A response surface methodology for optimizing the non-gluten over ripe canistel powder formulation

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Abstract. Over ripe canistel fruit contains high sugar and carotenoids. As it is an under-utilized fruit, it can be preserved by making it into powder beverage. In the processing of over ripe canistel powder, it is required an amount of water, maltodextrin and tricalsium phosphate (TCP). The objective of this study was to obtain the optimum formula of over ripe canistel powder using vacuum drier. Optimization was done using Design-Expert 7.0 Response Surface Methodology (RSM) software. Formula used in this study consisted of water 53-63%, maltodextrin 10-20%, and TCP 0-2%, with total with total 75% from the total interaction formulas and canistel fruit 25%. Responses of moisture content, yield, and flowability were analyzed. The formula of over ripe canistel powder significantly affected the yield and flowability, but did not significantly affect the moisture content. The optimum formula given by the Design-Expert 7.0 RSM software was 53.94% water, 19.06% maltodextrin, 2% TCP, with desirability value 0.729. The optimum formula gave prediction response values moisture content 4.37%, yield 26.12%, and flowability 30.14%. Results of verification for the optimum formula of over ripe canistel powder were moisture content 4.62%, yield 28.40%, and flowability 31.68%.

1. Introduction

Canistel (*Pouteria campechiana*) is a plant originating from Mexico that can grow in tropical and subtropical climate such as Indonesia (Kanak and Bakar, 2018). In Indonesia, canistel fruit is commonly found in West Java, especially in Padalarang and Cirebon. Canistel fruit contains alkaloids, tannins, flavonoids, steroids, and terpenoids (Mehraj et al. 2015). Based on Pertiwi et al. (2018), over-ripe canistel fruit has a softer texture, contains lower starch and more sugar than full slip canistel fruit. Over ripe canistel fruit can be utilized as canistel powder. The canistel powder produced will have a longer shelf life and can be used as a mixture of cake-making, cookies, ice cream and non-gluten food sources (Kanak and Bakar, 2018).

The principle of processing fruit into fruit powder is to reduce the moisture content in fruit juice to a certain extent so that microbial growth and enzyme activity can be prevented (Shishir and Chen 2017). Utilization of over-ripe canistel fruit into powder is done by drying process. One suitable drying method for agricultural products that is sensitive to high temperature but able to maintain the product quality is vacuum drying (Ando et al. 2018; Ramos et al. 2019).

The high sugar content in the over-ripe canistel will cause stickiness during drying. In an effort to reduce stickiness in canistel powder, it is necessary to add filler such as maltodextrin to produce non-sticky powder (Ramos et al. 2019), whereas to improve flowability and to inhibit clumping of fruit powder, tricalcium phosphate (TCP) is added (Aguilar et al. 2018).

In this study, the optimum formula for water concentration, maltodextrin and TCP used in the production of over-ripe canistel powder was determined. Determination of the three ingredients concentration was carried out using Response Surface Methodology (RSM). According to Rosidah et al. (2017), the advantage of using the RSM method is that it is faster and informative compared to the approach of one classic variable or a complete factorial design.

2. Material and Methods

This research method was based on Myres et al. (2016) which had four stages. The stages were determining the formula, making the product according to the formula, testing the product responses and data analysis, and finally the formula optimization and verification of the optimum formula.

2.1 Formula determination

The formulation process was carried out using design expert DX 7.0 (trialversion) from stat ease with mixture design D-optimal. The research factors were water concentration (A) in fruit pulp making, the concentration of maltodextrin (B) as filler material and concentration of TCP (C) as anticaking agent. The lower and upper limit on each factor were (A) 53-63%, (B) 10-20%, (C) 0-2%. In which the amount of (A+B+C) was 75% while the 25% remain was over-ripe canistel fruit flesh. In this method, thirteen formulas were chosen by the design expert program randomly according to the statistical method or research design used (Table 1).

Table 1. Optimization of over-ripe canistel powder formulation using RSM

Formulation	(A) Water (%)	(B) Maltodextrin (%)	(C) TPC (%)
1	55.245	17.756	1.999
2	59.806	15.194	0.000
3	53.004	20.000	1.996
4	63.000	10.669	1.331
5	53.004	20.000	1.996
6	55.000	20.000	0.000
7	56.726	18.274	0.000
8	58.132	14.868	2.000
9	61.207	13.043	0.750
10	63.000	12.000	0.000
11	59.440	13.560	2.000
12	57.699	16.359	0.942
13	63.000	10.669	1.331

Source: d*esign expert* output (2018).

2.2 The production of over-ripe canistel powder

Canistel powder was made using vacuum drying method. In the amount of 25% of over-ripe canistel flesh was mixed with water, maltodextrin, and TCP with the concentration of each formula to produce canistel fruit pure. Canistel fruit pure was then vacuum dried at a temperature of 60ºC and a pressure of 65-70 cmHg until it produced over-ripe canistel powder.

2.3 Product response test and its data analysis

The product response parameters tested in this study were moisture content (AOAC 1995), yield (AOAC 1995) and flowability (Shishir *et al*., 2014). The data were then processed by the

Design Expert 7.0 program with RSM statistical design of mixture design D-optimal method. The design expert program provided choice of polynomial model for each response, those were mean, linear, quadratic, cubic, and special cubic.

Based on the instructions from the application of design expert 7.0, there were three stages to accomplish polynomial equation, which was based on sequential model sum of squares [Type I], lack of fit tests, and model summary statistics. The sum of squares would choose the highest order of polynomial equation from one response variable whose variance analysis (ANOVA) gave significantly different result. Lack of fit tests would choose the order of the highest polynomial equation which gave the results that were not significantly different in terms of deviations in the response. The model summary statistics would select the order of the polynomial equation which contributed on the maximum "Adjusted R-squared" and "Predicted R-squared" values.

A response variable could be said significantly different or significant at the 5% significance level if the p value "prob > F" result of the variance analysis (ANOVA) was smaller than 0.05. The response variable whose significantly different result in the analysis of the variance could be used as a prediction model because the test variable had a significant effect on the response of the formula. Furthermore, the model considered most suitable would be displayed in contour plot in the form of two-dimensional (2-D) or three-dimensional (3-D) graph.

2.4 Optimization and selection of best formula

The optimization process was conducted to obtain a formula producing the optimal response according to the desired optimization target. The optimization value achieved was known as the desirability value which was indicated by the value in the interval of 0 - 1. The higher the desirability value, the higher the suitability of the formula obtained to achieve the optimal formula with the desired response variable. In determining the optimization target, scaling of interest was done for the desired goal. This scaling was called importance if it was selected from 1 (+) to 5 (+++++) depending on the importance of the response variable in question. The more positive signs given indicated the importance of the higher response variable. Based on the predetermined optimization target, the program design expert would provide the optimum formula solution which would then be proceed to the verification stage to ensure the correct prediction of the selected formula response values and the results of the laboratory research responses obtained.

3. Results and Discussion

The results of the response analysis based on the 13 formulas of trial design and the responses of the Design Expert 7.0 program with the RSM statistical design of the mixture design D-optimal method are shown in Table 2.

Table 2. Response of over-ripe canistel powder formula

Formula	Factor			Response		
	Water (%)	Maltodextrin (%)	TCP (%)	Moisture content (%)	Yield (%)	*Flowability* (%)
1	55.245	17.756	1.999	3.636	24.249	23.457
2	59.806	15.194	0	3.134	24.244	35.135
3	53.004	20	1.996	4.715	29.038	31.149
4	63	10.669	1.331	4.358	17.388	43.892
5	53.004	20	1.996	4.702	28.052	39.922
6	55	20	0	4.108	23.849	37.012
7	56.726	18.274	0	4.987	24.853	39.867
8	58.132	14.868	2	4.436	17.537	22.704
9	61.207	13.043	0.75	4.828	20.362	32.456
10	63	12	0	5.509	18.103	36.061
11	59.44	13.56	2	3.299	14.830	30.725
12	57.699	16.359	0.942	4.657	23.159	33.333
13	63	10.669	1.331	4.435	16.964	41.851

Sumber: *output design expert* (2018).

3.1 Moisture content of canistel powder

Moisture content is the amount of water contained in the material expressed in percent. Moisture content in food ingredients also determines the freshness and durability of these foods. The moisture content response test results ranged from 3.13% -5.51% (Table 2). Wong et al (2015) and Sangamitrha et al (2015) stated that fruit powder containing moisture content that less than 5% can prevent enzymatic reactions and damages caused by microbial activity. The results of the analysis carried out by the expert design program produced the polynominal model of the response of moisture content, which is shown in Table 3 and the three-dimensional graph forms are shown in Figure 1.

Tabel 3. ANOVA of moisture content response

Source	**Sum of** Squares	df	**Mean** Square	**F** Value	**p-value** Prob > F	
Model	0	0				
Residual	5.49	12	0.46			
Lack of Fit	5.49	10	0.55	360.19	0.0028	*Significant*
Pure Error	3.05E-03	2	1.52E-03			
Cor Total	5.49	12				
Std. Dev.	0.68	R-Squared		0		
Mean	4.37	Adj R-Squared		0		
C.V. %	15.48	Pred R-Squared		-0.1736		
PRESS	6.44	Adeq Precision				

Based on Table 3, the polynominal model of the moisture content response was the mean. The results of analysis of variance (ANOVA) at the 5% significance level indicated that lack of fit of the model produced was significant. This was indicated by the smaller value of lack of fit than 0.05 (<0.0028) and F-value of 360.19.

The significant value of lack of fit shows that there is no conformity of moisture content response data and the model. A significant lack of fit indicates that variations in the replication of mean values are smaller than variations in design points from predicted values (Susilo, 2011).

The predicted R-squared value was negative, which was -0.1736. Negative predicted R-squared value indicate that the overall mean gives a better prediction for the moisture content response. The model produced for the response of moisture content was only made based on the mean value so that the equation was obtained as follows:

Moisture content (%) =+4.36961...(1)

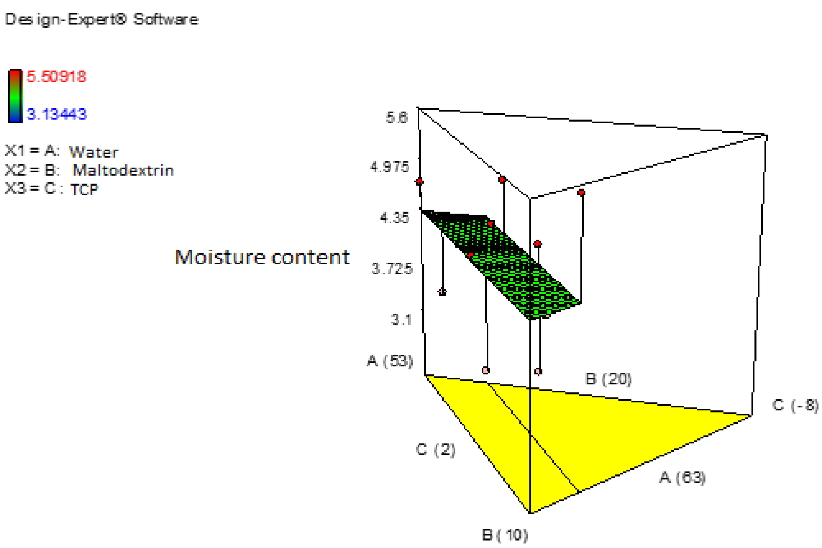

Figure 1. Three dimension graph of moisture content response testing

The countour plot graph in Figure 1 illustrates how the combination of components does not affect each other with the value of the moisture content response. The colors that look the same in all countour chart areas shows that the measured response values are the same in all combinations of the measured components. This is because the chosen polynomial model is the mean, so that the moisture content response value is considered the same in each combination. The response of moisture content also looks flat on each combination of components measured. The response rate of moisture content is considered not significantly different in each combination among components. The concentration of water used in making fruit pulp does not significantly affect the moisture content response. This is caused by the drying process with a vacuum dryer at the same temperature and pressure making the water in the material optimally evaporated to produce non-significant different powder's moisture content.

3.2 The yield of canistel powder

Yield is a ratio of the weight of powder produced and the weight of the canistel fruit flesh used. Calculation of yield is based on the dry weight of the material. The drying causes decline of moisture content in the material, resulting the decreasing of powder yield. The high yield indicates that the amount of powder obtained is increasing. The yield response test results reveals the values in the range of 14.83-29.04% (Table 2). ANOVA statistical analysis in Table 4 demonstrates the polynomial model provided by Design-Expert 7.0 software.

Table 4. ANOVA of yield response

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	239.12	6	39.85	48.06	< 0.0001
Linear Mixture	184.82	2	92.41	111.44	< 0.0001
AB	12.83	1	12.83	15.47	0.0077
AC	0.29	1	0.29	0.35	0.5763
BC	0.75	1	0.75	0.9	0.379
ABC	14.35	1	14.35	17.3	0.0059
Residual	4.98	6	0.83		
Lack of Fit	4.4	4	1.1	3.82	0.2183
Pure Error	0.58	2	0.29		
Cor Total	244.09	12			
Std. Dev.	0.91	R-Squared		0.9796	
Mean	21.74	Adj R-Squared		0.9592	
C.V. %	4.19	Pred R-Squared		0.8501	
PRESS	36.58	Adeq Precision		19.441	

Table 4 shows that the more maltodextrin added, the higher the yield obtained. This is due to the addition of maltodextrin induces in the addition of total solid found in maltodextrin, so that it can increase the total solid of over-ripe canistel powder. The higher the total solid in the dried material, the higher the yield produced (Badarudin 2006).

Based on the results of the analysis carried out by the design expert program, the polynominal model of the yield response is special cubic. Analysis of variance operated in the design expert program with the mixture design d-optimal method on the yield response value of the formulas made reveals that the model made is significant. This means that the formula made has a significant effect on the yield response, so that the response value can be used for the optimization process to obtain a product with optimum characteristics.

F-value of lack of fit was 3.82, with the p value of 0.2183 indicating that the lack of fit is not relative significant to pure error. The insignificant value of lack of fit is a requirement for a good model. The insignificant lack of fit reveals the suitability of the yield response data and the model.

The predicted R-squared and adjusted R-squared value for the yield response are 0.8501 and 0.9592, respectively, which indicate that the predicted data and actual data for yield response are included in the model at 85.01% and 95.92%. Adequate precision for the yield response is 19,441 which illustrates the amount of signal to noise ratio. Adequate precision values greater than 4 (19,441) reveal adequate signal, so that this model can be utilized as a design space guide. Based on the analysis, the model is qualified as a good model, so that it is expected to provide good predictions. Equation (2) of the polynomial for yield response is as follows:

Yield (%) = -0.54357A - 9.03602B - 282.31868C + 0.21309AB + 4.38195AC + 13.04752BC

- 0.19301ABC..(2)

In which, A = concentration of water, B = concentration of maltodextrin, C = concentration of TCP, AB = influence of water and maltodextrin interaction, AC = influence of water and TCP interaction, BC = influence of maltodextrin and TCP interaction, ABC = influence of water, maltodextrin and TCP interaction.

Based on the equation, it can be seen that beside being influenced by three components of material (water, maltodextrin, and TCP), yield is also influenced by the interaction among the three components. The response of the yield will decrease with the increase in the amount of water concentration, maltodextrin and TCP and the interaction of the three ingredients. This is indicated by a negative value coefficient. The randemen response will increase along the increasing of water and maltodextrin interaction, water and TCP and maltodextrin and TCP interaction. This is shown by a positive coefficient.

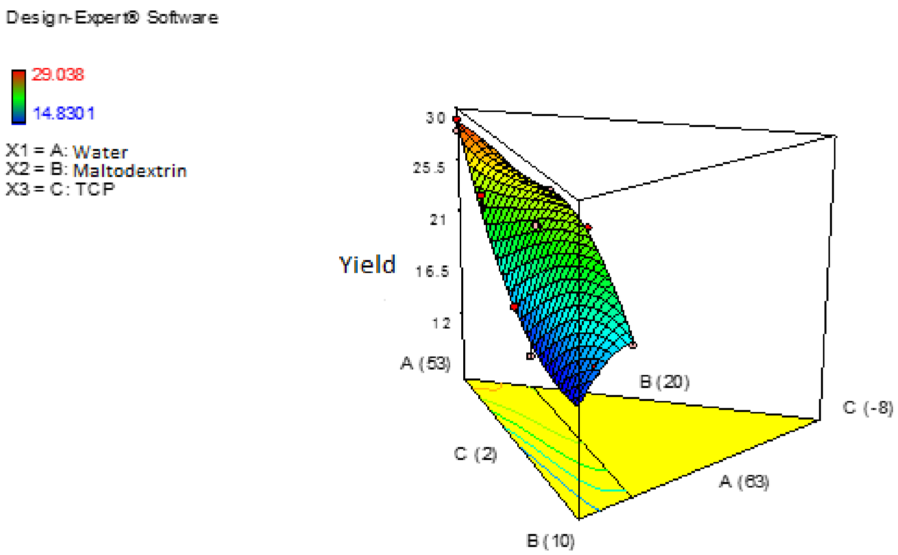

Figure 2. Three dimension graph of yield response testing

The countour plot graph in Figure 2 illustrates how the combination of components affects the response value. Different colors on the contour graph plot show the yield response value. The blue color shows the lowest yield response value, which is 17.0952%. The red color shows the highest yield response, which is 26.5484%. Lines consisting of points on the contour graph plot show a combination of the three components with different amounts that produce a yield response. Differences in surface height show different response values for each combination of formula components. The low area shows a low yield response value while the high area shows a high yield response value.

3.3 Flowability of canistel powder

The addition of anticaking agent into over-ripe canistel powder aimed to maintain the flow property of powder to keep it free-flowing or easily flow. Flowability is the ability of a solid grain or powder to flow or called ease of flow. The physical and chemical properties of a powder are very dependent on the surrounding environmental factors, namely particle size, storage time, temperature, and fat content which can result in clumping (Ganesan et al. 2008).

Flowability response test results range from 22.70-43.89 (Table 2). The lowest flowability test value is 22.70 derived from formula 8. The lowest flowability value is included in the fairly easy flow category. While the highest flowability test value of 43.89 comes from formula 4 and falls into the category of very poor flow. The average value of the flowability response is 34.43 with a standard deviation of 6.21. The lowest flowability value is included in the category that is quite easy to flow because it is indeed the nature of hygroscopic fruit powder. Based on the analysis, the polynomial model of flowability response is special cubic. The results of the variance analysis (ANOVA) are found in Table 5.

Table 5. ANOVA of flowability response

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F
Model	424.49	6	70.75	5.5	0.0285
Linear Mixture	105.5	2	52.75	4.1	0.0754
AB	245.27	1	245.27	19.07	0.0047
AC	2.63	1	2.63	0.2	0.6671
BC	6.39	1	6.39	0.5	0.5072
ABC	64.26	1	64.26	5	0.0668
Residual	77.17	6	12.86		
Lack of Fit	36.61	4	9.15	0.45	0.775
Pure Error	40.56	2	20.28		
Cor Total	501.66	12			
Std. Dev.	3.59	R-Squared		0.8462	
Mean	34.43	Adj R-Squared		0.6923	
C.V. %	10.42	Pred R-Squared		-0.6705	
PRESS	838.01	Adeq Precision		7.149	

Table 5 demonstrates that the recommended model (special cubic) is significant with the p value "prob> F" smaller than 0.05 (0.0285). This means that the formula made has a significant effect on the response of flowability, so that the response value can be used for the optimization process, namely to obtain products with optimum characteristics.

F-value of lack of fit is 0.45 with p value 0.775 indicating that lack of fit is not relative significant to pure error. The insignificant value of lack of fit is a requirement for a good model. This insignificant lack of fit shows the suitability of the data flowability response and the model.

The predicted R-squared and adjusted R-squared values for flowability responses are -0.6705 and 0.6923, respectively. Negative predicted R-squared value indicates that the overall mean gives a better prediction for flowability response. Adequate precision for flowability response is 7.149 which shows the amount of signal to noise ratio. The value of adequate precision greater than 4 (7,149) demonstrates an adequate signal so that this model can be used as a design space guide. Based on the analysis that has been done, the model is qualified as a good model, so that it is expected to provide good prediction. Equation (3) polynomial for flowability response is as follows:

$$\text{Flowability (\%)} = 0.19179A - 1.96233B - 379.61631C + 0.090864AB + 6.59772AC + 23.13948BC - 0.40851ABC \quad (3)$$

Based on the equation, it can be seen that beside being influenced by three components of material (water, maltodextrin, and TCP), flowability is also influenced by interactions among the three ingredients. Flowability response will increase along with the increasing amount of water concentration; interaction of maltodextrin, water and maltodextrin; interaction of water and TCP; and interactions of maltodextrin and TCP. This is indicated by a positive value coefficient. The flowability response will decrease along with the increasing of maltodextrin, TCP; the interaction among water, maltodextrin and TCP. This is indicated by a negative coefficient. Flowability value will be greatly influenced by TCP because the TCP variable has the highest negative coefficient value (good flowability has a low value).

The easier the powder flows, the easier the powder will segregate in the mixture because the particle movement is very high. The worse the nature of the flow of a powder, the more cohesive the powder and not easily segregated (Harnby and Edwards, 1992). Anticaking agent will coat food particles in the form of powder and absorb excess water so that the properties of powder granules can be maintained (Oelmuller and Grinschgl, 1998). In addition, the use of anticaking agent in powder products can reduce tension among particles increasing bulk density of powder products (Iskandar, 2001).

The countour plot graph in Figure 3 illustrates how the combination among components influences the flowability response value. Different colors in the contour graph plot show flowability response value. Blue shows the lowest flowability response value, which is 22.70. The red color shows the highest flowability response, which is 43.89. Lines consisting of points on the contour graph plot show a combination of the three components with different amounts that produce the same flowability response. Differences in surface height show different response values for each combination of formula components. Low area reveals low flowability response value while high area shows high flowability response value.

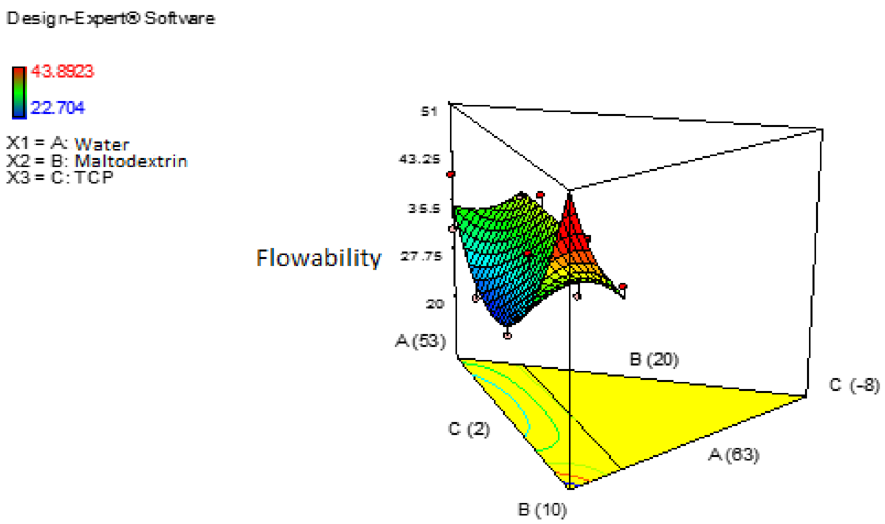

Figure 3. Three dimension graph of *flowability* response testing

3.4 Optimization of the selected formula with Design-Expert 7.0 Software

The selected formula is the optimal solution or formulation predicted by the design expert of mixture design d-optimal method based on the analysis of the response namely yield, moisture content, and flowability. The accuracy of the formulation and the value of each response can be seen in desirability. Desirability is the accuracy degree of optimal solution or formulation outcome. The closer to the value of one, the higher the accuracy value of the formulation. Therefore, it can be concluded that the desirability value that has approached 1.00 has a fairly high accuracy. The description of the elements optimized with each element having different goals and weighting interests is shown in Table 6.

Table 6. Description of optimized process and response parameter elements

Name	**Goal**	**Lower Limit**	**Upper Limit**	**Lower Weight**	**Upper Weight**	**Importance**
Water	is in range	53.00	63	1	1	3
matodextrin	is in range	10.66	20	1	1	3
TCP	is in range	0	2	1	1	3
Moisture content	is in range	3.13	5.50	1	1	5
Yield	maximize	14.83	29.03	1	1	4
flowability	minimize	22.70	43.89	1	1	3

Based on Table 6, the moisture content response is optimized with goal in range and importance (+++++) because for powder products, moisture content is an important parameter. Changes in moisture content will cause changes in microbiological, chemical and physical quality. The powder response is optimized by goal maximize and importance (++++). This is reasonable because the higher the yield, the better the economic calculation. Flowability responses are optimized with goal minimize and importance (+++). The lower the flowability value, the better the flow of powder, then the easier the flow of powder.

Table 7. The optimum formula solution

No	Water	Matodextrin	TCP	Moisture content	Yield	*Flowability*	*Desirability*
1	53.939	19.061	2	4.369	26.122	30.145	0.729
2	53.004	20	1.996	4.369	28.904	35.084	0.683
3	56.780	17.702	0.519	4.369	25.148	34.222	0.595

Source: *output design expert* (2018).

Based on the stages of optimization that have been done, the design expert program provides three optimum formula solutions that can be seen in Table 7. Formula 1 has a desirability value of 0.729, formula 2 is 0.683 and formula 3 is 0.595. By taking notice of the three optimum formula solutions, it is said that formula 1 has the highest desirability value, so it is recommended by the design expert program (selected). This indicates that due to the results of the optimization that has been done, formula 1 obtains the desired optimization target. Based on the recommendation, formula 1 will be proceed to the verification stage because it has the highest desirability value.

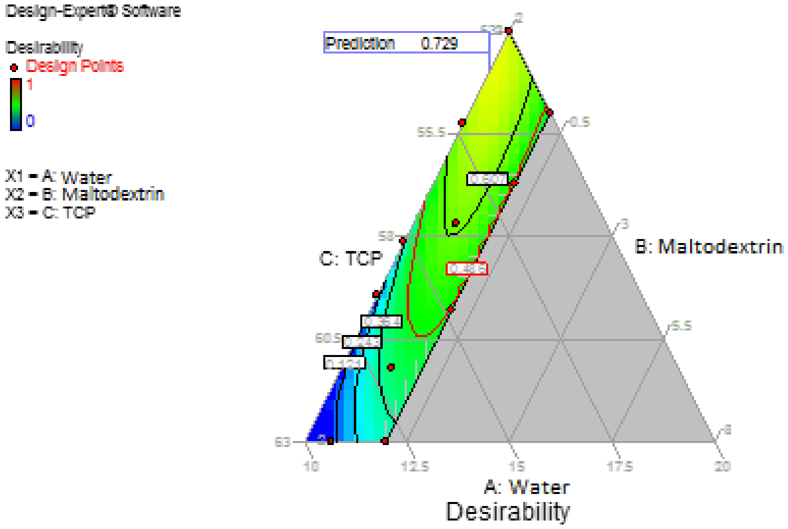

Figure 4. *Countour plot* of *desirability* test

Figure 4 presents the results of the optimum formula solution presented in the form of two-dimensional contour plot using a prediction model for moisture content, yield and flowability response. The value on the contour line is a combination of three components, namely water, maltodextrin and TCP those results in achieving desirability values. The lowest desirability value is shown in blue and the highest is indicated by green.

3.5 Selected product verification

After the formula optimization step was done by using the design expert program, then the verification phase was conducted. This verification phase aimed to prove the predictions of the response value of the optimum formula solution provided by the design expert program. From the verification stage, the actual response value would be obtained which would then be compared with the predicted response generated by the design expert program.

Beside predicting the response value of each given optimum formula solution, the design expert program also provides confident interval (CI) and prediction interval (PI) for each predicted response value at a significance level of 5%. Confident interval is a range that shows the average expected measurement results at a certain level of significance, in this case 5%. Prediction interval is a range that shows the prediction of the actual value of the results of measurements of individual responses at a certain level of significance, in this case 5%. The results of the verification carried out along with predictions from each response can be seen in Table 8.

Table 8. Prediction and results of response value verification for optimum formula solution

Response	DX Prediction	Verification Result	95% CI low	95% CI high	95% PI low	95% PI high
Moisture content	4.3696	4.62	**3.96**	**4.78**	2.84	5.90
Yield	26.1225	28.40	24.93	27.31	**23.60**	**28.65**
Flowability	30.1452	31.58	**25.46**	**34.83**	20.20	40.10

Source: *output design expert* (2018).

In Table 8, you can see the verification data with predictions made by the design expert program. The values of the verification result obtained were almost close to the predicted value. The results of the verification of the three responses still met 95% CI and 95% of predicted PIs. The difference among the results of verification and prediction is due to the variation of the process. This shows that the optimization of the canistel powder formula using RSM of mixture design D-optimal method according to the results of the actual analysis. The obtained equation is considered as still good enough to determine the optimum formula and the obtained response.

4. Conclusions

Over-ripe canistel powder formula significantly influences the yield response and flowability, but it does not significantly affect the moisture content response. From the results of calculations carried out in the design expert program with mixture design d-optimal method, it has been obtained that the optimum over-ripe canistel powder formula has a desirability value in the amount of 0.729. The optimum formula consisted of 53.94% water, 19.06% maltodextrin, 2% TCP and 25% over-ripe canistel fruit pulp. The optimum formula produces a prediction of the response value of moisture content of 4.37%, yield of 26.12% and flowability of 30.14%. Based on the results of laboratory testing verifications carried out on the optimum through over-ripe canistel powder formula, the moisture content response value was in the amount of 4.62%, yield was 28.40% and flowability was 31.68%.

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