



Full Length Research Article

Changes in Chemical Composition of Betung Bamboo (*Dendrocalamus asper*) after Alkali Immersion Treatment under Various Immersion Times

Rio Ardiansyah Murda¹, Sena Maulana^{1,2*}, Adesna Fatrawana³, Silvia Uthari Nuzaverra Mayang Mangurai⁴, Soleh Muhamad⁵, Wahyu Hidayat⁶, Yazid Bindar⁷

¹ Forestry Engineering Program Study, Institut Teknologi Sumatera (ITERA). South Lampung, 35551, Indonesia

² Research and Innovation Center for Advanced Materials, Institut Teknologi Sumatera (ITERA). South Lampung, 35551, Lampung, Indonesia

³ Forestry Program Study, Faculty of Agriculture, Khairun University. Maluku Utara, 97719, Indonesia

⁴ Faculty of Forestry, University of Tanjungpura. Jl. Prof. Hadari Nawawi, Pontianak, 78124, West Kalimantan, Indonesia

⁵ Furniture Production Engineering, Polytechnic of Furniture and Wood Processing Industry. Jalan Wanamarta Raya, Kawasan Industri Kendal, Central Java, 51371, Java, Indonesia

⁶ Department of Forestry, Faculty of Agriculture, University of Lampung. Jalan Sumantri Brojonegoro, Bandar Lampung, 35145, Lampung, Indonesia

⁷ Chemical Engineering, Institut Teknologi Bandung. Jalan Ganesha 10, 40132, Bandung, Indonesia

* Corresponding Author. E-mail address: sena.maulana@rh.itera.ac.id

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ABSTRACT

This research aimed to analyze the change in chemical composition of the betung bamboo (*Dendrocalamus asper*) strands after alkali immersion treatment under various immersion times. The bamboo culms were converted into strands with the target length, width, and thickness of 70, 25, and 0.5 mm, respectively. Strands were alkali immersion-treated with 1% NaOH solution for 1, 2, and 3 h. Preparation of powder for chemical component analysis refers to the Technical Association of the Pulp and Paper Industry (TAPPI) standard T 264 cm-07 regarding the preparation of wood test samples for chemical analysis. Structural and non-structural bamboo strand chemical components such as holocellulose, α -cellulose, hemicellulose, klason lignin, and extractives were analyzed. The results showed that alkali immersion treatment decreased the hemicellulose content from 21.55% before treatment to 20.30% after 3 h immersion. Thus, it changed the holocellulose and α -cellulose composition. Alkali immersion treatment also changed the extractive substances dissolved in cold water, hot water, 1% NaOH, and ethanol-benzene solution. The decrease in hemicellulose, molecular weight lignin, and extractive substances would be beneficial for bamboo-oriented strand board manufacture to improve strand adhesion, dimensional stability, mechanical properties, and durability against biological agents attack.

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1. Introduction

Indonesia has abundant natural resources. Bamboo is one of Indonesia's natural resources that have very high potential. Generally, bamboo is utilized for various items, including furniture, food, beverages, animal feed, toothpaste, and building structural components (Liese and Köhl 2015). The bamboo working process is also relatively easy because bamboo is easy to split, cut, and shape (Febrianto et al. 2017). In addition, bamboo is a plant that can be harvested at the age of 3-5 years, so the plant is classified as a fast-growing plant (Subekti et al. 2014). Bamboo has

several advantages as a building material. It is strong, light, versatile, environmentally friendly, inexpensive, and has high productivity (Sharma et al. 2014). However, bamboo also has several weaknesses, such as susceptibility to deterioration factors, poor dimensional stability, large variations in physical properties between the base, middle, and ends, limited diameter, and the presence of cavities in the bamboo stem, which makes it thinner than wood (Febrianto et al. 2017; Sharma et al. 2014).

Bamboo can be converted into biocomposite products. Conversion of bamboo into biocomposite products is a promising solution to overcome its weaknesses. One of the biocomposite products that can be produced using bamboo as the raw material is oriented strand board (OSB). Bamboo is very suitable to be used as raw material for OSB because of the nature of bamboo, which has very high tensile strength but low splitting toughness, which will be very beneficial when converting bamboo into strands (Febrianto et al. 2017). Our previous studies showed that bamboo-oriented strand board (BOSB) had better properties (Adrin et al. 2013; Febrianto et al. 2012, 2015, 2017; Maulana et al. 2021b) than wood-based OSB (Akrami et al. 2014; Bufalino et al. 2015). BOSB with good properties is produced with relatively expensive methylene diphenyl diisocyanate (MDI) adhesive. A cheaper adhesive alternative to replace MDI is phenol-formaldehyde (PF) adhesive. However, BOSB with PF required a higher adhesive content than BOSB made with MDI (Adrin et al. 2013). Therefore, it is necessary to conduct pretreatment on bamboo strands to increase the efficiency of adhesive levels and overcome the other problems in BOSB manufacturing that use PF. The issues are the high content of extractive substances in bamboo, which prevents adhesive penetration (Pizzi and Kumar 2019), and low dimensional stability due to amorphous components such as hemicellulose (Fatrawana et al. 2019). Therefore, several studies have found a solution by steam modification treatment at 126°C under 0.14 MPa pressure (Adrin et al. 2013; Febrianto et al. 2015; Maulana et al. 2017). Steam modification treatment on the bamboo strand can remove low molecular weight components such as hemicellulose and extractives so that BOSB has better properties such as a better gluing process, better dimensional stability, and better resistance to deterioration factors (Fatrawana et al. 2019; Maulana et al. 2017, 2021a; Maulana 2018). However, the steam modification treatment requires a lot of energy, tools, and time. Thus, a more practical, cheaper, and faster method is needed to overcome the problems in the manufacturing of BOSB. Alkali immersion treatment with 1% NaOH on bamboo strands could be the alternative way to improve its properties.

Alkali immersion treatment is more practical and cheaper than steam modification treatment, particularly to degrade the hemicellulose and extractives of bamboo strands. On the other hand, alkaline conditions caused by alkaline treatment can accelerate the curing process of PF adhesive, which is categorized as alkaline-curing resin adhesive (Maulana et al. 2021a). Several studies have reported that alkali immersion treatment on lignocellulosic material changes its chemical composition. Alkali treatment with 1, 2, and 4% NaOH at 30°C for 30 minutes degraded the content of hemicellulose, lignin, and extractives of the oil palm frond (Jasmi et al. 2014). Maulana et al. (2021a) reported that alkali washing with 1, 2, 3, 4, and 5% NaOH after steam treatment affected the chemical composition of the bamboo strands. In the other study, Setiawan et al. (2019) reported that alkali immersion treatment with 5 and 10% NaOH solution for 24 h increased oil palm fiber's surface roughness properties and thermal resistance. This research aimed to analyze the change in chemical composition of the bamboo strands after alkali immersion treatment under

various immersion times. The data from this research will be valuable to determine the optimum bamboo strand immersing time to produce BOSB with good physical and mechanical properties.

2. Materials and Methods

2.1. Materials

The research material is 4-year-old betung bamboo (*Dendrocalamus asper*) from Sukabumi district, West Java, Indonesia. Sodium hydroxide (NaOH), ethanol-benzene, distilled water, sulfuric acid (H₂SO₄), and acetic acid (CH₃COOH) for chemical components analysis.

2.2. Bamboo Preparation

The bamboo culms were cut, and the outer and inner skin was removed. The bamboo culms were converted into strands with the target length, width, and thickness of 70, 25, and 0.5 mm, respectively. Strands were alkali immersion-treated with 1% NaOH solution for 1, 2, and 3 h.

2.3. Chemical Component Analysis

The powder from the milling operation was sieved to achieve particles of 40-60 mesh size. Preparation of powder for chemical component analysis refers to the Technical Association of the Pulp and Paper Industry (TAPPI) standard T 264 cm-07 regarding the preparation of wood test samples for chemical analysis (TAPPI 1997).

2.3.1. Holocellulose content determination

The determination of holocellulose content refers to (Browning 1967). The powder sample of 2 g was placed in a 250 ml Erlenmeyer and then added with 80 ml of distilled water, 1 g of sodium chlorite (NaClO₂), and 0.5 ml of glacial acetic acid. The sample was heated at 70 - 80°C using a water bath. Every 1 h from the reaction time, 1 g of NaClO₂ and 0.5 ml of acetic acid were added for 4 repetitions. After the residue was whitish, the sample was filtered and washed using hot distilled water and 25 ml of 10% acetic acid. The sample was oven-dried at 103 ± 2°C for 24 h and weighed until the dry weight was constant. The calculation of holocellulose content was determined by the following equation:

$$\text{Holocellulose content (\%)} = \frac{W_1}{W_0} \times 100\% \quad (1)$$

where W_1 is the oven-dried weight of holocellulose (g), and W_0 is the oven-dried weight of the sample (g).

2.3.2. α -cellulose content determination

Holocellulose sample of 1.5 g was placed in a 250 ml Erlenmeyer, and 10 ml of 17.5% NaOH was added at 20°C and stirred until the sample was wholly wetted. 5 ml of 17.5% NaOH was added into Erlenmeyer every 5 minutes. The addition was carried out 3 times so that the total volume of 17.5% NaOH was 25 ml. The sample was allowed to stand for 30 minutes, then 33 ml of distilled water was added and allowed to stand again for 1 h at 20°C. The sample was filtered and washed with hot distilled water before being used. After that, the sample was rinsed three times with 10%

acetic acid, followed by hot distilled water until it was acid-free. The sample was oven-dried at $103 \pm 2^\circ\text{C}$ for 24 h and then weighed until the dry weight was constant. The determination of α -cellulose content was calculated by the following equation:

$$\alpha\text{-cellulose content (\%)} = \frac{W_2}{W_0} \times 100\% \quad (2)$$

where W_2 is the oven-dried weight of α -cellulose (g), and W_0 is the oven-dried weight of sample powder (g).

2.3.3. Klason lignin content determination

Determination of klason lignin content refers to the standard of TAPPI (2021) with modifications (Lin and Dence 1992). The extractive free sample weighing 500 mg was placed in a 50 ml beaker, and 5 ml of 72% sulfuric acid was added gradually while stirring. The samples were kept in the bath at $20 \pm 1^\circ\text{C}$ for 3 h and went occasionally. The sample was transferred to a 500 ml Erlenmeyer and diluted to a concentration of 3% sulfuric acid by adding water to a total volume of 196 ml. The sample was heated in an autoclave for 30 minutes at 121°C . A filter glass was used to filter the sample, dried at $103 \pm 2^\circ\text{C}$ for 24 h, and then weighed until the dry weight was constant. Determination of klason lignin content was calculated by the following formula:

$$\text{Klason lignin content (\%)} = \frac{W_3}{W_0} \times 100\% \quad (3)$$

where W_3 is the oven-dried weight of klason lignin (g), and W_0 is the oven-dried weight of sample powder (g).

2.3.4. Determination of cold water-soluble extractive content

The sample of 2 g was placed in a 500 ml Erlenmeyer, and 300 ml of distilled water was added. Extraction was carried out at $23 \pm 2^\circ\text{C}$ for 48 h. Next, the sample was filtered and washed with 200 ml of cold distilled water. Finally, the sample was dried at $103 \pm 2^\circ\text{C}$ to constant dry weight (TAPPI 1999).

2.3.5. Determination of hot water-soluble extractive content

The sample of 2 g was put into a 250 ml Erlenmeyer, and 100 ml of hot distilled water was added. Samples were extracted for 3 h at 100°C . The sample was filtered and washed with 200 ml of hot water. Samples were dried at $103 \pm 2^\circ\text{C}$ to constant dry weight (TAPPI 1999).

2.3.6. Determination of 1% NaOH-soluble extractive content

The measurement of the concentration of dissolved extractives in 1% NaOH refers to the standard TAPPI T 212 om-02 (TAPPI 2002). A sample of 2 g was put into an Erlenmeyer, and 100 ml of 1% NaOH solution was added. The Erlenmeyer was placed in a water bath with the water level in the bath above the water in the Erlenmeyer for 60 minutes. The solution was heated for 10, 15, and 25 minutes while stirring with a glass stirrer. The material was washed twice with hot water and twice with 25 ml of 10% acetic acid. The samples were then rinsed in hot water until it became acid-free. Samples were dried at $103 \pm 2^\circ\text{C}$ until the dry weight was constant.

2.3.7. Determination of ethanol-benzene-soluble extractive content

The measurement of extractive content refers to the standard of TAPPI T 204 cm-07 (TAPPI 2007). A sample of 2 g was placed in a lead paper whose weight is known. The sample was put into Soxhlet and extracted with the ethanol-benzene solution with a ratio of 1:2 for 6-8 h. The sample was washed with ethanol and aerated. The sample was dried at $103 \pm 2^\circ\text{C}$ until the dry weight was constant. The content of extractives was calculated by the following formula:

$$\text{Extractives content (\%)} = \frac{W_0 - W_4}{W_0} \times 100\% \quad (4)$$

where W_4 is the oven-dried weight of the sample after extraction (g), and W_0 is the oven-dried weight of sample powder before extraction (g).

2.4. Data Analysis

This study's experimental design was a simple Completely Randomized Design (CRD) with a single factor as alkali immersion times. The data obtained in this research were analyzed statistically using variance (ANOVA) followed by Duncan's Multiple Range Test (DMRT) to determine a significant difference within the factor's level. The statistical analysis was conducted using International Business Machines (IBM) SPSS Statistics Software 22.

3. Results and Discussion

3.1. Holocellulose Content

The holocellulose content in bamboo ranged from 71.61 to 72.19% (Fig. 1). The highest holocellulose content was found in untreated bamboo, and the lowest was found in the bamboo with alkali immersion treatment for 3 h. There was a decrease in the value of holocellulose content due to alkali immersion treatment for 1, 2, and 3 h, although it had no significant effect. This phenomenon was indicated by the results of the ANOVA test that there was no significant effect due to alkali immersion treatment with 1% NaOH for 1, 2, and 3 h.

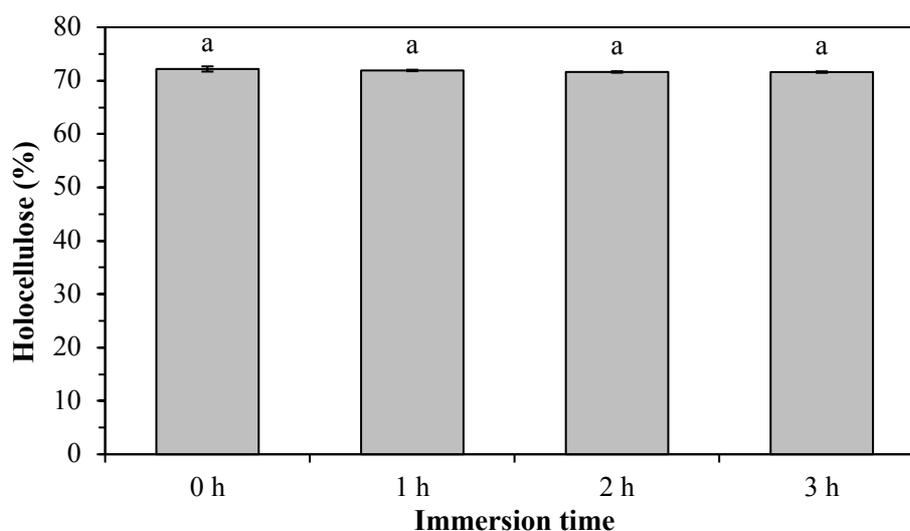


Fig. 1. Holocellulose content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

Holocellulose consists of cellulose and hemicellulose. Holocellulose content, which tends to increase, correlates with increased relative α -cellulose content caused by hemicellulose degradation due to alkaline treatment (Jasmi et al. 2014). Similar results were reported by Maulana et al. (2021a) that alkali washing treatment with 1-5% NaOH after steam treatment slightly increased the level of α -cellulose due to a decrease in hemicellulose content. Thus, this phenomenon caused a reduction in holocellulose content.

3.2. α -cellulose Content

The value of α -cellulose content varied from 50.64 to 51.31% (Fig. 2). The value of α -cellulose content slightly increased due to alkali immersion treatment for 1, 2, and 3 h. Based on the results of ANOVA ($P \leq 0.05$), alkali immersion treatment with 1% NaOH had not significantly affected the α -cellulose of the betung bamboo.

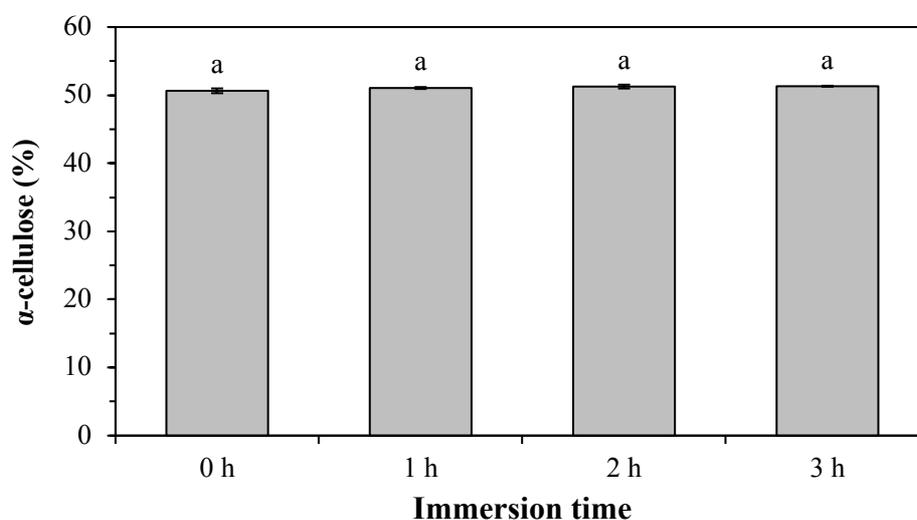


Fig. 2. α -cellulose content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

The α -cellulose had a sturdy structure, so its levels were unaffected by the alkaline immersion treatment with 1% NaOH. However, the relative levels of α -cellulose tend to increase due to hemicellulose degradation after immersion treatment with 1% NaOH for 1, 2, and 3 h. Several previous studies reported that alkaline treatment could degrade hemicellulose and low molecular weight substances (Jasmi et al. 2014; Maulana et al. 2021a). Degradation of hemicellulose and low molecular weight components increased the crystallinity of lignocellulosic material fibers (Abdal-hay et al. 2012). Setiawan et al. (2019) reported that X-ray diffraction results showed that the crystallinity of palm coir fiber and banana midrib reached its maximum value in alkaline treatment with 5% NaOH. A similar result had reported by Cao et al. (2012) that the crystallinity of flax fibers improved due to alkaline treatment. This phenomenon was thought to be related to the increasing crystallinity of cellulose. According to Zhang et al. (2013), NaOH solution swelled the structure of cellulose polymers even at room temperature conditions.

3.3. Hemicellulose Content

The hemicellulose content of betung bamboo ranged from 20.30 to 21.55% (**Fig. 3**). The lowest value of hemicellulose content was found in betung bamboo with alkali immersion treatment for 3 h. According to statistical analysis, alkali immersion treatment under various immersing-time significantly affected hemicellulose content. The DMRT results showed a significant difference between each treatment. In addition, the results show that alkali immersion treatment stained hemicellulose content with a higher intensity at a longer immersion time.

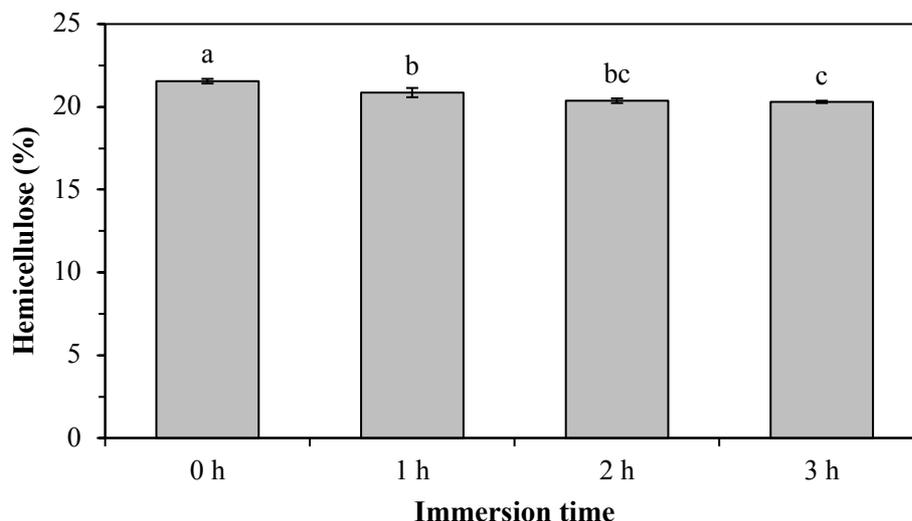


Fig. 3. Hemicellulose content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

The hemicellulose content decreased due to alkali immersing treatment for 1, 2, and 3 h. A similar result was reported by [Fatrawana et al. \(2019\)](#) that hemicellulose content decreased by steam and washing treatment with water and 1% NaOH solution. [Reddy et al. \(2013\)](#) reported that hemicellulose content of alkali-treated century fiber decreased from 22.24 to 7.01% due to its amorphous structure. In the other study, alkali treatment with 5% and 10% NaOH for 24 h caused the degradation of lignin, hemicellulose, and other contaminants ([Setiawan et al. 2019](#)). Hemicellulose is an amorphous polysaccharide component that causes low dimensional stability of BOSB ([Fatrawana et al. 2019](#)). The hemicellulose degradation improved the mechanical properties, hygroscopic properties, water absorption capacity ([Fatrawana et al. 2019](#)), crystallinity enhancement, and wettability properties improvement ([Cao et al. 2012](#)).

3.4. Klason Lignin Content

Fig. 4 shows that the klason lignin content ranged from 28.05 to 28.57%. The highest klason lignin content was found in alkali-immersed betung bamboo with 1% NaOH for 3 h. Meanwhile, the lowest klason lignin content was found in the untreated betung bamboo. The alkali immersion with 1% NaOH under various immersing times had a significant effect ($P \leq 0.05$) on the content of the klason lignin component. The DMRT results showed a significant difference between each treatment. The alkali immersion treatment for 1, 2, and 3 h tend to decrease the klason lignin content value.

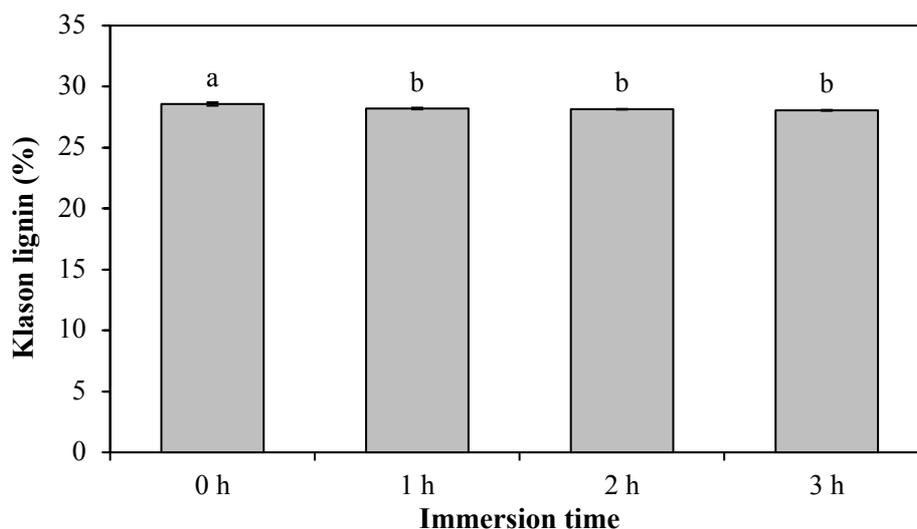


Fig. 4. The lignin content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

The decrease in klason lignin levels was caused by the alkalization process due to alkaline treatment with NaOH solution (Setiawan et al. 2019). NaOH solution could cause the delignification process of lignocellulosic material (Xu et al. 2020). Several studies reported that alkaline treatment decrease lignin content, especially at high concentration (Maulana et al. 2021a; Yang et al. 2019). Another study found a similar outcome reported by Tong et al. (2018) that alkaline immersion treatment with 10% NaOH for 24 h partially removed the lignin content. This study shows that klason lignin content decreased event at a low concentration of alkali and shorter immersion time. The decrease in lignin content was such an advantage for biocomposite material. Partially lignin removal may alter fiber's tensile and surface roughness properties (Hashim et al. 2017; Setiawan et al. 2019; Tong et al. 2018). Thus, the fibers were easier to bond with the adhesive.

3.5. Cold Water-Soluble Extractives Content

Cold water-soluble extractives in betung bamboo ranged from 2.16 to 5.83% (Fig. 5). The highest extractive content value was found in untreated betung bamboo. The lowest value of extractive substances was found in betung bamboo treated with alkali immersion with 1% NaOH solution for 3 h of immersion. The results of the ANOVA test showed that the alkali immersion treatment with 1% NaOH solution had a significant effect ($P \leq 0.05$) on the concentration of dissolved extractives in cold water. The DMRT revealed that each treatment element differed significantly.

Cold water-soluble extractive substances experienced a significant decrease. This phenomenon was because of the low molecular weight component in extractives easily degraded by alkaline treatment (Maulana et al. 2021a). Substances generally dissolved in cold water consist of tannins, gums, sugars, and salts (Chauhan et al. 2020). The most effective alkali immersion treatment with 1% NaOH to degrade cold-water-soluble extractives was immersed for 3 h. Thus, the decrease in cold-water-soluble extractives content of alkali immersed betung bamboo is correlated with the length of immersion time.

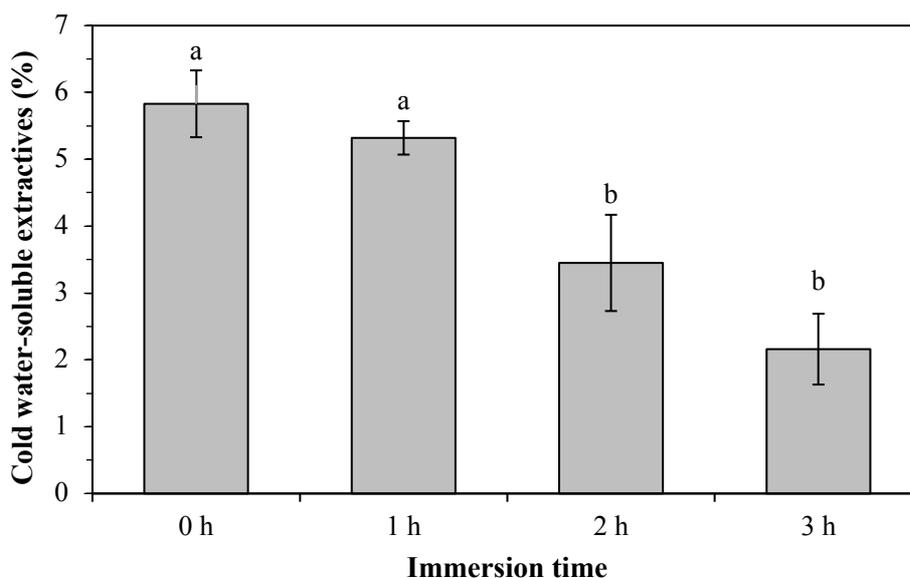


Fig. 5. Cold water-soluble extractives content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

3.6. Hot Water-Soluble Extractives Content

Fig. 6 shows the hot water-soluble extractive content in betung bamboo. The highest extractive content value was found in untreated betung bamboo (7.37%). The lowest value of extractive substances was found in betung bamboo treated with alkali immersion with 1% NaOH solution for 3 h of immersion (4.62%). The results of the ANOVA test showed that the alkali immersion treatment with 1% NaOH solution had a significant effect ($P \leq 0.05$) on the concentration of dissolved extractives in hot water. The DMRT revealed that each treatment element differed significantly.

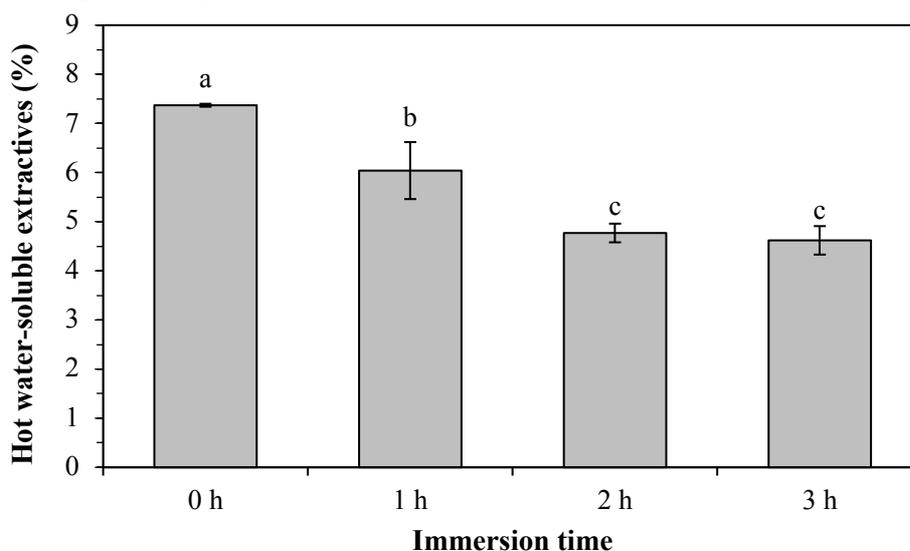


Fig. 6. Hot water-soluble extractives content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

Organic compounds dissolved in hot water consist of fats, waxes, alkaloids, and phenols (Chauhan et al. 2020). These extractives had a negative effect on bamboo strands as the BOSB material. In addition, starch is a component consumed by deterioration factors such as termite and powder post beetle (Febrianto et al. 2014). Therefore, the degradation of hot water-soluble extractives may alter the durability properties of the betung bamboo.

3.7. 1% NaOH-Soluble Extractives Content

1% NaOH-soluble extractives content ranged from 20.35 to 25.32% (Fig. 7). The highest extractive content value was found in untreated betung bamboo. The lowest value of extractive substances was found in betung bamboo treated with alkali immersion with 1% NaOH solution for 3 h of immersion. The results of the ANOVA test showed that the alkali immersion treatment with 1% NaOH solution had a significant effect ($P < 0.05$) on the concentration of dissolved extractives in 1% NaOH solution. The DMRT showed a significant difference between the alkali immersed betung bamboo and the untreated betung bamboo. However, there were no significant differences between the alkali immersion treatment for 1, 2, and 3 h.

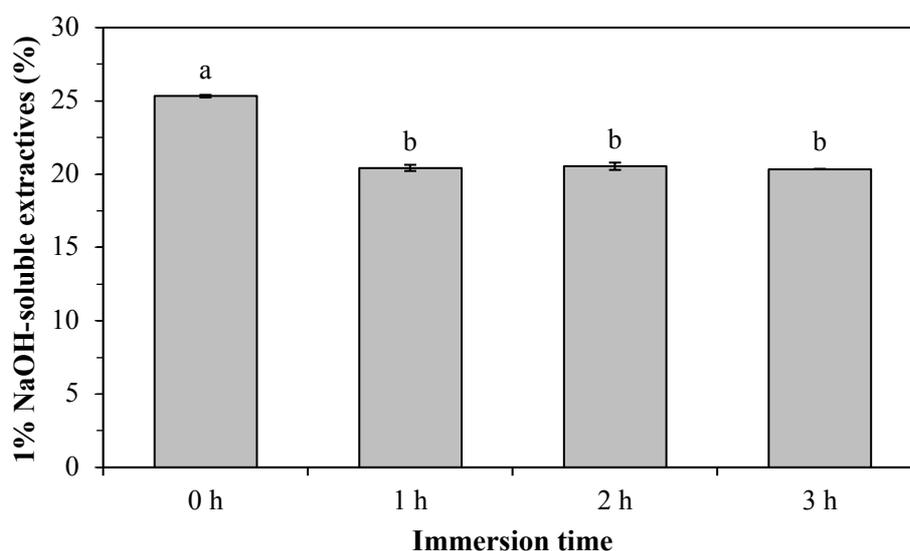


Fig. 7. 1% NaOH-soluble extractives content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

Alkali immersion treatment with 1% NaOH under various immersing times decreased the value of 1% NaOH-soluble extractives content. Pirayesh et al. (2012) reported that extractives had an adverse effect on adhesive curing and caused poor internal bonding strength. Determination of the dissolved extractive content of 1% NaOH can be used to estimate the presence of carbohydrates and low molecular weight components and the level of degradation of wood chemical components by deterioration factors (Murda et al. 2018).

3.8. Ethanol-Benzene-Soluble Extractives Content

Ethanol-benzene-soluble extractives in betung bamboo ranged from 3.91–7.90% (Fig. 8). The highest extractive content value was found in untreated betung bamboo. The lowest value of

extractive substances was found in betung bamboo treated with alkali immersion with 1% NaOH solution for 3 h of immersion. The results of the ANOVA test showed that the alkali immersion treatment with 1% NaOH solution had a significant effect ($P \leq 0.05$) on the concentration of dissolved extractives in ethanol-benzene solution. The DMRT showed a significant difference between the alkali immersed betung bamboo and the untreated betung bamboo. However, there were no significant differences between the alkali immersion treatment for 1, 2, and 3 h.

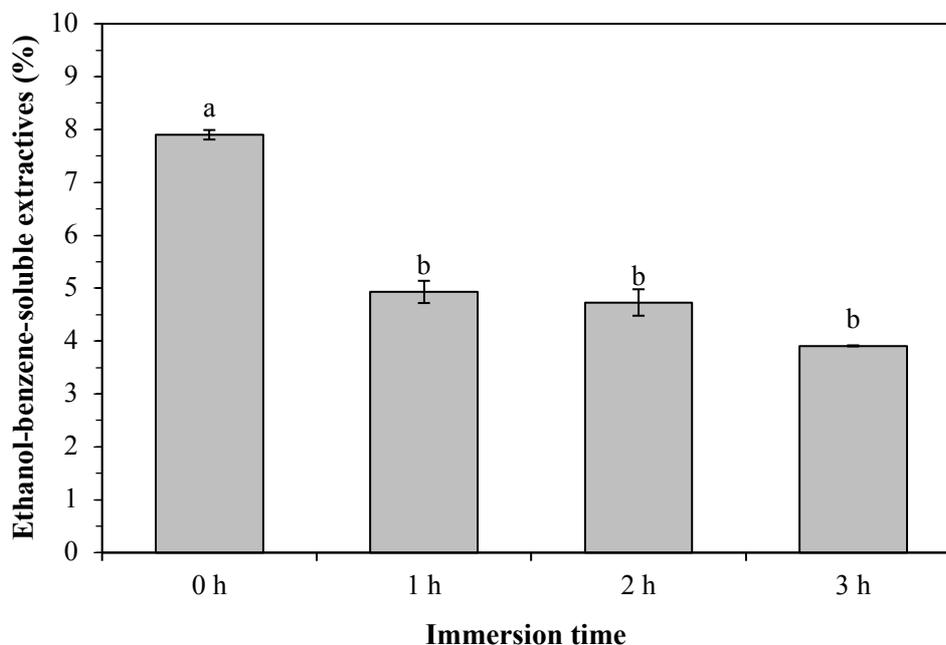


Fig. 8. Ethanol-benzene-soluble extractives content of betung bamboo due to alkali immersion treatment at various immersion times (Different letters show significant differences between treatments according to Duncan's multiple range tests at a 5% confidence interval. Error bars show standard deviation).

The extractive chemicals dissolved in ethanol-benzene were terpenoids to phenolic compounds (Murda et al. 2018). Extractive substances can inhibit the gluing process (Pizzi and Kumar 2019). The degradation of extractive substances of betung bamboo has several benefits for BOSB manufacturing, such as good adhesion, higher dimensional stability, better mechanical properties, and more durability to deterioration factors attack (Fatrawana et al. 2019; Maulana et al. 2021a, 2017; Maulana 2018; Murda et al. 2018). The most effective immersing time in alkali treatment with 1% NaOH to decrease the ethanol-benzene-soluble extractives content was 1 h.

4. Conclusions

Alkali immersion treatment with 1% NaOH solution changed the chemical composition of betung bamboo. Holocellulose content tends to decrease due to the reduced proportion of hemicellulose content. On the other hand, the relative levels of α -cellulose increased due to reduced hemicellulose and lignin levels. Extractive substances experienced the most significant changes among betung bamboo's chemical components. Extractive substances degraded significantly due to alkaline immersion treatment. The decrease in hemicellulose, molecular weight lignin, and extractive substances would be beneficial for bamboo-oriented strand board

manufacture to improve strand adhesion, dimensional stability, mechanical properties, and durability against biological agents attack.

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