Optimisation on the production of biodegradable plastic from starch and cassava peel flour using response surface methodology

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Abstract. Biodegradable plastic is starch-based plastic that can be naturally decomposed by microorganisms. Cassava is one of the primary starch-producing plants. The increasing amount of cassava production increases cassava peel waste. Cassava peel has 50% of the starch content in the tuber. The factors that are affecting the production of biodegradable plastics are temperature and drying duration. This study aimed to obtain the optimum temperature and drying time to produce biodegradable plastics from starch and flour of cassava peel waste. Response Surface Methodology (RSM) with a Central Composite Design (CCD) method was employed. The experimental design included two factors and three responses. The first factor was the drying temperature (40 °C, 50 °C, and 60 °C) and the second factor was the drying duration (5 hours, 6 hours, and 7 hours). The responses measured were tensile strength, elongation, swelling and biodegradability. The study found that the optimum condition of the process was at the drying temperature of 57.79 °C and drying duration of 5 hours. At this optimum condition, the biodegradable plastic produced has the tensile strength of 2554.65 N/m²; elongation of 16.67%; and swelling of 124.17%. Biodegradation testing for 12 days resulted in a mass reduction of 58.30%.

1. Introduction

At present, the most often used type of packaging is plastic, but it is difficult to be degraded by microorganisms in the environment. In normal environmental conditions, to degrade several grams of plastic, it takes about one hundred years [1]. To overcome these problems, it is necessary to innovate environmentally friendly plastics, one of which is biodegradable plastic packaging. Biodegradable plastic is a plastic based on biological compounds so that it can be degraded by microorganisms in the environment [2]. The biological compounds that are often used to make biodegradable plastics are starch.

Cassava has a starch content of 34%, where 83% is amylopectin and 17% is amylose [3]. According to data from Statistics Indonesia, cassava production in Indonesia reached 24 million tons in 2015 [4]. The waste of cassava peel produced increased with the high cassava production, which estimated that each kilogram of cassava can produce 15-20% of cassava peel waste [5]. Therefore, based on production data of cassava in 2015, it is estimated that amount of cassava peel waste in Indonesia can reach 3.6-4.8 million tons. Thus, this is certainly a great potential to increase the cassava value by making starch-based biodegradable plastics from cassava peel.
There are several factors that can affect the making of biodegradable plastics, including drying temperature and drying time [5-8]. If the drying temperature was too high or the drying time was too long, the bioplastic particles can undergo physiochemical structural changes to become tighter and more homogeneous. This inhibits microorganisms to decompose plastic constituent particles. However, if the drying temperature was too low or the drying time was too fast, microorganisms can only break down a few particles of bioplastics due to its unfinished structural formation [5].

Several studies have reported that biodegradable plastics can be made from cassava starch and cassava peel flour [7, 9]. Previous study found that the best biodegradable plastic products were made from 100% cassava starch due to its optimum gelatinisation capacity [7]. Another study demonstrated that the optimal results of biodegradable plastic from cassava peel starch obtained were at drying temperature of 50°C for 6 hours, giving the biodegradability rate of 51.18% [9]. This study aimed to determine the optimum temperature and drying time for making biodegradable plastics from starch and cassava peel flour to obtain plastic with optimal mechanical properties and swelling.

2. Materials and Method

2.1. Preparation of cassava peel flour
Cassava peels were obtained from the local market in Malang, East Java, Indonesia. The cassava peels were removed from the brown outer skin. About 1.4 kg cassava peels were washed then cut into small pieces with the size of 1 x 1 cm. The sample preparation was following the procedures described in Pulungan et al. [7]. The sample was blanched for 5 minutes at 80-85°C, dried in tunnel dryer for 4 hours, crushed by blender, and then sifted with a 60-mesh sieve.

2.2. Preparation of biodegradable plastic
The preparation of making biodegradable plastic was following the procedures described by Pulungan et al. [7], with modification. Chitosan 2% (w / v) was mixed with 50 ml of 1% acetic acid then stirred with a spatula at 100°C for 15 minutes until chitosan gel was obtained. About 60% cassava starch and 40% cassava peel flour were mixed with 100 mL of distilled water and manually stirred at 65°C for 30 minutes to produce a starch suspension. The starch suspension was stirred and heated at 65°C for 5 minutes to form a gel. Chitosan gel, starch gel, and glycerol 3% (v / v) were mixed while heated at 65°C and stirred with the spatula for 10 minutes to form a biodegradable plastic gel. About 150 mL biodegradable plastic gels were molded in the Teflon with a size of 17 x 27 cm. The samples were dried in the oven according to the experimental design.

2.3. Experimental design
A Central Composite Design (CCD) in Response Surface Methodology (RSM) design was used with the drying temperature (X₁) and drying time (X₂) as variables. The drying temperature levels were 40°C, 50°C, 60°C and the drying time were 4, 5, and 6 hours. The experiment was conducted at 13 experimental points. The data obtained from this research were processed using Design Expert 7.5 program to obtain the optimal value of biodegradable plastic from cassava starch and peel flour. The responses measured were tensile strength, elongation, and swelling. The sample obtained from the optimum treatment was then selected for further analysis of biodegradability test, FTIR, and SEM.

3. Results and Discussion

3.1. Properties of the biodegradable plastic
The results of the biodegradable plastic’s mechanical properties and swelling in various conditions are shown in Table 1. This study demonstrated that biodegradable plastic with the highest tensile strength (3981.56 N/m²) was resulted from the treatment of drying temperature at 60°C and drying time of 5 hours. Similar result was obtained for the treatment with 50°C drying temperature and 4.59 hours drying time. However, when using drying temperature of 50°C for 6 hours, the resulted biodegradable plastic has the lowest tensile strength of 617.83 N/m². The similar trends were also observed for elongation parameters. Where the highest elongation value was resulted from the treatment of 60°C
for 5 hours or 50°C for 4.59 hours, giving the value of 16.25%. The lowest elongation was again obtained from treatment of 50°C for 6 hours with the value of 2.5%. The lowest elongation value was possibly due to the uneven thickness of the plastic which affects the mechanical properties of the biodegradable plastic. The highest swelling was obtained from the treatment of drying temperature at 64.14°C for 6 hours, with the value of 215.1%. While, the lowest swelling value was 131.3% resulted from treatment with drying temperature of 40°C dan drying time of 5 hours. This result was in agreement with Suderman et al. [6] that drying temperature and time have impact on the mechanical properties of the biodegradable plastic.

3.1.1. Tensile strength
The ANOVAs results of biodegradable plastic tensile strength can be seen in Table 2. The model has p > 0.05, indicating that the quadratic model was not significant. The model demonstrates that drying temperature, drying time, and the interaction of both factors had no significant effect on the tensile strength of biodegradable plastic. R² value was 0.5324 which showed that 53.24% of the data supporting the model. Based on this, the polynomial equation in the form of actual variables can be written as follows:

$$Y = 20643.98 + 61.46X_1 - 6616.97X_2 - 45.85X_1X_2 + 2.44X_{12} + 702.66X_{22}$$  \hspace{1cm} (1)$$

### Table 1. Mechanical properties and swelling of biodegradable plastic in various conditions

| Code | Variable | Actual Variable | Response |
|------|----------|-----------------|----------|
| X₁   | X₂       | Drying Temperature (°C) | Drying Time (hours) | Tensile Strength (N/m²) | Elongation (%) | Swelling (%) |
| -1   | -1       | 40              | 5         | 1843.68              | 7.5            | 131.3        |
| 1    | -1       | 60              | 5         | 3981.56              | 16.25          | 172.6        |
| -1   | 1        | 40              | 7         | 2147.69              | 8.75           | 193.3        |
| 1    | 1        | 60              | 7         | 2451.70              | 10             | 166.7        |
| -1.414 | 0       | 35.68           | 6         | 2147.69              | 8.75           | 208.4        |
| 1.414 | 0       | 64.14           | 6         | 2147.69              | 8.75           | 215.1        |
| 0    | -1.414   | 50              | 4.59      | 3981.56              | 16.25          | 176          |
| 0    | 1.414    | 50              | 7.41      | 2147.69              | 8.75           | 186.2        |
| 0    | 0        | 50              | 6         | 3373.54              | 7.5            | 171          |
| 0    | 0        | 50              | 6         | 1843.68              | 7.5            | 198.4        |
| 0    | 0        | 50              | 6         | 1843.68              | 7.5            | 201.6        |
| 0    | 0        | 50              | 6         | 617.83               | 7.5            | 198.4        |

### Table 2. The ANOVA results of biodegradable plastic’s tensile strength

| Source          | Sum of Squares | df | Mean Square | F Value | p-value  | p-value  |
|-----------------|----------------|----|-------------|---------|----------|----------|
| Model           | 7.009E+006     | 5  | 1.402E+006  | 1.59    | 0.2771   | Not significant |
| A-Temperature   | 7.454E+005     | 1  | 7.454E+005  | 0.85    | 0.3878   | Not significant |
| B-Time          | 1.823E+006     | 1  | 1.823E+006  | 2.07    | 0.1930   | Not significant |
| AB              | 8.408E+005     | 1  | 8.408E+005  | 0.96    | 0.3607   | Not significant |
| A²              | 4.148E+005     | 1  | 4.148E+005  | 0.47    | 0.5143   | Not significant |
| B²              | 3.435E+006     | 1  | 3.435E+006  | 3.91    | 0.0887   | Not significant |
| Residual        | 6.155E+006     | 7  | 8.793E+005  |         |          |          |
| Lack of Fit     | 9.792E+005     | 3  | 3.264E+005  | 0.25    | 0.8565   | Not significant |
| Pure Error      | 5.176E+006     | 4  | 1.294E+006  |         |          |          |
| Cor Total       | 1.316E+007     | 12 |             |         |          |          |
The effect of drying temperature and time on the tensile strength of the biodegradable plastic can be seen in Figure 1. In Figure 1a, the outer line of the contour plot shows the highest tensile strength, and the deeper line shows the decrease in the tensile strength. Contour colors from red to blue indicates that the value of tensile strength was decreasing. From Figure 1b, it shows that increases in the drying temperature to more than 60°C and decreases in the drying time to lower than 5 hours, causing an increase in the tensile strength of the biodegradable plastics produced. Similarly, Utomo et al. [10] found that increasing drying temperature and lowering drying time have positive impact on increasing the tensile strength of the biodegradable. This was possibly because a high temperature can promote the particle to form a strong binding and homogenous structure in the biodegradable plastic [5]. Furthermore, the addition of chitosan may also contribute to the mechanical properties of the biodegradable plastic. Chitosan and starch were found to be able to form hydrogen bonds, due to a high temperature used, thus creating a strong chemical bond in the plastic [10]. However, if the drying time was too long, the tensile strength tended to decrease due to a greater evaporation of chitosan [9, 10].

![Figure 1](image1.png)

**Figure 1.** Response surface contour (A) and three-dimensional plots (B) biodegradable plastic tensile strength

### 3.1.2. Elongation

The ANOVA analysis results of biodegradable plastic elongation is shown in Table 3. Drying temperature and the interaction between both factors have no significant effect on the elongation of the biodegradable plastic. However, drying time had a significant effect on the biodegradable plastic’s elongation. The $R^2$ value was 0.7721, indicating that 77.21% of the data was supporting the model. Thus, the polynomial equation in the form of actual variables was as follows:

$$Y = 91.83 + 0.13X_1 - 28.58X_2 - 0.19X_1X_2 + 0.01X_{12} + 3.00X_{22} \quad (2)$$

![Table 3](image2.png)

| Source        | Sum of Squares | df | Mean Square | F Value | p-value     |     |
|---------------|----------------|----|-------------|---------|-------------|-----|
| Model         | 123.43         | 5  | 24.69       | 4.74    | 0.0328      | Significant |
| A-Temperature | 12.50          | 1  | 12.50       | 2.40    | 0.1651      | Not significant |
| B-Time        | 30.45          | 1  | 30.45       | 5.85    | 0.0462      | Significant |
| AB            | 14.06          | 1  | 14.06       | 2.70    | 0.1442      | Not significant |
| A^2           | 8.80           | 1  | 8.80        | 1.69    | 0.2345      | Not significant |
| B^2           | 62.61          | 1  | 62.61       | 12.03   | 0.0104      | Significant |
| Residual      | 36.43          | 7  | 5.20        |         |             |     |
| Lack of Fit   | 16.43          | 3  | 5.48        | 1.10    | 0.4477      | Not significant |
| Pure Error    | 20.00          | 4  | 5.00        |         |             |     |
| Cor Total     | 159.86         | 12 |             |         |             |     |
The effect of drying temperature and time on the elongation of biodegradable plastic is shown in Figure 2. In Figure 2a, the outer line of the contour plot shows the highest elongation and the deeper line shows a decrease in the elongation. Contour colors from red to blue indicates that the value of elongation decreased. In Figure 2b, it can be seen that if the drying temperature was raised to more than 60°C and the drying time was reduced to less than 5 hours, the elongation was increasing. Such behavior was possibly due to effect of drying condition to the mechanical properties. In this study, it shows that an increase in the elongation of the biodegradable plastic was triggered by an increase in the drying temperature and by a reduction in the drying time. A high drying temperature was found to cause water evaporation, which solidified and homogenized the structure of biodegradable plastic particle [10]. However, a high drying temperature or drying time was also found to reduce the elongation ability of the biodegradable bioplastic, possibly due to the evaporation of the glycerol added [9, 10].

![Figure 2](A) ![Figure 2](B)

**Figure 2.** Response surface contour (A) and three-dimensional plots (B) biodegradable plastic elongation

3.1.3. **Swelling**

The ANOVA analysis results of biodegradable plastic elongation can be seen in Table 4. In the table it is known that, the model has p> 0.05 so that the quadratic model was not significant. The drying temperature, drying time, and the interaction of both independent variables have a non-significant effect on the swelling of biodegradable plastics. \( R^2 \) value was 0.5452 which showed that the model supporting data was 54.52%. Based on this, the polynomial equation in the form of actual variables was as follows:

\[
Y = -863.84 + 9.32X1 + 263.64X2 - 1.70X1X2 + 0.01X12 - 14.16X22
\]

(3)

| Source         | Sum of Squares | df | Mean Square | F Value | p-value     |
|----------------|----------------|----|-------------|---------|-------------|
| Model          | 3306.80        | 5  | 661.36      | 1.68    | 0.2572      | Not significant |
| A-Temperature  | 73.06          | 1  | 73.06       | 0.19    | 0.6797      | Not significant |
| B-Time         | 621.72         | 1  | 621.72      | 1.58    | 0.2493      | Not significant |
| AB             | 1152.60        | 1  | 1152.60     | 2.93    | 0.1309      | Not significant |
| A²             | 9.40           | 1  | 9.40        | 0.024   | 0.8816      | Not significant |
| B²             | 1395.31        | 1  | 1395.31     | 3.54    | 0.1019      | Not significant |
| Residual       | 2757.96        | 7  | 393.99      |         |             |               |
| Lack of Fit    | 2074.92        | 3  | 691.64      | 4.05    | 0.1050      | Not significant |
| Pure Error     | 683.04         | 4  | 170.76      |         |             |               |
| Cor Total      | 6064.76        | 12 |             |         |             |               |

**Table 4.** The ANOVA results of biodegradable plastic’s swelling
The effect of drying temperature and time on the swelling of biodegradable plastic can be seen in Figure 3. In Figure 3a, the outline of the contour shows the highest swelling value and the deeper shows a decrease in the swelling value. The contour colour from red to blue shows the swelling value was decreasing. Figure 3b indicates that increasing the drying temperature to more than 60°C with the drying time in the range of 5-6.5 hours, resulted in an increase in the swelling values. But, if the drying temperature was less than 40°C and the drying time was more than 7 hours, the swelling value was also found to increase. This result indicates that the higher the drying temperature and the longer the drying time can increase the swelling value of the biodegradable plastic. This study in agreement with Suryati et al. [5] and Harsojuwono et al. [11] who reported that the swelling values of the biodegradable plastic tends to increase with the increasing temperature and times of drying process.

![Figure 3](image_url)

**Figure 3.** Response surface contour (A) and three-dimensional plots (B) of biodegradable plastic swelling

### 3.1.4. Optimization of the biodegradable plastic’s process parameters

Determination of the optimum point was based on the criteria and objectives of the factor and the desired response. After optimization, verification was then carried out. Table 5 shows a comparison of predictive values and research results. The error rate value of each responses was high, possibly due to uneven thickness of biodegradable plastic during the molding process which resulted a high difference in tensile strength, elongation and swelling ability. A manual molding process was reported to influence on the thickness of the biodegradable plastic, thus impacting its mechanical properties [10, 12].

| Actual Variable | Response |
|-----------------|----------|
| **Drying Temperature (°C)** | **Drying Time (hours)** | **Tensile Strength (N/m²)** | **Elongation (%)** | **Swelling (%)** |
| Prediction      | 57.79    | 5       | 3582.97   | 14.57    | 187.51  |
| Verification    | 57.79    | 5       | 2554.65   | 16.67    | 124.17  |
| Error rate value (%) |         |         | 40.25     | 12.60    | 51.01   |

### 3.2. Biodegradation testing

The results from the biodegradation test are shown in Figure 4, which indicating a reduction in biodegradable plastic mass of 58.30% for 12 days period. Prolonged the biodegradability test to 30 days has no impact on the mass reduction, due to a limited availability of nutrient. Previous study has reported that microbial growth is affected by the lack of nutrients available for microbes causes the microbes to die [13]. Furthermore, a decrease in mass in bioplastic indicates that the biodegradation process was occurred, where the greater mass reduction means a faster degradation of biodegradable plastic [14].
3.3. FTIR and SEM analysis

FTIR spectrum is illustrated in Figure 5. Indeed, biodegradable plastic has the same functional group as its constituent components (i.e. glycerol, chitosan, etc.). The functional groups are C-H alkanes (2850-2970 cm\(^{-1}\)), C-H aromatic rings (690-900 cm\(^{-1}\)), and C-H alkenes (675-995 cm\(^{-1}\)), O-H hydroxyl (3200-3600 cm\(^{-1}\)). The hydroxyl-OH group in biodegradable plastic is believed to originate from glycerol \[15\].

Another additive widely used to make biodegradable plastic was chitosan. Chitosan has a functional group N-H amide/amine. In the FTIR spectrum, the N-functional group is in the wave range 3300-3500 cm\(^{-1}\). Allegedly N-H forms hydrogen bonds so that the functional group -NH shifts to the -OH functional group. –NH group can overlap with the –OH group because of the hydrogen bond in the wave number range of 3400 cm\(^{-1}\). From the FTIR spectrum, it shows that the O-H functional group in biodegradable plastic shifts to be narrower compared to that of the cassava starch and peel flour. Therefore, it was assumed that a new compound has been formed \[16\].

The results of SEM analysis can be seen in Figure 6, demonstrating that the structure of biodegradable plastic was not evenly distributed. Some part of the biodegradable plastics still has rough parts, while other parts were smooth. This rough plastic structure was possibly originated from the cassava peel fibre due to imperfect destruction. Furthermore, pores were also seen in biodegradable plastic’s structure, which causing a high swelling due to absorption of water through these pores. This study found that both drying temperature and time has impact on the structure of the biodegradable plastic (i.e. the pores and uneven surfaces). A study has reported that the higher temperature and longer time of drying, causing the biodegradable plastic to dry faster and to evaporate the water faster, resulting the material particles to move upwards and to fuse the inter-cell layer \[5\]. Some starch granules and air bubbles were also observed, possibly due to inhomogeneous mixing and stirring process, as well as the imperfect molding process which causes the formation of air cavities.
4. Conclusions

The optimum condition of the making of biodegradable plastic from cassava starch and peels flour was at drying temperature of 57.79°C and drying time of 5 hours. This optimum combination resulted the biodegradable plastics with the mechanical properties of tensile strength (2554.65 N/m²), elongation (16.67%), and swelling (124.17%). The biodegradability of the resulted plastic was 58.30% mass reduction in 12 days. The FTIR test indicating that the biodegradable plastic had the same functional group as its constituent components. The SEM of biodegradable plastic, based on SEM analysis, revealed a porous and rough structure due to starch granules formulation and some air bubbles.

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