor in plants and to control phytopathologies. This work aimed to study the influence of spray drying process variables with the use of maltodextrin as carrier on the characteristics of the powder, by using a central composite rotational design (CCRD). The independent variables were maltodextrin concentration (X_1 , 0 to 30 % w/v), coffee leaf extract concentration (X_2 , 2 to 32 % w/v), inlet air temperature (X_3 , 180 to 250 °C) and air flow rate (X_4 , 3.5 to 5.5 m³min⁻¹). The response variables were collection efficiency (η), moisture content (MC), phenolic compounds content (Ph), soluble solids (S) content, wettability (We) and particle size (Me). The results showed that the combination of a higher concentrations of coffee leaf extract (X_2) (32%) and lower inlet air temperatures (X_3) (180°C) make the best drying performance. This process condition lead to a powder with higher Ph, S, We, Me and lower MC. Therefore, the use of high air flow rates (X_4) increase the collection efficiency (η) of process and the use of low maltodextrin concentration (X_1) lead to better preservation of phenolic compounds content (Ph) on coffee leaf extract powdered.*

Index terms: NEFID, resistance inductor, CCRD, maltodextrin.

SECAGEM POR SPRAY DRYING DE EXTRATO DE FOLHA DE CAFÉ

RESUMO: *A formulação de extrato de folhas de café foi utilizada como indutor de resistência em plantas e no controle de fitopatologias. Objetivou-se, neste trabalho, estudar a influência das variáveis do processo de secagem por spray drying, com o uso de maltodextrina como agente carreador, nas características do pó, através do uso de um delineamento central composto rotacional (DCCR). As variáveis independentes foram a concentração de maltodextrina (X_1 , 0 a 30 % p/v), concentração de extrato de folhas de café (X_2 , 2 a 32 % p/v), temperatura de entrada do ar (X_3 , 180 a 250 °C) e taxa de fluxo de ar (X_4 , 3.5 a 5.5 m³min⁻¹). As variáveis resposta foram eficiência de coleta, conteúdo de umidade (MC), conteúdo de compostos fenólicos (Ph), conteúdo de sólidos solúveis (S), molhabilidade (We) e tamanho da partícula (Me). Os resultados mostraram que a combinação de altas concentrações de extrato de folha de café (X_2) (32%) e baixas temperaturas de entrada de ar (X_3) (180°C) proporcionaram as melhores performances de secagem. Essa condição de processo produz pós com maiores Ph, S, We, Me e menores MC. Além disso, o uso de altas taxas de fluxo de ar (X_4) aumentam a eficiência de coleta do processo e o uso de baixas concentrações de maltodextrina (X_1) promovem uma melhor preservação dos compostos fenólicos (Ph) nos pós de extrato de folha de café.*

Termos para indexação: NEFID, indutor de resistência, DCCR, maltodextrina.

1 INTRODUCTION

Brazil is the biggest coffee producer and exporter with approximately 30 % of the world production (FOOD AND AGRICULTURE ORGANIZATION CORPORATE STATISTICAL DATABASE - FAOSTAT, 2015). Besides, it is the biggest exporter of extracts, essences and coffee concentrates. The vegetables extracts are used for treating plant diseases. Coffee leaf extract has shown a great performance on protecting coffee trees and other plants (CARVALHO et al., 2008; MEDEIROS et al., 2009).

Coffee leaf extract formulation (NEFID) is a liquid and diluted product, storage at low temperatures (MEDEIROS et al., 2009; PEREIRA et al., 2008) that activate defense-related genes in plants. The possibility of converting this extract to

a solid product carries out the advantages of better transport, conservation and handling conditions. Drying is an appropriated operation for lowering the moisture content, volume and weight of biological products (CORRÊA et al., 2011, 2012; FANTE et al., 2011; ISQUIERDO et al., 2013).

Spray drying is a process commonly used for drying liquid products like coffee, milk, fruit juices, pharmacological products, among others (AL-MANSOUR; AL-BUSAIRI; BAKER, 2011; BOWEY et al., 2013; CAL; SOLLOHUB, 2010; CARNEIRO et al., 2013; CATELAM; TRINDADE; ROMERO, 2011; FANG; BHANDARI, 2012; FERRARI et al., 2013; MARQUES et al., 2014b; TONON et al., 2009). The process consists in a high temperature exposure that promotes a quickly drying. The spray drying

^{1,2,3}Universidade Federal de Lavras/UFLA - Departamento de Ciência dos Alimentos/DCA - Cx. P. 3037- 37.200-000 - Lavras - MG
jefferson@dca.ufla.br, keamendonca@msn.com, leoreis@ig.com.br

⁴Universidade Federal de Lavras/UFLA - Departamento de Fitopatologia/DFP - Cx. P. 3037- 37.200-000 - Lavras - MG - mlucio@dfp.ufla.br

⁵Universidade Federal de Lavras/UFLA - Departamento de Engenharia/DEG - Cx. P. 3037- 37.200-000 - Lavras - MG guialves@gmail.com

is recommended to products with thermo sensible substances and flavor and aromatic products. Among the carriers, maltodextrin is largely used by its low cost, high solubility, low viscosity and high protection against oxidation (CARNEIRO et al., 2013; FERRARI et al., 2013).

There is to a lack of published results on spray drying of extract of coffee leaf. This work aimed to evaluate the influence of inlet air temperature, air flow rate, coffee leaf extract concentration and maltodextrin concentration on the characteristics of this powder.

2 MATERIAL AND METHODS

2.1 Material

The coffee leaf extract acquired from the coffee (*Coffea arabica* L. cv. Mundo Novo), from Lavras, INCT-CAFÉ/UFLA, Brazil. The soluble compounds were extracted at 120 °C and 1.2 atm in distilled water. The solution was concentrated in a vacuum evaporator (50 °C, 600 mm de Hg), filtered in a 400 mesh sieve and stored at -20 °C (MEDEIROS et al., 2009). Before spray drying, the product was heated at 40 °C and mixed, for 15 min, with maltodextrin DE 10 according to the defined proportions in the experimental design.

2.2 Spray dryer

The spray dryer was in a pilot scale with cylindrical chamber height 1.05 m, 0.80 cm diameter and total height 2.40 m (Labmaq do Brasil LTDA., model SD 5.0, Ribeirão Preto, Brazil). The equipment could work with air flow rate until 5.5 m³min⁻¹, inlet air temperature from ambient to 250 °C, product flow rate until 5 L h⁻¹ and compressed air flow rate until 100 L min⁻¹. The dried product was collected in a 20 cm Lapple cyclone coupled to the spray-dryer. The spray drying was performed with 5 L h⁻¹ of product flow rate in all treatments.

2.3 Experimental design

A central composite rotational design (CCRD) was applied to determine the influence of spray drying process variables on characteristic of powder. A 2⁴ factorial design was used, consisting of 16 assays (-1.00 and +1.00 levels), 4 central points and 8 axial points (-2.00 and +2.00 levels), resulting in an orthogonal distribution for 28 experiments (RODRIGUES; IEMMA, 2012). The maximum and minimum values of each level were established by pre-tests. Such pre-tests emphasized

the common range of process variables (flow rate and temperature) and concentration of the wall and core materials, maltodextrin and coffee leaf extract, respectively (BOTREL et al., 2014; MURALI et al., 2014). The independent variables were maltodextrin concentration X₁ (% w/v), coffee leaf extract concentration X₂ (% w/v), inlet air temperature X₃ (°C) and air flow rate X₄ (m³min⁻¹), the factor levels are provided in Table 1. The responses variables were collection efficiency (η), moisture content (MC), phenolic compounds content (Ph), soluble solids content (S), wettability (We) and particle size (Me). The experimental data obtained were analyzed by the response surface regression procedure using the following second-order polynomial (Eq. (1)):

$$Y_i = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_4 X_4 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{44} X_4^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{14} X_1 X_4 + \beta_{23} X_2 X_3 + \beta_{24} X_2 X_4 + \beta_{34} X_3 X_4$$

where Y_i is the response variables presented in Table 2, β₀ is a constant, β₁, β₂, β₃ and β₄ are linear terms, β₁₁, β₂₂, β₃₃ e β₄₄ are quadratic terms. The other terms are interaction coefficients between the variables. The regression analysis and analysis of variance (ANOVA) of response variables of each experiment were analysed using Statistica (STAT-SOFT, 2010). The effects of the variables were considered statistically significant with p < 0.10.

2.4 Analytical Methods

The yield of the process was estimate for the collection efficiency (η), which was calculated as a ratio between the dehydrated dry weight of the powdered coffee leaf extract obtained to the dry mass of extract on the inlet solution (MARQUES et al., 2014b).

The moisture content was gravimetrically determined in an oven at 105 °C (ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS - AOAC, 2010).

The wettability test was based on the an adaptation of the Marques et al. (2014a) methodology, in which 1.0 g of powder was spread over 100 mL of distilled water at 25 °C without stirring. The test was performed in a 250 mL beaker. The time required for the powder particles to sediment, or sink and disappear from the surface of the water, was measured and used for a relative comparison between the samples.

TABLE 1 - Levels of independent variables of the CCRD for spray drying of coffee leaf extract formulation.

Variable	Level				
	-2	-1	0	1	2
Maltodextrin concentration [%, m/V]	0.0	7.5	15.0	22.5	30.0
Coffee leaf extract concentration [%, m/V]	2.0	9.5	17.0	24.5	32.0
Inlet air temperature [°C]	180.0	197.5	215.0	232.5	250.0
Air flow rate [m ³ min ⁻¹]	3.5	4.0	4.5	5.0	5.5

The solubility was determined using the method described by Cano-Chauca et al. (2005) in which 100 mL of distilled water was transferred to a beaker and agitated in a homogenizer at 2500 rpm. Then, 1 g of powder (dry basis) was gently added to the water, and the agitation was maintained for five minutes. The solution was transferred to a tube and centrifuged for five minutes at 2600 rpm. One aliquot (25 mL) of the supernatant was transferred to a petri dish and dried for five hours at 105 °C. The percent solubility (mass of powder/volume of solution) was calculated based on the weight difference.

The particle size distribution was measured using a laser light diffraction instrument (Mastersizer 2000, model Hydro 2000 MU; Malvern Instruments, Malvern, UK). A small powder sample was suspended in isopropyl alcohol at 92.8% under agitation for 10 minutes (MARQUES et al., 2014b). Particle size distribution was monitored, 5 measurements were done with 10 seconds for each sample. The granulometric composition was determined using the theory of Fraunhofer and the model of spherical particle were used.

The determination of phenolic compounds content was according to the method Folin-Denis, proposed by AOAC (2010).

3 RESULTS AND DISCUSSION

The results obtained according to a rotatable central composite design of the coffee leaf extract powder were provided in Table 2 and the coefficients of the statistical model (Equation 1), on Table 3. According to Rodrigues and Lemma (2012), the statistical model was able to predict response variable behavior only for particle size diameter (Me) ($F_{calc} > F_{tab}$), with satisfactory determination coefficient ($R > 0.70$). The other response variables were discussed based on their statistic significant effects.

The collection efficiency (η) was in the

range 31.73 to 67.62 %. It was observed that η was significantly positively dependent on air flow rate in the linear term and negatively dependent on interaction coffee leaf extract concentration and air flow rate. Increasing air flow rate, the relation air/wet particles were increased and the drying is improved and it is easier to obtain the dried particles. According to Wang and Langrish (2009), the collection efficiency of a spray-drying process is highly affected by loss of material that adheres to the walls of the spray equipment. Besides the studied variables, collection efficiency is still function of the type of the formulation, dimensions of the chamber and the operational properties (solid content, viscosity and inlet air temperature) (TRUONG; BHANDARI; HOWES, 2005). Vardin and Yasar (2012), in spray drying of pomegranate juice with maltodextrin, obtained collection efficiency from 2.3 to 76.3 % and observed that materials with great sugar content like fruits could be adhered to the dryer walls, with lower collection efficiency. The tests 9 and 14 presented the higher collection efficiency (67.62 and 65.87 %, respectively). In test 9, maltodextrin concentration was low (7.5 %), what means lower production cost and higher proportion coffee leaf extract/maltodextrin in the dried product.

Moisture content (MC) was from 0.6 to 11.08% (b.u.) (Table 2). Similar results were observed by several authors. Tonon et al. (2009) for açai juice, observed MC ranged from 0.64% to 2.89 % with drying inlet air temperatures between 138 and 202 °C. Marques et al. (2014b) observed MC of 1.49 to 2.59 % for spray dried green corn pulp with drying temperatures of 143.2 to 176.8 °C. The main goal of drying operation is to obtain products with low MC. It means that the negative effect with respect to moisture content are desirable, as the linear effect of coffee leaf extract concentration. The higher the coffee leaf extract concentration, the lower the MC, due the decrease of proportion air flow/product, which increase the driving force for drying. Marques et al. (2014b)

noted that for a pulp concentration higher than 45% (w/w), the MC of green corn pulp powered was reduced. Grabowski, Truong and Daubert (2008) studied the spray drying of sweet potato puree and observed that higher concentrations

of solids in the feed resulted in the production of powders with lower MC. In those cases, increasing the solids content in the feed process reduced the MC available for evaporation, diminishing MC in the dried powder.

TABLE 2 - Spray dried coffee leaf extract formulation collection efficiency (η), moisture content (MC), phenolic compounds content (Ph), soluble solid content (S), wettability (We) and particle size median distribution (Me) obtained according to a rotatable central composite design.

Test	Codified independent variables				Response variables					
	X ₁	X ₂	X ₃	X ₄	η [%]	MC (w.b.) [kg kg ⁻¹]	Ph [%]	S [%]	We [s]	Me [μ m]
1	-1	-1	-1	-1	50.12	4.81	13.52	95.02	1378	15.42
2	+1	-1	-1	-1	48.57	5.29	6.28	94.20	1005	14.66
3	-1	+1	-1	-1	44.20	2.26	10.46	98.06	1941	21.49
4	+1	+1	-1	-1	37.90	2.34	10.67	95.96	2374	27.80
5	-1	-1	+1	-1	47.73	3.42	6.46	94.36	2403	18.16
6	+1	-1	+1	-1	43.50	3.78	7.01	93.90	2665	16.13
7	-1	+1	+1	-1	57.27	2.53	9.40	95.24	2478	28.70
8	+1	+1	+1	-1	57.73	0.60	9.21	97.18	3509	28.23
9	-1	-1	-1	+1	67.62	4.03	8.19	94.16	1550	13.85
10	+1	-1	-1	+1	57.90	3.87	6.30	87.96	1550	15.83
11	-1	+1	-1	+1	49.73	1.95	9.75	96.80	2771	23.27
12	+1	+1	-1	+1	48.91	2.60	8.08	97.22	1130	22.76
13	-1	-1	+1	+1	54.41	2.71	8.09	93.14	375	16.73
14	+1	-1	+1	+1	65.87	1.04	7.76	96.74	1943	18.57
15	-1	+1	+1	+1	36.63	2.31	11.10	98.00	5230	28.88
16	+1	+1	+1	+1	44.99	2.18	10.20	94.50	3290	31.59
17	-2	0	0	0	49.10	2.11	7.86	94.60	1268	17.97
18	+2	0	0	0	50.14	1.18	7.68	96.02	2416	19.05
19	0	-2	0	0	31.73	8.15	9.15	90.30	1835	24.57
20	0	+2	0	0	40.92	1.58	10.53	97.06	3470	28.80
21	0	0	-2	0	40.82	4.66	8.89	92.84	1823	17.32
22	0	0	+2	0	49.37	11.08	7.88	86.50	1542	60.32
23	0	0	0	-2	35.93	4.44	9.03	94.20	2103	20.81
24	0	0	0	+2	50.33	2.26	9.81	96.56	2179	24.68
25	0	0	0	0	49.00	2.85	8.97	96.48	2008	25.58
26	0	0	0	0	46.52	2.16	8.41	96.66	2762	23.21
27	0	0	0	0	47.27	2.96	10.52	93.48	3010	20.54
28	0	0	0	0	45.75	2.72	11.14	95.10	1761	24.73

X₁ maltodextrin concentration (% w/v), X₂ coffee leaf extract concentration (% w/v), X₃ inlet air temperature (°C) and X₄ air flow rate (m³min⁻¹).

TABLE 3 - Statistic effects for collection efficiency (η), moisture content (MC), phenolic compounds content (Ph), percentage of soluble solid content (S), wettability (We) and particle size median distribution (Me)

Coefficient	η [%]	MC (w.b.) [kg kg ⁻¹]	Ph [%]	S [%]	We [s]	Me [μ m]
β_0	47.1350	2.6720	9.7616	95.4300	2385.2500	18.4025
β_1	-0.0216	-0.3467	-0.9848*	-0.3566	136.3330	0.5697
β_{11}	3.6675	-1.0370	-0.9794*	0.4875	-223.2920	-2.5309
β_2	-3.3316	-2.1100*	1.5008*	3.0833*	1093.6670*	6.1104*
β_{22}	-2.9812	0.5716	0.0558	-0.3275	181.9580	-0.7502
β_3	1.6924	0.3546	-0.5053	-0.7500	636.000* ⁰	8.0155*
β_{33}	1.4050	2.0746*	-0.6700	-2.3325*	-303.0420	4.8137*
β_4	5.6533*	-0.7238	-0.1647	-0.0566	19.8330	-0.2117
β_{44}	0.4225	-0.1869	-0.1542	0.5225	-73.7921	-1.3004
β_{12}	0.7175	-0.0422	0.7948	0.0800	-446.7500	1.5348
β_{13}	4.3050	-0.5521	1.2137*	1.2851	312.7501	-1.3568
β_{14}	2.6125	-0.0391	0.2341	-0.5300	-420.7501	0.2353
β_{23}	3.5725	0.6898	0.7401	-1.2400	548.5000	1.3503
β_{24}	-9.0900*	0.8702	0.2882	0.6951	519.0001	0.6346
β_{34}	-5.9625	0.0209	1.7091*	1.1000	-65.0000	-0.0086
R ²	0.5930	0.6422	0.6873	0.6232	0.6116	0.7277
F _{calc}	1.3013	1.4781	2.0655	1.4269	1.4470	2.3044

Values followed by * are significant at $p < 0.10$. β_x are coefficients of statistical model, β_1 maltodextrin concentration (% w/v), β_2 coffee leaf extract concentration (% w/v), β_3 inlet air temperature ($^{\circ}$ C) and β_4 air flow rate ($\text{m}^3\text{min}^{-1}$).

The contribution of inlet air temperature to higher MC in the quadratic term could be due to the formation of a solid outer crust in the particle because of the rapid evaporation of water at the early drying stages when high inlet air temperatures are used. This crust difficult the remove of the water from the inner to the outlet (HASSAN; MUMFORD, 1993; KIM; CHEN; PEARCE, 2009). High inlet air temperatures and flow rates also resulted in increased moisture content due to the rapid contact of the product in the drying chamber (MARQUES et al., 2014b).

According to Hoge Kamp and Schubert (2003), the wettability (We) could be defined as the capability of a powder to be penetrated by a liquid by capillary forces. On one hand, the We could be aid by small particles (the smaller the particle, the higher the surface area). On the other hand, small particles could present smaller porosities, which makes We more difficult (FERRARI et al., 2012; TONON et al., 2009). In other words, particle diameter should be analyzed with the type of the material, its chemical composition and its microstructure, porosity, density and the

presence of amphipathic substances on the surface (MARQUES et al., 2014b). The obtained extract presented We from 375 to 5230 s. The significant effect of the coffee leaf extract concentration and of the inlet air temperature on the linear term, for both, were positive for We. The use of higher inlet air temperatures during drying undergo thermal degradation of the components, which may produce hydrophobic substances and increase the We (BHANDARI et al., 1993). Even though, the time for the particle to be wet is an arbitrary term, Marques et al. (2014a) adopted 300 seconds as adequate. The trials performed at low inlet air temperature (197.5° C) and product concentration (9.5 %, m/V) presented the smaller values of wettability (trials 13 and 2, 375s and 1005s, respectively), what is desirable for an instantaneous powder.

Solubility (S) is connected to the reconstitution of the powdered product and is defined as the dissolution capability of the solid product in a liquid. The perceptual of soluble solids ranged from 86.50 to 98.06 %. Trials 3 and 15 showed better outcome with respect to

this variable, close to those found for mango juice (CANO-CHAUCA et al., 2005) and salvia tea (ŞAHIN-NADEEM et al., 2013) that used maltodextrin as carrier. The favorable experimental conditions of these tests were low levels of maltodextrin (7.5 %) and high concentration of product (24.5 %). In test 3, additionally, low inlet air temperature (197.5 °C) and air flow (4 m³min⁻¹) were used. The effect of the coffee leaf extract concentration in the solution to be dried was positive for increasing the S, representing an advantage for the process, increasing the productivity by increasing the concentration of product. Furthermore, the quadratic effect of inlet air temperature was negative, contributing to the decrease of the percentage of soluble solids. Higher concentrations of coffee leaf extract in the solution to be dried may have aided in the process, causing an easier release of the powder. With opposite effect, high inlet air temperatures may have caused rigid crusts in powder particles by rapidly removed from the surface, making the output of the nuclei thus damaging the solubility of the material. In spray drying using maltodextrin as carrier of salvia tea (ŞAHIN-NADEEM et al., 2013) and watermelon juice (QUEK; CHOK; SWEDLUND, 2007), negative effect was also observed for the inlet air temperature on the percentage of soluble solids. Those authors argue that, by increasing temperature, the agglomeration of the particles is reduced, with consequently reduced S. Based on that study, higher inlet air temperatures lead to obtaining a material with higher moisture content by crust formation on the surface of the particle. The formation of the crust and highest moisture content are two factors pointed out by several authors (QUEK; CHOK; SWEDLUND, 2007; TONON et al., 2009; VARDIN; YASAR, 2012) as responsible for decreased S.

The content of phenolic compounds (Ph) ranged from 6.28 to 13.52 %. The trials 1 and 28 showed the best results. While the trial 28 is a central point in the experimental design, the trial 1 corresponds to the lower levels for all factors. The preservation of phenolic compounds content on powdered coffee leaf extract presented better results with use of low concentration of maltodextrin (7.5 %), leading to a product with less carrier. Furthermore, the low temperature (197.5 °C) and air flow (4 m³min⁻¹) can be energetically favourable.

The linear and quadratic effects of maltodextrin content in relation to the Ph were negative. This decrease is due to increased concentration of maltodextrin opposite the concentration of the remaining components of the product. The issues of the amount of maltodextrin and the proportion of maltodextrin/ coffee leaf extract in the final product should be evaluated in more detail based on the protection against oxidation of phenolic compounds which carrier presence can provide the final product (LOPEZ et al., 2009).

The linear effect of coffee leaf extract concentration on the product to be dried was positive, contributing to increase the phenolic compounds content of the final product, which means that when working with high concentrations of coffee leaf extract, in addition to increasing productivity is obtained a product with a higher Ph.

The distribution of the volume fraction of the average particle diameter of the coffee leaf extract powder obtained in the conditions established at central point of the DCCR (using 15% w/v of maltodextrin concentration, 17% w/v of coffee leaf extract concentration, 215°C of inlet air temperature and 4.5 m³min⁻¹ air flow rate) is shown in Figure 1. The distribution was in a bimodal shape in which only about 4.5 % of the particles presented size in the range 0.3 to 3 µm. The largest volume fraction of the particles (approximately 9 %) was correspondent to particles with 18 to 25 µm. The bimodal distribution and the range of diameter distribution was in accordance with the ones obtained with products assoluble coffee and açai juice (ESTEVEZ, 2006; TONON et al., 2009). Tonon et al. (2009) in a study of spray drying of açai juice using DE 10 maltodextrin as carrier also found a bimodal distribution of particle diameter. The range in that work was from 0.1 to 41 µm, with an average diameter of 10.94 µm. Those values and behaviour were similar to those obtained in this work. Esteves (2006) obtained soluble coffee particles with diameters between 60 to 400 µm. Thus, the particles obtained in this work are much smaller than those found commercially compared to instant coffee and closer to those found in fruit juices using maltodextrin as carrier.

The statistical model of the particle diameter was represented by Equation 2 (correlation coefficient, R² = 0.73), where the median diameter, Me [µm], and the variables are coded: X₂, the coffee leaf extract concentration in the solution to be dried and X₃, the inlet air temperature.

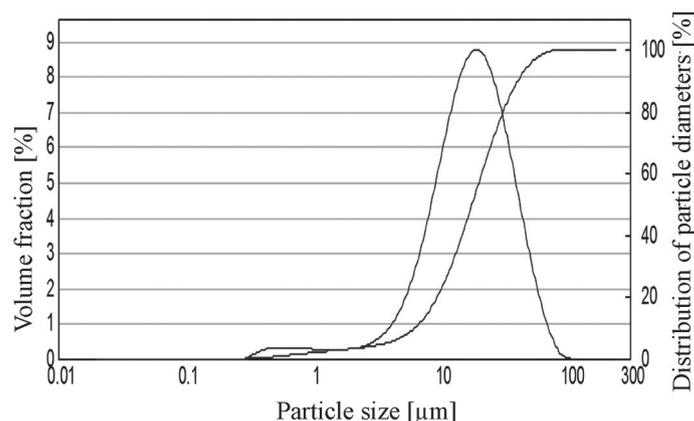


FIGURE 1 - Distribution of the particle diameters and volume fraction of the coffee leaf extract powder according to particle size obtained in the following experimental conditions: 15% w/v of maltodextrin concentration, 17% w/v of coffee leaf extract concentration, 215°C of inlet air temperature and 4.5 m³min⁻¹ air flow rate.

$$Me = 18.4025 - 1.26549X_2 + 3.05521X_3 - 0.10588X_3^2$$

The linear effects of the product concentration and inlet air temperature were positive for the particle size, with a consequent increase of values of the median diameter of particles. With the increase on the inlet air temperature or concentration of the coffee leaf extract in the product to be dried, there was an increase in viscosity of the solution and hence an increase in the size of the particle atomization. Moreover, inlet air temperature influences positively in particle size due to the increased drying rate by providing more energy, with more rapid formation of the structure, not allowing the particles undergo shrinkage during drying. Tonon et al. (2009), studying açai powder juice, found that the rise in inlet air temperature causes an increase of the average particle size due to the greater expansion caused by higher temperatures.

3.1 Better spray drying condition

Operationally, the best spray drying condition for coffee leaf extract using maltodextrin as carrier was the combination of a higher concentrations of extract (32%) and lower inlet air temperatures (180°C) yielded the best drying performance, contributing to produce spray dried powder with higher phenolic compounds content, soluble solids, wettability capacity and lower moisture content and particle size. The same considerations were reported by Marques et al. (2014b).

4 CONCLUSION

The results indicate that the combination of a higher concentrations of coffee leaf extract (32%) and lower inlet air temperatures (180°C) yield the best drying performance. These process conditions lead to a powder of coffee leaf extract using maltodextrin as carrier with high phenolic compounds content, soluble solids, wettability capacity and lower moisture content and particle size.

5 REFERENCES

- AL-MANSOUR, H. E.; AL-BUSAIRI, B. H.; BAKER, C. G. J. Energy consumption of a pilot-scale spray dryer. **Drying Technology**, Singapore, v. 29, n. 16, p. 1901-1910, 2011.
- ASSOCIATION OF OFFICIAL ANALYTICAL CHEMISTS. **Official methods of analysis of the Association of Official Analytical Chemists**. 17th ed. Gaithersburg, 2010.
- BHANDARI, B. R. et al. Flavor encapsulation by spray drying: application to citral and linalyl acetate. **Journal of Food Science**, Chicago, v. 57, n. 1, p. 217-221, 1992.
- BOTREL, D. A. et al. Influence of wall matrix systems on the properties of spray-dried microparticles containing fish oil. **Food Research International**, Toronto, v. 62, p. 344-352, 2014.
- BOWEY, K. et al. Characterization of biologically active insulin-loaded alginate microparticles prepared by spray drying. **Drug Development and Industrial Pharmacy**, London, v. 39, n. 3, p. 457-465, 2013.

- CAL, K.; SOLLOHUB, K. Spray drying technique: I., hardware and process parameters. **Journal of Pharmaceutical Sciences**, Singapore, v. 99, n. 2, p. 575-586, 2010.
- CANO-CHAUCA, M. et al. Effect of carriers of microstructure of mango powder spray drying and its functional and characterization. **Innovative Food Science and Emerging Technologies**, Heverlee, v. 6, n. 4, p. 420-428, 2005.
- CARNEIRO, H. C. F. et al. Encapsulation efficiency and oxidative stability of flaxseed oil microencapsulated by spray drying using different combinations of wall materials. **Journal of Food Engineering**, New York, v. 115, n. 4, p. 443-451, 2013.
- CARVALHO, T. M. B. et al. Avaliação de extratos vegetais no controle de *Oligonychus ilicis* (McGREGOR, 1917) (*Acari: Tetranychidae*) em laboratório. **Coffee Science**, Lavras, v. 3, n. 2, p. 94-103, 2008.
- CATELAM, K. T.; TRINDADE, C. S. F.; ROMERO, J. T. Water adsorption isotherms and isosteric sorption heat of spray-dried and freeze-dried dehydrated passion fruit pulp with additives and skimmed milk. **Ciência e Agrotecnologia**, Lavras, v. 35, n. 6, p. 1196-1203, nov./dez. 2011.
- CORRÊA, J. L. G. et al. The influence of ethanol on the convective drying of unripe, ripe, and overripe bananas. **Drying Technology**, Singapore, v. 30, n. 8, p. 817-826, 2012.
- CORRÊA, J. L. G. et al. Drying of pineapple by microwave-vacuum with osmotic pretreatment. **Drying Technology**, Singapore, v. 29, p. 1556-1561, 2011.
- ESTEVES, B. N. **The influence of the mechanical spray drying process on particle size and bulk density of instant coffee**. 2006. 86 p. Master (Dissertation in Chemical Engineering) - Universidade de São Paulo, São Paulo, 2006.
- FANG, Z.; BHANDARI, B. Comparing the efficiency of protein and maltodextrin on spray drying of bayberry juice. **Food Research International**, Charlestown, v. 48, n. 2, p. 478-483, 2012.
- FANTE, C. et al. Drying of plums (*Prunus sp.*, c.v. Gulfblaze) treated with KCl in the field and subjected to pulsed vacuum osmotic dehydration. **International Journal of Food Science and Technology**, London, v. 46, p. 1080-1085, 2011.
- FERRARI, C. C. et al. Influence of carrier agents on the physicochemical properties of blackberry powder produced by spray drying. **International Journal of Food Science and Technology**, London, v. 47, n. 6, p. 1237-1245, 2012.
- _____. Storage stability of spray-dried blackberry powder produced with maltodextrin or gum arabic. **Drying Technology**, Singapore, v. 31, n. 4, p. 470-478, 2013.
- FOOD AND AGRICULTURE ORGANIZATION CORPORATE STATISTICAL DATABASE. **Final 2014 data and preliminary 2015 data for 5 major commodity**. Available at: <<http://faostat.fao.org/site/339/default.aspx>>. Accessed at: 7 Aug 2015.
- GRABOWSKI, J. A.; TRUONG, V. D.; DAUBERT, C. R. Nutritional and rheological characterization of spray-dried sweet potato powder. **LWT- Food Science and Technology**, Zürich, v. 41, n. 2, p. 206-216, 2008.
- HASSAN, H. M.; MUMFORD, C. J. Mechanisms of drying of skin-forming materials: III., droplets of natural products. **Drying Technology**, Singapore, v. 11, n. 7, p. 1765-1782, 1993.
- HOGKAMP, S.; SCHUBERT, H. Rehydration of food powders. **Food Science and Technology International**, Valencia, v. 9, p. 223-235, 2003.
- ISQUIERDO, E. P. et al. Drying kinetics and quality of natural coffee. **Transactions of the ASABE**, Michigan, v. 56, n. 3, p. 1003-1010, 2013.
- KIM, E. H. J.; CHEN, X. D.; PEARCE, D. Surface composition of industrial spray-dried milk powders: 2., effects of spray drying conditions on the surface composition. **Journal of Food Engineering**, New York, v. 94, n. 2, p. 169-181, 2009.
- LOPEZ, O. D. H. Características del aceite de semillas de Cucurbita pepo L. microencapsulado mediante secado por aspersion con maltodextrina y goma arábica. **Latin American Journal of Pharmacy**, Buenos Aires, v. 28, n. 4, p. 628-632, 2009.
- MARQUES, G. R. et al. Application of maltodextrin in green corn extract powder production. **Powder Technology**, Schofield, v. 263, p. 89-95, 2014a.
- _____. Spray drying of green corn pulp. **Drying Technology**, Singapore, v. 32, n. 7, p. 861-868, 2014b.

- MEDEIROS, F. C. L. et al. Defense gene expression induced by a coffee leaf extract formulation in tomato. **Physiological and Molecular Plant Pathology**, East Lansing, v. 74, n. 2, p. 175-183, 2009.
- MURALI, S. et al. Encapsulation of black carrot juice using spray and freeze drying. **Food Science and Technology International**, London, v. 21, n. 8, p. 604-612, 2014.
- PEREIRA, R. B. et al. Coffee berry husk extract, thyme essential oil and acibenzolar-S-methyl in the control of brown eye spot of coffee tree. **Pesquisa Agropecuária Brasileira**, Brasília, v. 43, n. 1, p. 1287-1296, 2008.
- QUEK, S. Y.; CHOK, N. K.; SWEDLUND, P. The physicochemical properties of spray-dried watermelon powders. **Chemical Engineering and Processing**, Delft, v. 46, n. 5, p. 386-392, 2007.
- RODRIGUES, M. I.; LEMMA, A. F. **Experimental design and process optimization**. Campinas: Casa do Pão Editora, 2012. v. 1.
- ŞAHIN-NADEEM, H. et al. Influence of inlet air temperature and carrier material on the production of instant soluble sage (*Salvia fruticosa* Miller) by spray drying. **LWT - Food Science and Technology**, Zürich, v. 52, n. 1, p. 31-38, 2013.
- STAT-SOFT. **Statistica**: data analysis software system. Version 8.0. New York, 2010. Disponible at: <<http://www.statsoft.com>>. Accessed in: 10 Sept. 2015.
- TONON, R. V. et al. Physicochemical and morphological characterization of açai (*Euterpe oleraceae* Mart.) powder produced with different carrier agents. **International Journal of Food Science and Technology**, London, v. 44, n. 10, p. 1950-1958, 2009.
- TRUONG, V.; BHANDARI, B. R.; HOWES, T. Optimization of co-current spray drying process of sugar-rich foods: part I, moisture and glass transition temperature profile during drying. **Journal of Food Engineering**, New York, v. 71, n. 1, p. 55-65, 2005.
- VARDIN, H.; YASAR, M. Optimisation of pomegranate (*Punica Granatum* L.) juice spray-drying as affected by temperature and maltodextrin content. **International Journal of Food Science and Technology**, London, v. 47, n. 1, p. 167-176, 2012.
- WANG, S.; LANGRISH, T. A. G. A review of process simulations and the use of additives in spray drying. **Food Research International**, Charlestown, v. 42, n. 1, p. 13-25, 2009.