

Effect of Alkali-Washing at Different Concentration on the Chemical Compositions of the Steam Treated Bamboo Strands¹

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ABSTRACT

The objective of this study was to investigate the effect of alkali-washing with different sodium hydroxide concentrations on the chemical compositions of steam-treated Betung bamboo strand. Strands were subjected to steam treatment at 126 °C for 1 h under 0.14 MPa pressure and followed by washing with 1-5% sodium hydroxide solution for 30 sec. The alteration of structural and non-structural chemical components content of bamboo strands was evaluated. Steam and washing treatments with various concentrations of sodium hydroxide solution considerably reduced the extractive content of bamboo strands, and the cell wall chemical components of the strand in the small degree. FTIR analysis showed noticeable changes in peaks related to hemicellulose and lignin. The relative crystallinity increased significantly after steam and washing treatment with sodium hydroxide up to 3% concentration. SEM Images showed smooth and clean strands surface after washing with 3% sodium hydroxide.

Keywords: betung bamboo strand, chemical components, extractives, sodium hydroxide concentration, steam modification

1. INTRODUCTION

Bamboo is a potential alternative raw material for

wood resources due to its rapid growth, abundant amount, growing widespread, and high tensile strength (Liese, 1987). Generally, bamboo has a small diameter

¹ Date Received June 10, 2020, Date Accepted December 8, 2020

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with tubular shape and thin culm, thus the utilizations of solid bamboo are inflexible and more appropriate to be raw material for composites. However, bamboo has a high content of starch and others extractives which may responsible for low dimensional stability and bonding property of the bamboo composite (Febrianto *et al.*, 2015).

Among the bamboo species, Betung bamboo has a high potential to be used for board manufacturing and light-construction due to its big culm size. It can grow as high as 30 m with diameters of about 14 cm at the bottom part (Park *et al.*, 2018) and is distributed in tropical regions such as Indonesia, Malaysia, Thailand, Vietnam, and so forth (Dransfield and Widjaya, 1995). Up to date, there are many our studies on the composite properties using the bamboo materials (Febrianto *et al.*, 2015; Maulana *et al.*, 2017; Fatrawana *et al.*, 2019).

In order to improve the properties of bamboo composite, pre-treatment of bamboo strands can be applied to reduce the undesirable chemical component in the bamboo strands. Steam treatment at high temperatures can degrade low molecular weight substances and convert the free sugar groups into furan resins (Rowell, 2004). In the relatively low temperature at around 120 °C, steam treatment is known to reduce the extractive content of bamboo up to 3% and significantly increase the physical and mechanical properties of OSB (Maulana *et al.*, 2017). However, during steam treatment, the extractive substances can be deposited and accumulated on the surface of strands. This circumstance can affect the bonding process by inhibiting the penetration of adhesive into the cell wall (Pizzi, 1983).

Sodium hydroxide solution may washing the chemical components on the surface of bamboo strands, which expects the enhancement of the adhesion properties of bamboo composites. Therefore, this study tried to investigate the effect of washing treatment using sodium hydroxide solution after steam treatment on the chemical compositions of bamboo strands.

2. MATERIALS and METHODS

2.1. Materials

The Betung bamboo culms were harvested from Sukabumi District, West Java, Indonesia. Bamboo strands were prepared from the internode of bamboo culm without the outer layer part and node using a sharp knife and scissor with target size ($70 \times 25 \times 0.7$) mm³. All chemicals used in this experiment were analytical grade.

2.2. Methods

2.2.1. Steam and Washing Treatment

Steam and washing treatment were carried out according to Maulana (2019). Steam treatment was conducted in an autoclave at 126 °C for 1 hour under 0.14 MPa pressure. Washing treatment after steam treatment was carried out with sodium hydroxide solution at the concentrations of 1%, 2%, 3%, 4%, and 5% for 30 sec. Steam-treated sample without washing (0%) was also prepared as a comparison. The treated strands were air-dried and then oven-dried for 36 hours to get moisture content below 5%.

2.2.2. Chemical components analyses

Holocellulose and α -cellulose content were determined by the Browning methods (Browning, 1967) and lignin was by the method of Dence (1992). Extractives soluble in cold and hot water were determined according to TAPPI T-207 cm-99 (TAPPI, 1999). Extractives soluble in Ethanol-benzene determination method referred to ASTM D-1107-96 (ASTM, 2013). Solubility in 1% NaOH solution was determined according to TAPPI T-212 om-02 (TAPPI, 2002). The pH value was determined according to SNI 06-6989.11-2004 (BSN, 2004).

The bamboo powder was used for chemical composition analyses using the ATR method in the range of

400-4000 cm^{-1} with an FT-IR spectrometer (Perkin Elmer Inc., USA) installed in the central laboratory of Kangwon National University, Korea.

2.2.3. X-ray diffraction analysis and scanning electron microscopy observation

An X-ray diffractometer (DMAX 2100V, Rigaku, Japan, 40 kV, 40 mA) equipped with a Cu target ($\lambda = 0.1542 \text{ nm}$) was used for measuring crystalline properties. The relative crystallinity and crystal width of betung bamboo strands were analyzed using Seagal's method (Seagal *et al.*, 1959) and Scherrer's equation (Burton *et al.*, 2009). To investigate the surface morphology of the Betung bamboo strands, the strands surface was gold-coated with a Gressington sputter coater (ULVAC G-50DA) and observed with a scanning electron microscope (JSM-5510, JEOL, Japan, 15 kV).

2.3. Data analysis

The experiment was a completely randomized design with one factor: sodium hydroxide concentration of six levels. Each treatment was carried out in four replications. The data obtained were statistically analyzed using analysis of variance (ANOVA). Furthermore, Duncan's multiple range test was carried out if there was a significant influence.

3. RESULTS and DISCUSSION

3.1. Chemical properties

Holocellulose contents of the steam-treated bamboo strands decreased with increasing alkali concentration in washing treatments as shown in Fig. 1. The significant decrease of holocellulose mainly occurred in the samples washed with 4% NaOH solution. The statistical analyses showed that the washing treatment had a significant effects ($P < 0.01$) to the contents of holocellulose, α -cellulose, and hemicellulose.

Holocellulose content might be decreased mainly due to the degradation of hemicellulose. Hemicellulose is an amorphous polymer with a low degree of polymerization, and more susceptible to the degradation by alkali (Fengel and Wegener, 1984). Alpha-cellulose was slightly changed in alkaline washing treatments at concentrations of 2%, and remain stable at higher concentrations of up to 5% NaOH. The proportion of α -cellulose in holocellulose increased slightly with decreasing of hemicellulose content. In the previous

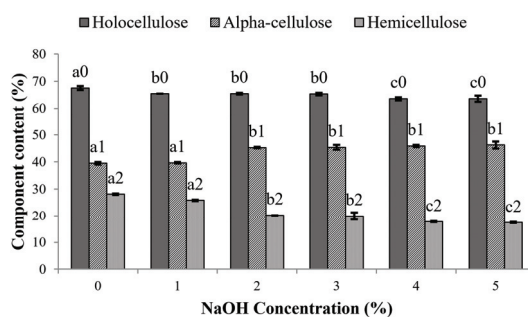


Fig. 1. Holocellulose components in alkali-washed Betung bamboo strands after steam treatment. Different letters at the same number show significant differences between treatments according to Duncan multiple range tests at 5% confidence interval. Error bars show standard deviation.

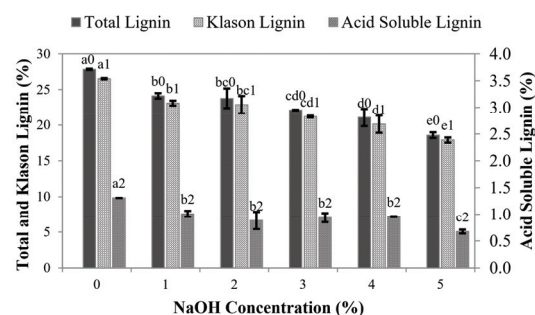


Fig. 2. Lignin contents in the alkali-washed Betung bamboo strands after steam treatment. Different letters at the same number show significant differences between treatments according to Duncan multiple range tests at 5% confidence interval. Error bars show standard deviation.

study, it was reported that hemicellulose content decreased by steam and washing treatment, thus increasing the proportion of alpha-cellulose content (Maulana *et al.*, 2018). The NaOH solution is able to swell the structure of cellulose polymer even at room temperature conditions (Zhang *et al.*, 2013). Changes in the chemical properties of holocellulose may alter the mechanical properties, hygroscopic properties, and the water absorption capacity of the materials.

Lignin content of the bamboo strands decreased by alkali washing treatment as shown in Fig. 2. The results of the analysis of variance ($\alpha = 0.05$) showed that the alkali treatment had a highly significant effect ($P < 0.01$) on the lignin content of Betung bamboo strand. The higher alkali concentration, the lower lignin content of the strands. Jung *et al.* (2017) also found that alkali treatment of steamed barley husk reduced lignin content. Alkali treatment at higher concentration produces lower lignin content (Yang *et al.*, 2019).

In this research, acid soluble lignin of the bamboo strands also decreased by alkali washing treatment. The changes of acid-soluble lignin content indicated that the composition of lignin was changed to some extent as a result of partial delignification. Acid-soluble lignin produced during Klason lignin procedure was affected by syringyl unit (Matsushita *et al.*, 2004). In alkali condition syringyl unit is more easily de-

graded than guaiacyl units (Nawawi *et al.*, 2017), which is due to syringyl is more reactive than guaiacyl (Tsutsumi *et al.*, 1995). Thus the decrease of acid soluble lignin could be caused by syringyl unit degradation. Lignin plays a role as a supporting component for mechanical properties of plant cells. The changes in lignin content and its chemical properties will affect the mechanical properties of the lignocellulose material.

Table 1 shows the soluble contents in the bamboo strands in cold and hot water, 1% alkali and ethanol-benzene solutions. The highest content of extractives of bamboo strand was shown in 1% NaOH solution. It might be due to high starch content in bamboo (Fatrawana *et al.*, 2019). On the solubility in 1% NaOH, the extractives in the alkali-washed bamboo strands was lower than the unwashed strands except washing at 5% NaOH solution, suggesting that the degradation of chemical components on the surfaces of the bamboo strands occurred even during short alkali-washing treatment. The bonding quality of bamboo composites can be improved owing to the decrease of extractive substances of alkali-washed bamboo strand. Pizzi (1983) explained that the extractive substances may inhibit adhesive penetration into the wood substrates during bonding process.

The pH value of unwashed Betung bamboo strands was 5.79 and then increased by alkali-washing treat-

Table 1. Solubility of extractive substances in the steamed and alkali-washed bamboo strands

Extractives	NaOH concentration (%)					
	0	1	2	3	4	5
Soluble in:						
Cold water (%)	15.12±0.19 ^a	14.47±0.75 ^b	8.77±0.01 ^c	7.66±0.01 ^d	7.93±0.41 ^d	8.14±0.05 ^d
Hot water (%)	17.54±0.43 ^{ab}	16.37±0.67 ^{ab}	15.75±0.84 ^{ab}	14.14±4.62 ^a	20.08±1.51 ^{bc}	22.43±5.32 ^{bc}
1% NaOH (%)	33.36±0.06 ^a	31.93±0.55 ^a	29.97±3.70 ^a	30.16±5.99 ^a	32.51±7.80 ^a	34.15±2.89 ^a
Ethanol-benzene (%)	17.50±0.81 ^a	15.39±0.32 ^{bc}	14.75±0.84 ^c	13.97±0.03 ^d	15.51±0.19 ^{bc}	15.58±0.00 ^b

Different letters on the same row show significant differences between treatments according to Duncan multiple range test at 5% confidence interval.

ment as shown in Fig. 3. Acidity could be influenced by polyphenols and organic acids present in woody materials (Krilov and Lasander, 1988). Organic acids in wood were found as acetic acid, formic acid, polyphenols and as acetyl groups in hemicellulose, which are easily hydrolyzed into free acids (Nawawi *et al.*, 2012). Since alkali washing treatment decrease extractives content and degrade hemicellulose, it is expected that organic acids were reduced and followed by increasing the pH value. In addition, the pH values that tend to be alkaline might be caused by the residual of NaOH after washing treatment. The higher concentration of NaOH residues on the strand surface could result in higher pH value. The results of the analysis of variance ($\alpha = 0.05$) showed that the treatment had a very significant effect ($P < 0.01$) on the pH value. In the bonding process, the higher pH value is an appropriate condition for the curing process us-

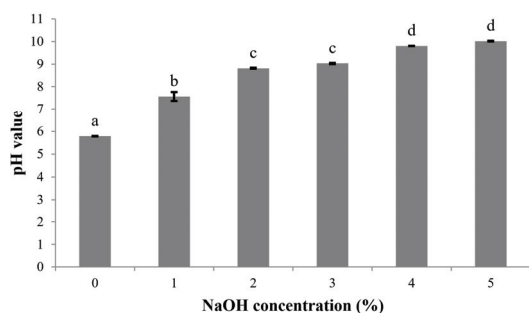


Fig. 3. pH value in the alkali-washed bamboo strands after steam treatment.

ing phenol formaldehyde (PF), which is categorized as an alkaline-curing resin. It is known that the curing process of PF resin is effective in the pH value above 9 (Pizzi and Stephanou, 1993).

Different letters show significant differences between treatments according to Duncan multiple range test at 5% confidence interval. Error bars show standard deviation.

3.2. FTIR analysis

Fig. 4 shows the FTIR spectra of the alkali-washed bamboo strands after steam treatment. The corresponding peak assignments are summarized in Table 2. The changes in the spectra after alkali washing at different concentrations were found at the peak at 1728 cm^{-1} .

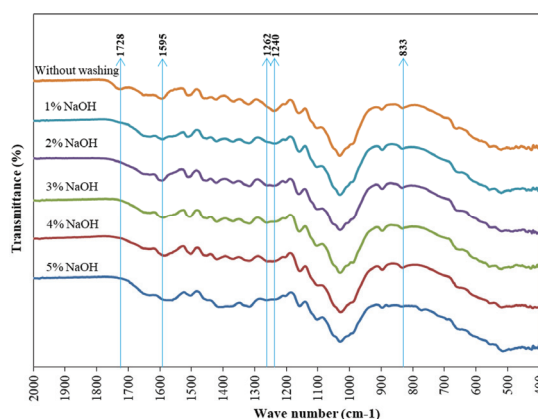


Fig. 4. FTIR spectra of the alkali-washed bamboo strands after steam treatment.

Table 2. Peak assignment of FTIR spectra of alkali-washed bamboo strands after steam treatment.

Component	Wavenumber (cm^{-1})	Peak Assignment
Carbohydrate	1728	Unconjugated C=O in hemicellulose (xylans) (Cheng <i>et al.</i> , 2013; Tomak <i>et al.</i> , 2013; Lee <i>et al.</i> , 2019)
Lignin	1595	Aromatic ring and C-H bond (Cheng <i>et al.</i> , 2013)
	1262	Guaiacyl ring breathing with C-O stretching (Cheng <i>et al.</i> , 2013)
	1240	C-OH aromatic phenyl (Cheng <i>et al.</i> , 2013)
	833	Lignin C-H (Cheng <i>et al.</i> , 2013; Tomak <i>et al.</i> , 2013)

The absorbance at 1728 cm^{-1} attributed to C=O stretching vibration from hemicellulose (xylan) disappeared by alkali washing. The peaks at 1595 cm^{-1} related to aromatic skeletal vibration in lignin also decreased. The peak at 1240 cm^{-1} in unwashed bamboo strand may be the result of approximately equal intensities overlapping at 1270 cm^{-1} (related to G ring lignin) and 1230 cm^{-1} (C-C, C-O, C=O stretch), which is (Faix 1991; Cheng *et al.*, 2013). The peak at 1249 is the characteristics of Graminae lignin, which consist of p-hydroxyphenyl, guaiacyl, and syringyl unit, presence in the bamboo. However, the peak intensity at 1240 cm^{-1} has decreased after washing treatment and followed by appearance peak at 1262 cm^{-1} corresponding to guaiacyl lignin in the alkali washed samples. These changes may be related to the decrease of acid soluble lignin value due to syringyl unit in the alkali treated samples. As mentioned above, syringyl unit is more reactive and easily degraded by alkali, compared to guaiacyl unit. Another change is showed by a decrease of peak intensity at 833 cm^{-1} after washing with 5% NaOH. This peak is indicative for p-hydroxyphenyl lignin unit (Faix 1991).

3.3. Crystalline characteristics and morphology

The relative crystallinity and crystallite width of the Betung bamboo strands slightly increased by alkali-

Table 3. Crystallinity and crystallite width of the alkali-washed bamboo strands after steam treatment

Alkali washing treatment with different NaOH concentration (%)	Crystallinity (%)	Crystallite width (nm)
Without washing	76 (0.4)	3.65 (0.02)
1	78 (1.2)	3.66 (0.02)
2	79 (2.1)	3.99 (0.02)
3	81 (2.3)	4.12 (0.03)
4	78 (1.1)	3.68 (0.03)
5	78 (1.4)	3.90 (0.01)

li-washing as shown in Table 3. The increase in crystallinity can probably be attributed to the degradation of hemicelluloses (Tang *et al.*, 2019). Andersson *et al.* (2005) reported that the increase in the size of cellulose crystallites was due to the changes in porosity of the cell wall by the degradation of the amorphous part of wood during heat treatment. Kim *et al.* (2018) and Kim and Kim (2019) also reported that heat treatment increased the relative crystallinity of *Paulownia* wood. Higher crystallinity could increase the mechanical properties such as stiffness, MOE and MOR of the OSB with the bamboo strands.

Fig. 5 shows the SEM images of the bamboo strands at various treatments. In unwashed bamboo strands, the degraded products after steam treatment, such as starch granules were scattered throughout the strand surface. Starch granules can usually be found in the bamboo culm, mostly in parenchyma cells (Ito *et al.*, 2015). These products on the strand surface were reduced after alkali-washing treatment, showing clean and smooth surface. The surface morphology of the bamboo strands might be caused by the degradation of lignin and hemicellulose and could affect the bonding property for the composite material.

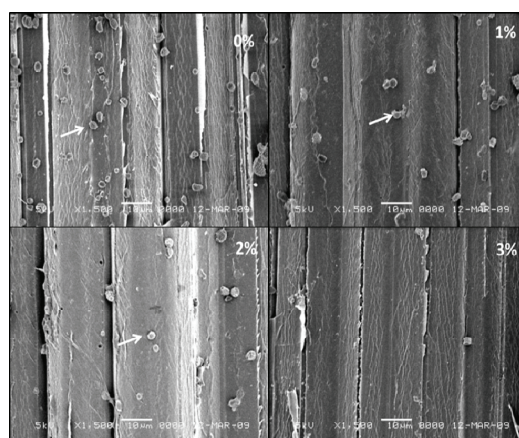


Fig. 5. SEM images of the alkali-washed bamboo strands (1500x).

4. CONCLUSION

Steam followed by washing with NaOH solution affected the change of chemical components of Betung bamboo strand. Washing with higher NaOH concentration affected more severely to the hemicellulose and lignin reduction. The acid soluble lignin content was decreased and syringyl unit degradation was found in FTIR spectra. The extractive content of strand decreased by and the pH value increased by alkali washing treatment. Alkali-washing treatment increased the relative crystallinity and crystalline width of the bamboo strands. The degraded products after steam treatment on the strand surface were reduced by alkali washing treatment, resulting in the smooth and clean surface.

ACKNOWLEDGMENT

This research was also supported by Science and Technology Support Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Science and ICT (MSIT) (NRF-2019K1A3A9 A01000018) and also supported by Basic Science Research Program through NRF funded by the Ministry of Education (No. 2016R1D1A1B01008339 and 2018R1A6A1A03025582).

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