CARPATHIAN JOURNAL OF FOOD SCIENCE AND TECHNOLOGY

journal homepage: **http://chimie-biologie.ubm.ro/carpathian_journal/index.html**

OPTIMIZATION OF SPRAY DRYING FOR COCONUT MILK POWDER USING RESPONSE SURFACE METHODOLOGY AND INVESTIGATION OF THE POWDER PROPERTIES

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https://doi.org/10.34302/crpjfst/2022.14.4.9

Article history:
Received
3 August 2021
Accepted
29 September 2022
Published
December 2022

Keywords:
Coconut milk-powder;
Optimization;
Response Surface Methodology;
Spray drying.

ABSTRACT

The main objective of this work was to optimize the spray drying of coconut milk using response surface methodology (RSM). Maltodextrin was added to the feed coconut milk as the carrier agent in the laboratory scale experiments and the same were designed using Box-Behnken design approach. The effect of the chosen input variables on the responses namely product yield, moisture content, hygroscopicity, and angle of repose of the powders were evaluated using 3D surface plots. Among the chosen input variables of the process, the temperature of the inlet air in the spray drying chamber was found to significantly affect the characteristics of the powder. The flowability properties of the synthesized powders were experimentally obtained and the values were found to be satisfactory. The functional and morphological properties, and color of the powder produced at the optimal condition were compared to those of a commercially available coconut milk powder. For the synthesized powder of the optimal condition, an enhancement of 30°C for the glass transition temperature was seen over to that of the commercial powder.

1. Introduction

The storage of aqueous extract of coconut endosperm (commonly known as coconut milk) in its native liquid form is not suitable due to rancidity caused by microbial activity (Seow and Gwee 1997; Hettiarachchi et al. 2019). Coconut milk is an oil-in-water emulsion and very unstable due to insufficient quality and quantity of the protein mass and a high percentage of fat. These factors culminate to the separation of milk into two separate layers (creamy layer and dense aqueous serum) upon storage (Tangsuphoom and Coupland 2008; Maidannyk et al. 2020). Also, coconut milk is highly susceptible to spoilage (undesirable odor and taste) due to microbial activity, chemical deterioration due to lipid autoxidation, lipolysis, and oxidation of unsaturated fatty acids. Considering the importance of coconut milk in the food industry due to its nutritional importance, anti-cancerous and anti-inflammation properties, several methods were proposed to increase the shelf-life of coconut milk. For short-term storage, nevertheless, heat treatment alone is effective. Sterilization is an option for long-term storage but the physical instabilities within the milk due to long-term standing cannot be avoided (Ho et al. 2019).

In the past, spray drying or dehydration of coconut milk to powder form was shown as a commercially viable option for long-term storage with no major loss of the nutrients (Zafisah et al. 2018; Fournaise et al. 2020). Although some inevitable loss of favoring volatile components cannot be overruled in the drying process, almost all of the original milk attributes can be achieved under normal conditions when the milk is prepared from the powder.

Spray drying is considered as a better option for the drying thermo-sensible liquids due to shorter exposer times of the liquids to higher temperatures in the drying chamber with almost no thermal degradation of the milk constituents (Sosnik and Seremeta 2015; Voronin et al. 2021). The carrier agents added to the feed milk can effectively encapsulate the hydrophobic and hydrophilic active compounds and increase the shelf-life of the milk powder (Chávez-Servín et al. 2015).

In the spray drying of coconut milk, higher viscosity of the milk due to the presence of fat globules can result in the improper spraying and the nozzle clogging in the drying chamber. To avoid these undesirable features, carrier agents are added to the feed milk for the encapsulation of the fat content and to prevent the formation of lumps and avoid powder particles sticking to the wall of the drying chamber. Maltodextrin, lactose, whey protein, and Gum arabic are generally used as carrier agents for this purpose (Carlos et al. 2018). Further, addition of microencapsulating agents to the feed milk can increase the product yield up to 60% by weight (Simuang et al. 2004; Manikantan et al. 2015). Emulsifiers and stabilizers are also added to the precursor milk for the stability in avoiding the formation of larger oil droplets (Hassan 1985). As the desirable physio-chemical properties for the powder (stability, solubility, flow properties, particle size and shape, density, and compatibility, etc.) can be achieved in the spray drying process (Kim et al. 1996), the production of coconut milk powder using spray drying was highly recommended (Negizj and Lagergren 1995; Langrish and Fletcher 2001; Millqvist-Fureby 2003; Krishnan et al. 2005; Piatkowski and Zbicinski 2007).

Another important parameter of the powder is its glass transition temperature (Adhikari et al. 2005). It is defined as the temperature at which amorphous glassy powder changes to rubbery type (Le Meste et al. 2002). If the glass transition temperature of the powder is higher than the maximum storage temperature, then lumping of the powder particles can be prevented upon storage. Commonly, the sticky nature of the particles can be seen at 10 to 20°C above the glass transition temperature. Hence, higher glass transition temperatures are desired for longer shelf-life. The flow properties of the powder are also equally important in the design of handling machines (Prescott and Barnum 2000). The other important functional properties are morphology and density. The hydration properties (wettability, sink ability, dispersibility and solubility) of the powder must be desirable in the successful preparation of milk from the powder (Schober and Fitzpatrick 2005; Hammes et al. 2015).

For several engineering applications, response surface methodology (RSM) has been successfully proven as an effective option for the optimization of input parameters (Nwabueze 2010). RSM is a collection of several mathematical and statistical techniques and can be utilized to design new formulations in the development of useful products (Islam Shishir et al. 2016). The Central Composite Design (CCD) and Box-Behnken Design (BBD) are the most famous approaches and these methods are generally chosen based on the application of interest and operability. RSM was used to optimize the experimental parameters for the spray drying of high-value oyster byproducts and the development and utilization of aquatic shellfish byproducts (Chen et al. 2018). To optimize the spray drying of pink guava extract, RSM was used with central composite face-centered design of experiments (Islam Shishir et al, 2016).

The four salient parameters of the spray drying of coconut milk are feed flow rate, inlet temperature of the air, atomizer speed and the concentration of the carrier agent in the feed. At higher feed flow rates, the rate of evaporation drastically reduces. The formation of a rigid impermeable layer on the droplets hinders the uniform drying at higher temperatures inside the drying chamber. Due to high-fat content of the coconut milk, it is necessary to add carrier agents to ease the spray drying process.

The main objective of the proposed work was to find the optimal operating parametric conditions for the production of coconut milk powder of improved quality. Three input parameters namely inlet temperature, concentration of carrier agent and atomizer speed were selected for this purpose. Box-Behnken Design (BBD) was employed for the design of lab-scale experiments. Using RSM, the effect of the three input parameters were correlated with the product yield and three important properties of the powders namely angle of repose, hygroscopicity, and moisture content. The flow properties of the powders were experimentally calculated and presented. The morphology, angle of friction and glass transition temperature of the milk powder produced at the optimal conditions were compared to those of a commercial coconut milk powder sample.

2. Materials and methods

2.1. Materials

Coconuts of 8-9 months of age were collected and de-husked. The endosperms were made brown testa free and the white coconut endosperm flesh was thoroughly washed and grated. The grated coconut flesh was kept in a measuring jar of 1-liter capacity and the container (top open to atmosphere) was partially immersed in a hot water bath (50 °C) for 15 minutes with no physical contact of the hot water and coconut flesh. Thereby, the smoothened flesh was cooled to room temperature and mixed with distilled water (1:1 by mass). The mixture was taken on a cheesecloth, and the cloth was then wrapped followed by mechanically squeezing for the milk. The coconut milk was pasteurized at 72 °C for 1 min and cooled to room temperature. In the current work, a carbohydrate component namely Maltodextrin (DE=20) was employed as the carrier agent.

2.2. Experimental procedure

Spray drying experiments were performed in a spray mate JISL type spray dryer. The dryer can operate up to 250°C of the inlet air. In Figure 1, a schematic diagram and the working model of the spray dryer in the co-current operation mode was shown with the locations of the temperature measurements. Precisely, the hot air from the heater, through an air distributor, comes into contact with the sprayed coconut milk in the drying chamber. The inlet pressure of air was kept at 2 atm throughout the experiments.

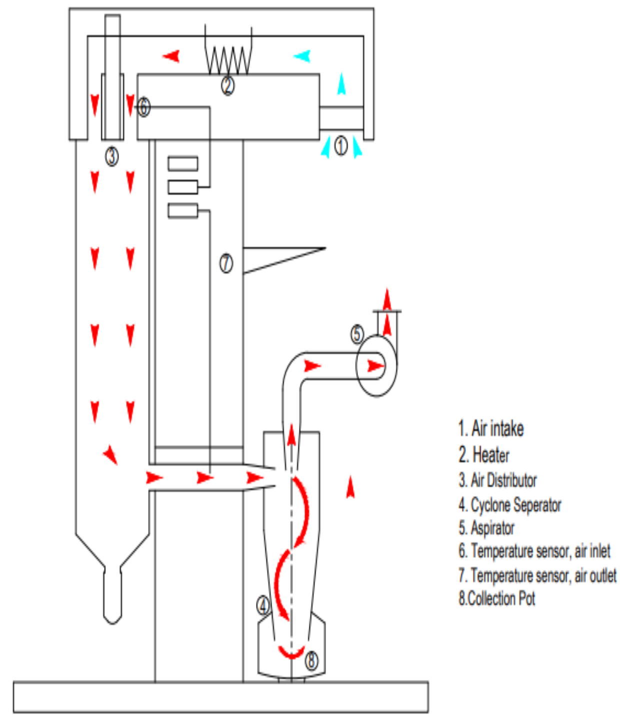


Figure 1. Schematic diagram of the airflow during spray drying

2.3. Characterization

The hygroscopicity of the milk powder was determined as follows. A sample of milk powder was placed in a desiccator for one week under a saturation solution of Na_2SO_4. The hygroscopic moisture was expressed as g of moisture per 100 g dry solids. The hygroscopicity was calculated as given below.

$$Hygroscopicity = \frac{(W_f - W_i)\times 100}{W_i \times \left(100 - \frac{(W_f - W_i)}{100}\right)} \quad (1)$$

where W_f and W_i are the final and initial weights of the powder. The moisture content of dried powder was determined gravimetrically by oven drying at 102°C for 2 hours (standard AOAC

1990). In general, lower values of hygroscopicity and moisture content are desired for the food powders.

The angle of repose is an indicator of the flowability of the powder. Smaller values of the angle of repose indicate better flowability of the powders and this parameter is a crucial one in the design of processing and packaging equipment. The angle of repose (Θ) of the powder was determined as the inverse of the tangent of the ratio of height (*h*) to the radius (*r*) of the cone of powder formed when 100 g of powder is poured through a funnel.

$$\Theta = tan^{-1}\left({}^{h}/_{r}\right) \quad (2)$$

The yield of coconut milk powder (in percentage) is expressed as

$$Yield, \text{wt\%} = \frac{W_2}{W_1} \times 100 \quad (3)$$

where W_2 = weight of coconut milk powder; W_1 = weight of coconut milk plus carrier agent. The moisture contents of the powder samples were evaluated with moisture Analyzer AND MX – 50 with a tolerance limit of 0.01 %.

The insolubility index is an indicator of the solubility of the powder in water while reconstitution. IDF Standard 129A method summarizes the evaluation of insolubility of powders. To calculate insolubility index, 10 g of dry powder is added to 100 mL of water at 50℃ with continuous mixing for 5 min. The suspension was stirred with a spatula and 50 mL was filled to a graduated 50 mL centrifuge tube and the tube was centrifuged at 5070 rpm. The same procedure was repeated after siphoning off the sediment-free liquid. The sediment in the bottom was dried at 70℃ overnight until it approaches a constant weight. The insolubility index is expressed as the weight of the sediment after drying (mg). Lower insolubility index values are desired for the powders.

Dispersibility is the ability of the powder to separate and form individual particles upon mixing. The dispersibility of the powder is calculated as,

$$\%\, Dispersibility = \frac{(10+a)\, TS}{a\left(\frac{100-b}{100}\right)} \quad (4)$$

where a = Powder in g, b = percentage of moisture content, TS =Total solids in the coconut milk.

The powder characteristic to quickly form a stable suspension can be depicted as the solubility time. It is expressed as the time taken for the uniform suspension formation after mixing. Bulk density (ρ_b) is a measure of the mass of powder occupying a unit volume that include the inter particle voids of the powder. Tapped density (g/cm^3) of powder is defined as the ratio of the mass of the powder to the volume occupied by the powder after it has been tapped for a defined period. The tapped density (ρ_t) of the powder can be calculated as $\rho_t = M/V_f$, where M and V_f are mass of powder in grams and tapped volume in milliliter or cm^3, respectively.

Using ρ_b and ρ_t values, the flowability characteristics of the powder such as Compressibility index (*Carr's Index*) and *Hausner ratio* are determined as given below.

$$Carr's\ Index = \frac{\rho_t - \rho_b}{\rho_t} \times 100 \quad (5)$$

$$Hausner\ ratio = \frac{\rho_t}{\rho_b} \quad (6)$$

The thermal characterization of the milk powders was performed using differential scanning calorimetry (DSC). The DSC Thermogram can be used to estimate the glass transition temperature of the powder. The morphology of the powders was evaluated in the scanning electron microscopy (SEM) imaging. It is very easy to visualize caking, bridge formation among the powder particles using this technique.

2.4. Box-Behnken Design of experiments

The spray drying experiments were systematically designed and the responses were statistically interpreted for the optimization of chosen independent variables. The three selected input variables namely inlet air temperature (*A*), atomizer speed (*B*), the concentration of the carrier agent (*C*) were considered as independent variables. Feed flow rates of the milk and inlet air were kept at their maximum allowable values. Three different coded levels (Low (-1), Medium (0), High (+1)) were selected for the Box-Behnken Design of experiments (DOE) approach. For each response, a quadratic polynomials correlating the independent variables was obtained. The experimental design matrix of the Box-Behnken design is depicted in Table 1. It shows the range of the values of independent variables and response variables of the performed experiments.

The constraints for the optimization were maximum product yield, minimum values of hygroscopicity and moisture content, and the angle of repose should be between 10° and 35°. The predicted and adjusted R^2 values, and coefficient of variation (*CV*) were evaluated to understand the lack of the model fit for the data. terms concerning the independent variables were generated ($p < 0.01$). The experimental data fitted for a response R_j and independent variables *A*, *B*, and *C* is given below.

The analysis of variance (ANOVA) against each response was generated and all the interaction

$$R_j = \beta_{0j} + \beta_{1j}A + \beta_{2j}B + \beta_{3j}C + \beta_{12j}AB + \beta_{13j}AC + \beta_{23j}BC + \beta_{11j}A^2 + \beta_{22j}B^2 + \beta_{33j}C^2 \quad (7)$$

For j = 1 to 4 where β_{0j} is a constant; β_{1j}, β_{2j}, and β_{3j} are the coefficients of linear regression; β_{12j}, β_{13j}, and β_{23j} are the coefficient of interaction & β_{11j}, β_{22j} and β_{33j} are the coefficients of quadratic regression.

3. Results and discussions

Several preliminary experiments were performed to identify the range of selected independent operating parameters. The suitable temperature range for the inlet air was found to be 160°-190°C. Watery product or insufficient drying condition was observed when the temperature of inlet air was kept below 160°C. insufficient drying was very much bothersome with the nozzle clogging in the spray drying chamber. At lower temperatures of the inlet air, stronger outer crust may not form on the powder particles. Above 190°C, the product color was dark due to overheating. Powders with desirable properties were produced when the maltodextrin (DE=20) concentration in the feed milk is in the range of 5% - 15% (w/w). Fig. 2a shows the sample containing coconut milk and MD (DE=20) before spray Drying. Fig 2b shows the dripping of milk in the spray drier column when the air inlet temperature is < 160°C.

Table 2 shows the experimental range and level of variables as per the Box-Behnken Design. The experiments were performed at different factor values, called levels. Each experiment involves the combinations of the levels of factors. In BBD, only three levels required to run an experiment and the three levels of independent variables are chosen were also depicted in Table 1.

Figure 2. a) Coconut Milk b) dripping of coconut milk for inlet air temperature <160°C

The experimental results were utilized to derive the polynomial equations. The optimal values of independent variables for the given constraints were obtained with the Response Surface Methodology (RSM) in Design Expert® software.

Table 1. Design matrix of the Box-Behnken design obtained from RSM

Std	Run	*A*	*B*	*C*	R_1	R_2	R_3	R_4
		ºC	w/w	rpm	g	deg	g/100g	%
16	1	0.00	0.00	0.00	5.1	32.005	32.01	6.8
3	2	-1.00	1.00	0.00	5.11	29.56	27.01	8.5
14	3	0.00	0.00	0.00	5.1	32.005	32.01	6.8
2	4	1.00	-1.00	0.00	8.21	25.1	37.1	6.8
1	5	-1.00	-1.00	0.00	8.1	29.61	28.6	8.2
9	6	0.00	-1.00	-1.00	5.6	31.27	32.87	6.75
13	7	0.00	0.00	0.00	5.1	32.005	32.01	6.8
5	8	-1.00	0.00	-1.00	5.1	29.4	27.1	6.3
7	9	-1.00	0.00	1.00	8.5	31.27	27.45	7.9
4	10	1.00	1.00	0.00	5.1	25.3	38.01	5.1
11	11	0.00	-1.00	1.00	8.15	31.27	27.4	7.56
8	12	1.00	0.00	1.00	5.1	25.1	37.1	5.32
15	13	0.00	0.00	0.00	4.98	32.005	32.01	6.8
12	14	0.00	1.00	1.00	5.1	31.55	31.9	7.25
10	15	0.00	1.00	-1.00	5.01	32.56	32.65	7.12
6	16	1.00	0.00	-1.00	4.25	25.73	38.05	5.62
17	17	0.00	0.00	0.00	5.1	32.005	32.01	6.8

A: Temperature; *B*: Concentration; *C*: Atomizer speed
R_1: Yield %, R_2: Angle of repose, R_3: Hygroscopicity, R_4: Moisture content

Table 2. The Experimental range and level of variables chosen for the Box-Behnken design

Variable, unit Symbol	Low (-1)	Center (0)	High (+1)
Inlet temperature, ºC	160	170	180
Maltodextrin concentration, w/w	5	10	15
Atomizer speed, rpm	1400	1700	2000

3.1. Product yield

The product yield results are plotted in Figure 3. As shown in Fig 3(a), for a fixed concentration of Maltodextrin, the yield decreased with the increase of inlet air temperature. For a fixed temperature, the effect of maltodextrin concentration on the yield was almost absent. Atomizer speed and inlet air temperature are having a significant impact on yield (Fig 3(b)). At the highest values of inlet temperature and atomizer speed (190ºC and 2000 rpm respectively), the maximum yield was seen. Similarly, maximum yield was obtained at the highest values of maltodextrin concentration and atomizer speed as depicted in Fig 3(c). The heat transfer rate increases and becomes effective with respect to the increase in the atomizer speed. Overall, the product yield was mainly quite sensitive with respect to inlet air temperature and atomizer speed. The polynomial equation in terms of coded factors for the product yield is given below.

$$R_1 = 5.10 - 1.65\,A + 0.041\,B + 0.04\,C - 0.078\,AB - 0.095\,AC + 0.1\,BC + 1.48\,A^2 - 0.051\,B^2 + 0.061\,C^2$$

ANOVA results of the fitted polynomials of the product yield are shown in Table 3. The model is significant as the Model F value is 468.84. There is only a 0.01% chance that a "Model F-value" of this large could occur due to noise. Since the values of "Prob > F" are less than 0.05, it indicates the significance of model terms. Hence, in the predicted polynomial, the terms encompassing *A*, *B*, *C* and *AB* are significant. A value of 0.9735 for the "Pred R-Squared" is in reasonable agreement with the "Adj R-Squared" of 0.9962. The well-dispersed nature of the data with better reproducibility and repeatability was seen as the *CV* (Coefficient of variation) less than 10 %.

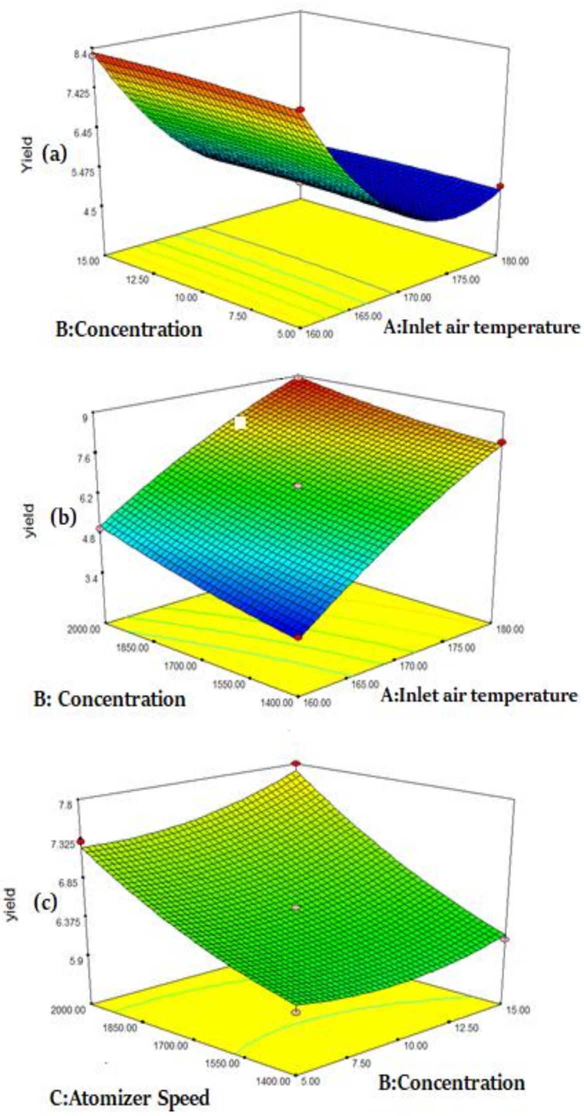


Figure 3. Response surface plots: effect of independent variables on the product yield

The probability value of 0.01 signifies the statistical stability of the responses and their agreement with a lack of fit value. The greater adequate precision value suggests that the respective model equations are valid for the experimental data. Figure 4 shows the experimental and predicted values of yield suggesting a close agreement between the predicted and actual values of the yield.

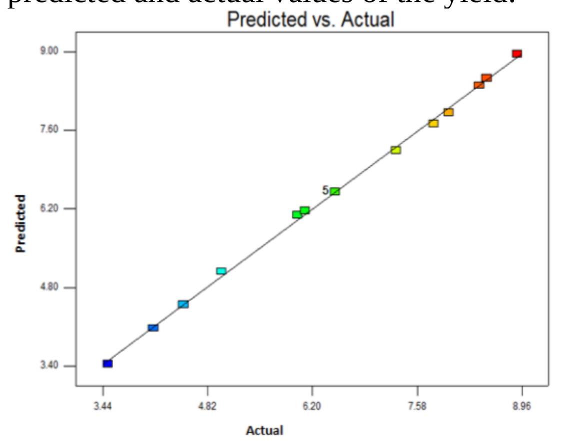


Figure 4. Experimental and predicted yield of powder

3.2. Angle of repose (Ɵ)

If the angle of repose value is less than 40°, the flow behavior of the powder is classified as an easily flowable type. In Figure 5, the variation of the angle of repose concerning the three independent variables is represented. The values of angle of repose were less than 40° in all the experiments. With respect to the inlet air temperature, the angle of repose exhibits a maximum value of 32.4 °C. The effect of atomizer speed and concentration of the carrier agent was quite insignificant as the variation in the value of Ɵ is very small as shown in Fig 4(a). The inlet air temperature has a significant effect on the angle repose in comparison to that of the other two input variables as shown in Fig 4(b) and 4(c). Maximum values of angle repose were observed approximately in the range of 165°-170°C and ANOVA results are satisfactory (data not shown). The angle of repose (Y_2) is written as

$$R_2 = 32.0 - 2.07\,A + 0.17\,B + 0.27\,C + 0.062\,AB - 0.20\,AC + 0.35\,BC - 4.26\,A^2 - 0.35\,B^2 - 0.19\,C^2$$

Table 3. Analysis of variance (ANOVA) for the fitted polynomial model of yield

Source	Sum of squares	df	Mean Square	F value	P-Value Prob > F
Model	31.30	9	3.48	468.84	<0.0001 significant
A-Inlet air temperature	21.81	1	21.81	2940.61	< 0.0001
B-Concentration	0.014	1	0.014	1.84	0.2176
C-Atomizer speed	0.013	1	0.013	1.73	0.2304
AB	0.024	1	0.024	3.24	0.1149
AC	0.036	1	0.036	4.87	0.0632
BC	0.044	1	0.044	5.95	0.0449
A^2	9.27	1	9.27	1249.62	< 0.0001
B^2	0.011	1	0.011	1.49	0.2616
C^2	0.016	1	0.016	2.13	0.1879
Residual	0.052	7	7.418×10^{-3}		
Lack of Fit	0.052	3	0.017		
Pure Error	0.000	4	0.000		
Cor Total	31.35	16			
Std. Dev	0.086	R^2	0.9983		
Mean	5.80	Adj R^2	0.9962		
C.V. %	1.48	Pred R^2	0.9735		
PRESS	0.83	Adeq Precision	54.291		

3. Hygroscopicity

Hygroscopicity is an important parameter for the long time storage and shelf life. The hygroscopicity values of food powders should generally be lesser. With a careful observation of Fig 6(a-c), one can see that the inlet air temperature had a great significance on the hygroscopicity of powder as the hygroscopicity increased with the increase of inlet air temperature of the spray drying. The increased removal of mositure quantity from the powder with the increase of inlet air temperature could be a reason for this trend. The hygroscopicity increased from 27% to 39 % for the temperature increase from 160℃ to 180℃ (Fig 6(a)). The effect of the other two independent variables was quite insignificant. The hygroscopicity is related to the three independent variables as given below

$$R_3 = 32.14 - 5.48\,A - 0.29\,B - 0.16\,C - 0.025\,AB + 0.23\,AC + 0.047BC + 0.88A^2 + 0.4B^2 - 0.24\,C^2$$

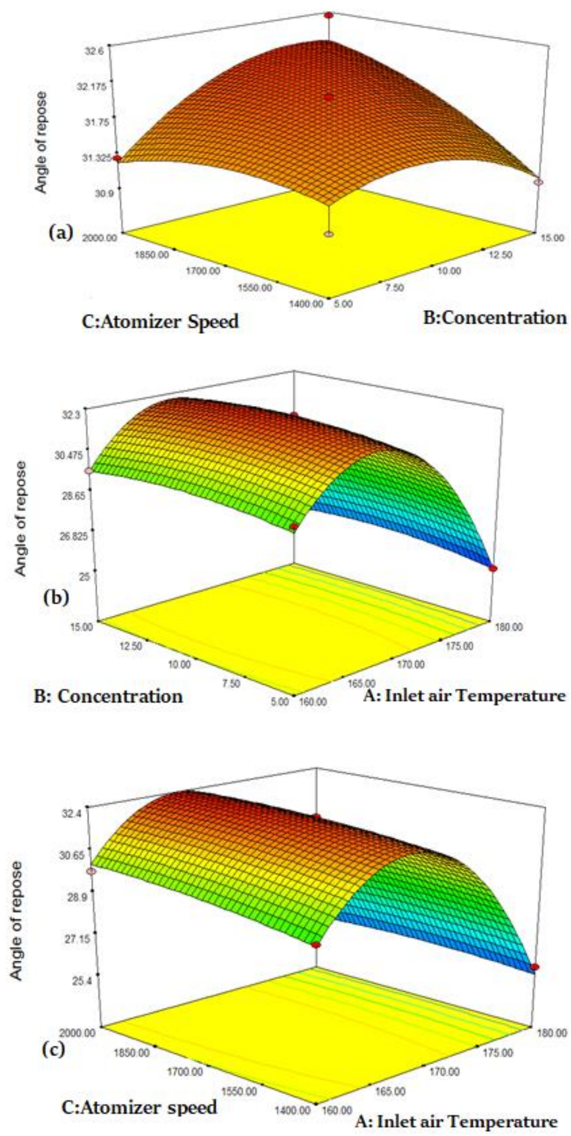


Figure 5. Response surface plots: effect of independent variables on the angle of repose of powder

3.4. Moisture content

The moisture content decreased with an increase in inlet air temperature (Fig 7(a) and Fig 7(b)). It is obvious since higher inlet air temperatures cause increased moisture removal from the powder. The effect of the other two input parameters was again not much pronounced as evident in Fig 7(c).

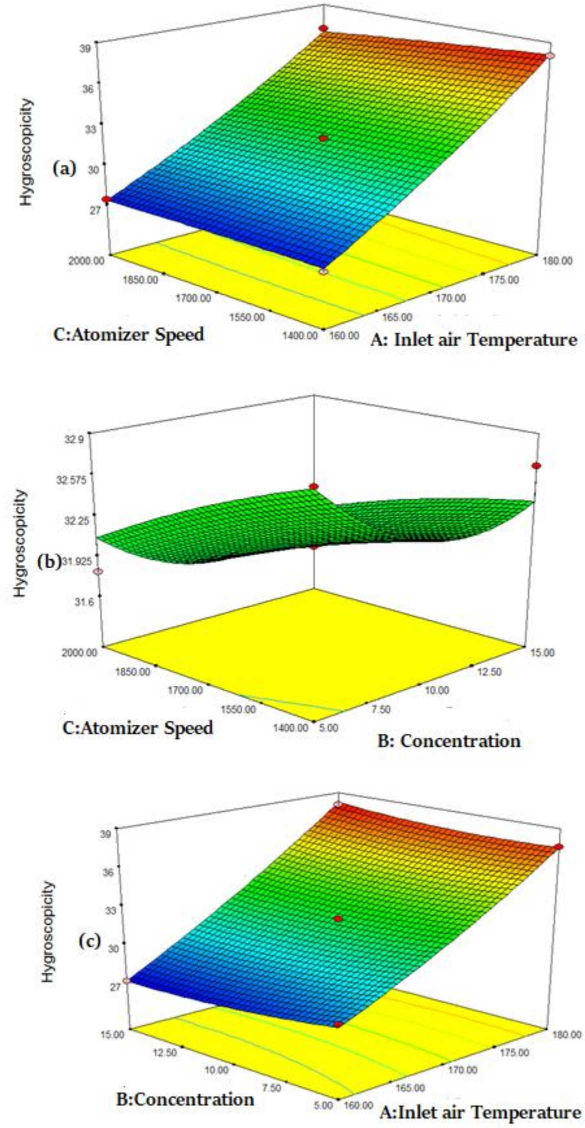


Figure 6. Response surface plots: effect of independent variables on Hygroscopicity of spray-dried coconut milk powder.

The equation for moisture content is obtained as given below.

$$R_4 = 6.80 - 2.42A + 0.028\,B + 0.029\,C - 0.99\,AB + 0.13\,AC + 0.068\,BC + 0.83\,A^2 - 0.29\,B^2 + 0.50\,C^2$$

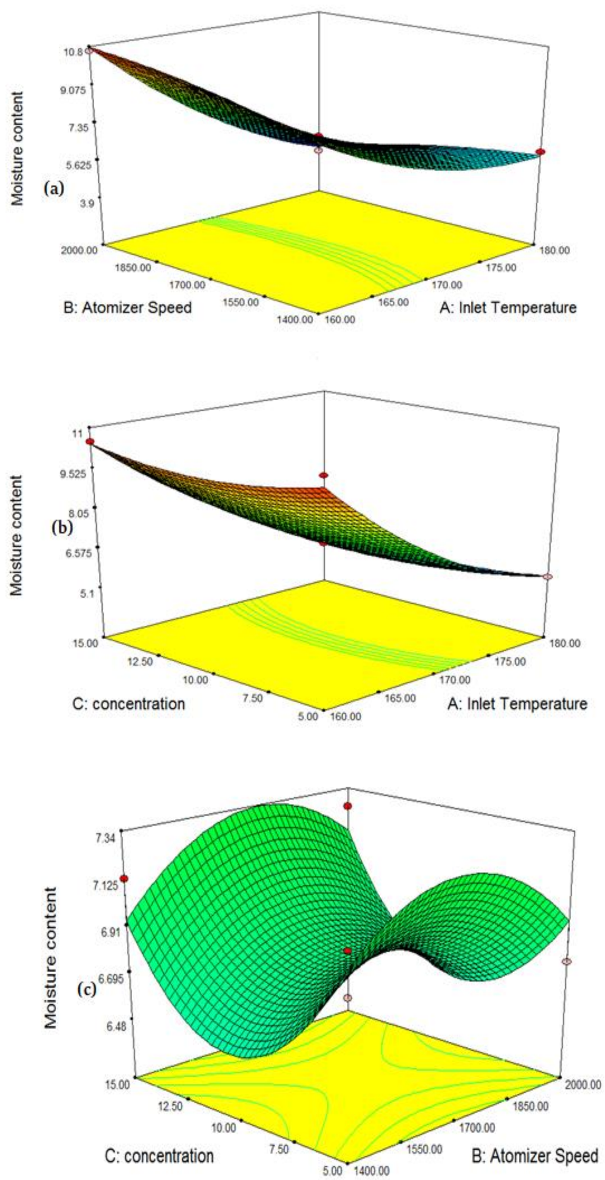


Figure 7. Response surface plots: effect of input variables on moisture content of spray-dried coconut milk powder.

3.5. Optimization

The overall desirability of the responses concerning the independent variables wass 0.94. The optimized values for the three independent variables are 160°C (inlet air temperature), 9.71w/w (maltodextrin concentration), 1400 rpm (atomizer speed). For these input values, the product yield, moisture content, angle response, and hygroscopicity are 8.14 g/200 mL of coconut milk, 8.35 wt%, 29.17°, and 27.01 respectively.

3.6. Flow roperties of the powder

In Table 4, the flow properties of the milk powders of the spray drying experiments were tabulated. It can be seen that an increase in the inlet air temperature increases the insolubility index values. Also, the higher inlet air temperatures result in the denaturing and can adversely effect the quality. The dispersibility values of the coconut milk powders were in the acceptable range of 80.55 to 96.2%. The obtained compressibility Index (*Carr's Index*) values were in the range of 0.5-3.6. The Hausner ratio values range from 1.19 to 1.22 The results show that the powders exhibits good flowability.

The physical and flow properties of the optimized powder, is compared to those of a commercial coconut milk powder sample. All the experiments were done in triplicates and the average values were tabulated in Table 5. The optimized powder exhibits desirable values of the dispersibility, flowability, and hygroscopicity. The values of the other parameters (moisture content, angle of repose, bulk density, tapped density) were comparable to those of the commercial powder.

3.7. Angle of internal friction

The flowability of the powders depends on many factors which include physical properties of the powder and the quality of the equipment used for handling, storing, and processing. Here, to calculate the angle of internal friction of the two powder samples (prepared and commercial), the direct shear method was used. This Method evaluates the shear force required to overcome the cohesive strength at different vertical loads. The shear stress (S) at the failure was plotted against the normal stress (σ) and the curve can be fitted to a linear equation as

$$S = C + \sigma \tan \Theta \quad (7)$$

where S: shear strength; σ: normal stress; C: cohesion; Θ: angle of shearing resistance.

Table 4. Flowability properties of the powder samples

S. No	**Inlet Temperature (°C)**	**Concentration (w/w)**	**Atomizer Speed (RPM)**	**Bulk (g/cm^3)**	**Tapped Density (g/cm^3)**	**Hausner Ratio**	**Compressibility Index**	**Solubility index (mg)**
1*	180	10	1400	0.214	0.219	1.023	2.283	14.25
2	180	10	2000	0.208	0.215	1.033	3.256	16.85
3	170	10	1700	0.317	0.417	1.460	23.98	10.01
4	170	10	1700	0.339	0.4	1.18	15.25	10.15
5	160	15	1700	0.3077	0.363	1.18	15.23	9.41
6	170	10	1700	0.384	0.465	1.211	17.42	10.12
7	160	5	1700	0.238	0.293	1.231	18.77	9.34
8	170	5	1400	0.333	0.377	1.132	11.67	10.23
9	180	5	1700	0.268	0.272	1.015	1.47	16.52
10	170	15	2000	0.379	0.413	1.09	8.232	10.03
11	170	5	2000	0.377	0.416	1.10	9.375	10.27
12	170	10	1700	0.384	0.408	1.06	5.88	10.16
13	160	10	2000	0.488	0.625	1.28	21.92	9.21
14	170	15	1400	0.476	0.625	1.31	23.84	10.28
15*	160	10	1400	0.212	0.236	1.11	10.17	9.18
16	180	15	1700	0.202	0.215	1.065	6.046	15.32
17	170	10	1700	0.235	0.289	1.23	18.685	10.15

* Dispersibility tests were conducted for the powders of these test runs (For (1) 80.79% and (15) 92.36%

Table 5a. The properties of commercial coconut milk powder and the powder synthesized at optimal conditions

Bulk density (g/cm^3)	Tapped Density (g/cm^3)	Hausner Ratio	Compressibility Index	Flowability Index	Solubility (sec)	Dispersibility (%)
0.215 (0.236)	0.302 (0.263)	1.40 (1.14)	28.80 (10.04)	0.216 (0.181)	298 (272)	94.3 (87.5)

Moisture content (%)	The angle of repose (Θ)	Hygroscopicity(g/100g)
9.56 (8.35)	30.2 (29.17)	31.2 (27.01)

The variation of shear stress with respect to normal stress values of the two samples were shown in Fig 8 (a) and 8(b). The obtained Θ values for both samples were close to 30° and it can be interpreted that the samples exhibit good flowability.

3.8 Color analysis

Color is a sensory attribute that can be evaluated as the measure of quality and consumer acceptability. Color analysis of both the optimized and commercial powder were evaluated values are represented by *L**, *a** and *b** using KONICA MINOLTA spectrophotometer.

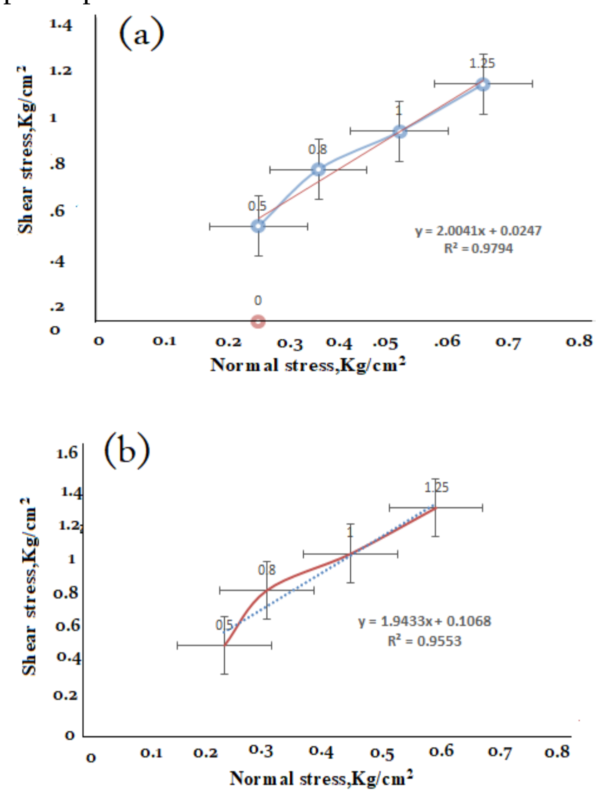


Figure 8. Plots showing the angle of friction for (a) optimized and (b) commercial powder**.**

Table 6 shows that the values of the both optimized and commercial powder are very much similar color attributes. The *L** values indicated that the colors of both powders were more aligned towards brightness. The negative and positive values of *a** and *b** imply lighter green and yellow shades of the powders respectively. The overall appearance of both powders was white and similar.

Table 6. The color analysis of the powders

Item	*L**	*a**	*b**
Optimized powder	70.66	-0.27	3.5
Commercial powder	70.89	-0.22	3.61

3.9. Differential Scanning Calorimetry (DSC)

Using differential scanning calorimetry (DSC), a plot of the temperature of powder sample versus heat flow (mW) difference between the sample and the reference was plotted. In the plot curve, a number of phase changes (glass transition, cooling crystallization, melting etc.) for the sample can be witnessed. After the glass transition, there would be the relaxation of enthalpy in the DSC curve. The DSC curves of two powder samples (commercial and optimized) are shown in Figure 9.

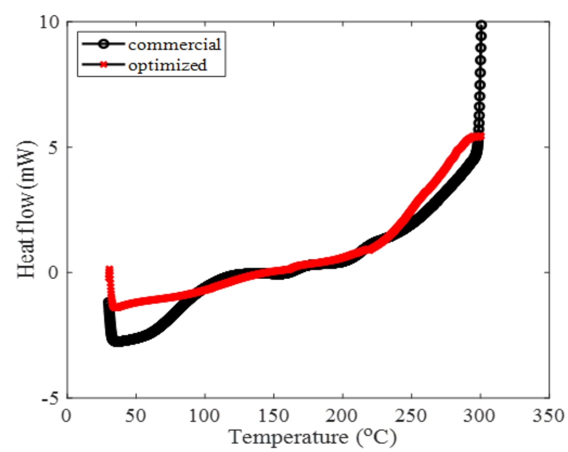


Figure 9. DSC Thermograms of commercial and optimized powder

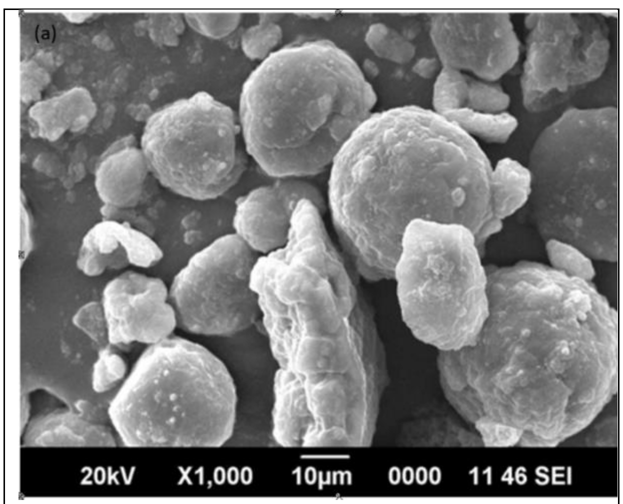
Glass transition temperatures of commercial and optimized powders were estimated to be around 120.3°C and 152°C respectively. The higher glass transition temperature of the optimized powder ensures its adaptability at the elevated temperatures with no loss of quality upon storage.

3.10. Scanning Electron Microscopy (SEM)

SEM images of the commercial coconut milk powder and the milk powder prepared at the optimum conditions are shown in Figure 9. The commercial and synthesized powders were imaged after 30 days of storage to study the structural stability of the powders. Figure 9 (a) shows that the particles of synthesized powder at the optimum conditions were globular with desirable structural stability. On the other hand, the commercial powder exhibited undesirable distorted structures with water bridges formed among the non-globular and irregular particles of the powder.

4. Conclusions

The operation of spray drying of coconut milk to milk powder was optimized using response surface methodology (RSM). Box-Behnken design of experiments approach was used for this purpose. The effect of input variables on the responses were analyzed using the 3D surface plots between the inputs and responses. In the analysis, it was found that inlet air temperature in the spray drying chamber has an significant effect on the physical properties of the powder. The solubility, flowability and dispersibility values of the powders synthesized were in the acceptable range.



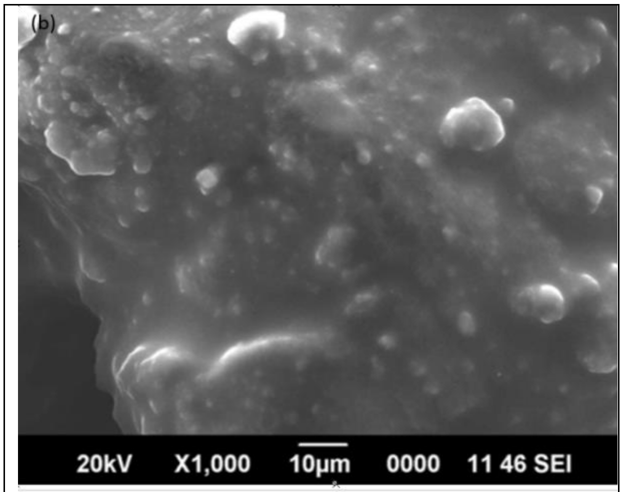


Figure 9. SEM images of (a) powder synthesized at optimum conditions and (b) commercial powder

The optimized values of the input parameters were obtained as follows: inlet temperature 160 °C, Maltodextrin concentration 9.71 w/w, atomizer speed 1400 rpm. Correspondingly, for the powder synthesized at the optimal conditions, the values of the four responses were as follows: 8.14 g/200 ml of milk (yield), 8.35 wt% (moisture content), 29.17° (angle of repose), and 27.01 (hygroscopicity). The properties of the powder prepared at the optimal conditions were compared with those of a commercial powder sample and found to be in god agreement. Moreover, the optimized powder exhibited superior structural stability.

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Acknowledgment

We gratefully acknowledge *Department of Food Technology*, *TKM Institute of Technology*, Kollam, Kerala, for the permission to perform the experiments in their laboratory.