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Original Article

Effect of mercerization on properties of mendong *(Fimbristylis globulosa)* fiber

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Abstract

The study is aiming at investigating the effects of alkali treatments on tensile properties, crystallinity, and functional group of mendong fiber. The experiment was conducted by soaking fibers in alkali solution containing NaOH concentrations of 2.5%, 5.0%, 7.5%, and 10% for 2 hours at ambient temperature. The tensile properties and structure of fiber were evaluated by single fiber tensile test and XRD method, respectively. The functional group and morphology of the fiber were observed by FTIR and SEM analysis. The results show that the crystalline structure properties of mendong fiber were changed during treatment by using alkali. The results indicate that the degree of crystalline and crystalline index of mendong fiber was increased by a mercerization process using the alkali solution having NaOH concentration of 7.5%, while the optimum tensile strength of 497 MPa was achieved at NaOH concentration of 5%.

Keywords: mendong fiber, mercerization, tensile strength, XRD, FTIR

1. Introduction

At present, the encouragement to replace the synthetic fiber with the natural fibers is increasing due to the environmental concern. The synthetic fibers are commonly used as an engineering material such as reinforcement in composite because of their excellent mechanical properties. Due to its non-biodegradable property, therefore, many efforts are made to find the alternative fiber as a replacement. Natural fiber is abundant, and the utilization as composites reinforcement has many advantages compared with synthetic fiber reinforced composites, which are biodegradable, renewable, low price, low density, easily separated and having non-abrasive character (Mu, Wei, & Feng, 2009; Jawaid & Khalil, 2011). Consequently, it has attracted the huge interest of scientists to explore new natural fibers for replacing synthetic fiber applications as composite reinforcement in different industrial sectors.

Agricultural crops by products are a promising source of natural fiber due to their abundance, renewability and inexpensive price (Reddy & Yang, 2009). Several alternatives of fiber sources from agricultural by-products including rice straw (Reddy & Yang, 2006), wheat straw, switch grass (Reddy & Yang, 2007a), pineapple leaves (Mishra, Mohanty, Drzal, Misra, & Hinrichsen, 2004), Indian grass (Liu,

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Mohanty, Askeland, Drzal, & Misra, 2004), sugar cane rind (Han & Wu, 2004), napier grass (Kommula *et al.*, 2016), and banana tree (Kiruthika & Veluraja, 2009) have been used to produce cellulose fibers. Some of these fibers such as wheat stalks, corn stalk, rice husk, sugar cane bagasse, pineapple leaves, and fruit peels have been widely used as reinforcement on polymer matrix composites (Reddy & Yang, 2007b).

Mendong (*Fimbristylis globulosa*) is a kind of grass, a family of Cyperaceae, including plants growing in wetlands, muddy areas, having enough water, and categorized as a cultivated crop. It can grow to a length of 100 – 150 cm and the production of mendong in Java island, Indonesia is estimated to be 14,000 tons per year (Suryanto, Irawan, Marsyahyo, & Soenoko, 2014a). Mendong fibers are traditionally used as rope material and fabrication of value-added products such as furniture components, handicrafts, and mats. This fiber appears to be a potential candidate for synthetic fiber replacement. It has enhanced the function from the traditional material to engineering material.

The main drawback for applying natural fiber in polymer composites is the incompatibility between the hydrophilic nature of natural fiber and hydrophobic properties of the polymer matrix, causing poor interfacial adhesion between fiber and matrix. The natural fibers are usually treated to improve its interfacial adhesion with the matrix. Common surface treatments for natural fibers to improve the fiber-matrix bonding are alkali and silane treatment, grafting methods, acetylation, and uses of chemical agents. Among these methods, alkali treatment may be considered to be the most economical technique (Ramadevi, Sampathkumar, Srinivasa, & Bennehalli, 2012). Many authors have reported that treatment using alkali has a positive effect on the mechanical strength and interfacial adhesion of various types of natural fibers such as flax (Karsli & Aytac, 2015), betel nut (Lazim, Salit, Zainudin, Mustapha, & Jawaid, 2014), hemp (Liu, You, Jin, & Yu, 2013), coir (Nam, Ogihara, Tung, & Kobayashi, 2011) and jute (Mwaikambo, 2009).

Mercerization is a process in which fiber/textile (typically cotton) are treated with an alkali (NaOH) solution to improve fiber properties such as strength, luster, shrinkage resistance, and dye affinity (Zuber, Zia, Bhatti, Ali, Arshad, & Saif, 2012). The mercerization process involves the use of an alkali solution at different concentrations, temperature, and time to treat natural fiber (Mbada, Aponbiede, Ause, & Alabi, 2016). It can hydrolyze and remove impurities inside the fiber such as lignin, wax, pectin, and others which are considered to be a hindrance for its adhesion to the matrix during composite fabrication (Ramadevi *et al.*, 2012). This process results in the change in structure and pores (Sun, Zhou, & Xing, 2016), microstructure, and morphology. The conformation of the cellulose chains and strength (Alam & Shamim, 2016) also occurs during mercerization, therefore, improve the interfacial shear stress of composite (Karsli & Aytac, 2015). The present study aims to investigate the effect of mercerization process toward the physical, chemical, morphological, and tensile properties of the mendong fiber.

2. Material and Methods

2.1 Material

The research material is mendong straw and its fibers. Samples were obtained from the cultivation land in Wajak district, Malang, East Java, Indonesia. The samples were selected in the harvest period of 5-6 months, and the length straw was 1-1.2 m. The mendong fibers contain 72.14% cellulose, 20.2% hemicellulose, 3.44% lignin, 4.2% extractive, and moisture of 4.2%–5.2% (Suryanto, Marsyahyo, Irawan, Soenoko, & Aminnudin, 2015).

2.2 Extraction of fiber

Mendong fibers were extracted mechanically. The wet mendong straw was cut off 60 cm long from the base, and the next piece to the top was removed. Mendong straw was pounded repeatedly and finally cleaned using water. Then, fibers were immersed in water for a week. Fibers were retrieved, cleaned and allowed to be dried up.

2.3 Mercerization process

The following procedure conducted the mercerization process: 2 gr mendong fibers were immersed in 100 ml NaOH solution (Merck) with various concentrations of 2.5%, 5%, 7.5%, and 10% for 2 hours at room temperature. Fibers were drained and rinsed by using the quads for five times, afterward, were dried and kept in plastic wrap in a dry box with 40% humidity (Suryanto, Marsyahyo, Irawan, & Soenoko, 2014b).

2.4 Morphological analysis

The morphological surface structure of the cellulose was observed by the Scanning Electron Microscope (SEM) (FEI, Inspect-S50 type) at 10.00 kV. Before SEM observation process, all specimens were coated with a layer of gold with a thickness of 10 nm (sputter coater, SC7-620 Emitech).

2.5 X-ray diffraction analysis

X-ray diffraction (XRD) analysis was performed under ambient condition by using PanAnalitical type X-Pert Pro Diffractometer system with CuKα radiation (λ = 1.54 Å). The finely powdered sample was used for XRD analysis to identify the crystal planes. The diffracted intensity of CuKα radiation was recorded between 2° and 80° (2θ angle range) at 40 kV and 30 mA. Crystalline Index (*CI*) and degree of crystalline (*%Cr*) were evaluated by Segal methods by using equation 1 and 2, respectively (Terinte, Ibbett, & Schuster, 2011).

$$CI = \frac{I_{(0\,0\,2)} - I_{am}}{I_{(0\,0\,2)}} x\ 100\% \quad (1)$$

$$\%Cr = \frac{I_{(0\,0\,2)}}{I_{(am)} + I_{(0\,0\,2)}}\ x\ 100\% \quad (2)$$

where $I_{(0\ 0\ 2)}$ is the largest intensity of the peak corresponding to the plane in the sample with the Miller indices (0 0 2) at 2θ angle in range 22-23º representing crystalline material and I_{am} is the intensity of diffraction of the non-crystalline material taken at an 2θ angle of about 18º in the valley among the peaks having minimum intensity, represents amorphous material in the cellulose fiber.

2.6 FTIR Analysis

FTIR spectra were recorded in Shimadzu IR Prestige-21, FTIR Spectrometer. The samples were prepared by using the KBr pellet technique. The background spectrum of KBr pellet was subtracted from the sample spectra. About 0.1 mg of the samples were ground into powder with high purity infrared-grade KBr powder (1 mg) and pressed into a pellet for measurement. IR spectra were recorded in the spectral range of 4000–400 cm^{-1} with a resolution of 2 cm^{-1} (Kiruthika & Veluraja, 2009).

2.7 Mechanical properties of fiber

Mechanical properties such as tensile strength and elastic modulus were determined by a tensile test performed ten times by using a single fiber test method in a tensile test machine with maximum load capacity of 5N. The fiber strains were measured by digital screw micrometer equipping in the tensile test machine with a crosshead speed of 3.5 mm/min at ambient temperature. A single fiber was prepared for the test within a card mounting, and the fiber cross-sectional area was determined through an optical microscope observation (Suryanto *et al.*, 2014b)

3. Results and Discussion

3.1 Morphology of fiber surface

Morphologies of mendong fiber immersed in alkali solution with a concentration of 2.5%, 5%, 7.5%, and 10% for 2 hours are shown in Figure 1. The result indicates that the amount of the noncellulosic material substance on the surface disappeared so the surface fiber cleaner (Figure1A). In addition, the surface was cleaner and noncellulosic material peeled from surface after the fibers were soaked in alkali with the concentration of 5% (Figure 1B). By increasing of alkali concentration, the topography of fiber surface is observed to be rougher (Figure 1C) since the outside layer was peeled off and many compounds were dissolved in the alkali results fiber bundle damage. Under 2 hours of 10% alkali soaking, the fiber is noticed to be broken (Figure 1D) causing the broken fibril-chain. Alkali penetrated the fibers and damaged the fiber structures shown by the decrease both of degree of crystalline and the crystal index of fiber (Figure 3).

3.2 Fiber structure

The cellulose and non-cellulose contents determine the structure, crystallinity and properties fiber (Reddy & Yang, 2006). The various models of crystal structure in cellulose structure are caused by conformation both of the inter- and intra-molecular hydrogen bonds through the hydroxyl group (Sheltami, Abdullah, Ahmad, Dufresne, & Kargarzadeh, 2012). The XRD patterns of mendong fiber in some stages of treatment with a various concentration of NaOH are shown in Figure 2.

XRD patterns mostly contain three peaks located at around 16°, 22°, and 35°. The peaks are related to the crystal planes of (0 1 1), (0 0 2), and (4 0 0) (El Oudiani, Chaabouni, Msahli, & Sakli, 2011). The peak is slightly observed to be wider approximately at 16°. It was the combination of the two peaks in the cellulose I_β (16.7° and 14.9°) or in the cellulose $I\alpha$ (16.8° and 14.3°), or both of them (Cheng, Varanasi, Li, Liu, Melnichenko, Simmons, & Singh, 2011). The main intensity peak is around 22° indicating the characteristic of the original cellulose (cellulose I) with the maximum peak around 22-23° (Le Troedec, Sedan, Peyratout, Bonnet, & Agnes, 2008). The amorphous compound is indicated by the lowest diffraction intensity at about 18°. The relatively low third peak around 34.5° is correlated with a quarter of cellobiose unit length and appeared along the fiber direction. This case is sensitive to the chain conformity being a fibril (Cheng *et al.*, 2011).

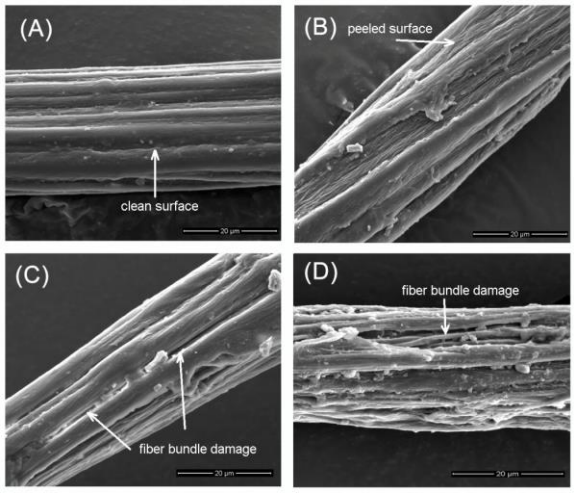

Figure 1. SEM images of mendong fiber morphology after soaking in alkali with the concentrations of 2.5% (a), 5% (b), 7.5% (c), and 10% (d).

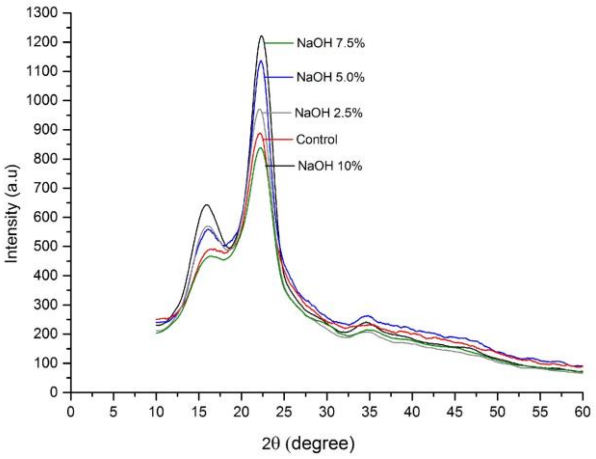

Figure 2. XRD patterns of mendong fiber soaked in alkali solution in various concentrations.

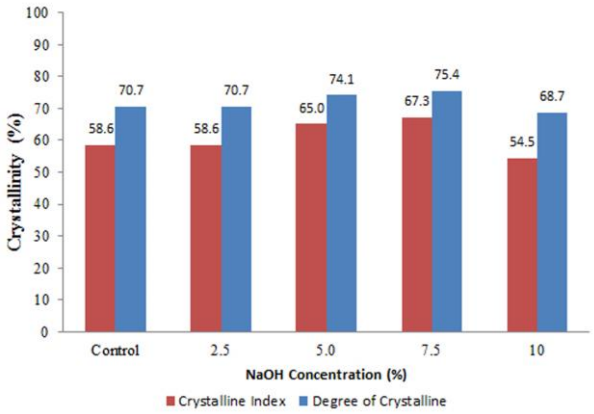

Figure 3. The influence of alkali concentration of the mercerization process on mendong fiber crystallinity.

The change of crystal parameter in mendong fiber as the result of a mercerization process with NaOH is shown in Figure 3. The degree of crystalline and crystalline index of untreated mendong fiber and mendong fiber soaked inside the solution containing 2.5%, 5.0%, 7.5%, and 10% of NaOH are 70.7%, 70.7%, 74.1%, 75.4%, 68.7% and 58.6%, 58.6%, 65.1%, 65.7%, 54.5%, respectively. The XRD peak on 22.6° is observed to be sharper by alkali treatment. The result reveals that the crystallinity was increased by an alkali treatment using higher concentration of NaOH up to 7.5%. With further NaOH concentration, the crystallinity is noticed to be reduced. The increase of crystallinity was caused by the loss of amorphous components like hemicellulose and lignin. However, these amorphous components could not be fully removed because the crystallinity of pure cellulose mendong is 86% (Suryanto, Fikri, Permanasari, Yanuhar, & Sukardi, 2018).

At low concentration of alkali (2.5%), NaOH amount was not sufficient for penetrating the crystal region of cellulose. Therefore, NaOH could only dissolve a small amount of amorphous compound. On the higher alkali concentration (5% and 7.5%), the NaOH solution was able to not only penetrate the crystals but also destruct both of inter- and intra-molecular hydrogen bond among the cellulose molecules and around crystal area. Amorphous fraction, such as hemicellulose, is a non-crystalline material having branch and less molecule weight compared to cellulose. It also has less ability to inhibit the alkali access inside the fiber cell relative to the cellulose (Knill & Kennedy, 2003). Consequently, this amorphous fraction was more easily hydrolyzed and the total number of amorphous compounds were decreased. Thus the crystallinity of the fiber was obviously increased.

The result also shows that the crystallinity degree and crystal index are significantly decreased by a mercerization process with the concentration 10%. However, the crystal size is increased. This case was possibly caused by penetration of alkali through crystal area to form cellulose soda resulting random cellulose cleavage on an alkali-accessible-molecular chain inside the crystal domain. The cellulose cleavage caused interference in the fiber crystal and was subsequently considered as an amorphous since they were not the part of the crystal anymore (Gümüskaya, Usta, & Kirci, 2003; El Oudiani *et al.*, 2011). This interference is proven by the reduction of the crystal size and index. In this level, the mercerization phenomenon conducted by using alkali was able to change not only the smooth morphology and structure of the fiber but also cellulose chain conformation to construct better packing of the cellulose chain. During this process, the alkali and water hydrate penetrated the cellulose fiber and swelled the structure evidenced by the increase in crystal size.

After the mercerization with NaOH 5% for 2 hours, there is no transformation of the mendong fiber crystal structure shown by the absence of the change of the angle of XRD pattern. However, the diffraction intensity is relatively increased after the soaking process in NaOH 5% solution. The increase of diffraction intensity shows that the more cellulose crystal was contained inside the fiber. It also demonstrates the increase of crystallinity from 70.71% to 74.1% due to the removal of amorphous content inside the fiber cellulose, like hemicellulose, lignin, and some other non-cellulose materials. Thus, it can re-arrange the fibril resulting better packing of cellulose chain (Symington, Opukuro, William, & Petrich, 2008).

3.3 The change of functional group of fiber surface

The chemical characteristic, the change of functional group, of mendong fiber caused by mercerization treatment was analyzed by using FTIR spectroscopy, and the result is shown in Figure 4. The mercerization process of the fiber caused the fiber cellulose to swell. It also removed the hemicellulose, lignin, and other polluters from the fiber surface. The infrared transmission pattern on untreated mendong fiber is different compared with the transmission pattern of the fiber soaked in alkali.

As shown in Figure 4, the wave number at a range between 3000 and 4000 cm^{-1} refers to alcohol (OH) stretching of the cellulose, hemicellulose, and lignin. While range between 2850 and 3000 cm^{-1} refers to C-H bond often observed in the group of an alkane. The peak intensity of C=O group inside ketone and carbonyl cluster are indicated in the range between of 1765 and 1715 cm^{-1} referring the hemicellulose compound. The peak of 1730 cm^{-1} is not feasible after mendong was soaked in the alkali. This phenomenon proves that hemicellulose compound was removed from the fiber after soaking in the alkali solution.

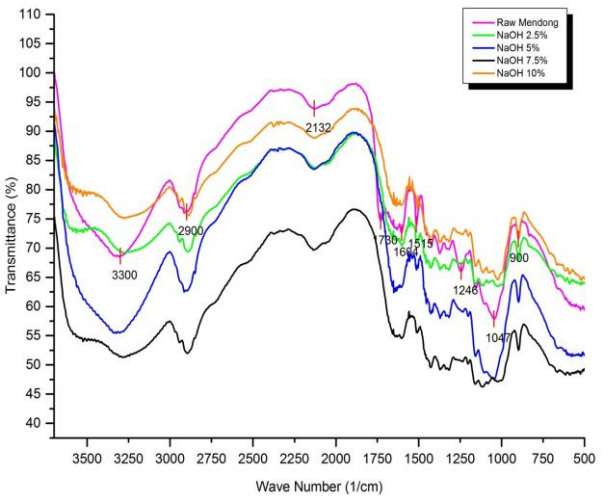

Figure 4. The infrared transmission patterns of functional group of mendong fiber by mercerization process.

The peaks in a range between 1450-1650 cm^{-1} refer to alcohol group and lignin aromatic structure of the fiber (C=C bond). The peaks at 1607 cm^{-1} are related to the absorption of molecular vibration of water molecules. The peaks at 1514 cm^{-1} show the existence of the lignin aromatic structure where the intensity was reduced after the mercerization process. The peaks in the range between 1300 and 1500 cm^{-1} characterize not only C-H bending but also O-H and C-O stretching of hemicellulose, cellulose, and alcohol group. The peaks at 1244 cm^{-1} relate to C–O stretching of lignin acetyl and its intensity is decreased after alkali treatment. This case indicates that the content of lignin in the mendong fiber was reduced after the mercerization process. The peaks in a range between 900 between 110 cm^{-1} refer to C-O group of the compound and C-C stretching of alkene group. The peaks in a range between 700 and 900 cm^{-1} relate to the existence of C-H bond of the aromatic group inside the lignin compound. Finally, the peaks at a range between 890-900 cm^{-1} are the characteristic of the β-glycoside link inside the cellulose and hemicellulose of the fiber (Moran, Alvarez, Cyras, & Vazquez, 2008; Marsyahyo, Soekrisno, Rohardjo, & Jamasri, 2008; Mahato, Prasad, & Mathur, 2009).

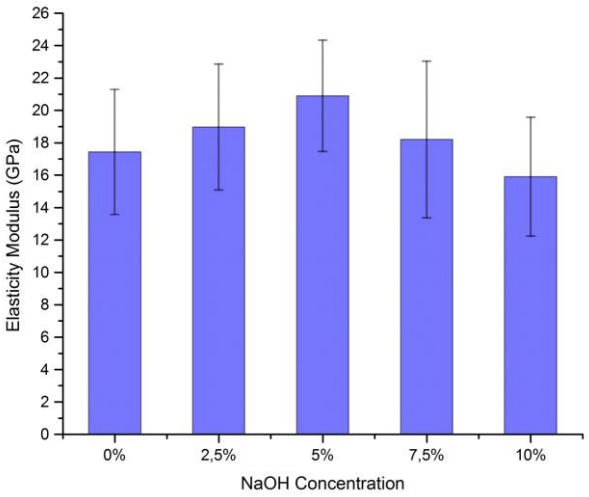

Figure 6. Elastic modulus of mendong fiber after mercerization process with different concentration of NaOH for 2 hours.

3.4 Tensile properties of fiber

Tensile properties of mendong fiber after mercerization process with different concentration of NaOH analyzed by the tensile test are shown in Figure 5 and 6.

The mercerization process for 2 hours led the change in diameter, tensile strength, and elastic modulus of the fiber. At NaOH concentration of 5%, the mendong fiber surface became cleaner, so, the average diameter was reduced, and the mechanical properties consisting of elastic modulus and tensile strength were increased. The mercerization process with NaOH concentrations of 2.5%, 5.0%, 7.5% and 10% shows both of tensile strength and its improvement of 469, 497, 457, 404 MPa and 3.8%, 10%, 1%, -10.6%, respectively. The similar phenomenon is also observed on elastic modulus having 19, 20.9, 18.2, and 15.9 GPa. The increase of fiber strength up to 5% of soaking was caused by some hemicelluloses dissolved by alkali conducting self-reconstruction to form better and unified fiber (Symington *et al.*, 2008). Thus, the crystallinity of the fiber was also increased. The increase of fiber crystallinity implied that a number of the non-crystalline compounds having a low elastic modulus, such as hemicellulose (8 GPa) and lignin (3 – 6.7 GPa), was reduced. The content of remaining cellulose crystal having high modulus (140 GPa) was in a higher percentage. Therefore, the tensile strength was gradually increased (Cousins, 1976; Gibson, 2012; El Oudiani *et al.*, 2011). The mercerization process with the alkali concentration beyond 5% affects in a reduction both of fiber tensile strength and elastic modulus. This phenomenon has occurred since the hemicellulose as the matrix acting as binder among fibers were over-dissolved. Therefore, the binder amount of microfibrils inside the fiber was consequently reduced. This over-reduction of hemicellulose also left the cavities inside the fiber separating microfibrils each other causing an imperfect cross-sectional area of the fiber and thus decreasing the strength when the fiber sustain the tensile load.

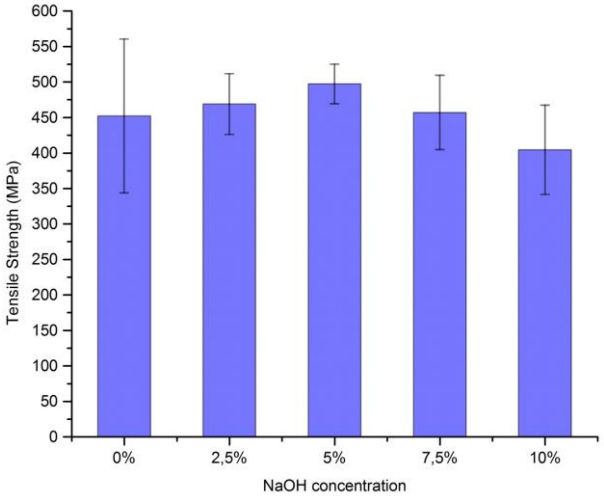

Figure 5. Tensile strength of fiber after mercerization process with different concentration of NaOH for 2 hours.

4. Conclusions

According to the physical, chemical, and mechanical analysis of mendong fiber treated by mercerization process using alkali solution (NaOH) with different concentration for 2 hours can be concluded that the mercerization process can improve the chemical, physical, and mechanical properties of mendong fiber where the optimum chemical properties were reached at alkali concentration of 7.5%, whereas, the optimum mechanical properties consisting of both tensile strength and elastic modulus were obviously achieved at alkali concentration of 5%.

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