Optimization on formulation of foamed overripe canistel powder using response surface methodology

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Abstract - Overripe canistel fruit was generally consumed and not yet widely utilized. The objective of this research was to study and determine the optimum formulation for water, egg albumin, and maltodextrin concentration in making canistel powder using the foam-mat drying method. Optimization was conducted by Response Surface Methodology (RSM) simple mixture of Design-Expert 7.0 software. The formulas used in this study were water of 50-55%, egg albumin of 15-20%, maltodextrin of 5-10% with a total of 75%, and overripe canistel fruit of 25%. The responses of yield, moisture, and beta carotene concentration were analyzed. The results showed that the higher the maltodextrin and the egg albumin concentration led to the higher the powder yield. The use of maltodextrin decreased moisture content and beta carotene concentration of canistel powder, while egg albumin increased them. In addition, the optimum solution was water of 50.00%, egg albumin of 16.88%, maltodextrin of 8.12%, and overripe canistel of 25%, which resulted in a yield of 54.90%, moisture content of 7.07%, and beta carotene of 2.65 mg.kg⁻¹. Validation of the optimum solution was a yield of 61.20%, moisture content of 7.09%, and beta carotene of 0.63 mg.kg⁻¹, which were within the 95% prediction interval low and high.

Index terms: Pouteria campechiana, overripe canistel fruit, foam-mat drying, surface analysis.

Otimização da formulação do pó canistel de sobreposição de espuma usando a metodologia de superfície de resposta

Resumo - Frutos maduros de canistel eram geralmente consumidos e ainda não amplamente utilizados. O objetivo desta pesquisa foi estudar e determinar a formulação ideal para a concentração de água, albumina de ovo e maltodextrina na produção de canistel em pó usando o método de secagem por espuma. A otimização foi conduzida pela simples mistura de Response Surface Methodology (RSM) do software Design-Expert 7.0. As fórmulas utilizadas neste estudo foram água de 50 a 55%, albumina de ovo de 15 a 20%, maltodextrina de 5 a 10%, com um total de 75%, e frutos de canistel maduros de 25%. As respostas de rendimento, umidade e concentração de betacaroteno foram analisadas. Os resultados mostraram que, quanto maior a concentração de maltodextrina e albumina de ovo, maior o rendimento de pó. O uso de maltodextrina diminuiu o teor de umidade e a concentração de betacaroteno do pó de canistel, enquanto a albumina de ovo aumentou. Além disso, a solução ideal foi água de 50,00%, albumina de ovo de 16,88%, maltodextrina de 8,12% e canistel maduro de 25%, o que resultou no rendimento de 54,90%, teor de umidade de 7,07% e betacaroteno de 2,65 mg.kg⁻¹. A validação da solução ótima foi o rendimento de 61,20%, teor de umidade de 7,09% e betacaroteno de 0,63 mg.kg⁻¹, que estavam dentro do intervalo de previsão de 95% baixo e alto.

Termos para indexação: Pouteria campechiana, frutos maduros de canistel, secagem por espuma, análise de superfície.
Introduction

Canistel fruit or Pouteria campechiana is a tropical plant from Southern Mexico, Belize, Guatemala, and El Salvador. This plant then spread to Central America, Caribbean, Southeast Asia, and Africa (CRANE; BALERDI, 2016). According to Mehraj et al. (2015), canistel fruit contains alkaloid, tannin, flavonoid, steroid, and terpenoid compounds. Full ripe canistel fruit is high in carbohydrate, carotene, and sugar contents. This fresh fruit is generally used as a mixture of ice cream, jam, milkshakes, cupcakes, and so on. Besides, canistel puree can be added into cake dough or ice cream as a flavor and used as a filler of pie (AWANG-KANAK; BAKAR, 2018). Overripe canistel fruit has a very soft texture, but the nutritional contents are better than the full ripe canistel fruit, such as higher protein and sugar. Making a powder of overripe canistel fruit using the drying process can provide broader utilization and improve the added value of this fruit.

Drying is the most commonly used method of increasing the shelf life of fruit and opening up other uses as food formulations. Paradagos (2014) processed canistel fruit into flour using conventional drying of sunlight. Pertiwi et al. (2020) made full ripe canistel fruit flour using a tray drying method. Besides, there is foam mat drying method that is often used in the manufacture of fruit powder including mango (WILSON et al., 2012; RAJKUMAR et al., 2007, KADAM et al., 2010), papaya (KANDASAMY; VARADHARAJU, 2014), lime (CHAND; PANDEY, 2012; DEGHANNYA et al., 2019), banana (SANKAT; CASTAIGNE, 2004; PRAKOTMAK et al., 2010; THUWAPANICHAYANAN et al., 2008), apple (RAHARITSIFA et al., 2006), jambolan (de CARVALHO et al., 2017), beetroot (NG; SULAIMAN, 2018), and starfruit (KARIM; WAI, 1999). The foam-mat drying method is a drying technique for heat-sensitive materials through foaming techniques by adding foaming agents (RAJKUMAR et al., 2007). Proteins, gums, and various emulsifiers (Glycerol monostearate, carboxymethylcellulose, and trichlorophenate) are commonly used as foaming agents. Also, stabilizers and fillers are other important factors in the foam-mat drying, for example, maltodextrin and methylcellulose. From these data, the conversion of high-sugar canistel fruit into its powder form using a foam mat drying method has not been well documented in the literature.

The objective of this research is to utilize the overripe canistel fruit as a powder and to optimize the concentration of water, egg albumin, and maltodextrin in the formulation of foamed over ripe canistel fruit powder using response surface methodology.

Material and Methods

Canistel fruits were obtained from Padalarang, West Java, Indonesia. The fruits were in the overripe condition (12-15 days after harvesting). Eggs and maltodextrin were obtained, respectively, from the local market and chemical store of PT. Setia Guna in Bogor, West Java, Indonesia. The experiment, which was based on Myers et al. (2016), consisted of four stages, namely determining the formula, making the product according to the formula, testing the product response, analyzing the data from the response results, and optimizing and verifying the optimum formula solution.

Determining the formula

The factors in this study were water (A, 50%-55%) in the manufacture of fruit puree, egg albumin (B, 15%-20%) as foaming material, and maltodextrin (C, 5%-10%) as a stabilizer and filler. The total of A + B + C = 75% of the formulation, while the remaining 25% was the overripe canistel fruit. Formulations were analyzed using Design-Expert 7.0 software, a simplex lattice design mixture. At this stage, a formula that was suitable for the desired optimization objectives was obtained from fourteen combinations of formulas that were provided by the Design-Expert 7.0 software (Table 1).

Table 1. Experimental combination of optimization on over ripe canistel powder using Response Surface Methodology (RSM).

| Formula | (A) Water (%) | (B) Egg albumin (%) | (C) Maltodextrin (%) |
|---------|---------------|---------------------|----------------------|
| 1       | 51.67         | 16.67               | 6.66                 |
| 2       | 52.50         | 17.50               | 5.00                 |
| 3       | 52.50         | 15.00               | 7.50                 |
| 4       | 53.33         | 15.83               | 5.83                 |
| 5       | 50.00         | 15.00               | 10.00                |
| 6       | 50.00         | 15.00               | 10.00                |
| 7       | 50.00         | 20.00               | 5.00                 |
| 8       | 55.00         | 15.00               | 5.00                 |
| 9       | 50.00         | 20.00               | 5.00                 |
| 10      | 50.00         | 17.50               | 7.50                 |
| 11      | 52.50         | 17.50               | 5.00                 |
| 12      | 50.83         | 18.33               | 5.83                 |
| 13      | 55.00         | 15.00               | 5.00                 |
| 14      | 50.83         | 15.83               | 8.33                 |
Making the product according to the formula
The obtained formulas were then produced according to the stages of making overripe canistel powder using the foam-mat drying method in an electric food dehydrator type MKS-DR10 (manufacturer of PT Toko Mesin Maksindo, Indonesia). The 250 grams of puree foam produced from the next formulation was poured into each pan of 34x34x2 cm. The puree foam thickness was ±2 mm in a pan. Then dried at a temperature of 45 °C for 7 hours, and every 2 hours 20 minutes, the placement of the tray was moved for stable drying conditions. After that, the dried puree was milled using disc mill type FFC-15 and sieved using 100 mesh siever.

Testing the product response and analyzing the data from the response results
The responses in this study were yield (AOAC, 2005), moisture content (AOAC, 2005), and beta carotene (NIELSEN, 1995). The response results were analyzed using Design-Expert 7.0 software with polynomial models. There were three stages of obtaining a suitable polynomial model or equation, including the sequential sum of squares model [Type I], lack of fit tests, and summary statistics model. After the appropriate polynomial model was obtained, analysis of variance (ANOVA) was used to determine the significance value of the model and to find out that each response was significantly different or not at the 95% of the confidence interval. Then the data was presented in the form of the response surface.

Optimizing and verifying the optimum formula solution
The optimization process was conducted to obtain the optimum formula. In determining the optimization target, the importance was indicated by the values of 1 (+) to 5 (++++) depending on the importance of the response. After optimization, Design-Expert 7.0 software provided some optimum formulation solutions and predictions of the responses. The selection of the optimum formula was based on the desirability value, which was close to 1. After that, the selected optimum formula solution was produced, and the response test was performed in the laboratory. The optimum formula response was verified by comparing the results of the actual data against the predicted value. The validation phase was to ensure the correctness of the obtained combinations and equations as well as the prediction of the responses.

Results and Discussion
The results of response analysis based on the mixture simplex lattice design of Response Surface Methodology (RSM) software can be seen in Table 2.

| Formula | Water (%) | Egg albumin (%) | Maltodextrin (%) | Yield (%) | Moisture content (%) | Beta caroten concentration (mg.kg⁻¹) |
|---------|-----------|-----------------|------------------|-----------|----------------------|-------------------------------------|
| 1       | 51.67     | 16.67           | 6.66             | 53.81     | 10.47                | 2.74                                |
| 2       | 52.50     | 17.50           | 5.00             | 46.21     | 11.17                | 0.00                                |
| 3       | 52.50     | 15.00           | 7.50             | 43.60     | 11.62                | 2.90                                |
| 4       | 53.33     | 15.83           | 5.83             | 40.32     | 10.72                | 2.06                                |
| 5       | 50.00     | 15.00           | 10.00            | 59.07     | 9.54                 | 1.87                                |
| 6       | 50.00     | 15.00           | 10.00            | 58.69     | 8.79                 | 2.24                                |
| 7       | 50.00     | 20.00           | 5.00             | 50.05     | 8.21                 | 1.52                                |
| 8       | 55.00     | 15.00           | 5.00             | 35.33     | 10.26                | 1.42                                |
| 9       | 50.00     | 20.00           | 5.00             | 46.11     | 12.37                | 1.63                                |
| 10      | 50.00     | 17.50           | 7.50             | 54.27     | 6.91                 | 2.56                                |
| 11      | 52.50     | 17.50           | 5.00             | 42.69     | 8.84                 | 1.53                                |
| 12      | 50.83     | 18.33           | 5.83             | 48.35     | 9.02                 | 3.02                                |
| 13      | 55.00     | 15.00           | 5.00             | 30.80     | 11.03                | 0.00                                |
| 14      | 50.83     | 15.83           | 8.33             | 48.11     | 8.64                 | 1.65                                |

Table 2. The responses of over ripe canistel powder formulas.
Canistel Powder Yield

The powder yield is a ratio of the amount of fruit powder against the initial fruit pulp in the formulation. The higher the yield leads to the more amount of powder produced. The canistel powder yield is in the range of 30.80 - 59.07% (Table 2). Statistical analysis can be seen in Table 3 shows the polynomial model provided by Design-Expert 7.0 software for the yield response is linear with equation (1).

\[ \text{Yield} (\%) = -0.49004A + 2.65983B + 4.24845C \quad (1) \]

where A = water, B = egg albumin, and C = maltodextrin.

Table 3 shows that the model is significant, with an F value of 35.78 with a probability of 0.01%. Also, it is known that all treatment factors have a significant effect on yield response. The model for the yield response has an R² of 0.8668, which indicates that 86.68% variability in the response can be explained by the model, with an insignificant lack of fit values. Besides, the adequacy precision value for the yield response is 15.779, which is greater than 4, indicates an adequate signal so that the model can be used as a design space guide.

| Source           | Sum of squares | df | Mean square   | F value | p-value  |
|------------------|----------------|----|---------------|---------|----------|
| Model            | 752.87         | 2  | 376.43        | 35.78   | < 0.0001 |
| Linear Mixture   | 752.87         | 2  | 376.43        | 35.78   | < 0.0001 |
| Residual         | 115.73         | 11 | 10.52         |         |          |
| Lack of Fit      | 91.40          | 7  | 13.06         | 2.15    | 0.2401   |
| Pure Error       | 24.33          | 4  | 6.08          |         |          |
| Cor Total        | 868.60         | 13 |               |         |          |

| Std. Dev.         | 3.24           | R-Squared   | 0.8668 |
| Mean             | 46.96          | Adj R-Squared | 0.8425 |
| C.V. %           | 6.91           | Pred R-Squared | 0.8007 |
| PRESS            | 173.13         | Adeq Precision | 15.7792 |
Canistel Powder Moisture Content

Table 4 shows the moisture content response of overripe canistel powder, which is in the range of 6.91 - 12.37% (wb). Based on the statistical analysis, the polynomial model given for the response of moisture content is in equation (2).

Table 4. Analysis of variance for moisture content.

| Source          | Sum of squares | df  | Mean square | F value | p-value |
|-----------------|----------------|-----|-------------|---------|---------|
| Model           | 16.44          | 5   | 3.29        | 2.00    | 0.1836  |
| Linear Mixture  | 6.48           | 2   | 3.24        | 1.97    | 0.2020  |
| AB              | 0.12           | 1   | 0.12        | 0.07    | 0.7976  |
| AC              | 3.83           | 1   | 3.83        | 2.33    | 0.1658  |
| BC              | 7.01           | 1   | 7.01        | 4.25    | 0.0731  |
| Residual        | 13.18          | 8   | 1.65        |         |         |
| Lack of Fit     | 1.24           | 4   | 0.31        | 0.10    | 0.9749  |
| Pure Error      | 11.94          | 4   | 2.98        |         |         |
| Cor Total       | 29.63          | 13  |             |         |         |

| Std. Dev.       | 1.2836         | R-Squared | 0.5551 |
| Mean            | 9.8269         | Adj R-Squared | 0.2770 |
| C.V. %          | 13.0623        | Pred R-Squared | -0.5222 |
| PRESS           | 45.0967        | Adeq Precision | 5.6062 |

Moisture Content (%) = -0.530A + 4.588B - 9.131C - 0.045AB + 0.308AC - 0.417BC  (2)

where A = water, B = egg albumin, C = maltodextrin, AB = Interaction of water and egg albumin, AC = Interaction of water and maltodextrin, BC = Interaction of egg albumin and maltodextrin. The analysis of variance shows that the lack of fit is not significant, which indicates the suitability of moisture content data with the model with R² of 55.5%. The value of adequacy precision for moisture content response is 5.606 so that the model can be used as a design space guide, that is expected to provide good predictions (MUTHUKUMAR et al., 2003).

The response of moisture content will increase along with an increase in egg albumin and the interaction of water and maltodextrin. Meanwhile, the moisture content will decrease with an increase in water,
maltodextrin, the interaction of water and egg albumin, and interaction of egg albumin and maltodextrin. These are in line with Ayu et al. (2016), which stated that the higher the maltodextrin is added, the lower the moisture content of instant green sambal. According to Siska and Wahono (2014), a high concentration of maltodextrin can absorb more water in synonymic drinks due to its hygroscopic properties. Water, which was absorbed by maltodextrin, was more volatile than the water in the tissue material so that the water evaporation process is easier and faster (ARIFIN, 2006). Also, Fabra et al. (2011) reported the addition of maltodextrin in the manufacture of noni pulp powder (Morinda citrifolia L) made lower powder’s moisture content. Avila et al. (2015) also explained the decrease in moisture content in sugarcane juice powder as a result of higher levels of maltodextrin at low drying temperatures. Ekpong et al. (2016) reported a pattern of decreased moisture content in the tamarind powder along with an increase in the maltodextrin levels. This is also in line with Sukri et al. (2018), which reported that the moisture content of pineapple powder was decreased with the addition of maltodextrin. The moisture content of overripe canistel powder can also be decreased with the interaction of water and egg albumin, and the interaction of egg albumin and maltodextrin. These interactions can increase the volume of the produced foam. Maltodextrin has a role in stabilizing the foam and increasing the foam volume so that the evaporation of water in the material will be easier and faster. Zubaedah et al. (2003) stated that the higher the foam concentration led to the larger the surface area and the greater the porous structures on the material, which impact to easier and faster evaporation process of water from the material. Response surface analysis for moisture content can be seen in Figure 2.

![Response surface analysis for moisture content response](image)

**Figure 2.** Response surface analysis for moisture content response

*Canistel Powder Beta Carotene*

The lack of fit of the adjusted model, which can be seen in Table 5, is not significant with $R^2$ of 57.84% and the adequacy precision more than 4, which indicates that there is sufficient compatibility of the beta carotene response data between the model and the values.
**Table 5.** Analysis of variance for beta carotene response

| Source            | Sum of squares | df | Mean square | F value | p-value |
|-------------------|----------------|----|-------------|---------|---------|
| Model             | 6.51           | 5  | 1.30        | 2.19    | 0.1545  |
| Linear Mixture    | 2.91           | 2  | 1.46        | 2.46    | 0.1474  |
| AB                | 0.04           | 1  | 0.04        | 0.07    | 0.7978  |
| AC                | 2.36           | 1  | 2.36        | 3.98    | 0.0811  |
| BC                | 0.85           | 1  | 0.85        | 1.44    | 0.2651  |
| Residual          | 4.74           | 8  | 0.59        |         |         |
| Lack of Fit       | 2.49           | 4  | 0.62        | 1.11    | 0.4624  |
| Pure Error        | 2.25           | 4  | 0.56        |         |         |
| Cor Total         | 11.25          | 13 |             |         |         |

|                  | Std. Dev. | R-Squared | Adj R-Squared | Pred R-Squared | Adeq Precision |
|------------------|-----------|-----------|---------------|----------------|----------------|
|                  | 0.7701    | 0.5784    | 0.3148        | -0.0920        | 4.1135         |

The equation for beta carotene response can be seen in equation (3).

\[
\text{Beta carotene} = -0.168A + 1.448B -13.412C - 0.027AB + 0.242AC + 0.145BC \tag{3}
\]

where \(A\) = water, \(B\) = egg albumin, \(C\) = maltodextrin, \(AB\) = Interaction of water and egg albumin, \(AC\) = Interaction of water and maltodextrin, \(BC\) = Interaction of egg albumin and maltodextrin. Equation (3) shows a negative relationship between maltodextrin and beta carotene of overripe canistel powder, where the higher the maltodextrin addition leads to the lower beta carotene in the powder. According to Quek et al. (2007), the increase in maltodextrin affected pigment content due to the dilution of pigment. This is in line with Oberoi and Sogi (2015), which explained that an increase in the maltodextrin levels would decrease lycopene and carotenoid levels in watermelon powder. Surface analysis of beta carotene levels can be seen in Figure 3.

![Design-Expert® Software](image)

**Figure 3.** Response surface analysis for beta carotene response
Selected optimum formula

The description of the optimized elements with each element has different goal and importance is shown in Table 6.

Table 6. Description of optimized process and response variables.

| Element          | Goal               | Lower Limit | Upper Limit | Lower Weight | Upper Weight | Importance |
|------------------|--------------------|-------------|-------------|--------------|--------------|------------|
| Water            | is in range        | 50          | 55          | 1            | 1            | 3          |
| Egg albumin      | is in range        | 15          | 20          | 1            | 1            | 3          |
| Maltodextrin     | is in range        | 5           | 10          | 1            | 1            | 3          |
| Yield            | maximize           | 30.80       | 59.07       | 1            | 1            | 4          |
| Moisture content | minimize           | 6.91        | 12.37       | 1            | 1            | 5          |
| Beta carotene    | is in range        | 0.00        | 3.02        | 1            | 1            | 3          |

Table 6 shows the treatments and responses in which each element has its own goal. The desired goals of the yield, moisture content and beta carotene are the highest yield value with the importance of 4 (+++), the lowest moisture content (minimize) with the importance of 5 (+++++), and the beta carotene (in range) with the importance of 3 (+++). These are due to the higher the yield will give the better economic value of the powder, the moisture content can affect the shelf life and the quality of the canistel powder, and the beta carotene value is high enough. After optimization, Design-Expert 7.0 software provides the optimum solution that has a desirability value of 0.917 with water of 50.00%, egg albumin of 16.88%, and maltodextrin of 8.12%. This solution gives prediction in yield of 54.90%, moisture content of 7.07%, and beta carotene of 2.65 mg.kg$^{-1}$. The response surface for the optimized or selected solution can be seen in Figure 4.

Figure 4. Response surface analysis for optimized solution

Validation of the selected optimum solution

Table 7 shows that the validation of the selected formulation gives a yield of 61.20%, moisture content of 7.09%, and beta carotene of 0.63 mg.kg$^{-1}$, where these are within the confirmation 95% prediction interval (95% PI low and 95% PI high). This low beta carotene is related to fruit’s initial beta carotene of 7.1 mg/kg with total carotenoid compounds of 226 mg/kg (Costa et al., 2010). In addition, Ndawula et al. (2004) reported that a dehydration process such as open sun drying could reduce the beta carotene in mango fruit until 94.2%. Demiray and Tulek (2017) also reported an increase in the rate of degradation of beta carotene content in carrot slices by increasing the drying temperature. The validation of moisture content has a good result with value as same as the predicted value and within the confirmation 95%
confidence interval (95% CI low and 95% CI high). This narrower range of CIs shows better optimization values (HEIBERGER; HOLLAND, 2004). Eventually, the data show that the obtained model and equations are good enough to determine the optimum formula and the responses.

**Table 7.** Prediction and validation of response in optimum solution.

| Response          | Prediction | Validation | 95% CI low | 95% CI high | 95% PI low | 95% PI high |
|-------------------|------------|------------|------------|-------------|------------|-------------|
| Yield             | 54.90      | 61.20      | 52.08      | 57.72       | 47.22      | 62.58       |
| Moisture Content  | 7.07       | 7.09       | 4.69       | 9.44        | 3.27       | 10.86       |
| Beta Carotene     | 2.65       | 0.63       | 1.22       | 4.08        | 0.37       | 4.93        |

CI = Confidence Interval, PI = Prediction Interval

**Conclusion**

The use of maltodextrin in the manufacture of canistel fruit powder increase the yield and decrease moisture content and beta carotene concentration with interactions between egg albumin and maltodextrin. The optimum formula for making canistel fruit powder was 50.00% water, 16.88% egg albumin, and 8.12% maltodextrin with yield, moisture content, and beta carotene concentration of 54.90%, 7.07%, and 2.65 mg.kg\(^{-1}\), respectively. Actual data from validation results still meet the predicted 95% prediction interval, and moisture content is within in 95% confidence interval.

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