Multi-response optimization of cellulose fiber isolation from tapioca solid waste and its characteristics

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Abstract. The tapioca-based starch industry produces solid waste in abundance that has not been used optimally, especially the cellulose fraction. This study aimed to optimize the H₂O₂ concentration and the process temperature of cellulose fiber isolation from tapioca solid waste. Statistical regression modeling and optimization of H₂O₂ concentration and process temperature using the response surface methodology. A central composite design (CCD) was applied for experimental design and analysis of the effect of H₂O₂ concentration and process temperature on multi-response characteristics of cellulose, consisting of whiteness index (WI), yield, and α-cellulose content. Cellulose fibers were characterized, including surface morphology, crystallinity degree, and thermal stability. The results showed that the H₂O₂ concentration and process temperature were significantly affected by WI, yield, and α-cellulose content. The maximum WI, yield, and α-cellulose content were 63.99%, 65.73% (w/w), and 78.31% (w/w), respectively, obtained from H₂O₂ concentration of 22.62% (v/v) and process temperature of 93.51 ºC. This cellulose has a relatively coarse fiber formation, with a high degree of crystallinity and thermal stability. Thus, cellulose from TSW might have a potential to be applied in broader fields.

1. Introduction
Starch from cassava tuber has been extensively applied in the food and non-food fields [1]. However, these production processes result in Tapioca Solid Waste (TSW). Approximately 10-15% TSW will be generated from the conversion process of fresh cassava into starch products [2]. TSW can cause environmental pollution problems due to the decomposition process. The decomposition process of organic matter has been known to produce various types of greenhouse gases that can result in global climate change [3], [4]. This problem certainly needs further handling, and the use of TSW as a source of cellulose raw material is an alternative solution.

Generally, the cellulose isolation from starchy materials is carried out through several stages, namely starch hydrolysis, delignification, and bleaching. Starch hydrolysis was carried out to degrade starch to produce lignocellulosic fibers [5]. Delignification was carried out to degrade the lignin and hemicellulose fractions from lignocellulose [6]. In the final stage, the bleaching process is carried out to remove chromophore compounds and residual lignin to increase the purity [7]. Currently, many researchers have reported and developed environmentally friendly bleaching processes, one of which is H₂O₂. Bleaching of sago frond fiber with 30% (v/v) H₂O₂, the temperature of 100 ºC, the ratio of material to a solution of 1:10 (w:v), for 1 hour resulted in yield and WI of 45.73% and 72.78%, respectively [8]. Bleaching jute fiber with 1% (v/v) H₂O₂, the temperature of 50 ºC, the ratio of material to a solution of 1:40 (w:v), for 30 minutes produces WI of 63.22% [9]. This study indicates that various factors influence
the bleaching process on different materials, and the conditions vary greatly, resulting in varying cellulose characteristics. For this reason, research on the optimum conditions for cellulose fibers isolation is essential.

Generally, studies that have been reported on the bleaching process with H₂O₂ under various conditions and types of materials were carried out without a mathematical optimization model approach. Meanwhile, this approach has not been reported for the bleaching process optimization of cellulose isolation from TSW. Optimizing the isolation process conditions is expected to simultaneously produce a characteristic response of cellulose with high whiteness, yield, and content of α-cellulose.

Currently, experimental designs with a multivariate analysis approach have been reported to analyze multiple variables simultaneously to evaluate the main effects, interactions between variables and determine the combination of each level to produce optimal process conditions [10]. The response surface methodology (RSM) combined with the desirability function has been widely used to design experiments and build models for determining the optimal conditions of multiple factors and responses [11]. In this regard, this study aims to optimize the concentration of H₂O₂ and temperature of the cellulose isolation process from TSW on the characteristics of WI, yield, and α-cellulose content. Cellulose obtained from optimum conditions was characterized by surface morphology, crystallinity degree, and thermal stability.

2. Materials and methods

2.1. Materials

The materials used in this study were NaOH (Merck), 50% v/v H₂O₂ (technical grade, Brataco), α-amylase, and amylglucosidase enzymes. TSW was obtained from the small-scale tapioca industry in Tanah Baru, Bogor, West Java. TSW was sun-dried until the moisture content reached about 12%, then ground and sieved to a size smaller than 40 mesh.

| Run number | Code    | H₂O₂ (% v/v) | T (°C) | WI (%)  | Yield (% w/w) | α-Cellulose (% w/w) |
|------------|---------|--------------|--------|---------|--------------|---------------------|
| 1          | -1      | 10           | 80     | 54.24   | 70.36        | 53.18               |
| 2          | 1       | 30           | 80     | 57.59   | 62.52        | 55.99               |
| 3          | -1      | 10           | 100    | 61.57   | 65.06        | 62.77               |
| 4          | 1       | 30           | 100    | 63.22   | 57.79        | 82.28               |
| 5          | -1.414  | 5.86         | 90     | 50.78   | 71.88        | 51.09               |
| 6          | 1.414   | 34.14        | 90     | 66.27   | 58.77        | 79.04               |
| 7          | 0       | -1.414       | 20     | 75.86   | 53.94        | 60.91               |
| 8          | 0.1414  | 20           | 104.14 | 68.78   | 54.12        | 76.75               |
| 9          | 0       | 20           | 90     | 58      | 65.03        | 75.04               |
| 10         | 0       | 20           | 90     | 62.43   | 67.93        | 77.28               |
| 11         | 0       | 20           | 90     | 65      | 70.63        | 74.15               |
| 12         | 0       | 20           | 90     | 61.93   | 69.74        | 70.85               |

2.2. Experimental design and data analysis

The study was designed using RSM-CCD with two independent variables, namely the H₂O₂ concentration (X₁) and the process temperature (X₂), and three responses as the dependent variable, namely WI (Y₁), yield (Y₂), and α-cellulose content (Y₃). The design matrix of the CCD and the response value of each cellulose characteristic is presented in Table 1. Design-Expert software version 7.0 is used for Analysis of Variance (ANOVA) analysis, mathematical models design, graphs, and analyzing the optimal conditions of each response. Furthermore, an optimization method with a desirability function determines the optimal conditions for multiple responses.
2.3. Cellulose fiber isolation

Isolation of cellulose fibers from TSW was carried out by starch hydrolysis, delignification, and bleaching [3]. TSW (30% w/v) was heated at 95-100°C until the starch fraction was gelatinized, then 1.2 mL/kg (600 U/mL) α-amylase enzyme was added and stirred periodically for 1 hour. After that, the mixture's temperature was lowered to 55°C, added the enzyme amylloglucosidase 1.2 mL/kg, and stirred periodically for 1 hour. Next, the mixture is pressed to separate the liquid fraction from the fibers. The fiber was dried to a moisture content of about 10% w/w to produce TSW-destarched. After that, the fiber (10 g) was delignified with 100 mL of 10% (w/v) NaOH solution at 100°C for 2 hours. Next, the fibers were rinsed to neutral pH and dried to produce delignified fibers (DF). Furthermore, DF (10 g) was bleached using 100 mL of H₂O₂ solution at a concentration and temperature according to the process conditions in the experimental design (Table 1). The bleaching process was carried out for 1 hour with 600 rpm stirring. This process produces cellulose fibers, characterized according to the predetermined variables. Isolation of α-cellulose was carried out by dissolving cellulose fibers into 500 mL of 17.5% (w/v) NaOH at 80°C, stirring at 600 rpm for 30 minutes. Next, the mixture was filtered and washed until the fiber filtrate reached a neutral pH. α-cellulose was dried in an oven at 100°C for 24 hours.

2.4. Sample analysis

TSW was analyzed for its chemical composition, including moisture content, protein, lipid, ash, carbohydrates, starch, cellulose, hemicellulose, and lignin. Meanwhile, cellulose fibers were analyzed for WI, yield [12], and α-cellulose content [13]. Cellulose was characterized morphologically by scanning electron microscopy (SEM), crystallinity degree by X-ray diffraction (XRD), and thermal stability by thermogravimetric analysis (TGA).

3. Results and discussion

3.1. Tapioca solid waste characteristics

TSW contained water content of 12.32 ± 0.78%, protein of 0.94 ± 0.77%, fat of 1.07 ± 0.02%, ash of 0.79 ± 0.01%, and carbohydrates (by difference) of 84.88 ± 0.80% [3]. In addition, TSW also contains starch, lignin, hemicellulose, and cellulose at 60.1%, 2.53%, 8.89%, and 10.04%, respectively. The lignin, hemicellulose, and cellulose content increased after the starch was enzymatically hydrolyzed (TSW-destarched). Then, the lignin and hemicellulose content decreased, followed by increased cellulose content after TSW was delignified. The chemical compositions of TSW, TSW-destarched, and DF are presented in Table 2. Increasing the cellulose content indicated that the alkaline solution could degrade the lignin and hemicellulose fractions so that the purity of the cellulose was increasing. The TSW contains relatively high cellulose so that it can be used as a source of natural fiber. Several researchers have reported that TSW contains cellulose of 20% [14], 15% to 50% [15], and 10% to 15% [16]. The difference in cellulose content was caused by the different varieties and growing conditions of cassava, the efficiency of the process conditions, and the method of cellulose isolation.

Table 2. Composition (% w/w) analysis of TSW, TSW-destarched, and delignified fiber (DF)

| Composition                  | TSW   | TSW-destarched | DF    |
|------------------------------|-------|----------------|-------|
| Moisture                     | 12.32 | 9.89           | 10.77 |
| Protein                      | 0.94  | -              | -     |
| Lipid                        | 1.07  | -              | -     |
| Ash                          | 0.79  | 5.54           | 1.26  |
| Carbohydrate (by difference) | 84.88 | -              | -     |
| Starch                       | 60.10 | -              | -     |
| Lignin                       | 2.53  | 25.21          | 10.98 |
| Hemicellulose                | 8.89  | 24.54          | 11.30 |
| Cellulose                    | 10.04 | 30.45          | 55.57 |
3.2. Optimization of cellulose fiber isolation

3.2.1. The effect of the isolation process conditions on the characteristics of cellulose

The results of the quadratic model analysis of the effect of each independent variable on each characteristic response of cellulose fiber are presented in Table 3. A good model is a model that has a lack of fit value that is relatively insignificant to the pure error. The quadratic model of all responses has an insignificant lack of fit value (p>0.05), which means that the model has met the requirements to describe experimental data. The value of the determination coefficient (R²) shows the contribution of the influence of independent factors (X) on the experimental response (Y). The R² values of the WI, yield, and α-cellulose regression models were 0.7875, 0.9120, and 0.8873, respectively. This value means that the H₂O₂ concentration and process temperature influence the characteristics changes of WI, yield, and α-cellulose by 78.75%, 91.20%, and 88.73%, respectively. ANOVA analysis showed that the H₂O₂ concentration and process temperature in linear form (X₁, X₂) had a significant effect (p<0.05) on WI, Yield, and α-cellulose. Meanwhile, in the quadratic form (X₁², X₂²), the H₂O₂ concentration only had a significant effect on the α-cellulose content, and the process temperature only had a significant effect on yield (p<0.05). Meanwhile, the interaction between the H₂O₂ concentration and process temperature (X₁X₂) did not significantly affect all responses (p>0.05).

Table 3. Analysis by ANOVA for the quadratic models of whiteness index (WI), yield, and α-cellulose (α-Cell) content

| Source     | Sum of Squares | df | Mean Square | F value | p-value |
|------------|----------------|----|-------------|---------|---------|
|            | WI Yield α-Cell |    |             | WI Yield α-Cell | WI Yield α-Cell |
| Model      | 257.12         | 3  | 85.71       | 4.40    | 9.45    | 0.04* 0.00* 0.01* |
| X₁         | 90.46          | 2  | 45.23       | 7.74    | 20.1    | 0.03* 0.00* 0.00* |
| X₂         | 143.89         | 2  | 71.95       | 12.31   | 14.55   | 0.01* 0.02* 0.01* |
| X₁X₂       | 0.72           | 1  | 0.72        | 0.06    | 2.93    | 0.81 0.91 0.14  |
| X₁²        | 22.03          | 1  | 2.20        | 1.88    | 8.84    | 0.22 0.38 0.02* |
| X₂²        | 1.22           | 1  | 1.22        | 0.10    | 2.18    | 0.76 0.00* 0.19  |
| Residual   | 70.14          | 6  | 11.69       | 1.80    | 5.68    | 0.32* 0.58** 0.09*** |
| Lack of Fit| 45.06          | 3  | 15.02       | 1.80    | 7.12    | 0.32* 0.58** 0.09*** |
| Pure Error | 25.09          | 3  | 8.36        | 0.03    | 0.38    | 0.03* 0.58** 0.09*** |
| Cor Total  | 327.26         | 11 | 29.75       | 1.80    | 7.12    | 0.32* 0.58** 0.09*** |

Note: *: significant (p<0.05), ns: non-significant

The results of multiple regression analysis of the correlation between the independent variables (X) to each response (Y) obtained quadratic polynomial equations presented in equations (1), (2), and (3). A positive value of the regression coefficient indicates a synergistic effect, while a negative value indicates an antagonistic effect [17]. The regression model equation shows that the WI value increases directly with the increasing H₂O₂ concentration and isolation process temperature (equations (1)). This result has been confirmed from the counter plot graph in Figure 1a. The increase in WI value was caused by the decreasing levels of lignin and hemicellulose in line with the increasing solution concentration and process temperature [18]. The WI value is influenced by the material's high and low lignin levels [19]. The higher the lignin content in cellulose, the lower the WI value.

\[ Y₁ (WI) = -33.441 + 1.459 X₁ + 1.294 X₂ - 0.004 X₁X₂ - 0.019 X₁² - 0.004 X₂² \]  (1)
\[ Y₂ (Yield) = -289.336 - 0.199 X₁ + 8.331 X₂ + 0.001 X₁X₂ - 0.009 X₁² - 0.048 X₂² \]  (2)
\[ Y₃ (α-Cell) = -178.804 - 0.692 X₁ + 4.949 X₂ + 0.042 X₁X₂ - 0.057 X₁² - 0.028 X₂² \]  (3)
The yield and $\alpha$-cellulose content increased when the concentration of $\text{H}_2\text{O}_2$ decreased, but the temperature isolation process increased (equations (1), (2)). Changes in yield and $\alpha$-cellulose content are more significantly influenced by the process temperature, which is confirmed from the coefficient value of $X_2$, which is relatively larger than $X_1$. This condition has been confirmed from the counter plot graphs in Figure 1b and Figure 1c. This happens because the cellulose is degraded at concentrations of higher $\text{H}_2\text{O}_2$ solution, so combining a lower solution concentration with a high process temperature is thought to increase the yield and $\alpha$-cellulose content [20].

**Figure 1.** The counter plot of the optimization response of (a) WI, (b) yield, (c) $\alpha$-cellulose content, and (d) desirability

### 3.2.2. Multi-response optimization and model validation

Optimization was carried out to obtain the optimum conditions of the cellulose isolation process based on the regression model of some desired cellulose characteristics (desirability). The desired optimum condition is to maximize the value of WI, yield, and $\alpha$-cellulose in a predetermined range of process conditions. Table 4 shows the components of the optimized factors, their targets, minimum and maximum limits, and the level of importance in the optimization phase of the model. WI, yield, and $\alpha$-cellulose are responses with an importance level of 5 (++++)). Yield is a response that determines the efficiency of the isolation process and is related to economic value. Meanwhile, the value of WI and $\alpha$-cellulose determines the cellulose purity level, affecting the cellulose multiplication rate in various fields. The results of the optimization model analysis obtained the optimum conditions for the cellulose isolation process at a concentration of 22.62% $\text{H}_2\text{O}_2$ (v/v) and a temperature of 93.52°C. This condition resulted in cellulose characteristics with WI, yield, and $\alpha$-cellulose values of 63.99%, 65.72%, and 78.31%, respectively. This process condition has a relatively high desirability value of 0.748. The desirability value determines the level of accuracy of the results of the optimal solution. The desirability value close to one means the higher the optimization accuracy value. The contour plot graph of the desirability value at optimum conditions is presented in Figure 1d. WI values from several sources of cellulose fiber have been reported, including cotton 44.17% [21], jute 63.22% [9], and switchgrass...
30.13% [22]. Meanwhile, the yield value of energy cane bagasse is 36.20% [23] and sago frond 45.73% [8]. The α-cellulose content of date palm was 78.63% [13], empty fruit bunches 32.48% [12], and jackfruit leaves 7.27% [24]. Differences in the characteristics of cellulose can be caused by different types of raw materials and conditions of the isolation process.

Table 4. Components of factors, goals, limits and importance in multi-response optimization

| Factors       | Goals          | Lower Limit | Upper Limit | Importance |
|---------------|----------------|-------------|-------------|------------|
| H₂O₂ Cons. (% v/v) | is in range    | 10.00       | 30.00       | 3 (++++)   |
| Temperature (°C)   | is in range    | 80.00       | 100.00      | 3 (++++)   |
| WI (%)        | maximize       | 50.78       | 68.78       | 5 (++++++)  |
| Yield (%)     | maximize       | 54.12       | 71.88       | 5 (++++++)  |
| α-cellulose (%)| maximize       | 51.09       | 82.28       | 5 (++++++)  |

The validation results in Table 5 show that the isolation process under optimum conditions produces an actual value (validation) of the response which is still in the range between the 95% PI low and 95% PI high values and is not significantly different from the predicted value obtained from the optimization model. This value means that the isolation process conditions with H₂O₂ concentration and temperature treatment have been consistently applied to obtain the characteristics of WI, yield, and α-cellulose from cellulose fibers.

Table 5. The results of the prediction and validation values of the solution response values at optimum conditions

| Response | Prediction | Validation | 95% CI low | 95% CI high | 95% PI low | 95% PI high |
|----------|------------|------------|------------|-------------|------------|-------------|
| WI       | 63.99      | 65.44      | 59.92      | 67.98       | 54.66      | 73.24       |
| Yield    | 65.72      | 60.81      | 63.07      | 68.56       | 59.50      | 72.13       |
| α-cellulose | 78.31    | 75.28      | 72.44      | 83.94       | 64.94      | 91.44       |

3.3. Characteristics of cellulose fiber

TSW cellulose is in fiber bundles with a relatively rough surface and a diameter ranging from 7-30 µm (Figure 2a). Cellulose isolation by delignification and bleaching processes has been reported to cause changes in the morphology and dimensions of the fibers. This change was caused by the degradation of hemicellulose and lignin fractions. The alkaline delignification also causes the cellulose structure to swell [25]. XRD analysis showed that the cellulose fiber had type II cellulose with a crystallinity degree of 55.50%. Cellulose II was characterized by 20 peaks of the (1-10), (110), and (020) planes at 12.1°, 19.9°, and 21.7°, respectively (Figure 2b). Cellulose II was formed due to alkalization treatment using 17.50% NaOH in the α-cellulose isolation process. TGA/DTG analysis (Figure 2c) shows that cellulose has three degradation phases, namely phase I (65°C-110°C) is the evaporation phase, II (220°C-400°C) is the depolymerization phase, and III (>400°C) is the phase of char formation [26]. The maximum degradation temperature occurred at 351.51°C, with a weight loss of 44.34%. The characteristics of cellulose fibers from several materials have been reported, including date palm cellulose which has a crystallinity degree of 52.27%, and thermal stability of 200°C [13]. In addition, sisal fiber was 57.30% and 350°C [27], Agave gigantea was 64.60% and 363°C [28], respectively.
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4. Conclusion

The optimization model of H$_2$O$_2$ concentration and process temperature on multi-response characteristics of cellulose from TSW has been successfully designed. The regression model showed that the H$_2$O$_2$ concentration and process temperature significantly affected the characteristics of WI, yield, and α-cellulose content of cellulose fibers. The WI value increases if the H$_2$O$_2$ concentration and the process temperature increases. Meanwhile, the yield and α-cellulose content increased when the H$_2$O$_2$ concentration decreased, followed by increased process temperature. The optimum conditions of the TSW cellulose isolation process were obtained at an H$_2$O$_2$ concentration of 22.62% (v/v), and a temperature of 93.52°C resulted in the maximum value for WI (63.99%), yield (65.72%), and α-cellulose content (78.31%). Characteristics of α-cellulose have a relatively rough surface with a diameter of 8-22 µm, crystallinity degree of 55.50%, and a maximum degradation temperature of 351.51°C. Thus, TSW cellulose has the potential to be widely applied in various fields.

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Figure 2. The characteristics of cellulose (a) surface morphology by SEM magnification 500 X, (b) crystallinity degree by X-RD, and thermal stability by TGA/DTG.
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