Optimization of process temperature and time of vacuum drying for production of cashew apple (*Anacardium occidentale* L.) powder using response surface methodology

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Abstract. Cashew apple is a waste from cashew nut processing industry that is high in antioxidants. The cashew apple powder was dried using a vacuum dryer that aimed to create a functional product containing high antioxidant compounds. The objective of this study was to optimize vacuum drying conditions for the production of cashew apple powder using Response Surface Methodology (RSM). The effect of process temperature (55-65 °C) and time (9-15 h) on some characteristics (vitamin C content, antioxidant activity, water content, and color changes) of cashew apple powder were analyzed. A Central Composite Design was used to develop models for the responses. The optimum condition of the vacuum drying process was obtained at a drying temperature of 60.1 °C and drying time of 11.4 hours. The optimum combination resulted in cashew apple powder with vitamin C content of 118.5 mg/100g, antioxidant activity (IC50) of 70.6 ppm, water content of 11.4%, and total color difference (ΔE) of 8.8. A validation experiment was conducted at the obtained optimum condition and the results showed that no significant difference between the actual and predicted values based on paired t-test (p > 0.05). In conclusion, cashew apple powder is a good source of vitamin C and antioxidant compounds.

1. Introduction
Cashew (*Anacardium occidentale* L.) is primarily cultivated for its nut and produces a high amount of cashew apple as a byproduct. The utilization of cashew apple is very limited due to its highly perishable and astringent characteristics [1]. In contrast, the chemical components in cashew apple such as polyphenols, vitamin C, dietary fiber, organic acids, and minerals made it potential to be utilized as a functional food that will increase its economic value and reduce the waste from the environment [2]. In addition, the production of cashew nut in Indonesia has been increasing from 115,149 tons in 2010 to 137,580 tons in 2015 [3]. Cashew apple represents 90% total weight of cashew fruit, meaning that for every ton of cashew nut being produced there will be 10-15 tons of cashew apple being wasted [1]. Thus, there will be more than one million cashew apple wasted every year in Indonesia that can be utilized further to produce cashew apple powder.

A drying process is needed to evaporate the water content in the cashew apple. However, several research showed that conventional drying which uses high temperature and long drying time produces good quality of dried fruit product, but it will degrade and decrease the antioxidant compounds in dried
products [4,5,6]. Therefore, the drying process in this study was conducted using a vacuum dryer to preserve the valuable compounds in the cashew apple. The objective of this study was to optimize the vacuum drying process with temperature and drying time as the experiment factors for the production of cashew apple powder. Response Surface Methodology was used to determine the optimum condition of vacuum drying on the responses in vitamin C content, antioxidant activity (IC_{50}), water content, and total color difference (ΔE) for producing cashew apple powder. The optimum temperature and time of the vacuum drying process obtained from this study can be used for producing cashew apple powder that contains high vitamin C content and antioxidant activity.

2. Materials and Methods

2.1. Materials and reagents
Cashew apples were obtained from cashew plantation in Kediri, East Java, Indonesia with bright red color and 6-10 cm in length. The cashew apple was stored in a freezer (-16°C) before used. The drying process was carried out using a vacuum dryer. Blender type Kirin/KBB-250 PLI was used for processing cashew apple chips into powder. The reagents used for analyzing Vitamin C (ascorbic acid), antioxidant activity, crude protein content, and lipid content were DPPH 1 mM in methanol, methanol (p.a), aquades, ascorbic acid (p.a), concentrated H_2SO_4 (95%), NaOH 30%, H_3BO_3 3%, HCl 0.1 N, Kjeldahl tablet, petroleum ether, methyl red and phenolphthalein indicator.

2.2. Experimental design
The experiment was conducted using Response Surface Methodology (RSM) with Central Composite Design (CCD) under Design Expert 12 software. The factors or independent variables that were used consist of drying temperature with an upper limit of 65°C and lower limit of 55°C and drying time with an upper limit of 15 hours and a lower limit of 9 hours. Vitamin C, antioxidant activity (IC_{50}), water content and total color difference (ΔE) were analyzed as responses or dependent variables. The independent variables and their level in this research showed in Table 1.

| Independent Variables | Levels |
|-----------------------|--------|
| Temperature (X_1, °C) | -1.414 -1 0 +1 +1.414 |
| Time (X_2, hours)     | 52.93 55.00 60.00 65.00 67.07 7.76 9.00 12.00 15.00 16.24 |

2.3. Processing of cashew apple powder
Frozen cashew apples were thawed until the ice melts completely. Then, the apples were washed using tap water to remove dirt from the surface. After that, it was sliced using a stainless steel knife with 1.0-1.5 mm thickness. Sliced cashew apples weighed 400 g using an analytical balance (Denver Instrument M-310). The samples were dried using a vacuum dryer with the combination of drying temperature (52.93, 55, 60, 65, 67.07°C) and drying time (7.76, 9, 12, 15, 16.24 hours) with vacuum pressure at -56±2 cmHg suggested from Design Expert 12 software. Dried cashew apple chips grounded using blender at low velocity for 5 minutes. Afterward, cashew apple powder was sieved using a 60 mesh sieve.

2.4. Determination of ascorbic acid content
The ascorbic content was analyzed using the spectrometric method based on Kurniawati and Riandini method [7]. Ascorbic acid (2-10 mg/L) was diluted in aquades to prepare a stock solution for creating the calibration curve with 265.5 nm in wavelength. 2 g of samples then extracted in 20 ml of aquades. The solution was then diluted until the absorbance was under 1.0. The total ascorbic content was calculated in equation (1):
Vitamin C content (mg/100g) = \frac{c \times v \times DF}{M \times 100} \quad (1)

Where c is the concentration of the sample in mg/L, v is the volume of the aquades used in L, DF is the dilution factor, and M is the mass of the sample in g.

2.5. Determination of antioxidant activity (IC₅₀)
The antioxidant activity was determined based on decolorization stable free radical of 2,2-diphenyl-1-picrylhydrazyl (DPPH) in methanol solution following the method done by Nuraeni and Sembiring [8]. 0.02 g of sample were extracted using 20 ml methanol using shaker waterbath at 60°C for 30 min. The extract was then diluted with 1 ml of DPPH 1 mM in varying concentrations (20, 40, 60, 80, 100 ppm) in a 5 ml volumetric flask. The control used 1 ml of DPPH solution in 5 ml of methanol. The absorbance was measured using 517 nm wavelength and incubated for 30 min in a dark room before analysis. The percentage inhibition of DPPH was determined using the formula in equation (2). Afterward, the % inhibition (y) was plotted against sample concentration (x) to obtain the regression formula (y = ax + b). The IC₅₀ was calculated by substituting (y) with 50, then the concentration (x) is the IC₅₀ value.

\% inhibition = \frac{A_o - A_s}{A_s} \times 100\% \quad (2)

Where A₀ is the absorbance of the control and Aₚ is the absorbance of the sample.

2.6. Proximate analysis
The analysis of water, crude protein, lipid, ash, and carbohydrate content were conducted by using the method from AOAC [9].

2.7. Determination of total color difference (ΔE)
The samples and the fresh cashew apple were analyzed using Color Reader (Konica Minolta, Japan) to obtain the L*, a*, b* profile. The total color difference was measured using the formula in the equation (3):

\[ \Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (3) \]

Where ΔL is the differences in L* value between samples and fresh fruit, Δa is the differences of a* value in dried and fresh sample, and Δb is the differences of b* value between the sample and fresh fruit.

3. Results and Discussion

3.1. Optimization of process temperature and time of vacuum drying
The central composite design of response surface methodology was used to analyze the effect of independent variables on the dependent variables during the vacuum drying process for the production of cashew apple powder. The 13 runs of the experiment were suggested from Design Expert 12 software to observe vitamin C content, antioxidant activity (IC₅₀), water content, and total color difference (ΔE) to obtain the optimum condition (Table 2). The analysis of the responses was conducted in triplicates to rule out experimental error or bias.

3.1.1. Effect of temperature and time on vitamin C response
Vitamin C content of cashew apple powder from the vacuum drying process was obtained between 98.29-121.41 mg/100g. Analysis of Variance (ANOVA) on vitamin C suggested that the response
should be analyzed using the quadratic model ($R^2 = 0.9808$) with p-value <0.0001. The results of ANOVA (Table 3) show that variables A, B, $A^2$, and $B^2$ have a significant effect on Vitamin C ($\alpha=5\%)$ with p-value 0.001, 0.0002, <0.0001 and <0.0001 respectively. The variables AB or the interactions of two factors did not have any significant effect on Vitamin C with p-value of 0.7842. The polynomial equations of Vitamin C response with the quadratic model from the ANOVA are:

$$Vitamin\ C = +120.42-2.83A-3.66B + 0.21AB-5.61A^2-8.14B^2$$  \hspace{1cm} (4)

The correlation between the two factors on the response can also be seen in Figure 1. According to the equation and the surface curve, vitamin C content of cashew apple powder will increase coincide with increasing of process temperature and time until its optimum point but will decrease at higher process temperature and longer drying time. Vitamin C is highly sensitive to high-temperature environments. Exposing Vitamin C or ascorbic acid to high heat for a long time during the drying process can trigger an oxidation reaction that will alter its structure producing 2,3-diketogulonic acid that has no Vitamin C activity [10]. However, there was one case when the ascorbic acid content of dried or powdered product was reported to be higher than the unprocessed sample [11]. Ascorbic acid is considered as a solid in food that will not evaporate during the drying process, which makes the concentration of ascorbic acid in dried products, tend to be higher than the fresh sample.

**Table 2.** Coded and actual combination of central composite design.

| Treatment | X1   | X2   | Temperature ($^\circ$C) | Time (Hours) | Vitamin C (mg/100g) | IC$^{50}$ (ppm) | Water content (%) | ΔE |
|-----------|------|------|-------------------------|--------------|---------------------|----------------|------------------|----|
| 1         | -1.00| -1.00| 55.00                   | 9.00         | 114.19              | 77.16          | 15.35            | 4.23 |
| 2         | 1.00 | -1.00| 65.00                   | 9.00         | 106.54              | 86.68          | 10.24            | 11.01|
| 3         | -1.00| 1.00 | 55.00                   | 15.00        | 105.10              | 89.15          | 11.16            | 13.12|
| 4         | 1.00 | 1.00 | 65.00                   | 15.00        | 98.29               | 94.23          | 9.91             | 18.36|
| 5         | -1.41| 0.00 | 52.93                   | 12.00        | 112.74              | 90.82          | 13.52            | 5.23 |
| 6         | 1.41 | 0.00 | 67.07                   | 12.00        | 106.96              | 98.19          | 9.19             | 14.88|
| 7         | 0.00 | -1.41| 60.00                   | 7.76         | 109.02              | 72.68          | 13.76            | 6.04 |
| 8         | 0.00 | 1.41 | 60.00                   | 16.24        | 100.56              | 78.41          | 9.42             | 22.52|
| 9         | 0.00 | 0.00 | 60.00                   | 12.00        | 119.14              | 67.81          | 11.82            | 8.13 |
| 10        | 0.00 | 0.00 | 60.00                   | 12.00        | 120.17              | 62.94          | 11.34            | 9.73 |
| 11        | 0.00 | 0.00 | 60.00                   | 12.00        | 121.21              | 73.12          | 11.76            | 10.13|
| 12        | 0.00 | 0.00 | 60.00                   | 12.00        | 120.17              | 68.86          | 11.52            | 9.62 |
| 13        | 0.00 | 0.00 | 60.00                   | 12.00        | 121.41              | 71.02          | 11.79            | 8.64 |

**3.1.2. Effect of temperature and time on antioxidant activity (IC$^{50}$) response**

The antioxidant activity of cashew apple powder from the process was in the range of 62.94-98.19 ppm. According to Analysis of Variance (ANOVA) of antioxidant activity response, the model that fits for the response was the quadratic model ($R^2 = 0.9452$) with p-value 0.0003. Based on the ANOVA (Table 3), it shows that variables A, B, $A^2$, and $B^2$ give a significant effect on antioxidant activity ($\alpha=5\%)$ with each p-value of 0.0377, 0.0256, <0.0001 and 0.022. There no significant effect of the interactions of two factors (AB) on the response (p-value = 0.5415). The equations from ANOVA of antioxidant activity with the quadratic model is shown in equation (5):

$$IC^{50} = +68.75 - 3.13A - 3.46B - 1.11AB + 13.32A^2 + 3.84B^2$$  \hspace{1cm} (5)
The effect of the two factors on the response is provided in Figure 1. Based on the equation and the surface curve, antioxidant activity (IC$_{50}$) of cashew apple powder will decrease with the increase of vacuum drying temperature and time until its optimum point. However, the extended increase in the two factors above its optimum point will increase the IC$_{50}$ value of cashew apple powder.

The drying process can increase the antioxidant activity of the dried product. This is due to the damage in covalent bonds between phenolic acids and the cell wall of fruit [6]. The free phenolic acids are viable antioxidant compounds that neutralize free radical substances. Nonetheless, at a certain temperature and time of the drying process, the antioxidant activity started to decrease. This occurred as a result of enzyme activity that hydrolyzes the antioxidant compounds in food and the evaporation of volatile antioxidant during the drying process [12].

### 3.1.3. Effect of temperature and time on water content response

The water content of cashew apple powder was obtained at a range of 9.19-15.35%. In accordance with Analysis of Variance (ANOVA) on water content response, the model used for analysis was 2-factor interactions (2FI) ($R^2 = 0.9828$) with $p$-value <0.0001. Based on the results of ANOVA (Table 3) shows that variables A, B, and AB give a significant effect on water content response ($\alpha$=5%) with $p$-value <0.0001 of all variables. The polynomial equations of 2FI model according to the ANOVA on water content response is:

$$\text{Water content} = +11.60 - 1.56A - 1.33B + 0.97AB$$  \hspace{1cm} (6)

The relationship between the two independent variables on the response is shown in Figure 1. From the polynomial equation and the surface curve, it can be concluded that the water content of cashew apple powder will decrease when the process temperature and time of the vacuum drying process increased. The drying process that uses high temperature provides more available heat to evaporate free water in the sample [13]. Hence, the water content of the dried product that uses higher temperature and longer time for the drying process will be lower than the dried product from lower temperature and shorter time in the drying process.

**Table 3. ANOVA for each response.**

| Sources                        | Vitamin C | IC$_{50}$ | Water content | ΔE   |
|-------------------------------|-----------|-----------|---------------|------|
| Model                         | $<0.0001$ | 0.0003    | $<0.0001$     | $<0.0001$ |
| A-Temperature of vacuum drying process | 0.0010    | 0.0377    | $<0.0001$     | 0.0001  |
| B-Time of vacuum drying process | 0.0002    | 0.0256    | $<0.0001$     | $<0.0001$ |
| AB                            | 0.7842    | 0.5415    | $<0.0001$     | 0.5363  |
| $A^2$                         | $<0.0001$ | $<0.0001$ | -             | 0.5518  |
| $B^2$                         | $<0.0001$ | 0.0220    | -             | 0.0011  |

### 3.1.4. Effect of temperature and time on total color difference ΔE response

The ΔE value or total color difference of cashew apple powder from the vacuum drying process was obtained around 4.23-225.2. According to Analysis of Variance (ANOVA) on total color difference, the model that was used to observe the response was the quadratic model ($R^2 = 0.9701$) with $p$-value <0.0001. The results of ANOVA (Table 3) show that variables A, B and $B^2$ give a significant influence on ΔE ($\alpha$=5%) with $p$-value 0.0001, $<0.0001$, and 0.0011 consecutively. The variables AB or the interactions of two factors and $A^2$ have no significant influence on ΔE with a $p$-value of 0.5363 and
0.5518 respectively. The polynomial equations of $\Delta E$ response with a quadratic model based on the ANOVA are provided in equation (7):

$$\text{Total color difference (}\Delta E\text{)} = +9.25 + 3.21A + 4.94B - 0.38AB + 0.28A^2 + 2.39B^2$$  \hspace{1cm} (7)

The correlation of the process temperature and time on the response can be analyzed in Figure 1. Based on the polynomial equation and the surface curve it was indicated that increasing the process temperature and time of vacuum drying will increase the $\Delta E$ value. The temperature and time of the drying process affected the color profile of a sample which cause a decrease in $L^*$ value and an increase in $a^*$ and $b^*$ values. The increase in $a^*$ value in the dried product is caused by the browning reaction that occurred [14]. Non-enzymatic browning reaction occurred due to the oxidation process of ascorbic acid into furan or furfural compounds [15]. The enzymatic browning in the drying process mostly occurs as a result of degradation of phenolic compounds by polyphenol oxidase enzyme that produces melanoidins, nitrogen-containing compound that gives brown appearance due to phenol degradation by enzymes or heat [6]. At low temperature, the polyphenol oxidase enzyme is still actively degrading polyphenols into melanoidins, which give dark-brown color. With higher temperature, polyphenol oxidase denatured that reduces enzymatic browning. However, high temperature promotes oxidation reaction of ascorbic acid into furfural compounds that also contributes to the browning of the product.

![Figure 1](image)

**Figure 1.** Surface curve effect of process temperature and time towards vitamin C response (A), antioxidant activity ($\text{IC}_{50}$) response (B), water content response (C), and total color difference response (D).

According to the optimization process from Design Expert 12, the optimum condition of the vacuum drying process was 60.1 °C in drying temperature and 11 hours 40 minutes in drying time with a desirability value of 0.776. The optimum condition resulted in cashew apple powder with Vitamin C content of 118.5 mg/100g, antioxidant activity ($\text{IC}_{50}$) of 70.6 ppm, the water content of 11.4%, and total color difference ($\Delta E$) of 8.8. Then validation experiment was carried out with the optimum condition in triplicate and the results concluded that there were no significant differences between the prediction data
from Design Expert 12 and validation data based on paired t-test (p > 0.05). The characterization analysis of fresh cashew apple and optimum cashew apple powder is shown in Table 4.

### Table 4. Characterization of cashew apple and cashew apple powder.

| Analysis                        | Optimum cashew apple powder* | Fresh cashew apple* |
|---------------------------------|------------------------------|---------------------|
| Water content (%)               | 11.42 ± 0.53                 | 75.46 ± 1.09        |
| Ash content (%)                 | 2.75 ± 0.43                  | 2.28 ± 1.06         |
| Crude protein (%)               | 8.87 ± 0.40                  | 12.75 ± 0.66        |
| Fat content (%)                 | 2.22 ± 0.54                  | 4.31 ± 0.95         |
| Carbohydrate (by difference) (%)| 74.75 ± 0.92                 | 5.20 ± 1.67         |
| IC₅₀ (ppm)                      | 70.58 ± 1.40                 | 51.95 ± 0.72        |
| Vitamin C (mg/100g)             | 118.53 ± 1.24                | 101.59 ± 1.29       |
| Color                           |                              |                     |
| L                               | 65.77 ± 0.31                 | 72.30 ± 0.95        |
| a                               | +3.63 ± 0.25                 | +0.80 ± 0.36        |
| b                               | +27.70 ± 0.30                | +32.90 ± 0.72       |

*The values of the analysis were conducted in triplicate with standard deviation

Based on Table 4 the water content of optimum cashew apple powder decreased to 11.42% from 75.46% in fresh cashew apple. The drying process uses heat that evaporated water content [13]. The ash content increased in cashew apple powder from 2.28% to 2.75%. The decreased water content in the sample will increase solid components in the sample accordingly and hence the result [11]. The crude protein decreased during the drying process from 12.75% in fresh samples to 8.87% in powdered samples. The Maillard reaction during the drying process took place and therefore the protein were polymerized with reducing sugar-forming melanoidins [6]. The fat content of optimum cashew apple powder was lower than the fresh ones due to the evaporation of volatile compounds during the drying process such as carotenoid [16]. The carbohydrate content was increased in accordance with the decreased water content in the final product. The IC₅₀ value was increased in the cashew apple powder during the drying process due to the polymerization of polyphenols into melanoidins, also the evaporation of antioxidant compounds [12]. Vitamin C is solid and the content in the cashew apple powder was increased due to the decreased water content in the powder. The brightness (L) and the yellowness (b*) were decreased while the redness (a*) was increased during the drying process due to the browning reaction from the oxidation of Vitamin C into furan or furfural compounds and the degradation of polyphenols into melanoidins [6,15].

### 4. Conclusions

The drying process with high temperature and long drying time caused the degradation of antioxidant compounds in many fruits. A vacuum dryer was used to lower the process temperature and shorten the drying time in order to preserve the antioxidant compounds in cashew apple. The optimization of process temperature and time of vacuum drying for the production of cashew apple powder can produce a product that high in vitamin C and antioxidant activity. Based on this study, the optimum condition for the production of cashew apple powder was obtained at a temperature of 60.1 °C and time of 11.4 hours with vitamin C content of 118.5 mg/100g, antioxidant activity (IC50) of 70.6 ppm, the water content of
11.4%, and total color difference (ΔE) of 8.8. The cashew apple powder produced from this research contains a viable source of vitamin C and antioxidant compounds. Hence, the optimum process temperature and time from this research can be used for producing cashew apple powder that contains high antioxidant activity and Vitamin C content.

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