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Microencapsulation of hydrosoluble extract of babassu nut press cake

Microencapsulação do extrato hidrossolúvel da torta de amêndoas de babaçu

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Highlights:
Utilization of co-product generated in the processing of babassu almonds.
Application of complete factorial design to optimize the drying process.
Use of spray dryer to obtain of hydrosoluble extract of babassu nut press cake.

Abstract

Orbignya phalerata Mart. has great socio-economic value, originating various products and by-products; however, there are few studies about obtaining food products from of this raw material. In face of that reality, this study aimed to obtain powder extract from press cake of the babassu nut kernel, and to verify the influence of varying concentrations of Maltogill®10 and drying temperature on process yield through a factorial design, as well as to characterize the final product. Babassu nut used for pressing and acquisition of residual press cake and extract. Spray dryer was used to obtain the powder extract, which adopted a rotating central composite design (RCCD with 11 trials). The final product was characterized by analyses of moisture, solubility, hygroscopicity, water activity and bulk density. The experimental design showed significant influence ($p \leq 0.05$) for the variables inlet temperature in the linear and quadratic models, focusing only on the linear term and the interaction between them, the model adjusted in planning was considered predictive; the best productivity obtained (80.37%), was for the temperature of 95°C with encapsulating agent of 18%. With respect to the powder product analysis, low moisture values, hygroscopicity, and density were observed, as solubility and water activity within the recommended parameters.
Key words: Drying. Encapsulant. *Orbignya phalerata*. Planning.

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Resumo

O babaçu tem grande valor socioeconômico, e dá origem a diversos produtos e subprodutos, todavia poucos estudos destinados a obtenção de produtos alimentícios são encontrados, diante disso este trabalho objetivou obter extrato em pó proveniente da torta de amêndoas de coco babaçu, por meio de um planejamento fatorial, verificar a influência das variáveis concentração de Maltogill®10 e Temperatura de secagem sobre rendimento do processo, bem como, caracterizar o produto final. Foram utilizados amêndoas de coco babaçu, para prensagem, aquisição da torta residual e do extrato. Para obtenção do extrato em pó foi utilizado spray dryer, onde adotou-se um delineamento composto central rotacional (DCCR com 11 ensaios). O produto final foi caracterizado mediante análises de umidade, solubilidade, higroscopicidade, atividade de água e densidade aparente. O planejamento experimental mostrou influência significativa ($p \leq 0{,}05$) para as variáveis temperatura de entrada nos modelos linear e quadrático, concentração no termo linear, e interação entre elas, o modelo ajustado no planejamento foi considerado preditivo, o melhor rendimento obtido (80,37%), foi para temperatura de 95ºC com 18% de agente encapsulante. Com relação a análises do produto em pó, notou-se baixos valores de umidade, higroscopicidade e densidade aparente, bem como solubilidade e atividade de água dentro dos parâmetros recomendados.

Palavras-chave: Secagem. Encapsulante. *Orbignya phalerata*. Planejamento.

Introduction

Babassu (*Orbignya phalerata* Mart.) is one of the several raw materials produced in Brazil. Its main producing states are Ceará, Maranhão, Piauí and Tocantins (Companhia Nacional de Abastecimento, [CONAB], 2019).

This raw material has been used for various purposes, such as in cosmetic industries and coal production; however, the highlight product is oil. Studies mentioned that the main product obtained from babassu is oil, which is industrially produced with extraction from the kernel, which can also originate "babassu nut milk", but only in small-scale production (Soler, Muto, Armiliato, & Fukuzawa, 2013; A. P. S. E. Silva, 2011).

The extraction of these products originates a byproduct called residual press cake, which has nutritional characteristics. Studies performed with babassu pie found 20.60% crude protein, 5.81% ether extract, and 71.93% total carbohydrates and, despite having these attributes, this byproduct is only intended for animal feed production. In face of these properties, research on this co-product is necessary to implement it in human nutrition (Xenofonte, Carvalho, Batista, Medeiros, & Andrade, 2008; Oliveira Borges et al., 2017).

There is a growing interest in questions related to environmental impacts due to the imbalance caused by human action. Therefore, companies are forced to think of strategies to minimize these impacts. This includes food waste, which has been the subject of many studies focused to turning it into products, once it may be rich in nutrients, and safe for consumers (Fernandes & Mançú, 2016; Ishimoto, Harada, Branco, Conceição, & Coutinho, 2007).

Techniques such as drying are applied in alignment with this goal. Atomization stands out among such techniques, with the purpose of transforming fluid in powder. This process allows for greater stability and, therefore, consumers can purchase the product throughout the year (Anselmo, Mata, Arruda, & Sousa, 2006; Santos, Florêncio, Rocha, & Costa, 2014).

However, there are still some adjunct obstacles regarding its physical and chemical properties, as well as processing, taking place during the drying process. These may include tackiness, adhesion to equipment surfaces, and high hygroscopicity. An alternative to mitigate these limitations would be the encapsulation process, which aims to protect sensitive particles by isolating them from the external environment (Cano-Chauca, Stringheta, Ramos, & Cal-Vidal, 2005; Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007). Many studies reported application of encapsulation (Di Giorgio, Salgado, & Mauri, 2019; Fadini et al., 2019; Silva James, Castro, Freitas, & Nogueira, 2019; Souza, Rocha, Brito, Galvão, & Nunes, 2019).

Given the above, the present study aimed to attain an extract from the residual press cake of babassu nut kernels, and to investigate the influence of Maltogill®10 concentrations and drying temperatures on this process yields, being analyzed through a complete factorial design, in addition to characterize the final product by moisture, solubility, hygroscopicity, water activity and apparent density analyses.

Materials and Methods

Raw materials

The study was conducted at the Laboratory of Biomolecule Separation Processes and Food Dehydration, Federal University of Tocantins, in Palmas – TO, Brazil. Babassu (*Orbignya phalerata* Mart.) nut kernels were acquired in the city of São Miguel, Tocantins state.

Obtaining residual press cake

To obtain the residual press cake, kernels were selected, washed in running water to remove dirt, and then manually peeled to remove the film involving them. After that, they were weighed and bleached for lipase inactivation, as well as for facilitating homogenization. This process was conducted in an industrial blender (Metvisa, model LQL 6), with a rotation of 3450 rpm in a ratio of 2:1, with 2 parts hot water (85°C) and 1-part kernels, for a period of 15 minutes until uniform consistency. Afterwards, the material was filtered to separate solid and liquid portions. Finally, the remaining solid material is known as residual press cake.

Obtaining the extract

Newly, the same procedure was carried out to attain extract from the press cake. For that, 250 grams of the cake were weighed and homogenized at the same proportion (2:1). The mixture was then filtered with a fabric filter for removal of any large-sized solid particles to avoid damages to the dryer nozzle.

This resulting filtrate was called hydrosoluble extract from the residual press cake of babassu nut kernels and was ready for encapsulant dilution and subsequent drying. Next, the extract was diluted with an encapsulating agent using a homogenizer (model 712 Fisatom) at 1000 rpm for 5 minutes, as defined in the experimental design. After complete dissolution, the formulated solution was dried through atomization.

Experimental design

In order to check the influence of the variables, namely, Maltogill®10 concentrations and drying temperatures, on process yield, a rotating central composite design (RCCD) was used, 22 with 3 central points (level 0), and 4 axial points (levels ± α) totaling 11 trials. Table 1 shows the experimental design and the data monitored during the experiment, which was checked every 15 minutes.

Table 1
Experimental planning for powdered babassu nut extract from residual press cake, with variations in input air temperatures (T_{input}) and Maltogill®10 (MG) concentrations

	Independent variables				Monitored data			
	Real		Encoded					
Test	T_{ent} (ºC)	Conc. MG(%)	T_{ent} (ºC)	Conc. MG(%)	T_s(ºC) ±SD	T_a(ºC) ±SD	RM% ±SD	Yield (%)
1	75	10	-1	-1	70,66±1,15	27,15±1,20	74,3±2,12	40,54
2	95	10	+1	-1	81,33±2,30	26,76±0,70	78,13±2,34	40,52
3	75	18	-1	+1	70,33±0,57	27,03±0,20	78,7±0,36	36,64
4	95	18	+1	+1	82,66±2,30	27,7±0,30	74,13±1,28	80,37
5	70,9	14	-α	0	64,33±0,57	28,16±0,15	71,76±0,66	46,32
6	99	14	+ α	0	87,33±1,15	27,96±0,30	75,9±1,21	74,94
7	85	8,36	0	-α	72,00±2,00	24,43±025	70,43±2,17	39,60
8	85	19,64	0	+ α	72,00±2,00	26,9±0,26	76,66±0,05	57,08
9	85	14	0	0	72,00±0,00	26,83±0,20	76,00±0,43	41,65
10	85	14	0	0	72,00±0,00	27,5±0,36	75,85±0,37	45,10
11	85	14	0	0	72,00±0,00	27,1±0,00	72,76±0,76	45,25

In which: T_o is the output temperature; T_r is the room temperature; RM% is the relative moisture; and SD is the standard deviation. MG (%) = concentration of Maltogill ®10.

Obtaining the powder extract and process yield

Drying was conducted in spray dryer (LM MSD 0.5 of Labmaq brand from Brazil). During all the experiment, a pneumatic nozzle of 1.2 mm diameter was used, with feed flow rate of 0.2 L/h, air flow rate of 1.71 m^3/min, and drying air velocity of 30L/min.

Process yield was calculated based on sample soluble solids before drying. The result was expressed in percentage (%), and calculated according to equation 1:

$$Yield\ (\%) = (weight\ powder\ \times 100)\left[\frac{100}{\text{sample mass } \times \text{ sample ºBrix}}\right]$$

Analytical determinations of the powdered product

The powdered product had a better yield. It was evaluated for moisture, hygroscopicity, solubility, water activity and density. All analyses were performed in two replications.

Moisture and water activity

The moisture content was measured gravimetrically by drying the samples in an oven at 105 °C, and water activity was determined performed directly in water activity meter at 30 °C, using the Aqualab device, in triplicate (Association Off Official Agricultural Chemists [AOAC], 2005).

Solubility and hygroscopicity

Product solubility was determined by the method described by Eastman and Moore (1984), modified by (Cano-Chauca et al., 2005), with a few adaptations. About 0.5 g of sample was added into a flask containing 50 ml distilled water, under magnetic stirring of 1000 rpm for 5 minutes. This was followed by centrifugation at 4,500 rpm for 5 minutes. An aliquot of 12.5ml of supernatant was taken to an oven at 105 °C to constant weight, and solubility was calculated by weight difference, according to Equation 2:

$$Solubility\ (\%) = \left[\frac{Powder\ mass\ supernatant}{powder\ mass}\right] \times 100$$

And the powdered product hygroscopicity was evaluated according to the method proposed by Cai and Corke (2000), with some modifications. About 1 g of each sample was placed in an airtight container with a saturated NaCl solution (relative moisture between 69.8 to 76%) at 25 °C. After one week, samples were weighed.

Apparent density

Apparent density (ρ*ap*) was determined by transferring 1 g atomized powder from the babassu nut press cake extract into a measuring 10 ml cylinder. Subsequently, it was calculated as the ratio between the powder mass and its volume occupied (Goula & Adamopoulos, 2004a).

Statistical analysis

The experimental design results were evaluated by analyses of variance (ANOVA), and by multiple linear regression (response surface method, RSM). Second order polynomial models were generated to explain the influence of linear effects, and of the interaction between the variables Maltogill®10 concentration and drying temperature. Equation 3 was used to estimate regression coefficients. Software STATISTICA 7.0 was used for all statistical analyses.

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i<j}\sum \beta_{ij} x_i x_j$$

In which: Y is the predicted response; β0 is the intercept model; βi is the coefficient of linear effect; βii is the coefficient in the quadratic effect; βij is the coefficient of the interaction of effects; xi and xj are the independent variables.

Preliminary tests

Based on preliminary tests, a ratio of 2:1 was fixed (distilled water and residual babassu nut press cake), because this ratio showed better homogenization, and consequently efficiency in sample extraction.

Drying tests were carried out after establishing the ratio for extract obtainment. The first trial used only the extract obtained, i.e. without encapsulant, as for verifying raw material behavior in the machine without protective agent characteristics. In this case, there was no dust formation, resulting in product adherence to the drying chamber. The other trials were carried out to fix drying air inlet temperatures.

Results and Discussion

After the preliminary tests, drying air-inlet temperatures were established between 70.9 °C and 99 °C. Encapsulating agent concentrations varied between 8.36% and 19.64% for the studied samples.

The yield obtained in each drying trial of babassu nut kernel press cake extract is shown in Table 1, together with inlet temperature and encapsulant concentration, referring to the experimental design.

Process yield results ranged from 36.64 to 80.37%. These values refer to trial 3, whose drying temperature was 75 °C with 18% encapsulating agent; and trial 4 at a temperature of 95 °C and concentration of 18%, respectively (Table 1).

Table 2 shows the effect values at 95% confidence interval for the drying yield of babassu nut kernel extract from the residual press cake encapsulated with Maltogill®10.

Table 2
Estimated effect, standard error, coefficient (t), and significance level of yield values for samples encapsulated with Maltogill®10

Factor		Effect	Standard error	T(5)	p-value
Dry product	mean	44,0000	2,231864	19,71446	0,000006
	T_{ent} (L)	21,04620	2,733464	7,69946	0,000590
	T_{ent} (Q)	14,14625	3,253473	4,34805	0,007373
	$Conc._{mg}$ (L)	15,16761	2,733464	5,54886	0,002612
	$Conc._{mg}$ (Q)	1,85625	3,253473	0,57054	0,592992
	T_{ent} x$Conc._{mg}$	21,87500	3,865702	5,65874	0,002395

Values in bold were not significant at 95% confidence level.

By eliminating the non-significant factor and performing analysis of variance (ANOVA) using the F test, the significance of regression and lack of fit could be verified at 95% confidence level ($p \leq 0.05$), values shown in Table 3.

Table 3
Variance analysis of the adjusted model

Source of variation	GL	QM	$F_{calculated}$	$F_{tabulated}$	R^2
Regression	4	527,59	39,776	4,53	0,96366
Residue	6	13,26			
Lack of adjustment	4	17,82	4,29	19,25	
Pure error	2	4,14			
Total	10	218,99			

GL: Freedom degree; QM: Mean square.

Thus, the model equation obtained in the experimental design was obtained (Equation 4).

$$Yield\ (\%) = 44,87 + 10,52T_{entr\ (L)} + 6,80\ T_{entr\ (Q)}^2 + 7,58\ C_{Mg\ (L)} + 10,93\ T_{entr\ x}\ C_{Mg}$$

In which: T_{input} (L) and T_{input} (Q) are the linear and quadratic input air temperature during the drying process, respectively. The term C_{Mg} (L) indicates the Maltogill®10 concentration in the linear term.

Figure 1 shows the response surface generated by the proposed model for the powdered extract yield from the residual press cake of babassu nut kernels, encapsulated with Maltogill®10.

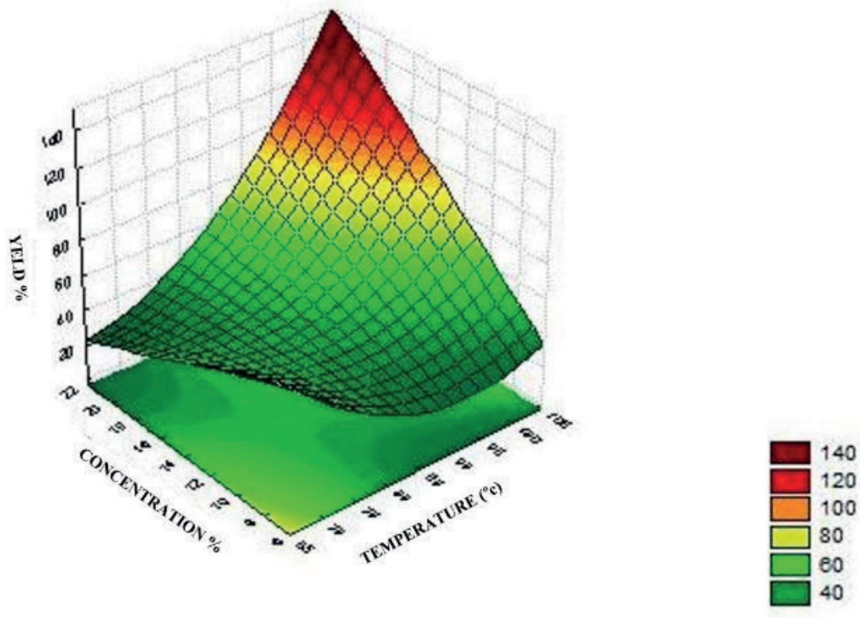

Figure 1. Response surface generated for the yield the process.

The final yield of encapsulated powder extract with Maltogill®10 showed significant influence ($p \leq 0.05$) for variables inlet temperature in both linear and quadratic models, and concentration only in linear term. For interaction between them, only the results for concentration in the quadratic term were not significantly influenced (p-value = 0.592992). Moreover, all effects were positive for the responses obtained.

The values observed have a direct connection with the heat and mass transfer process and its effectiveness. Studies performed Rocha point out that positive yields are related to this process, especially when higher temperature ranges are used (Rocha et al., 2014).

Furthermore, the significance of regression ($F_{calc} > F_{tab}$) and non-significance of the lack of fit ($F_{calc} > F_{tab}$), at the same confidence level, were established by ANOVA. According to the analysis, the model adjusted in planning is considered predictive, since the regression F_{calc} is eight times larger than the F_{tab}, according to (Rodrigues & Iemma, 2005). In order to consider a model predictive, its F_{cal} must be five times larger than the F_{tab}, confirming the results of this study.

The regression explains 96.36% of the variation, which is measured by the value of R^2, also correcting the mathematical model obtained, which is predictive.

An increase is observed through the response surface, possibly due to the additive effect of temperature, concentration and the interaction between them. In addition, there was a slight process increase due to the quadratic concentration model, which does not interfere with the current conditions. Thus, the process was optimized with higher temperature and concentration values, which brought about higher powdered extract yields from the residual press cake of babassu nut kernels.

This is possibly due to the drying temperature and glass transition temperature (T_g). When working at high drying temperatures, there are better effects in the heat and mass transfer process, and consequently drier particles. Studies performed atomized goat milk found that encapsulating agent and high temperatures increase process yield (Diniz et al., 2005).

The relationship of glass transition temperature and yield presents probable influence, which is the phase change of a material. Such temperature occurs through drastic changes in movement of polymer chains, bringing differences between the properties of gummy and vitreous states as a result (Leite, Murr, & Park, 2005). Babassu nut milk presents considerable levels of fatty acids, according to Arévalo-Pinedo et al. (2005), these compounds tend to reduce glass transition temperatures.

However, the addition of encapsulating agent increases T_g due to the addition of solids, i.e. it increases its molecular weight; consequently, there is higher yields at the process end. Studies recents observed similar behaviors (Largo Avila, Cortes Rodriguez, Velásquez, & José, 2015; M. A. Silva, Sobral, & Kieckbusch, 2006; Zotarelli, 2014).

Thus, increases in both drying temperature and glass transition temperature are seen to contribute to improved yields in the atomization process of a hydrosoluble extract of babassu nut kernel.

Regarding the powdered product, Table 4 elucidates the results of the analyses with atomized powder at 95 °C with 18% encapsulating agent, which means that the best results of the process were obtained under these conditions. The data presented correspond to the average of two replicates along with its respective standard deviation.

Table 4
Analysis of product powder

Analysis	Obtained data
Moisture (%)	0,97±0,00
Hygroscopicity (g water adsorbed/100g solid)	10,95±0,00
Solubility (%)	85,14± 2,63
Water activity (A_w)	0,142±0,00
Apparent density (g/cm^3)	0,10±0,00

Low moisture values were expected for the powdered product, and these were within the parameters established by legislation, whose criteria involve the protection against microorganism growth, whether deteriorating or pathogenic (Ministério da Sáude [MS], 2005).

The results obtained were consistent with those observed by Santana (2013) in atomization drying of babassu nut milk, which found values between 0.92 to 1.62% moisture. Studies with powdered guava obtained by a spray dryer observed a content of 5.69% moisture (Santos et al., 2014). Both studies used atomized samples with maltodextrin as a carrier agent. Fadini et al. (2019) evaluated microencapsulated fish oil by spray drying and obtained moisture content value of 3.10 (g/100 g). It is important to highlight that for good stability during storage, food-grade powders should have a moisture content below 3 g/100 (Thirundas, Gadhe, & Syed, 2014).

With respect to hygroscopicity, the values obtained demonstrate the efficacy of the carrier agent used, and it provided better protection against environment moisture adsorption, which makes the product less hygroscopic.

The results observed may be related to the used encapsulant concentration (18%), and its addition entails less hygroscopicity products. The data were consistent if compared to hygroscopicity tests carried out by Barbosa and Rodriguez-Hernandez for atomization of fruit powder and prickly pear juices, obtaining values from 20.85 to 25.32% and 18 to 23%, respectively Barbosa (2010) and

Rodríguez-Henández, González-García, Grajales-Lagunes, Ruiz-Cabrera and Abud-Archila (2005).

The results also corroborate those found by Cai and Corke (2000), who found the same effect in microencapsulation of betacyanin extracted from amaranthus by atomization. Their data showed reduction greater than 50% in hygroscopicity of the betacyanin produced with the addition of maltodextrin, which was not observed without the encapsulant.

The powdered extract showed good solubility. If related to water content and dryer operating conditions, it increases as water content decreases (Goula & Adamopoulos, 2005b).

The values found corroborate studies by Oliveira, Tonon, Nogueira and Cabral (2013) with drying of atomized strawberry pulp, which had solubility values from 87.15% to 91.44%.

Water activity was found to be lower than 0.2. This result is favorable since the presence of free water in the product makes it susceptible to degradation and consequent reduced useful life.

Studies reported similar values, of 0,18 in optimization of the production of double-shell microparticles containing fish oil (Fadini et al., 2019) and atomization drying of propolis. Highlighting that water activities for atomized products should be lower than 0.6 (F. C. Silva et al., 2013).

In face of the above, this study presented satisfactory results. It is worth noting that lower water activity values guarantee product stability. In addition, they provide other benefits such as conditioning to room temperature, substances that are sensitive to light, heat or oxidation, besides volume reduction and even greater availability throughout the year Azeredo (2008) and Oliveira et al. (2013).

The powdered extract from residual press cake of babassu nut kernels had low apparent density. This result may be explained by the carrier agent concentration since it decreases with its addition. Maltogill®10 is obtained by starch hydrolysis, and the more starch is hydrolyzed, the lower its density, because of the bonds which are broken, resulting in smaller and lighter chains (Ferrari, Germer, Alvim, Vissotto, & Aguirre, 2012).

The results observed here are similar to those of Astolfi, Souza, Reipert, & Telis (2005) who studied encapsulation of passion fruit juice and reported values between 0.63 to 0.76 (g/cm^3) and near those of Santana, in atomization drying of pequi pulp and babassu nut milk, who observed values between 0.35 to 0 42, and 0.39 to 043 (g/cm^3), respectively (Santana, 2013).

Conclusions

The study indicated that trial 4, at 95°C and Maltogill®10 concentration of 18%, was considered the most suitable for atomization of an extract from residual press cake of babassu nut kernels. Under these conditions, the obtained data reached a yield of 80.37%. Planning was significant ($p \leq 0.05$) for the variable inlet temperature in linear and quadratic models, and for concentration only in the linear term, as well as the interaction between them. Moreover, the model fitted in planning was considered predictive. Regarding final product analyses, moisture and water activity were within parameters recommended for dry products. The final powder showed good solubility, low hygroscopicity, and low apparent density. In so being, the elucidated results may have been favored by the encapsulant concentration used in the drying process.

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