Application of taguchi method for selection parameter bleaching treatments against mechanical and physical properties of agave cantala fiber

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Abstract. The characterized agave cantala fiber in this research came from Sumenep, Madura, Indonesia was chemically processed using sodium hydroxide (NaOH) and hydrogen peroxide (H₂O₂) solution. The treatment with both solutions is called bleaching process. Tensile strength test of single fiber was used to get mechanical properties from selecting process of the various parameter are temperature, PH and concentration of H₂O₂ with an L9 orthogonal array by Taguchi method. The results indicate that PH is most significant parameter influencing the tensile strength followed by temperature and concentration H₂O₂. The influence of bleaching treatment on tensile strength showed increasing of crystallinity index of fiber by 21%. It showed by lost of hemicellulose and lignin layers of fiber can be seen from waveforms changes of 1735 (C=O), 1627 (OH), 1319 (CH₂), 1250 (C-O) by FTIR graph. The photo SEM showed that the bleaching of fibers causes the fibers more roughly and clearly than untreated fibers.

1. Introduction

Agave Cantala (cantala) fiber is one of the prospective tropical plants to produce fibers from their leaves. It has high fiber strength, resistant to high salinity and is renewable after harvested every two years. These plants grow well on dry and rocky land like Sumenep, Madura, Indonesia [1]. Chemical structure of cantala fibers contains hemicellulose, lignin and extractive substance as amorphous regions and cellulose as a crystalline region. The chemical process effectively decreases impurities of fibers such as hemicellulose and lignin. It changes the surface topology of fibers and their crystallographic structure. Bleaching is immersing the fibers in alkali solution with reacting hydrogen peroxide (H₂O₂). It process reduces lignin content of natural fibers termed as delignification. Hydrogen peroxide as a substitute of chlorine is widely used in pulp industry for bleaching because it oxides is poisonous and can damage environment [2]. Hydrogen peroxide safer and must conserve energy than another oxide cause if react with water (H₂O) will change to form a hydronium ion (H₃O⁺) and perhydroxyl ion (HO₂⁻) [3]:

\[
\text{H}_2\text{O}_2 + \text{H}_2\text{O} \rightarrow \text{H}_3\text{O}^+ + \text{HO}_2^-
\]
The research has done by Tutus (2014) on bleaching will increase white index and mechanical strength on pulp than using chlorine [4]. Hashem et al. (2010) developed research on bleaching for cotton fibers. These process more active in alkaline medium at PH≈10.3-10.8. Taguchi is one of the designs of experiment method for selection of parameter and their level. This method is simple and efficient to find the optimum reaction from a mixture of sodium hydroxide and hydrogen peroxide at the best temperature. Optimum mixture and condition process of solution effect increase value of mechanical properties from natural fiber. The bleaching process is reaction phase liquid with the absorption of hydroxyl ion in the surface of the fiber and increasing rate of the process by adding sodium hydroxide. The following is activation of hydrogen peroxide [5]:

\[
H_2O_2 + H_2O \leftrightarrow HO_2^- + H_3O^+
\]

(1)

Parameters that effect of bleaching are a concentration of \(H_2O_2\), temperature, and PH. All those parameters are very important against the quality of fibers such as brightness, tensile strength, and elongation [6].

2. Materials and methodology
2.1. Chemical material and process
Chemical treatment conducted by alkalization (NaOH(liquid) with purity of 48%) and bleaching (\(H_2O_2(liquid)\) with purity of 50%). Those products obtained from Brata Chemical Yogyakarta Indonesia. Pretreatment process of fiber is immersing 5% NaOH at 100°C for 1 hour (1:50) w/w of fiber and solution. After that continue by bleaching (NaOH + \(H_2O_2\)) at PH 10, 11 and 12. The concentration of hydrogen peroxide used is 1, 2 and 3%. Time of process is 45°C, 60°C and 75°C.

2.2. Tool
The main tools to prepare this experiment are erlenmeyer glass tube 500 ml (Iwaki), Hotplate magnetic stirrer (U-Ten 78-1), measure glass 1000 ml (Iwaki), digital weigher, PH tester (Milwaukee), Thermometer.

2.3. Single fiber tensile test
A single fiber of cantala tensile test was conducted according to the ASTM D3379-75 [7] standard using a Tenso Lab-168 E strength tester. For every single fiber of cantala was glued on paper frame 20 mm gage length. The tensile test was performed using load cell 300 N at crosshead speed 5 mm/min. The supporting paper frame was cut in the middle. The dimension of single fiber tensile test sample is as presented in Figure 1.

Figure 1. Tensile test specimen ASTM D3379-75

Figure 2. Set up for the fiber tensile test
The tensile strength is obtained from ratio of peak force to the cross-sectional area of plane perpendicular to the fiber axis that expressed by equation 3 [7];

$$\sigma = \frac{F}{A}$$

(3)

Where F is a force for failure (N), A is area of fiber diameter (mm$^2$)

2.4. Description of Taguchi method

Taguchi method developed with an orthogonal array form to design efficient experiments, analyst and minimize the number of experiments [8, 9 and 10]. The result selected is L9 orthogonal array has three control parameters are A (Temp), B (PH) and C (% H$_2$O$_2$) and their levels. The identifications show the parameter and level affect the response (tensile strength of single fiber). The result of experiments transforms into S/N ratio which are measure the deviation from characteristic quality. There are three choices of S/N ratio, i.e. Larger is better, Nominal is best and Smaller is better. This research choosing the larger is better that expressed by equation 4 [10];

$$S/N \text{ ratio} = -10\log_{10} \frac{1}{n} \sum_{i=1}^{n} \frac{1}{y_i^2}$$

(4)

2.5. Fiber characterization

XRD (X-Ray diffraction) that tool was used to study physical properties of the crystalline material. Type of XRD is Rigaku Miniflex diffractometer 600 operate condition 40 KW, 15 mA. Sample of fiber was scanned in 2 theta ranges varying from 3$^\circ$ to 40$^\circ$. The crystalllinity index ($I_c$) of cellulose can be calculated using equation 5 [12];

$$I_c = \left( \frac{I_{002}}{I_{amorph}} \right) \times 100\%$$

(5)

Where $I_{002}$ is the peak intensity (maximum intensity) located at two-theta is around 22$^\circ$ which represent the crystalline cellulose. $I_{amorph}$ is the valley intensity (minimum intensity) located at two-theta is around 18$^\circ$ which represent amorphous cellulose. FTIR (Fourier Transform Infra Red) spectroscopy is an appropriate tool to study chemical structure of cantala. Type of FTIR spectrometer is Paragon 1000 PC Perkin Elmer. Shape and wave number shows the bond of chemical structure in region 900 cm$^{-1}$ to 4000 cm$^{-1}$. SEM (Scanning Electron Microscopy) photographs of fibers surfaces before and after bleaching treatment were taken by JEOL JSM-6510 LA instrument.

3. Results and discussion

3.1. Chemical effect of bleaching process

Bleaching process of cantala fiber decreases the percentage of hemicellulose, lignin and other extractives. Lowering the content of these impurities increase the percentage of cellulose, surface roughness and index whiteness of fibers [14, 16]. Chemical treatment also causes fibrillation on fiber. It reduces fiber’s diameter from 200±60 μm to 60±30 μm.
Figure 3. Untreated fibers  

Figure 4. Bleached fibers  

Figure 5. Size distribution of diameter cantala fibers after bleaching

Cantala fibers after bleaching are resulting various diameter of 30-90 µm (0.030-0.090 mm). 108 samples were grouped into intervals class as much as 6 class. Frequency distribution of average cantala fibers diameter are uniformly located between 30-60 µm (0.030±0.060 mm) can be seen in Figure 5.

| Experiment Number | A Temp (°C) | B PH | C % H₂O₂ | Measured Tensile Strength | Calculated S/N ratio for tensile strength |
|-------------------|------------|------|----------|---------------------------|----------------------------------------|
| 1                 | 45         | 10   | 1        | 621.33                    | 55.81                                  |
| 2                 | 45         | 11   | 2        | 520.75                    | 54.25                                  |
| 3                 | 45         | 12   | 3        | 521.91                    | 53.90                                  |
| 4                 | 60         | 10   | 2        | 799.33                    | 57.85                                  |
| 5                 | 60         | 11   | 3        | 489.58                    | 53.72                                  |
| 6                 | 60         | 12   | 1        | 545.50                    | 54.68                                  |
| 7                 | 75         | 10   | 3        | 732.25                    | 57.27                                  |
| 8                 | 75         | 11   | 1        | 441.91                    | 52.57                                  |
| 9                 | 75         | 12   | 2        | 396.91                    | 51.55                                  |

Table 1 shows the experimental results for ultimate tensile strength for the single fiber of cantala and the corresponding S/N ratio using Eq. (4). The mean S/N ratio for each level of the bleaching parameter is summarized and called the mean S/N ratio for ultimate tensile strength.
Table 2. Response mean S/N ratio for ultimate tensile strength parameter and significant interaction

| Symbol | Bleaching parameter | Mean S/N ratio | Level 1 | Level 2 | Level 3 | Max-min | Rank  |
|--------|---------------------|----------------|---------|---------|---------|---------|-------|
| A  | Temperature °C      |                | 54.66   | 55.42   | 53.80   | 1.62    | 2     |
| B  | PH                  |                | 56.98   | 53.51   | 53.38   | 3.60    | 1     |
| C  | % H₂O₂              |                | 54.36   | 54.55   | 54.97   | 0.61    | 3     |

Table 2 shows the greatest value of max-min S/N ratio is 3.60 indicate that parameter PH most influence the fiber tensile strength. Temperature is a second parameter (1.62) and third is a concentration of hydrogen peroxide (0.61). PH, temperature and concentration above the optimum parameter causing increased rate of decomposition significantly and the unutilized HOO⁻ anion so rapid becomes unstable reaction so may damage the fibers.

Figure 6 shows result mean of single-to-noise (S/N) graph for the tensile strength of cantala fiber with Taguchi method. The optimum parameters of bleaching process are temperature 60°C, PH 10 and 3% H₂O₂ (A2-B1-C3). Those process caused the rate of evolution of perhydroxyl ion is equal to the rate of consumption. The ultimate tensile strength single fiber of cantala increase from 520 MPa (raw material) into 800 MPa after bleaching with parameter A2-B1-C3.

3.2 Fiber characterization

Fiber characterization X-ray diffraction shows that choosing the optimum parameter of bleach treatment that are temperature 60°C, PH 10 and 3% H₂O₂ (v/v) can increase tensile strength and crystallinity index (Ic) of cantala fibers. Increase value of Ic from 64.5% to 78% indicate that non cellulose component (lignin, hemicellulose and extractive substance) in fibers decrease significantly. This research similar to the Hay et al. (2012) that chemical modification of date palm fibers (DPF) can effectively increase the ultimate tensile stress and elongation [12, 13].
FTIR spectra can be seen in Figure 8, natural fibers waveform especially show four main chemical bond there are 1735 (C=O) bond of carbonyl C=O stretching of carboxylic acid or ester (lignin and hemicellulose), 1319 CH2 Wagging (lignin), 1250 stretching of acetyl C-O (lignin), 1627 OH (water content). Change waveform numbers from sharp valley to wide peaks indicate loss of chemical bond C=O, CH2 and C-O indicate the reduction of non cellulosic component [15]. The change of waveform at 1627 show that decrease water content in fibers causes the hydrophilic into hydrophobic properties. Hydrophilic nature is a major problem for all cellulose fibers if used reinforcement in plastics [13].

Figure 9 show SEM images were taken with 500 x optical zoom to produce topography of cantala fiber untreated, the picture that the bundle of fiber does not appear because still tied and coated by non cellulosic component in fiber. These fibers after bleaching treatment can be seen in Figure 10 shows the surface of fibers more clearly, roughly and diameter of fibers became tiny.

4. Conclusion
1. In this study, Taguchi parameter design was successfully used to optimize a parameter of the bleaching process. It was found that bleaching process with temperature 60°C, PH 10 and 3% H2O2 (A2-B1-C3) lead to higher tensile strength.
2. Optimum bleaching with parameter A2-B1-C3 shows increase mechanical strength and physical properties indicated by increasing crystallinity index (Ic) and decreasing percentage of water content in cantala fibers.
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