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Optimization of Drying Process for Production Red Ginger Granulated Palm Sugar Using Response Surface Methodology

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Abstract

Palm sugar is used as a natural sweetener that is added to food and beverages. The nutritional content in palm sugar can be enriched with the addition of antioxidants derived from red ginger (Zingiber *officinale var. rubrum).* The antioxidant activity of red ginger is 75.61% higher than that of emprit ginger and elephant ginger. The problem that arises during the production process of granulated palm sugar is the drying process that has not considered chemical characteristics, especially water content. The requirement for water content of palm sugar according to SNI 01-3743-1995 is a maximum of 3%. Moisture content is the main parameter that determines the quality of granulated palm sugar to long shelf life. Water content can affect other chemical characteristics such as sucrose, reducing sugar, ash content, calories, protein, fat, and carbohydrates. This optimization is using RSM (Response Surface Methodology) CCD model (Central Composite Design) on Software Statistica 10 with 20 treatments. The independent variables used were time (4-6 hours), material weight (100-300) grams, and material size (10-26 mesh). Data processing with Statistica 10 software resulted in the optimum water content condition of 2.9019%, with the drying process conditions covering 6.68 hours, material weight 368.18 grams, and material size 31.45 mesh. Validation was carried out to test the accuracy of the optimization results from Statistica 10 Software. The validation of the moisture content results obtained a value of 2.9016%, with an error percentage of 0.0003%. The chemical characteristic test was applied to the granulated palm sugar as a result of optimization so that the value of sucrose (96.5967%) was obtained, reducing sugar (6.0434%), ash content (1.8660%), calories (379.93%), protein (2,4268%), fat (0.3972%), and carbohydrates (91,5379%).

INTRODUCTION

Granulated palm sugar is one of the natural sweeteners that is rich in benefits and is consumed by the community as a food or drink sweetener (Pratama, et al., 2020). Granulated palm sugar has a lower glycemic index value than cane sugar, which is 35% (Wilberta et al., 2021). In addition, granulated palm sugar also contains several compounds useful for the body, such as calories, carbohydrates, fat, protein, and calcium (Fatriani et al., 2019). The content of beneficial compounds in granulated palm sugar can be enriched by adding antioxidants obtained from spices, one of which is red ginger (Zingiber officinale var. rubrum) (Wulandari et al., 2021). Red ginger can increase the body's response to fighting viruses. Including the chikungunya virus, pharyngitis, and COVID-19 (Assegaf et al., 2020; Kaushik et al., 2020; Sinamo & Hutabarat, 2021).

Granulated palm sugar has a shelf life of 1 year, longer than jaggery sugar, lasting for two weeks. The shelf life of granulated palm sugar is influenced by the water content (Ritonga et al., 2020). The drying process is an essential part of the production of granulated palm sugar to obtain a minimum water content. Optimum drying operating conditions will produce granulated palm

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sugar with minimum moisture content (Meldayanoor et al., 2019).

In general, the conventional drying process of granulated palm sugar still use the sunlight. The disadvantages of this drying method are the poor physical quality of sugar and high water content (Muhandri et al., 2020). These weaknesses can be overcome by determining the optimum operating conditions of the granulated palm sugar drying process. Parameters that are considered to produce granulated palm sugar water content according to SNI 01-3743-1995 standards are drying time, material weight, and material size using the Response Surface Methodology (RSM) method. So that, it fulfill the water content standard according to SNI 01-3743-1995, which is a maximum of 3 %. The purpose of this study was to determine the effect of optimum drying operating conditions on granulated palm sugar.

MATERIALS AND METHODS

Materials

The materials used in this study were palm sugar obtained from the Palm Sugar Production Center Limbangan and red ginger obtained from the Karang Ayu Market, Semarang. Materials used for analysis include 30% HCl (Merck), 45% NaOH (Merck), 30% NaOH (Merck), Luff's reagent, aquades, 20% KI (Merck), Na-thio 0.1 N, H_2SO_4 26 ,5%(Merck), starch indicator, arsenomolybdate reagent, concentrated H_2SO_4(Merck), 2% boric acid, 0.01 N HCl (Merck), 30% HCl (Merck).

The tools used in this study were oven, thermometer, analytical scale, wooden stirrer, frying pan, stove, coconut shell, sieve, baking sheet, grater, measuring cup, filter cloth, mesh sieve, brush, and spoon. The tools used for analysis include vochdoos, 100 mL Erlenmeyer, 100 mL volumetric flask spectrophotometer (UV Vis Shimadzu), test tube, water bath, furnace (Thermolyne 1000), and a cup.

Methods

This research was conducted in several stages, namely the production of red ginger granulated palm sugar, optimization with RSM (Response Surface Methodology) using Statistica 10 software, and testing the chemical characteristics of red ginger granulated palm sugar.

Red Ginger Granulated Palm Sugar Production

The red ginger that was cleaned is grated using a grater to extract the juice. Red ginger juice is separated from starch deposits through a filtering process. The red ginger juice is then cooked until it boils. The boiling red ginger juice is added with palm sugar that has been cut into pieces. Cooking is done until the sugar crystallizes. The granulated palm sugar that has been formed is sieved using a sieve to obtain granulated palm sugar based on material size. The palm sugar that passed the filter then dried using an oven to fullfil water content standard set by SNI, a maximum of 3%.

Optimization With RSM (Response Surface Methodology)

Optimization with RSM is based on research that has been done by (Mariana 2017). The RSM method is used to obtain data based on time, material weight, and material size so the water content of granulated palm sugar samples fulfill the SNI standards is a maximum of 3% (SNI, 1995).). The experimental design was carried out using the Central Composite Design (CCD) with 20 trials using the Statistica 10 software.

Moisture Test

The water content during drying of granulated palm sugar is calculated based on the following gravimetric equation (Normilawati et al., 2019).

$$\%\text{moisture level} = \frac{(W1 + W) - W2}{W} x\ 100\% \qquad (1)$$

Where, W is the weight of granulated palm sugar sample before drying (grams), W1 is the weight of vochdoos after drying (grams) and W2 is the weight of vochdoos + granulated palm sugar sample after drying (grams).

Sucrose Test

50 ml of the sample filtrate was put into an Erlenmeyer added with 25 ml of distilled water and 10 ml of 30% HCl. Heat the solution on a water bath at 200°C. The solution is neutralized with 45% NaOH then diluted to a specific volume so that the solution contains 15-60 mg of reducing sugar. A total of 25 ml of the solution was taken and put into an Erlenmeyer, then added 25 ml of Luff Schoorl's solution. The solution in the Erlenmeyer was

cooled rapidly, and 15 ml of 20% KI was added, then carefully added 25 ml of 26.5% H2SO4 solution. The iodine is titrated with a 0.1 N Na-thiosulfate solution using 2-3 ml starch indicator. Calculation of sucrose level using the Eq. (2)

$$\begin{aligned}&\text{Sucrose level } = \\&(\text{Reducing sugar content after inversion} \\&- \text{Reducing sugar content after inversion}) \\&\times\ 0.95\end{aligned} \qquad (2)$$

Reducing Sugar Test

Testing for reducing sugar was carried out using the Nelson Somogyi method (Haryanti, 2020). A total of 1 ml of a solution containing 0.02-0.08 mg/mL of glucose solution and 1 ml of Nelson's reagent was put into a test tube. Heated in a water bath for 20 minutes and then cooled. The solution that has been cooled is added with 1 mL of Arsenomolybdate reagent, then shaken until the Cu2O precipitate dissolves. After the precipitate dissolves, add 7 mL of distilled water, then shake until homogeneous. Optical Density (OD) was read by spectrophotometry at 540 nm wavelength.

Ash Level Test

Testing the ash content is carried out at temperatures above 450 °C (Yenrina et al., 2015). 3 - 5 grams of the sample is placed on a cup and put into an ashing furnace. The heating temperature is carried out in the first stages of 400 °C then the second stage is heated to 550 °C. The sample which has become ash is cooled in a desiccator. Ash content is calculated based on the Eq. (3).

$$\text{Ash level} = \frac{\text{C} - \text{A}}{\text{B} - \text{A}} \text{x } 100\% \qquad (3)$$

Where, A is the weight of empty cup, B is the weight of cup + sample and C is the weight of cup + sample (after ashing)

Calorie Value Test

The caloric value is calculated using the bomb calorimeter test (Sogandi, 2019). Calculation of heat of combustion based on the Eq. (4).

$$\Delta \text{U}_T = \frac{\text{C}\Delta\text{T} - \Delta\text{U}_1 - \Delta\text{U}_2}{\text{m}} \qquad (4)$$

Where, ΔU_T is heat of combustion of the sample (cal/g) and C is heat capacity of the calorimeter (cal/°C).

Protein Level Test

Protein level test using Kjeldahl method to determine protein levels (Siska & Apri, 2021). 0.4 grams of granulated palm sugar sample was put into a 100 ml Kjeldahl flask, added 25 ml of concentrated H_2SO_4, and heated for 2 hours. Then cooled and diluted with a 100 ml volumetric flask to the limit. 5 mL of the solution was put into a distillation and added 5 mL of 30% NaOH with a few drops of phenolphthalein indicator and distilled for 10 minutes. 10 ml of the distillation solution was added with 10 ml of 2% boric acid and then titrated with 0.01N HCl solution. The protein content is calculated based on the Eq. (5)

$$\text{Protein level} = \frac{(V_1 - V_2) \times N \times 0{,}014 \times fk \times 100}{\text{w}} \qquad (5)$$

Where V_1 and V_2 are the volume of HCl used for titration of sample and blank solutions, W is the sample weight, N is the normality of HCl and fk is the conversion factor.

Fat Level Test

1-2 grams of sample is weighed and put into a beaker glass. 30 ml of 25% HCl and 20 ml of water were put into a beaker glass, and then some boiling stones were added. The beaker glass was covered with a watch glass and boiled for 15 minutes. After that, the liquid is filtered in hot conditions by washing using hot water to prevent the acid reaction from occurring again. The filter paper and its contents were dried in an oven at 100-105 degrees.

The dried samples were extracted with hexane solvent for 5-6 hours at 80°C. The extracted solution was then distilled to separate the fat extract from the hexane solvent. The fat extract was dried in an oven at 100-105, then cooled and weighed. Calculation of fat content based on the Eq. (6).

$$\%\text{fat} = \frac{\text{mass of extract}}{\text{mass of sample}} \text{x } 100\% \qquad (6)$$

Carbohydrate Test

1 mL of the sample filtrate was added with 25 mL of distilled water. The filtrate and distilled water mixture were heated with a water bath for 1 hour. After that, the sample was dripped with three drops of PP and NaOH with 50% to neutral pH concentration. The neutral solution was diluted into

a 100 mL volumetric flask, shaken, and filtered to obtain the filtrate. The filtrate formed was taken 0.05 mL with a pipette and then added 0.45 mL of distilled water and 0.5 mL of Nelson's reagent. The solution was then heated for 10 minutes and cooled. The cooled solution was added with 4 mL of arsenomolybdate reagent, then vortexed and incubated for 30 minutes to read the absorbance. The absorbance value will be used to calculate the carbohydrate content. Calculation of carbohydrate content with Eq. (7)

$$\%\text{Carbohydrate} = \frac{X\left(\frac{mg}{L}\right) \times fp \times \text{ Total Volume (L)}}{w \times 100} \times 100\% \quad (7)$$

Where, X is the concentration (mg/L), fp is the dilution factor and w is the initial sample weight (kg).

RESULTS AND DISCUSSION

The influence between variables consisting of time, material weight, and material size was used to determine the optimum moisture content of the red ginger granulated palm sugar drying process. The data processing results with statistical software 10 to obtain a relationship between the observed value and predicted value.

The results of the percentage error that obtained were analyzed by MAPE (Mean Absolute Percentage Error). So that a value of 2.58% was obtained. This value indicates that the experiments carried out are very accurate. The value of the data accuracy category on MAPE is as follows:

< 10% = very accurate
10-20% = good
20-50% = reasonable
>50% = inaccurate
(Maricar, 2019)

Based on Table 1. the values have shown the accuracy between the predicted and observed so it can be analyzed to know equations of the mathematical model. The data that were obtained as shown in Table 2.

Based on Table 2, the R^2 value is 0.956. It shows that the model used is accurate because it is close to 1. Adjust R^2 of 0.917 indicates that the relationship between the independent and response variables is getting stronger. The p-value for the time factor (X_1) is 0.8892, and the weight of the material (X_2) is 0.4193, which shows no significant effect on the water content response because the p-value is > 0.05. Based on the interaction effect and significance, the response equation is shown in Eq. (8).

$$\begin{aligned} Y = {} & 2.02379 + 0.00629X_1 + 0.03774X_2 \\ & -0.136877X_3 - 0.18784X_1{}^2 + 0.31622X_2{}^2 \\ & + 0.13226X_3{}^2 + 0.46205X_1X_2 \\ & +0.48250X_1X_3 + 0.30515X_2X_3 \end{aligned} \quad (8)$$

Equation 8 is mathematical modeling in a polynomial equation of order 2 with significant coefficients on the model as in Table 2. The above equation is not the final equation because there are still insignificant effects. The linear factors of time (X_1) and weight of the material (X_2) must be neglected so that Eq. (8) is simplified to Eq. (9)

$$\begin{aligned} Y = {} & 2.02379 - 0.136877X_3 - 0.18784X_1{}^2 \\ & +0.31622X_2{}^2 + 0.13226X_3{}^2 + 0.46205X_1X_2 \\ & +0.48250X_1X_3 + 0.30515X_2X_3 \end{aligned} \quad (8)$$

The interaction effect between the variables obtained was validated using ANOVA, as shown in Table 3.

ANOVA determines the significance value of the independent variables that affect the dependent variable. The error tolerance limit (α) used is 5% or 0.05 so that the confidence level (1-0.05) = 0.95 or 95%. Based on Table 3, the time factor (X_1) and material weight (X_2) have a significance value of 0.88917 and 0.41932. So, these two factors do not affect the value of the optimum water level. However, the quadratic factor of time (X_1^2) and weight of material (X_2^2) had a significant effect on the optimum moisture content because it had a p-value of <0.05, namely 0.00642 and 0.00064. The interaction factor between time variables on the weight of material (X_1X_2), time on the size of the material (X_1X_3), and weight of the material on the size of the material (X_2X_3) has a significant effect on the optimum water level because it has a p-value <0.05, which is 0.00043; 0.00035; and 0.00284.

In addition to the significance test, it is necessary to do a model fit test (Lack of Fit). Lack of fit means a discrepancy so that if H_0 is accepted, then there is no discrepancy in the model, so it means that the model is appropriate (Pertiwi, 2018)

The hypothesis used in this test is as follows:

H_0= There is no lack of fit in the model
H_1 = There is a lack of fit in the model

Table 1. Value of Observed and Predicted Moisture Content

Run	Value			*Response Observed*	*Response Predicted*	*% Error*
	X_1(hour)	X_2(grams)	X_3(mesh)			
1	4	100	10	2.90	2.83	2.56
2	4	100	26	1.91	1.90	0.33
3	4	300	10	2.05	2.10	2.13
4	4	300	26	1.83	1.78	2.62
5	6	100	10	1.81	1.89	4.13
6	6	100	26	1.94	1.93	0.86
7	6	300	10	2.05	2.08	1.62
8	6	300	26	2.63	2.73	3.85
9	3.32	200	18	1.69	1.75	3.75
10	6.68	200	18	1.87	1.76	5.45
11	5	31.82	18	2.41	2.44	1.09
12	5	368.18	18	2.57	2.50	2.51
13	5	200	4.55	2.36	2.33	1.41
14	5	200	31.45	2.10	2.10	0.24
15	5	200	18	2.10	2.02	3.72
16	5	200	18	2.02	2.02	0.34
17	5	200	18	2.14	2.02	5.26
18	5	200	18	1.96	2.02	3.30
19	5	200	18	1.94	2.02	4.14
20	5	200	18	1.98	2.02	2.28

Table 2. Effect Estimation for Polynomial Equations of Order 2

Factor	*Effect*	*Std.Err.*	t	p
Mean/Interc.	2.0238	0.0323	62.5862	0.00000
X_1	0.0063	0.0429	0.1466	0.8892 ($p>0.05$)
X_1^2	-0.1878	0.0418	-4.4971	0.0064
X_2	0.0377	0.0429	0.8796	0.4193 ($p>0.05$)
X_2^2	0.3162	0.0418	7.5704	0.0006
X_3	-0.1369	0.0429	-3.1898	0.0243
X_3^2	0.1323	0.0418	3.1665	0.0249
X_1X_2	0.4621	0.0561	8.2417	0.0004
X_1X_3	0.4825	0.0561	8.6064	0.0003
X_2X_3	0.3052	0.0561	5.443	0.0028
R^2	0.95662	Adjust R^2	0.91758	

For the model to fit, it is necessary to reject H_1 so that H_0 is accepted. In Table 3, the F table value is 1.50524 < F count is 4.77247, and the p table value is 0.33228, which is greater than 0.05, so the conditions for H_0 are accepted. It means that the model is appropriate and there is no model discrepancy so that the second-order polynomial equation model can be accepted.

Determination of Optimum Conditions

The profile and desirability determine the optimum conditions for the drying process. We used it to predict the optimum condition of the process to obtain valuable data for the validation process. The interaction of the three variables is known for its optimum condition by drawing a straight line vertically. The optimum conditions for

Table 3. Results of ANOVA Analysis with Statistica 10 Software

Factor	SS	df	MS	F	p
X_1	0.00014	1	0.00014	0.02149	0.88917 ($p>0.05$)
X_1^2	0.12713	1	0.12713	20.22358	0.00642
X_2	0.00486	1	0.00486	0.77377	0.41932 ($p>0.05$)
X_2^2	0.36026	1	0.36026	57.31085	0.00064
X_3	0.06396	1	0.06396	10.17489	0.02427
X_3^2	0.06303	1	0.06303	10.02642	0.02491
X_1X_2	0.42698	1	0.42698	67.92509	0.00043
X_1X_3	0.46561	1	0.46561	74.07078	0.00035
X_2X_3	0.18623	1	0.18623	29.62641	0.00284
Lack of Fit	0.04731	5	0.00946	1.50524	0.33228
Pure Error	0.03143	5	0.00629		
Total SS	18.151	19			
R^2	0.95662	Adjust R^2	0.91758		

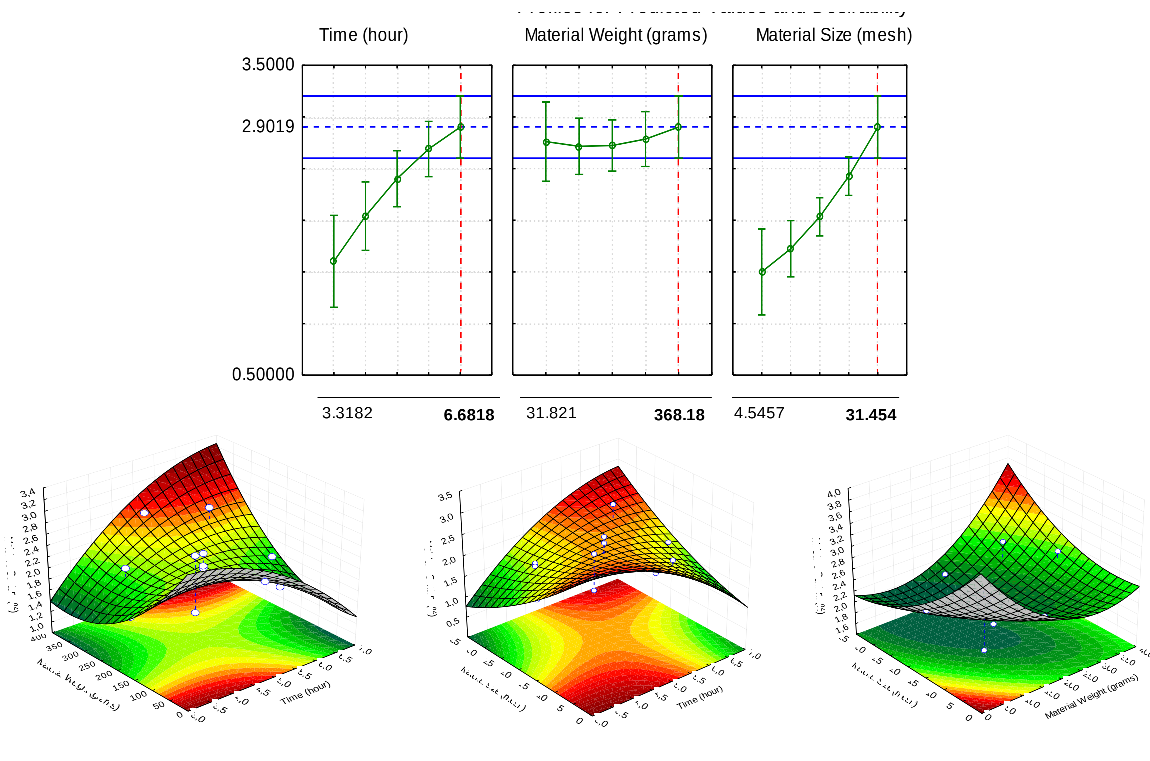

Figure 1. Optimum Condition of Drying Process on Moisture Level

the drying process of granulated palm sugar are shown in Figure 1.

A desirability value of 1 indicates that the goal solution has been fulfilled. While a desirability value of 0 indicates that the response must be discarded (Nurmiah et al., 2013). The prediction model produces a moisture response of 2.9019% with an optimum condition of 6.68 hours, material weight 368.18 grams, and material size 31.45 mesh. The surface plot shows that the optimization of time, material size and material weight can affect the moisture content of granulated palm sugar.

Validation of Optimum Conditions for Drying Process Optimization Results

The optimum condition of granulated palm sugar was validated by running process and testing the water level. It aims to determine the

Table 4 Validation Results of Predicted Value and Observed Value of Software Statistics

Variable	Optimum Value Based on Statistica 10 Software	Water Level of Granulated palm Sugar	
		Predicted value	Observed value
X_1	6.68 hour		
X_2	368.18 gram	2.9019%	2.9016%
X_3	31.45 mesh		
	% Error	0.0003%	

Table 5. Chemical Characteristics of Red Ginger Granulated Palm Sugar Test Results

Chemical Characteristics	Test Results	Quality qualification SNI 01-3743-1995	Information
Sucrose	96.5967%	Minimum 90%	Fulfilled
Sugar reduction	6.0434%	Minimum 6%	Fulfilled
Ash level	1.8660%	Maximum 2%	Fulfilled
Caloric	379.8951 kcal	379.93 kkal	Fulfilled
Protein	2.4268%	2.45%	Fulfilled
Fat	0.3972%	0.41%	Fulfilled
Carbohydrate	91.5379%	91.61%	Fulfilled

match between the observed values from running validation and the predicted values from optimization with RSM. The validation value of the optimization results is shown in Table 4.

Based on the data in Table 4, the difference in the response value between the prediction software and the actual data is 0.0003%. The difference in value is less than 5% so that the validation results can be accepted. It proves that the test results' water content matches the program's predicted value (Mariana, 2017).

Chemical Characteristics of Red Ginger Granulated Palm Sugar Optimum Validation Results

The chemical characteristics observed include sucrose, sugar reduction, ash level, calories, protein, fat, and carbohydrates are presented in Table 5.

The sucrose level of the test results showed a value of 96.5967%. This value is under the quality requirements of SNI 01-3743-1995, which is at least 90%. A significant sucrose value affects the durability of granulated palm sugar because it is associated with a low content of reducing sugar, so it is more durable during storage (Zuliana et al., 2016). It is appropriate because the reducing sugar content in palm sugar ant optimization results shows a low value of 6.0434%. This value is close to the minimum condition stated in SNI 01-3743-1995, at least 6%.

Low reducing sugar levels indicate the quality of the granulated palm sugar produced is good, because the sugar becomes stable and does not melt quickly (Indahyanti et al., 2014). The ash content of the test results shows a value of 1.8660%. It follows the quality requirements of SNI 01-3743-1995, which is a maximum of 2%. The ash level is related to the preservative level added in palm sugar. The higher the ash level in palm sugar, indicates the presence of mineral additives, one of which is preservatives added during the cooking process (Haryanti, 2020). Other chemical characteristics that are not a quality requirement for palm sugar in accordance with SNI 01-3743-1995 include calories, protein, fat, and carbohydrates. The results of the test on these characteristics are calories (379.8951 kcal), protein (2.4268%), fat (0.3972%), and carbohydrates (91.5379%). This value has met the quality of the Banten Agricultural Technology Assessment Agency (Heryani, 2016).

CONCLUSION

The second order polynomial equation with RSM has an R^2 value of 0.95662 with the simplified equation. The optimum operating condition of the drying results showed a response of water content of 2.9016% with a percentage error of 0.0003%, affecting the chemical characteristics of granulated palm sugar so that it fulfill the quality requirements of SNI SNI 01-3743-1995, sucrose (96.5967%), sugar reduction (6.0434%), ash level

(1.8660%), calories (379.8951 kcal), protein (2.4268%), fat (0.3972 %) and carbohydrates (91.5379%).

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